

Data collection

Enraf–Nonius CAD-4T 3215 reflections with
 diffractometer on rotating $I > 2\sigma(I)$
 anode source $R_{\text{int}} = 0.026$
 ω scans $\theta_{\text{max}} = 27.42^\circ$
 Absorption correction: $h = -6 \rightarrow 9$
 ψ scan (North, Phillips $k = 0 \rightarrow 22$
 & Mathews, 1968) $l = -17 \rightarrow 17$
 $T_{\text{min}} = 0.600$, $T_{\text{max}} = 0.670$ 3 standard reflections
 4026 measured reflections frequency: 60 min
 3703 independent reflections intensity decay: 5%

Refinement

Refinement on F^2 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $R(F) = 0.029$ $\Delta\rho_{\text{max}} = 0.41 \text{ e } \text{\AA}^{-3}$
 $wR(F^2) = 0.071$ $\Delta\rho_{\text{min}} = -0.34 \text{ e } \text{\AA}^{-3}$
 $S = 1.066$ Extinction correction: none
 3703 reflections Scattering factors from
 240 parameters *International Tables for*
 H atoms: see below *Crystallography* (Vol. C)
 $w = 1/[\sigma^2(F_o^2) + (0.0471P)^2$
 $+ 1.1696P]$
 where $P = (F_o^2 + 2F_c^2)/3$

Table 1. Selected geometric parameters (\AA , $^\circ$)

Ni1—S1	2.1519 (6)	S2—C20	1.736 (2)
Ni1—S2	2.1587 (6)	N1—C7	1.302 (3)
Ni1—N1	1.8992 (17)	N1—C8	1.436 (3)
Ni1—N2	1.9014 (15)	N2—C13	1.432 (2)
S1—C1	1.728 (2)	N2—C14	1.297 (3)
S1—Ni1—S2	81.85 (2)	S2—Ni1—N1	168.99 (5)
S1—Ni1—N1	97.08 (5)	S2—Ni1—N2	96.78 (5)
S1—Ni1—N2	168.93 (5)	N1—Ni1—N2	86.34 (7)

The crystal used for data collection was cut to size in inert oil from a cluster of cube-shaped crystals. Attempts to use a face-indexed correction for absorption (de Meulenaer & Tompa, 1965) were unsatisfactory in detail but indicated a transmission range of 0.54–0.67. A more reasonable correction was possible on the basis of three close-to-axial ψ scans with the North, Phillips & Mathews (1968) method as implemented in *PLATON97* (Spek, 1990). The correction range was scaled on the value of T_{max} given above. H atoms were taken into account at calculated positions (restrained to C—H = 0.95 \AA) and refined riding on their carrier atoms with $U_{\text{iso}} = 1.2U_{\text{eq}}$ of their attached atom. The structure was checked for possible higher symmetry using the *MISSYM* algorithm as implemented in *PLATON97*. The molecular twofold axis does not coincide with a lattice translation.

Data collection: locally modified *CAD-4 Software* (Enraf–Nonius, 1989). Cell refinement: *SET4* (de Boer & Duisenberg, 1984). Data reduction: *HELENA* (Spek, 1997). Program(s) used to solve structure: *DIRDIF96* (Beurskens *et al.*, 1996). Program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997). Molecular graphics: *PLATON97*. Software used to prepare material for publication: *PLATON97*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: BM1141). Services for accessing these data are described at the back of the journal.

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Bis(4-benzylpiperidinium) Tetrachloro-mercurate

ROLAND SPENGLER,^a FATMA ZOUARI,^b ABDELHAMID BEN SALAH^b AND HANS BURZLAFF^a

^a*Institut für Angewandte Physik, Lehrstuhl für Kristallographie, Universität Erlangen-Nürnberg, Bismarckstraße 10, D-91054 Erlangen, Germany, and* ^b*Faculté des Sciences, Département de Chimie, Université de Sfax, Rte. de Soukra, 3038 Sfax, Tunisia. E-mail: roland.spengler@krist.uni-erlangen.de*

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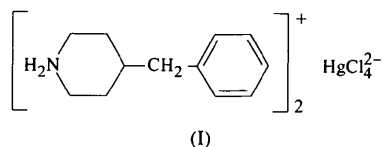
Abstract

The title compound, (C₁₂H₁₈N)₂[HgCl₄], contains a distorted tetrachloromercurate tetrahedron, with Hg—Cl distances ranging from 2.424 (3) to 2.614 (2) \AA .

Variations can be attributed to the presence of hydrogen bonding. The anions and cations are connected by N—H...Cl bonds, building a two-dimensional network.

Comment

The title structure forms part of our investigation into a class of compounds showing interesting structural and physical properties (Spengler, Zouari, Ben Salah & Burzlaff, 1997). The present compound, (I), is an example of a four-coordinated Hg atom.



The title compound contains two benzylpiperidinium cations and one HgCl_4^{2-} anion in the asymmetric unit (Fig. 1). The Hg atom is surrounded by four Cl atoms forming a distorted tetrahedron. The distribution of the Hg atoms forms a nearly A-centred cell. The tetrahedra are connected into a two-dimensional graphite-like network close to the (0yz) plane by hydrogen bonds *via* the NH_2 groups of the piperidinium ring (Fig. 2 and Table 2). The significantly longer Hg—Cl11 distance of 2.614 (2) Å is related to two short H...Cl hydrogen-bond distances (2.17 and 2.29 Å). The Hg—Cl14 distance of 2.450 (3) Å is related to one short and one longer hydrogen-bond distance (2.22 and 2.70 Å).

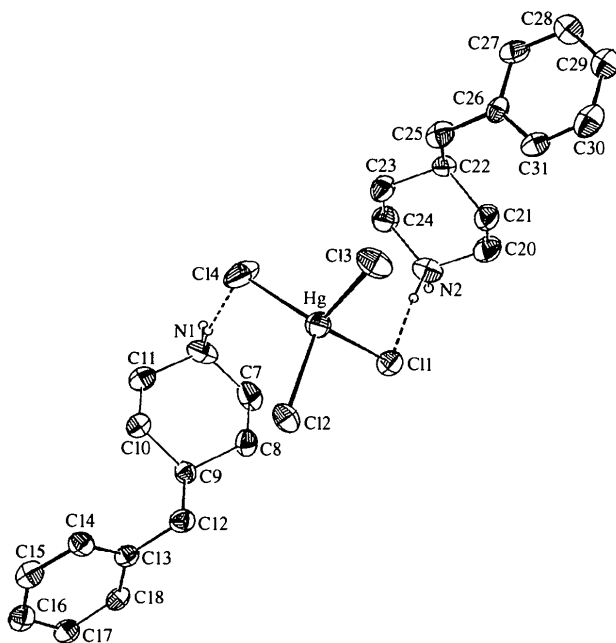


Fig. 1. Formula unit of the title compound in the crystal. Displacement ellipsoids correspond to 35% probability.

The distortion of the tetrahedron is more marked than in other related compounds (Spengler, Zouari, Ben Salah & Burzlaff, 1997; Ben Salah, Bats, Fuess & Daoud, 1983; Ben Salah, Bats, Kalus, Fuess & Daoud, 1982).

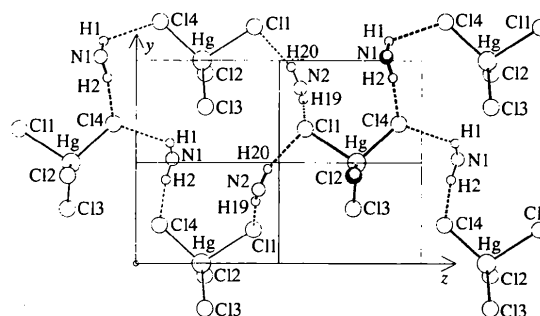


Fig. 2. Hydrogen contacts plotted along the [100] direction; only the NH_2 groups of the cations are shown. Spheres are shown with arbitrary radii.

Experimental

The title complex was obtained by mixing benzylpiperidinium chloride and mercury chloride (stoichiometric ratio 2:1) in an acetone-ethanol solution. After a few weeks, non-hygroscopic crystals of (I) are obtained by slow evaporation. The chemical analysis agrees with the formula. The determination of the crystal size and shape was performed using a self-constructed automated two-circle goniometer with a CCD camera (Zimmermann, Lange & Burzlaff, 1995).

Crystal data

$(\text{C}_{12}\text{H}_{18}\text{N})_2[\text{HgCl}_4]$

$M_r = 694.97$

Monoclinic

$P2_1$

$a = 13.603 (1) \text{ \AA}$

$b = 8.383 (1) \text{ \AA}$

$c = 12.198 (1) \text{ \AA}$

$\beta = 97.66 (1)^\circ$

$V = 1378.5 (2) \text{ \AA}^3$

$Z = 2$

$D_x = 1.674 \text{ Mg m}^{-3}$

D_m not measured

Mo $K\alpha$ radiation

$\lambda = 0.7107 \text{ \AA}$

Cell parameters from 24 reflections

$\theta = 6.1\text{--}17.9^\circ$

$\mu = 5.987 \text{ mm}^{-1}$

$T = 293 \text{ K}$

Prismatic plate

$0.33 (1) \times 0.23 (1) \times 0.12 (1)$

Colourless

Data collection

Rebuilt Philips PW1100 diffractometer (Gomm, 1993)

$\theta/2\theta$ scans

Absorption correction:

Gaussian, by integration (Busing & Levy, 1957)

$T_{\min} = 0.300$, $T_{\max} = 0.513$

4896 measured reflections

4342 independent reflections

3928 reflections with

$F > 2\sigma(F)$

$R_{\text{int}} = 0.048$

$\theta_{\text{max}} = 24.0^\circ$

$h = 0 \rightarrow 15$

$k = -9 \rightarrow 9$

$l = -13 \rightarrow 13$

4 standard reflections

every 100 reflections

intensity decay: 3.6%

Refinement

Refinement on F $R = 0.040$ $wR = 0.040$ $S = 1.45$

3928 reflections

280 parameters

H atoms not refined

 $w = 1/[\sigma^2(F_{obs}) + 0.02F_{obs}]$ $(\Delta/\sigma)_{max} = 0.01$ $\Delta\rho_{max} = 1.17 e \text{ \AA}^{-3}$ $\Delta\rho_{min} = -0.93 e \text{ \AA}^{-3}$

Extinction correction: none

Scattering factors from *International Tables for X-ray Crystallography* (Vol. IV)

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Table 1. Selected geometric parameters (\AA , $^\circ$)

Hg—Cl3	2.424 (3)	Hg—Cl4	2.450 (3)
Hg—Cl2	2.430 (3)	Hg—Cl1	2.614 (2)
Cl2—Hg—Cl3	115.39 (9)	Cl1—Hg—Cl3	107.7 (1)
Cl4—Hg—Cl3	115.8 (1)	Cl1—Hg—Cl2	99.18 (8)
Cl4—Hg—Cl2	112.3 (1)	Cl1—Hg—Cl4	104.16 (9)

Table 2. Hydrogen-bonding geometry (\AA , $^\circ$)

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
N1—H1 \cdots Cl4 ⁱ	0.96	2.70	3.342 (4)	125
N1—H2 \cdots Cl4	0.96	2.22	3.159 (6)	166
N2—H19 \cdots Cl1	0.96	2.17	3.131 (4)	174
N2—H20 \cdots Cl1 ⁱⁱ	0.96	2.29	3.207 (4)	159

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

The origin was fixed by setting the y coordinate of the Hg atom to zero. The absolute structure was determined by anomalous dispersion; the opposite enantiomorph resulted in a weighted R value of 0.085. All H-atom positions could be determined from the difference Fourier map. However, the calculated positions at a distance of 0.96 \AA were used in the final refinement, with a common isotropic displacement parameter, since the calculated distances were more reasonable than the refined ones. The residual density shows six maxima with values ranging from 1.0 to 1.2 $e \text{ \AA}^{-3}$ close to the Hg atom at distances of 1.1 to 1.2 \AA . A similar situation is found for the minima. All refinements were performed using a Levenberg–Marquardt least-squares technique (Spengler, Zimmermann & Burzlaff, 1994; Zimmermann & Spengler, 1995; Spengler & Zimmermann, 1996).

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* (Spengler & Zimmermann, 1996). Molecular graphics: *CRYSTAN* (Burzlaff & Rothammel, 1988). Software used to prepare material for publication: *CRYSTAN* (Burzlaff & Rothammel, 1988).

Supplementary data for this paper are available from the IUCr electronic archives (Reference: JZ1221). Services for accessing these data are described at the back of the journal.

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A New Gallium Phosphate Templated by Tris(2-aminoethyl)amine: $[Ga(HPO_4)(PO_4)(OH)] \cdot [(C_2H_7N)_3N] \cdot H_2O$

FABIEN SERPAGGI, THIERRY LOISEAU AND GÉRARD FÉREY

Institut Lavoisier – UMR CNRS 173, Université de Versailles Saint-Quentin-en-Yvelines, 78035 Versailles CEDEX, France.
E-mail: loiseau@chimie.uvsq.fr

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Abstract

The title compound, bis(μ -phosphato- $O:O'$)bis[(hydrogenphosphato- O)(hydroxo)gallium] tris(2-aminoethyl)amine hydrate, was hydrothermally synthesized, in the presence of tris(2-aminoethyl)amine (tren), from a non-aqueous mixture in which dimethyl sulfoxide was used as solvent (423 K, 24 h, autogeneous pressure). Its structure consists of isolated hexameric units composed of two GaO_4 and four PO_4 tetrahedra, these units being separated by the tren and water molecules.

Comment

Since the synthesis of a new series of microporous aluminium phosphates (Wilson, Lok, Messing, Cannan & Flanigen, 1982), a large number of novel three-dimensional open-framework compounds has been characterized. Phosphate-based materials are generally prepared from aqueous hydrothermal systems in the presence of an organic amine or a quaternary ammonium as a templating agent. Recent works report the synthesis from organic or mixed solvents in which the water is a minor component (Huo & Xu, 1990). The control of the water content in combination with the use of both a non-aqueous solvent and a mineralizing agent has led to large-crystal materials with dimensions in the size range 0.4–5.0 mm (Kuperman, Nadimi, Oliver, Ozin, Garcés & Olken, 1993). These studies mainly concern the use of alcoholic solvents such as glycols or linear-alkyl alco-