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Benzylammonium Bis[dichloromercury(II)] Chloride

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Abstract. $C_7H_{10}N^+.2HgCl_2.Cl^-$, $M_r = 686.6$, monoclinic, $C2/c$, $Z = 8$, $a = 17.370$ (5), $b = 6.891$ (2), $c = 24.735$ (7) Å, $\beta = 104.06$ (2)°, $V = 2872$ (1) Å^3, $D_c = 3.18$, $D_m = 3.1$ (1) Mg m^{-3}, μ(Mo $K\alpha$) = 22.69 mm^{-1}, single-crystal diffractometer data up to sin $\theta/\lambda = 0.65$ Å^{-1}, $R_w(F) = 0.056$. The structure consists of $C_6H_5CH_2NH_3^+$, $HgCl_2$ and Cl^- groups. The distorted $HgCl_2$ groups have average Hg–Cl = 2.282 (4) and 2.300 (4) Å. The Cl–Hg–Cl angles are 166.6 (2) and 169.9 (1)°. Long Hg–Cl bonds [2.917 (6)–3.094 (7) Å] form a chain structure along b.

Introduction. Colorless crystals of the title compound were obtained from a solution of $C_6H_5CH_2NH_3^+.Cl^-$ and $HgCl_2$ in ethanol. A crystal 0.12 × 0.16 × 0.37 mm was selected. Precession photographs showed the space group to be either Cc or $C2/c$; the latter was accepted during the refinement. Data were collected in two quadrants of reciprocal space, up to sin $\theta/\lambda = 0.65$ and 0.60 Å^{-1}, on a Syntex $P2_1$ diffractometer with Nb-filtered Mo $K\alpha$ radiation. 5873 reflections were obtained of which 3229 were independent. Background corrections were made (Blessing, Coppens & Becker, 1974). Three standard reflections observed after every 60 reflections showed small long-term fluctuations up to about 5%. The data were rescaled with respect to the standards.

An absorption correction was applied (transmission range 0.030 to 0.125). A weight was assigned to the reflections according to $w(I) = [\sigma^2(I)_{counting} + (0.03I)^2]^{-1}$. The equivalent reflections were averaged. The internal consistency was $R = \sum |I - \langle I \rangle|/\sum I =$ 0.043. 587 reflections with $\langle I \rangle < 0$ were not used in the analysis. The structure was determined by the Patterson method. A complication arose as the structure contains a pseudo-mirror plane about $y = 0$ and 0.5. The deviation from the mirror symmetry is given by the reflections with l odd, all of which are weak. An inspection of a Patterson synthesis with l odd data only, however, resulted in the directions of the shifts of the atoms from the pseudo-symmetry plane. No H atoms could be located. For those attached to C, positions were calculated and included in the structure factor calculation.

An isotropic extinction correction was made. The structure refined to $R(F) = 0.103$, $R_w(F) = 0.056$ and $S = [\sum (|F_o| - |F_c|)^2/(NO - NV)]^{1/2} = 2.23$. A final difference synthesis showed features between -3.3 and $+3.7$ e Å^{-3} at a distance of 1.05 Å from the Hg atoms. Otherwise no features >1.0 e Å^{-3} were found. Scattering factors were taken from *International Tables for X-ray Crystallography* (1974), anomalous scattering factors for Hg and Cl from Cromer & Liberman (1970). The calculations were performed with the XRAY system (Stewart, Kruger, Ammon, Dickinson & Hall, 1972) on the Univac 1108 computer of the University of Frankfurt. Positional parameters are given in Table 1,* bond lengths and angles in Table

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 35337 (23 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Positional parameters*

	x	y	z
Hg(1)	0·37143 (3)	0·4876 (1)	0·22286 (3)
Hg(2)	0·78544 (3)	0·4869 (1)	0·15547 (3)
Cl(1)	0·0843 (2)	0·5005 (10)	−0·2037 (2)
Cl(2)	0·1942 (2)	0·0132 (9)	−0·1302 (2)
Cl(3)	0·4572 (2)	0·5138 (8)	−0·1901 (2)
Cl(4)	0·3317 (2)	0·5331 (8)	−0·0900 (2)
Cl(5)	0·2536 (3)	0·2589 (6)	−0·2475 (3)
N	0·4026 (7)	−0·008 (3)	−0·1679 (6)
C(1)	0·4150 (9)	0·039 (3)	−0·1070 (7)
C(2)	0·5010 (8)	0·015 (3)	−0·0764 (7)
C(3)	0·5308 (10)	−0·167 (3)	−0·0602 (9)
C(4)	0·6055 (15)	−0·184 (4)	−0·0337 (13)
C(5)	0·6557 (10)	−0·037 (5)	−0·0227 (9)
C(6)	0·6268 (14)	0·146 (5)	−0·0371 (13)
C(7)	0·5514 (13)	0·170 (3)	−0·0667 (11)

Table 2. *Bond distances (Å) and angles (°)*

Hg(1)–Cl(2)	2·298 (4)		N–C(1)	1·51 (2)
Hg(1)–Cl(3)	2·302 (3)		C(1)–C(2)	1·51 (2)
Hg(1)–Cl(5)	2·917 (6)		C(2)–C(3)	1·38 (3)
Hg(1)–Cl(5′)	2·935 (6)		C(3)–C(4)	1·31 (3)
Hg(1)–Cl(3′)	3·270 (4)		C(4)–C(5)	1·33 (4)
Hg(2)–Cl(1)	2·289 (3)		C(5)–C(6)	1·37 (4)
Hg(2)–Cl(4)	2·275 (4)		C(6)–C(7)	1·35 (3)
Hg(2)–Cl(5″)	3·077 (7)		C(7)–C(2)	1·37 (3)
Hg(2)–Cl(5‴)	3·094 (7)			

|				
Cl(2)–Hg(1)–Cl(3)	169·9 (1)		Cl(4)–Hg(2)–Cl(5″)	104·0 (2)
Cl(2)–Hg(1)–Cl(5)	92·5 (2)		Cl(4)–Hg(2)–Cl(5‴)	102·8 (2)
Cl(2)–Hg(1)–Cl(5′)	89·6 (2)		Cl(5″)–Hg(2)–Cl(5‴)	68·0 (2)
Cl(2)–Hg(1)–Cl(3′)	90·8 (1)		N–C(1)–C(2)	111 (1)
Cl(3)–Hg(1)–Cl(5)	95·8 (2)		C(1)–C(2)–C(3)	120 (2)
Cl(3)–Hg(1)–Cl(5′)	98·4 (2)		C(1)–C(2)–C(7)	121 (2)
Cl(3)–Hg(1)–Cl(3′)	79·1 (1)		C(3)–C(2)–C(7)	119 (1)
Cl(5)–Hg(1)–Cl(5′)	72·2 (1)		C(2)–C(3)–C(4)	119 (2)
Cl(5)–Hg(1)–Cl(3′)	143·2 (1)		C(3)–C(4)–C(5)	124 (2)
Cl(5′)–Hg(1)–Cl(3′)	144·5 (1)		C(4)–C(5)–C(6)	118 (2)
Cl(1)–Hg(2)–Cl(4)	166·6 (2)		C(5)–C(6)–C(7)	120 (2)
Cl(1)–Hg(2)–Cl(5″)	87·7 (2)		C(6)–C(7)–C(2)	119 (2)
Cl(1)–Hg(2)–Cl(5‴)	87·6 (2)			
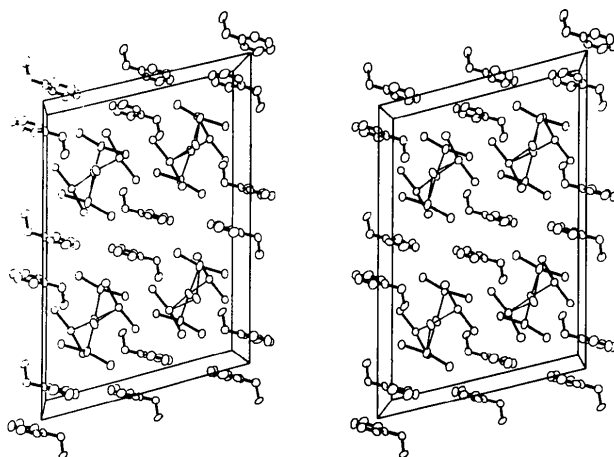
Fig. 1. Stereoscopic view of the structure. The thermal ellipsoids correspond to the 50% probability surfaces.

2. A stereoscopic view of the molecular packing is shown in Fig. 1.

Discussion. Hg^{II} chloride complexes show a variety of different Hg^{II} coordinations (see, *e.g.*, Sandström & Liem, 1978, and references therein). In the title compound each Hg atom is essentially in the form of a discrete HgCl$_2$ molecule. The average short Hg–Cl lengths are 2·300 and 2·282 Å for the two HgCl$_2$ groups. This is only a small elongation compared with the 2·252 (5) Å in gaseous HgCl$_2$ (Kashiwabara, Konaka & Kimura, 1973).

Both Hg atoms have two long bonds to Cl(5) atoms, ranging from 2·917 (6) to 3·094 (7) Å, which form a linear structure in the *b* direction. Hg(1) has a fifth contact with a Cl atom of 3·270 (4) Å. However, a contact of this length is no longer considered to contribute significantly to the bonding (Grdenić, 1965). The angles of 166·6 (2) and 169·9 (1)° in the HgCl$_2$ groups result from a distortion of this group by the long Hg–Cl bonds. Similar values have been found for HgCl$_2$ complexes and HgCl$_2$ solutions (Sandström, 1978). Distorted pseudo HgCl$_2$ molecules are also found in NH$_4$HgCl$_3$ (Harmsen, 1938) and NH$_4$HgCl$_3$.H$_2$O (Sagisawa, Kitahama, Kiriyama & Kiriyama, 1974). In these cases four long Hg–Cl bonds disturb the HgCl$_2$ molecule, resulting in longer short Hg–Cl bonds [2·34 and 2·37 (1) Å respectively]. Only in the HgCl$_2$ crystal, where no long Hg–Cl contacts <3·40 Å occur, has an essentially undisturbed HgCl$_2$ group been found with an Hg–Cl bond of 2·25 Å (Grdenić, 1950).*

The benzylammonium group occupies the spaces between the Hg$_2$Cl$_5$ chains. The N atom has five contacts with Cl atoms which might correspond to hydrogen bonds (Table 3). As no H atoms could be located, no definite conclusions can be made about the hydrogen-bonding scheme.

* *Editorial note:* see also Subramanian & Seff (1980).

Table 3. N···Cl *contacts*

N···X	N···X (Å)	C(1)–N···X (°)
N···Cl(1)^a	3·24 (1)	162 (1)
N···Cl(5)^b	3·39 (1)	112 (1)
N···Cl(5)^a	3·40 (1)	128 (1)
N···Cl(1)^c	3·48 (1)	109 (1)
N···Cl(3)^d	3·51 (1)	112 (1)

Symmetry code

(a)	$\frac{1}{2} - x, -\frac{1}{2} + y, -\frac{1}{2} - z$	(c)	$\frac{1}{2} + x, -\frac{1}{2} + y, z$
(b)	$x, \quad y, \quad z$	(d)	$x, -1 + y, z$

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Lithium [(R,S)-N,N'-Ethylenediaminedisuccinato]cobaltate(III) Trihydrate

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Abstract. $Li[Co(C_{10}H_{12}N_2O_8)].3H_2O$, $C_{10}H_{12}CoN_2O_8^-.Li^+.3H_2O$, $M_r = 408\cdot2$, monoclinic, $P2_1/c$, $a = 9\cdot624\,(10)$, $b = 12\cdot788\,(6)$, $c = 12\cdot049\,(6)$ Å, $\beta = 94\cdot85\,(6)°$, $U = 1477\cdot6\,(1\cdot8)$ Å^3, $Z = 4$, $D_m = 1\cdot80\,(2)$, $D_x = 1\cdot83$ Mg m^{-3}, Mo $K\alpha$ ($\lambda = 0\cdot7107$ Å), $\mu = 1\cdot28$ mm^{-1}. The final R is 0·074 for 1689 independent observed reflections. The Co atoms of the $[Co_2\{(R,S)\text{-edds}\}_2]$ dimeric complex anion are bonded octahedrally to the two N atoms and one O atom from each of the four carboxylate arms of the two complexing species. The complex anions are held together by LiO_4 tetrahedra and hydrogen bonds.

Introduction. Preparation, chemistry and preliminary results on the crystal structure of $Li[Co\{(R,S)\text{-edds}\}].3H_2O$ were given by Pavelčík & Majer (1977). In this paper full crystallographic data are presented. The crystal used for the structure determination was of an approximate cubic form {100}, {011} with an edge dimension of 0·2 mm. The intensities of 3785 independent reflections ($2\cdot9° \leq 2\theta \leq 55°$) were collected on a Syntex $P2_1$ diffractometer with graphite-monochromated Mo $K\alpha$ radiation and the θ–2θ technique at a scan rate varying from 4·88 to 29·3° min^{-1} in 2θ. The background was measured at each end of the scan for one half of the reflection scan time. Two standards, monitored after every 94 reflections,

showed that no correction for instrumental instability or crystal decay was required. 1689 reflections with $I > 1\cdot96\sigma(I)$ were considered as observed (only 45%). A value of $0\cdot698\sigma(I)$ was assigned to the weak unobserved reflections with $I < 0\cdot698\sigma(I)$. The intensities were corrected for Lorentz and polarization factors. No corrections for absorption or extinction were made. The structure was solved by three-dimensional Patterson and electron density Fourier syntheses. H atoms were found for the $[(R,S)\text{-edds}]^{4-}$ anion, but not for the water molecules. The structure was refined by block-diagonal least squares with anisotropic thermal parameters for the non-hydrogen and isotropic thermal parameters for the H atoms. The function $\sum w(|F_o| - |F_c|)^2$ was minimized; a weighting scheme $w^{-1} = \sigma^2(|F_o|) + (C|F_o|)^2$, where $\sigma(|F_o|)$ is derived from counting statistics and $C = 0\cdot05$, was employed. C was adjusted so that constant values of $\langle w(|F_o| - |F_c|)^2 \rangle$ were obtained in different $|F_o|$ intervals. The final residual R ($= \sum|\Delta F|/\sum|F_o|$) was 0·074 for the observed reflections used in the refinement and 0·166 including the zero-weighted reflections. Corresponding weighted residuals R_w [$= (\sum w|\Delta F|^2/\sum w|F_o|^2)^{1/2}$] were 0·083 and 0·114. The maximum peak in the final difference synthesis was 0·38 e Å^{-3}. Scattering factors were taken from *International Tables for X-ray Crystallography* (1968). All crystallographic calculations were performed with the NRC program package (Ahmed, 1970) on a Siemens 4004/150 computer. Atomic coordinates for the non-hydrogen

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