Eu—O(*W*3)	2.484 (5)	Na—O(*W*5)	2.40 (1)
Eu—N(1)	2.695 (7)		
O(1)—Eu—O(3)	127.8 (2)	O(*W*1)—Eu—O(*W*2)	72.3 (2)
O(1)—Eu—O(5)	78.3 (2)	O(*W*1)—Eu—O(*W*3)	73.8 (2)
O(1)—Eu—O(7)	139.7 (2)	O(*W*1)—Eu—N(1)	130.1 (2)
O(1)—Eu—O(*W*1)	74.6 (2)	O(*W*1)—Eu—N(2)	130.9 (2)
O(1)—Eu—O(*W*2)	78.0 (2)	O(*W*2)—Eu—O(*W*3)	75.2 (2)
O(1)—Eu—O(*W*3)	143.4 (2)	O(*W*2)—Eu—N(1)	71.8 (2)
O(1)—Eu—N(1)	65.0 (2)	O(*W*2)—Eu—N(2)	138.6 (2)
O(1)—Eu—N(2)	77.7 (2)	O(*W*3)—Eu—N(1)	126.9 (2)
O(3)—Eu—O(5)	141.3 (2)	O(*W*3)—Eu—N(2)	138.1 (2)
O(3)—Eu—O(7)	70.9 (2)	N(1)—Eu—N(2)	67.7 (2)
O(3)—Eu—O(*W*1)	139.4 (2)	O(5$^{\rm i}$)—Na—O(7)	139.8 (3)
O(3)—Eu—O(*W*2)	79.8 (2)	O(5$^{\rm i}$)—Na—O(*W*1$^{\rm i}$)	68.3 (2)
O(3)—Eu—O(*W*3)	70.9 (2)	O(5$^{\rm i}$)—Na—O(*W*3)	77.8 (2)
O(3)—Eu—N(1)	63.2 (2)	O(5$^{\rm i}$)—Na—O(*W*5)	125.7 (3)
O(3)—Eu—N(2)	89.3 (2)	O(5$^{\rm i}$)—Na—O(*W*4)	114.9 (3)
O(5)—Eu—O(7)	71.7 (2)	O(7)—Na—O(*W*1$^{\rm i}$)	81.2 (2)
O(5)—Eu—O(*W*1)	68.3 (2)	O(7)—Na—O(*W*3)	70.5 (2)
O(5)—Eu—O(*W*2)	138.1 (2)	O(7)—Na—O(*W*4)	87.6 (4)
O(5)—Eu—O(*W*3)	106.2 (2)	O(7)—Na—O(*W*5)	84.9 (3)
O(5)—Eu—N(1)	126.2 (2)	O(*W*1$^{\rm i}$)—Na—O(*W*3)	78.0 (2)
O(5)—Eu—N(2)	66.9 (2)	O(*W*1$^{\rm i}$)—Na—O(*W*4)	163.3 (5)
O(7)—Eu—O(*W*1)	116.3 (2)	O(*W*1$^{\rm i}$)—Na—O(*W*5)	102.0 (4)
O(7)—Eu—O(*W*2)	141.8 (2)	O(*W*3)—Na—O(*W*4)	86.6 (4)
O(7)—Eu—O(*W*3)	72.5 (2)	O(*W*3)—Na—O(*W*5)	155.2 (3)
O(7)—Eu—N(1)	113.4 (2)	O(*W*4)—Na—O(*W*5)	89.3 (5)
O(7)—Eu—N(2)	66.1 (2)		
O(1)···O(*W*3$^{\rm ii}$)	2.764 (8)	O(8)···O(*W*7)	2.72 (1)
O(2)···O(*W*2$^{\rm ii}$)	2.696 (9)	O(*W*1)···O(*W*7$^{\rm v}$)	2.772 (8)
O(3)···O(*W*1$^{\rm i}$)	2.721 (8)	O(*W*3)···O(*W*7$^{\rm v}$)	2.719 (8)
O(4)···O(*W*2$^{\rm i}$)	2.684 (9)	O(*W*4)···O(*W*6$^{\rm iii}$)	2.80 (2)
O(4)···O(*W*6)	2.76 (1)	O(*W*4)···O(*W*8$^{\rm v}$)	2.71 (2)
O(6)···O(*W*6$^{\rm iii}$)	2.78 (1)	O(*W*7)···O(*W*8)	2.70 (2)
O(6)···O(*W*8$^{\rm iv}$)	2.96 (2)	O(*W*8)···O(*W*8$^{\rm vi}$)	2.69 (4)
O(8)···O(*W*5)	2.69 (1)		

Symmetry codes: (i) $\frac{1}{4}+x, \frac{3}{4}-y, z-\frac{1}{4}$; (ii) $x-\frac{1}{4}, \frac{3}{4}-y, \frac{1}{4}+z$; (iii) $x-\frac{1}{2}, y, z-\frac{1}{2}$; (iv) $1-x, \frac{1}{2}-y, \frac{1}{2}+z$; (v) $\frac{5}{4}-x, \frac{1}{4}+y, \frac{1}{4}+z$; (vi) $\frac{3}{2}-x, \frac{1}{2}-y, z$.

The structures were solved by Patterson and Fourier techniques using *DIRDIF* (Beurskens *et al.*, 1992). All non-H atoms were refined anisotropically (*SAPI*91; Fan, 1991). H atoms were not located. Data collection and cell refinement: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Data reduction: *TEXSAN* (Molecular Structure Corporation, 1993). Molecular graphics: *ORTEP*II (Johnson, 1976).

Lists of structure factors, anisotropic displacement parameters and bond distances and angles involving non-H atoms have been deposited with the IUCr (Reference: MU1164). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Phenylpiperazinium Trichloromercurate

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Abstract

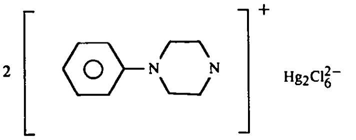
The structure of *N*-phenylpiperazinium trichloromercurate, $(C_{10}H_{15}N_2)[HgCl_3]$, consists of isolated bitetrahedral $[Hg_2Cl_6]^{2-}$ units, di-μ-chloro-bis(dichloromercury), sharing one edge, with two short bonds of 2.346 (7) and 2.365 (7) Å, and two long bonds of 2.852 (5) and 2.624 (7) Å. The organic cation, $(C_{10}H_{15}N_2)^+$, is located between the $[Hg_2Cl_6]^{2-}$ units. The phenylpiperazinium groups are connected to the Hg_2Cl_6 dimer by hydrogen bonds to the Cl atoms.

Comment

The reaction of substituted ammonium halogenides with metal halogenides leads to a series of compounds of the general formula RMX_3, with R being an alkyl or aryl substituent, M a divalent metal and X = Cl, Br, I.

These substances exhibit interesting structural and physical properties such as ferroelectricity {*e.g.* $[N(CH_3)_4][HgBr_3]$ (White, 1963) and (CH_3NH_3)-$[HgCl_3]$ (Ben Salah, Bats, Kalus, Fuess & Daoud, 1982)}.

Mercury(II) compounds exhibit a strong tendency to form almost linear *L*—Hg—*L* units. Crystal structures of this type with coordination number 2 are, therefore, the most familiar group; structures with coordination numbers 3 (trigonal pyramidal), 4 (tetrahedral), 5 (trigonal bipyramidal) and 6 (octahedral) are other common types of mercury(II) structures and complexes. The present work on the title compound, (I), is part of studies of the structures of complexes with cyclic diamines.



(I)

The structure of $(C_{10}H_{15}N_2)[HgCl_3]$, (I), consists of centrosymmetrical dimers, $[Hg_2Cl_6]^{2-}$, with two contiguous Hg atoms being bridged by two Cl atoms (Fig. 1). Each Hg atom is surrounded by four Cl atoms forming a tetrahedral structure in which there are two short Hg—Cl bonds to Cl(2) and Cl(3) subtending an angle of 148.5 (3)° and two long Hg—Cl bonds to Cl(1) and Cl(1^i) [symmetry code: (i) $1 - x, y, \frac{3}{2} - z$] subtending an angle of 91.9 (2)°. Similar coordination is found in $[As(C_6H_5)_4][HgCl_3]$ (Chaari, Ben Salah, Jouini & Daoud, 1989). The Hg—Cl bonds are similar to those found in the structure of (I). In (I), the cation and the anion are held together by a hydrogen bond, N—H···Cl (Fig. 2).

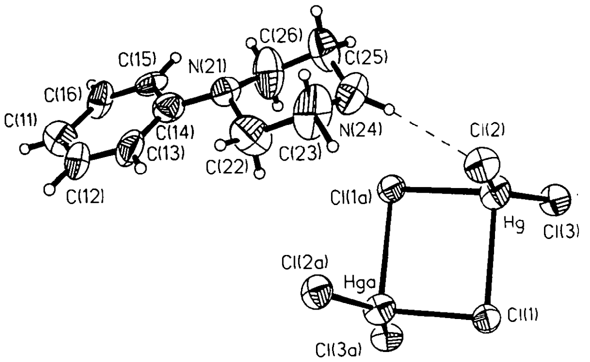


Fig. 1. The molecular structure of $(C_{10}H_{15}N_2)[HgCl_3]$ showing the atom labelling. The hydrogen bond is shown by a dashed line. Ellipsoids are drawn at 50% probability.

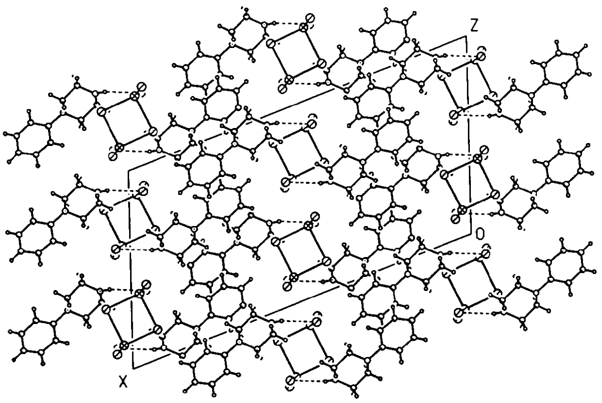


Fig. 2. Crystal packing projected along the *b* axis. Large circles represent Hg atoms (cross-hatched) or Cl atoms. C atoms are shown as circles. Hydrogen bonds are drawn as dashed lines.

Experimental

Single crystals of phenylpiperazinium trichloromercurate were obtained by mixing a solution of phenylpiperazinium chloride and mercury chloride in acetone–ethanol, in the stoichiometric ratio 2/1. Slow evaporation at room temperature leads to colourless prismatic crystals. Chemical analysis was in good agreement with the formula $C_{10}H_{15}Cl_3HgN_2$. The crystal density D_m was measured by pycnometry.

Crystal data

$(C_{10}H_{15}N_2)[HgCl_3]$
$M_r = 470.2$
Monoclinic
$C2/c$
$a = 25.975$ (3) Å
$b = 8.013$ (2) Å
$c = 14.051$ (2) Å
$\beta = 110.34$ (2)°
$V = 2742.2$ (6) Å^3
$Z = 8$
$D_x = 2.278$ Mg m^{-3}
$D_m = 2.261$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 5–15°$
$\mu = 11.785$ mm^{-1}
$T = 299$ K
Prismatic
0.43 × 0.35 × 0.16 mm
Colourless

Data collection

Siemens *P*4 diffractometer
$\omega/2\theta$ scans
Absorption correction: analytical
$T_{min} = 0.0828$, $T_{max} = 0.1758$
2509 measured reflections
2414 independent reflections
1112 observed reflections [$F > 6.0\sigma(F)$]

$R_{int} = 0.0177$
$\theta_{max} = 25°$
$h = 0 \rightarrow 30$
$k = 0 \rightarrow 9$
$l = -16 \rightarrow 15$
3 standard reflections monitored every 250 reflections
intensity decay: 5.2%

Refinement

Refinement on F
$R = 0.0529$
$wR = 0.0615$
$S = 1.14$
1112 reflections
146 parameters
H atoms refined as riding, fixed U_{iso}
$w = 1/[\sigma^2(F) + 0.0010F^2]$
$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max} = 1.76$ e Å^{-3}
$\Delta\rho_{min} = -1.31$ e Å^{-3}
Extinction correction: $F^* = F[1 + (0.002\chi \times F^2/\sin 2\theta)]^{-1/4}$
Extinction coefficient: $\chi = 0.00034$ (3)
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
Hg	0.5236 (1)	−0.3888 (1)	0.6359 (1)	0.050 (1)
Cl(1)	0.5748 (2)	−0.3931 (9)	0.8514 (4)	0.046 (2)
Cl(2)	0.5417 (3)	−0.1002 (9)	0.6315 (5)	0.050 (3)
Cl(3)	0.5441 (3)	−0.6636 (8)	0.6038 (5)	0.049 (3)
C(11)	0.179 (1)	0.134 (3)	0.686 (2)	0.059 (1)
C(12)	0.229 (1)	0.209 (4)	0.731 (2)	0.083 (2)
C(13)	0.273 (1)	0.199 (3)	0.699 (2)	0.061 (1)
C(14)	0.2680 (9)	0.109 (3)	0.612 (2)	0.050 (1)
C(15)	0.2171 (9)	0.028 (3)	0.560 (2)	0.043 (10)
C(16)	0.1747 (10)	0.040 (3)	0.598 (2)	0.053 (1)
N(21)	0.3121 (7)	0.094 (2)	0.575 (1)	0.040 (8)
C(22)	0.364 (1)	0.122 (4)	0.649 (2)	0.078 (1)
C(23)	0.411 (2)	0.133 (4)	0.604 (2)	0.081 (1)
N(24)	0.4084 (10)	−0.016 (3)	0.545 (2)	0.069 (1)
C(25)	0.355 (1)	−0.045 (4)	0.467 (2)	0.074 (1)
C(26)	0.311 (1)	−0.054 (4)	0.513 (3)	0.088 (2)

Table 2. *Selected geometric parameters* (Å, °)

Hg—Cl(1)	2.852 (5)	Hg—Cl(2)	2.365 (7)
Hg—Cl(3)	2.346 (7)	Hg—Cl(1$^{\rm i}$)	2.624 (7)
Cl(1)—Hg$^{\rm i}$	2.624 (7)	C(11)—C(12)	1.37 (4)
C(11)—C(16)	1.42 (4)	C(12)—C(13)	1.35 (5)
C(13)—C(14)	1.38 (4)	C(14)—C(15)	1.42 (3)
C(14)—N(21)	1.42 (3)	C(15)—C(16)	1.38 (4)
N(21)—C(22)	1.41 (3)	N(21)—C(26)	1.47 (4)
C(22)—C(23)	1.55 (5)	C(23)—N(24)	1.44 (4)
N(24)—C(25)	1.45 (3)	C(25)—C(26)	1.48 (5)
Cl(1)—Hg—Cl(2)	90.9 (2)	Cl(1)—Hg—Cl(3)	98.0 (2)
Cl(2)—Hg—Cl(3)	148.5 (3)	Cl(1)—Hg—Cl(1$^{\rm i}$)	91.9 (2)
Cl(2)—Hg—Cl(1$^{\rm i}$)	102.8 (2)	Cl(3)—Hg—Cl(1$^{\rm i}$)	107.0 (2)
Hg—Cl(1)—Hg$^{\rm i}$	88.1 (2)	C(12)—C(11)—C(16)	113.8 (3)
C(11)—C(12)—C(13)	126.7 (3)	C(12)—C(13)—C(14)	118.6 (2)
C(13)—C(14)—C(15)	118.7 (2)	C(13)—C(14)—N(21)	121.4 (2)
C(15)—C(14)—N(21)	119.9 (2)	C(14)—C(15)—C(16)	119.6 (2)
C(11)—C(16)—C(15)	122.5 (2)	C(14)—N(21)—C(22)	114.6 (2)
C(14)—N(21)—C(26)	116.3 (2)	C(22)—N(21)—C(26)	111.7 (2)
N(21)—C(22)—C(23)	113.9 (2)	C(22)—C(23)—N(24)	106.7 (2)
C(23)—N(24)—C(25)	114.3 (2)	N(24)—C(25)—C(26)	111.1 (2)
N(21)—C(26)—C(25)	111.3 (2)		

Symmetry code: (i) $1 - x, y, \frac{3}{2} - z$.

Preliminary oscillation and Weissenberg photographs showed a monoclinic crystal with systematic absences for the chosen cell consistent with the space group *C*2/*c*. Data reduction, structure solution and refinement were carried out using *SHELXTL/PC* (Sheldrick, 1993). The structure was solved by direct methods and refined successfully in the space group *C*2/*c*. Full-matrix least-squares refinement was carried out by minimizing $\Sigma w(F_o - F_c)^2$. The non-H atoms were refined anisotropically whereas H atoms were fixed isotropically to convergence. One H atom was not included.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: NA1108). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Tetrakis(μ-DL-alanine-*O*:*O'*)octaaquadierbium(III) Hexaperchlorate

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Abstract

The structure of the title compound, $[\mathrm{Er_2(C_3H_7NO_2)_4(H_2O)_8](ClO_4)_6}$, consists of dimeric $[\mathrm{Er_2(DL\text{-}alanine)_4(H_2O)_8}]^{6+}$ cations and perchlorate anions. The four alanine molecules act as bridging ligands linking two $\mathrm{Er^{3+}}$ ions through their carboxyl O atoms. Each $\mathrm{Er^{3+}}$ ion is also coordinated by four water molecules to complete eightfold coordination in a square antiprism fashion. The perchlorate anions and the methyl groups of the alanine ligands are disordered.

Comment

Structural investigations of rare earth ion–amino acid complexes are of interest with respect to both the understanding of the biological effects of rare earth elements and the enrichment of lanthanide coordination chemistry. Previous studies have revealed that in these complexes the amino acid ligands are bonded to the rare earth ions ($\mathrm{Ln^{3+}}$) through their carboxyl groups, while the amino groups are protonated and are not involved in the metal-ion coordination. The complexes usually adopt one of three types of structure, namely, dinuclear dimer, chain or network polymer. We report here the structure of an erbium complex with DL-alanine, (I), as part of crystallographic studies of rare earth ion–amino acid complexes.

The title compound crystallizes in the triclinic form having a dimeric unit similar to that in its monoclinic form reported by Zeng & Pan (1992). The disorder of the ligands and the perchlorate anions in the present structure could be responsible for the change in crystalline form.

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