

Absorption correction:  $R_{\text{int}} = 0.097$   
 by integration (Stoe & Cie, 1996d)  $\theta_{\text{max}} = 25.24^\circ$   
 $T_{\text{min}} = 0.010$ ,  $T_{\text{max}} = 0.083$   $h = -15 \rightarrow 15$   
 6843 measured reflections  $k = -10 \rightarrow 11$   
 2131 independent reflections  $l = -13 \rightarrow 13$   
 Intensity decay: none

#### Refinement

Refinement on  $F^2$  Weighting scheme: see below  
 $R[F^2 > 2\sigma(F^2)] = 0.026$   
 $wR(F^2) = 0.063$   $(\Delta/\sigma)_{\text{max}} = 0.002$   
 $S = 1.006$   $\Delta\rho_{\text{max}} = 1.049 \text{ e } \text{Å}^{-3}$   
 2131 reflections  $\Delta\rho_{\text{min}} = -0.709 \text{ e } \text{Å}^{-3}$   
 121 parameters Extinction correction: none  
 H atoms treated by a Scattering factors from  
 mixture of independent *International Tables for*  
 and constrained refinement *Crystallography* (Vol. C)

Data are 90.3% complete to  $\theta = 25.24^\circ$ . The faces of the forms {111} and {001} were developed. Their distances from the centre of gravity were determined microscopically. For the face-indexed numerical absorption correction, the Gaussian integration routine implemented in X-RED (Stoe & Cie, 1996d) was applied. The structure was solved by the Patterson method and refined by full-matrix least-squares techniques. Weights  $w = \{1 - \exp[-9.0(\sin\theta/\lambda)^2]\}/[\sigma^2(F_o^2) + (0.014P)^2]$ , where  $P = (F_o^2 + 2F_c^2)/3$ , were used. All but two H-atom positions could be located in a difference Fourier map after refining positions and anisotropic displacement parameters of the heavier atoms. In the final stages of refinement, the H atoms were included in idealized positions. H atoms of the C—H groups were situated 0.93 Å from the parent C atom on the external bisector of the corresponding C—C—C angle (quasi-idealized positions neglecting a very small out-of-plane tilt due to Bi—arene bonding) and a riding model was applied. C—H distances of the methyl groups were set to 0.96 Å, and C—C—H and H—C—H angles to 109.5°. The H atoms of each group were allowed to ride on the parent C atom and to rotate around the C—C bond direction. The isotropic displacement parameters were kept equal to 120 and 150% of the equivalent isotropic displacement parameters of the ring and of the methyl-group C atoms, respectively. Only one significant electron-density maximum ( $1.05 \text{ e } \text{Å}^{-3}$  at 0.90 Å from Bi1) was found in the final difference Fourier map.

Data collection: EXPOSE (Stoe & Cie, 1996b). Cell refinement: CELL (Stoe & Cie, 1996a). Data reduction: INTEGRATE (Stoe & Cie, 1996c). Program(s) used to solve structure: SHELXS97 (Sheldrick, 1997b). Program(s) used to refine structure: SHELXL97 (Sheldrick, 1997a). Molecular graphics: SHELXTL-Plus (Sheldrick, 1991). Software used to prepare material for publication: SHELXL97.

This work was supported by the Fonds der Chemischen Industrie.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: JZ1297). Services for accessing these data are described at the back of the journal.

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*Acta Cryst.* (1998). **C54**, 1628–1631

### 1,3-Propanediammonium Tetrachloromercurate

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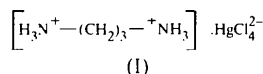
(Received 12 February 1998; accepted 11 June 1998)

#### Abstract

The title compound, (C<sub>3</sub>H<sub>12</sub>N<sub>2</sub>)[HgCl<sub>4</sub>], contains layers of tilted corner-sharing chloromercurate octahedra, with Hg—Cl distances ranging from 2.397 (2) to 2.839 (2) Å. These layers are interconnected by N—H···Cl bonds to the cations, building a three-dimensional network. The cation has a disordered propane chain.

### Comment

This investigation is the latest in a series of structure determinations of compounds formed by the reaction of substituted ammonium halides with metal halides. These compounds show interesting structural and physical properties (Spengler *et al.*, 1997). 1,3-Propanediammonium tetrachloromercurate, (I), is an example of a six-coordinated Hg atom.



The asymmetric unit contains two symmetry-independent Hg atoms at the centres of symmetry at (0,0,0) and  $(\frac{1}{2}, \frac{1}{2}, 0)$ , resulting in a centring of the *xy* plane. The Hg atoms are coordinated by Cl atoms in a tetragonally compressed octahedral arrangement (Table 2). Atoms Cl1 and Cl2 bridge the two independent Hg atoms, resulting in a two-dimensional network of corner-connected centred squares. Cl3 and Cl4 complete each centred square to form an octahedron. The corner-sharing octahedra are tilted and the lattice parameters *a*, *b* and  $\gamma$  are determined by the octahedral arrangement to a nearly tetragonal *xy* plane. The lattice parameters  $\alpha$ ,  $\beta$  and *c* are mainly correlated with the arrangement of the octahedra, since the octahedron situated at the centre of symmetry at (0,0,1) is nearly exactly above that at the centre of symmetry at  $(\frac{1}{2}, \frac{1}{2}, 0)$ , building a tetragonal network of octahedra.

The 1,3-propanediammonium cation connects the tetrachloromercurate layers, centring those squares that are not occupied by Hg atoms. The N atoms are connected to the Cl atoms *via* hydrogen bonding (Fig. 1 and Table 3). The Hg—Cl bonds to Cl1 and Cl2 are longer because they involve bridging halide atoms. The Hg—Cl1 distance is in good agreement with values found in the *International Tables for Crystallography* (1992, Vol. C). The Hg—Cl2 distance, however, is elongated, because of a single hydrogen bond to Cl2, which also leads to tilting of the octahedra. The bonds to the terminal atoms Cl3 and Cl4 are short. The difference in bond distances of these terminal Cl atoms is correlated with the hydrogen bonding to the cation. The distance of 2.528 (2) Å to Cl3 is longer than the literature values and this is due to two hydrogen bonds to two independent cations which are perpendicular to the bonding direction of Hg2—Cl3. The distance of 2.397 (2) Å to Cl4 is in good agreement with the values found in the literature for terminal Cl atoms. A similar situation for the bridging halides, with nearly equal distances and angles, can be found in Ben Salah *et al.* (1983). In the cited publication, however, the values for the terminal halides agree with the values in the *International Tables for Crystallography* (1992, Vol. C), but differ significantly from the present values.

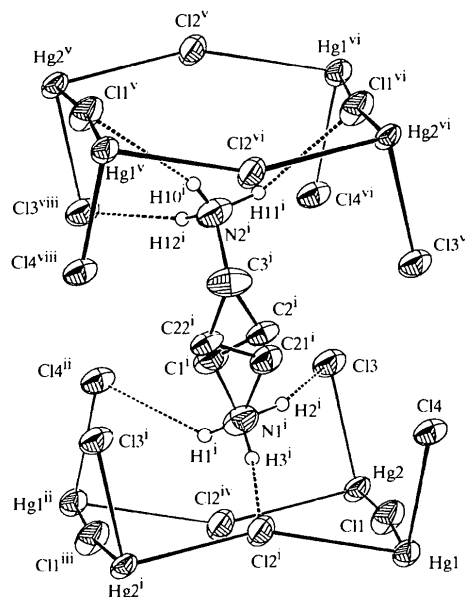


Fig. 1. The molecular structure of (I) showing the disorder of the cation. Displacement ellipsoids are shown at the 50% probability level and H atoms are shown as spheres of arbitrary radii [symmetry codes: (i)  $x - 1, y, z$ ; (ii)  $x, 1 + y, z$ ; (iii)  $-x, 1 - y, -z$ ; (iv)  $1 - x, 1 - y, -z$ ; (v)  $x - 1, y, 1 + z$ ; (vi)  $-x, -y, 1 - z$ ; (vii)  $-1 - x, -y, 1 - z$ ; (viii)  $-x, 1 - y, 1 - z$ ].

The structure is closely related to analogous copper halide compounds (Willett, 1990; Garland *et al.*, 1990) and to a cadmium halide compound previously reported by us (Groh *et al.*, 1997). The local neighbourhood of the layers is a mixture of the two kinds of related compounds: (i) the layer of corner-connected octahedra contains nearly equivalent distances in the plane and a shorter distance perpendicular to it, which is similar to the  $\text{CdCl}_4$  compound; (ii) in contrast to the  $\text{CdCl}_4$  compound, there are two contacts to bridging halides and only one to a terminal halide for N2, which is a similar situation to that in the  $\text{CuCl}_4$  compounds; (iii) the arrangement of both disordered cation chains is equivalent to the situation in  $[\text{CuCl}_4(\text{C}_5\text{H}_{16}\text{N}_2)]$  (Garland *et al.*, 1990) and could alternatively be described as a *trans-gauche* orientation (see torsion angles in Table 2); (iv) the similarities in the lattice parameters of all related compounds can be seen in Table 4.

### Experimental

The title complex was obtained by mixing a solution of 1,3-propyldiammonium chloride and mercury chloride in acetone-ethanol solution, in the stoichiometric ratio 1:1. After a few weeks, colourless non-hygroscopic crystals were obtained by slow evaporation. The chemical analysis was in good agreement with the formula.

#### Crystal data

$(\text{C}_3\text{H}_{12}\text{N}_2)[\text{HgCl}_4]$   
*M*<sub>r</sub> = 418.54

Mo K $\alpha$  radiation  
 $\lambda = 0.70926 \text{ \AA}$

## Triclinic

$P\bar{1}$   
 $a = 7.788(1) \text{ \AA}$   
 $b = 7.796(1) \text{ \AA}$   
 $c = 9.457(1) \text{ \AA}$   
 $\alpha = 68.11(1)^\circ$   
 $\beta = 72.90(1)^\circ$   
 $\gamma = 87.94(1)^\circ$   
 $V = 507.5(1) \text{ \AA}^3$   
 $Z = 2$   
 $D_x = 2.739 \text{ Mg m}^{-3}$   
 $D_m$  not measured

## Cell parameters from 27

reflections  
 $\theta = 7.2\text{--}9.4^\circ$   
 $\mu = 16.169 \text{ mm}^{-1}$   
 $T = 292 \text{ K}$   
 Parallelepiped  
 $0.39(1) \times 0.22(1) \times$   
 $0.05(1)$   
 Colourless

N1—C1—C2—C3  $-169(1)$  N1—C21—C22—C3  $176(1)$   
 C1—C2—C3—N2  $-73(1)$  C21—C22—C3—N2  $87(1)$   
 Symmetry codes: (i)  $x - 1, y, z$ ; (ii)  $1 + x, y, z$ .

Table 3. Hydrogen-bonding geometry ( $\text{\AA}, ^\circ$ )

$D\text{---}H\cdots A$	$D\text{---}H$	$H\cdots A$	$D\cdots A$	$D\text{---}H\cdots A$
N1—H1 $\cdots$ Cl4 <sup>i</sup>	0.96	2.59	3.366(4)	138
N1—H3 $\cdots$ Cl2	0.96	2.36	3.296(4)	164
N1—H2 $\cdots$ Cl3 <sup>ii</sup>	0.96	2.47	3.295(4)	144
N2—H12 $\cdots$ Cl3 <sup>iii</sup>	0.96	2.35	3.279(4)	163
N2—H10 $\cdots$ Cl1 <sup>iv</sup>	0.96	2.46	3.269(4)	141
N2—H11 $\cdots$ Cl1 <sup>v</sup>	0.96	2.52	3.339(4)	143

Symmetry codes: (i)  $1 + x, 1 + y, z$ ; (ii)  $1 + x, y, z$ ; (iii)  $1 - x, 1 - y, 1 - z$ ; (iv)  $x, y, 1 + z$ ; (v)  $1 - x, -y, 1 - z$ .

## Data collection

Rebuilt Philips PW1100  
 diffractometer (Gomm,  
 1993)  
 $\theta/2\theta$  scans  
 Absorption correction:  
 Gaussian by integration  
 (Busing & Levy, 1957)  
 $T_{\min} = 0.018, T_{\max} = 0.155$   
 1924 measured reflections  
 1804 independent reflections

1804 reflections with  
 $F^2 > 0$   
 $R_{\text{int}} = 0.042$   
 $\theta_{\text{max}} = 25^\circ$   
 $h = -8 \rightarrow 8$   
 $k = -8 \rightarrow 8$   
 $l = 0 \rightarrow 11$   
 5 standard reflections  
 every 200 reflections  
 intensity decay: 0.9%

## Refinement

Refinement on  $F^2$   
 $R(F) = 0.034$   
 $wR(F^2) = 0.048$   
 $S = 1.12$   
 1800 reflections  
 114 parameters  
 H-atom parameters  
 constrained  
 $w = 1/[\sigma^2(F_o^2)]$

$(\Delta/\sigma)_{\text{max}} = 0.02$   
 $\Delta\rho_{\text{max}} = 0.82 \text{ e \AA}^{-3}$   
 $\Delta\rho_{\text{min}} = -0.90 \text{ e \AA}^{-3}$   
 Extinction correction:  
 Larson (1970)  
 Extinction coefficient: 1424  
 Scattering factors from  
*International Tables for*  
*Crystallography* (Vol. C)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters ( $\text{\AA}^2$ )

	$x$	$y$	$z$	$U_{\text{eq}}$
Hg1	0	0	0	0.0320 (2)
Hg2	1/2	1/2	1/2	0.0288 (1)
Cl1	0.2619 (2)	0.2381 (2)	0.0074 (2)	0.0427 (7)
Cl2	0.7372 (2)	0.2422 (2)	0.0632 (2)	0.0425 (7)
Cl3	0.3485 (2)	0.4157 (2)	0.2958 (2)	0.0439 (7)
Cl4	-0.0603 (2)	-0.1730 (2)	0.2811 (2)	0.0439 (7)
N1	0.9500 (6)	0.4154 (8)	0.2405 (7)	0.044 (3)
C1†	0.800 (1)	0.367 (1)	0.396 (1)	0.044 (4)
C2†	0.756 (1)	0.165 (1)	0.479 (1)	0.040 (4)
C21‡	0.879 (6)	0.257 (8)	0.395 (6)	0.04 (2)
C22‡	0.682 (6)	0.283 (8)	0.459 (6)	0.04 (2)
C3	0.586 (1)	0.117 (1)	0.6180 (9)	0.057 (4)
N2	0.6058 (7)	0.1462 (9)	0.7572 (8)	0.054 (3)

† Site occupancy = 0.86 (1). ‡ Site occupancy = 0.14 (1).

Table 2. Selected geometric parameters ( $\text{\AA}, ^\circ$ )

Hg1—Cl4	2.397 (2)	Hg2—Cl3	2.528 (2)
Hg1—Cl2 <sup>i</sup>	2.821 (2)	Hg2—Cl2	2.701 (2)
Hg1—Cl1	2.839 (2)	Hg2—Cl1	2.775 (2)
Cl2 <sup>i</sup> —Hg1—Cl4	90.35 (6)	Cl1—Hg2—Cl3	88.69 (6)
Cl1—Hg1—Cl4	90.14 (6)	Cl1—Hg2—Cl2	90.61 (5)
Cl1—Hg1—Cl2 <sup>i</sup>	88.89 (5)	Hg1—Cl1—Hg2	174.33 (6)
Cl2—Hg2—Cl3	90.65 (6)	Hg1 <sup>ii</sup> —Cl2—Hg2	156.90 (9)

Table 4. Comparison of lattice parameters in related compounds

	Space group	$a$ ( $\text{\AA}$ )	$b$ ( $\text{\AA}$ )	$c$ ( $\text{\AA}$ )	$\beta$ ( $^\circ$ )	Layer separation	Reference
2D-CuCl <sub>4</sub>	$P2_1/a$	7.158	7.353	8.109	92.37	8.102	GEP
3D-CuCl <sub>4</sub>	$Pnma$	7.451	7.200	18.246	90	9.123	GEP
4D-CuCl <sub>4</sub>	$P2_1/a$	7.588	7.599	9.268	103.14	9.025	GEP
5D-CuCl <sub>4</sub>	$P2_1/n$	7.747	7.200	21.769	102.12	10.642	GEP
3D-HgCl <sub>4</sub>	$P\bar{1}$	7.788	7.796	8.470†	98.32†	8.367	This work
PA-CuCl <sub>4</sub>	$Pcab$	7.328	7.295	38.618	90	19.309	W
PA-CdCl <sub>4</sub>	$Pcab$	38.846	7.434	7.367	90	19.423	GSBZB

Notes:  $nD = n$ -alkanediammonium; PA = phenethylammonium; GEP = Garland *et al.* (1990); W = Willett (1990); GSBZB = Groh *et al.* (1997).

† The values for  $c$  and  $\beta$  cited for this work are related to a transformed nearly monoclinic isostructural cell (shown in Fig. 1).

The refined data set was cut because of primary beam collection at  $\sin\theta/\lambda < 0.074$ . The  $\bar{1}72$  reflection was omitted because of a measurement error. H-atom positions were refined using a riding model. The anisotropic displacement parameters of the C and N atoms were applied to the H atoms, multiplied by a factor of 1.2. Determination of the crystal size and shape was performed using a self-constructed automated two-circle goniometer with a CCD camera (Zimmermann *et al.*, 1995). Faces and distances (mm): (100) 0.193 (5), (100) 0.183 (5), (010) 0.300 (5), (010) 0.300 (5), (001) 0.120 (5), (00 $\bar{1}$ ) 0.023(5), (101) 0.175 (5), ( $\bar{1}0\bar{1}$ ) 0.175 (5), ( $1\bar{1}0$ ) 0.314 (5), ( $\bar{1}10$ ) 0.252 (5), ( $0\bar{1}1$ ) 0.217 (5), ( $\bar{1}12$ ) 0.185 (5), ( $112$ ) 0.213 (5), (025) 0.174 (5), (011) 0.294 (5), ( $0\bar{1}1$ ) 0.276 (5). The pseudo-centring vector due to the tetrachloromercurate anions resulted in an interesting triclinic Patterson map, which made it difficult to find the starting point for structure solution. When the Hg and Cl positions were calculated, the structure solution presented no further problems. The structure showed static disorder of the cation alkane chain. The refinement of a static disorder model, restraining the sum of the occupancy factors of chain C atoms to unity, remained stable and resulted in a value of 0.86 (1) for the restrained occupancy factor of the larger component (atoms C1 and C2; the minor components are C21 and C22). However, it was not possible to refine a split model of C3, since the positions are too close. In Fig. 1, it can be seen that the anisotropic displacement parameter of C3 in the split direction is increased, while the anisotropic displacement parameters for the N atoms show no obvious disorder. The residual density showed maxima and minima close to the Hg and Cl atoms.

All refinements were performed using a Levenberg–Marquardt least-squares model (Spengler & Zimmermann, 1998).

Data collection: local software (Gomm, 1993). Cell refinement: local software. Data reduction: local software. Program(s) used to solve structure: *CRYSTAN* (Burzlaff & Rothammel, 1988). Program(s) used to refine structure: *CRYSTAN*. Molecular graphics: *CRYSTAN*. Software used to prepare material for publication: *CRYSTAN*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: JZ1287). Services for accessing these data are described at the back of the journal.

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