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The Crystal Structure of $Hg_6O_4Cl_4$

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The structure of $Hg_6O_4Cl_4$ has been determined by symbolic addition and refined to $R = 0{\cdot}059$ for 1859 counter intensities. The crystals are monoclinic, space group $P2_1/c$, with $a = 10{\cdot}838$ (2), $b = 9{\cdot}317$ (2), $c = 11{\cdot}564$ (2) Å, $\beta = 108{\cdot}90$ (1)°, $Z = 4$. The asymmetric part of the unit cell contains six Hg atoms; four of them are two-coordinated in a nearly linear way [Hg–O: 2·03 (2)–2·10 (2), Hg–Cl: 2·364 (8) Å, O–Hg–O: 167·7 (8)–175·1 (7), O–Hg–Cl: 158·9 (5)°] and two are (2 + 1)-coordinated [Hg–O: 2·08 (2)–2·18 (2), 2·44 (2), 2·47 (2) Å]. All Hg and O atoms and one quarter of the Cl atoms form endless bands of the formula $[Hg_2(OHg)_4Cl^{3+}]_n$. The remaining Cl atoms are located in cavities in the structure at distances Hg–Cl ≥ 2·75 Å, indicating that the bonding is mainly ionic. The formula of the compound may be written $[Hg_2(OHg)_4Cl]Cl_3$. The stability relationships between $Hg_6O_4Cl_4$ and the previously investigated $Hg_3O_2Cl_2$ are studied. Solubility measurements indicate that $Hg_6O_4Cl_4$ is the stable phase at 25 °C. When heated to about 200 °C *in vacuo* the reaction $2Hg_3O_2Cl_2(s) \rightarrow Hg_6O_4Cl_4(s) \rightarrow Hg_5O_4Cl_2(s) + HgCl_2(s)$ takes place and above 270 °C, $Hg_5O_4Cl_2(s) \rightarrow 4HgO(s) + HgCl_2(s)$.

Introduction

On hydrolysis of dilute aqueous solutions of $HgCl_2$ by marble (Arctowski, 1895) at least four different compounds nHgO.$HgCl_2$ are formed, *viz.* those with $n = \frac{1}{2}$, 2 (two modifications) and 3. The present study deals with the structure of a compound with $n = 2$ but of the formula $Hg_6O_4Cl_4$ and may be the same compound as reported by Aurivillius (1954) with the formula $Hg_5O_4Cl_2$. The structures of the phases with $n = \frac{1}{2}$ (Hg_3OCl_4) and $n = 2$ ($Hg_3O_2Cl_2$) have been reported (Aurivillius, 1964*a*; Aurivillius & Stålhandske, 1974). A preliminary report of the present structure has been given (Aurivillius, 1971).

Experimental

Black, irregular crystals (dark red in transmitted light) were picked out from the faces of the marble stones. A preliminary single-crystal investigation by Weissenberg methods showed systematic absences $h0l$, $l \neq 2n$, and $0k0$, $k \neq 2n$, indicating space group $P2_1/c$. The formula was assumed to be $Hg_5O_4Cl_2$ and the structure was tackled by symbolic addition. The E map revealed, besides five high maxima, a sixth of nearly the same height, suggesting the presence of six Hg atoms. Least-squares refinements followed by difference maps revealed Cl and O atoms to give the formula $Hg_6O_4Cl_4$. A final refinement converged at $R = 0{\cdot}10$ (Mo $K\alpha$ radiation, 1157 independent reflexions). At that point new preparations of the compound were made. First $Hg_3O_2Cl_2$ was treated with 0·1 M HNO_3 (Weiss, Nagorsen & Weiss, 1954). The product consisted of two phases, one identical with that described by the previous authors as 4HgO.$HgCl_2$, isotypic with $Hg(OHg)_4Br_2$ (Aurivillius, 1965), the other identical with $Hg_6O_4Cl_4$. Then a mixture of HgO and $HgCl_2$ in the molar ratio 2:1, moistened with water, was heated in a gold capsule at 3·0 kbar and 250 °C for one week. The product was homogeneous and consisted of brilliant dark-red, small crystals identical with $Hg_6O_4Cl_4$. A new set of data was collected from a triangular plate of volume 0·00074 mm^3 on an Enraf–Nonius CAD-4 diffractometer equipped with a graphite monochromator and Mo $K\alpha$ radiation ($\lambda = 0{\cdot}71069$ Å). The intensities of 2534 unique reflexions with $3 < \theta < 27{\cdot}5°$ were collected with the ω–2θ scan technique and a scan interval $\Delta\omega = (0{\cdot}90 + 0{\cdot}50 \tan\theta)°$. The background was measured for one quarter of the scan time at each end of the interval. Two standard reflexions were measured at regular intervals. No systematic variation was detected. 675 reflexions with $I < 3\sigma(I)$, where $\sigma(I)$ was based on counting statistics, were considered as unobserved and deleted. The remaining 1859 reflexions were corrected for Lorentz, polarization and absorption effects. Transmission factors, evaluated by the numerical method, varied from 0·015 to 0·066. The cell dimensions were determined from powder photographs taken in a Guinier–Hägg focusing camera with Cu $K\alpha_1$ radiation ($\lambda = 1{\cdot}54051$ Å) and KCl ($a = 6{\cdot}2928$ Å at 22 °C) as an internal standard. Refinement

Table 1. *Crystal data*

$Hg_6O_4Cl_4$, FW 1409·4
Monoclinic, $P2_1/c$, $a = 10{\cdot}838$ (2), $b = 9{\cdot}317$ (2), $c = 11{\cdot}564$ (2) Å
$\beta = 108{\cdot}90$ (1)°, $V = 1104{\cdot}7$ Å^3, $Z = 4$
$D_m = 8{\cdot}44$, $D_x = 8{\cdot}47$ g cm^{-3}, μ(Mo $K\alpha$) = 857 cm^{-1}

of the cell parameters was by least-squares calculations. Some crystal data are given in Table 1.

Structure refinement

The structure derived from the preliminary data set was refined by full-matrix least-squares calculations with anisotropic temperature factors for Hg and Cl and isotropic for O. The function minimized was $\Sigma w_i(|F_o| - |F_c|)^2$, where $w_i^{-1} = \sigma^2(|F_o|^2)/4|F_o|^2 + 0{\cdot}002|F_o|^2$. A final refinement converged to $R = 0{\cdot}059$, $R_w = 0{\cdot}071$ and $g = 0{\cdot}13\,(2) \times 10^4$ where g is the extinction parameter. The averages of $w_i(|F_o| - |F_c|)^2$ were approximately constant as a function of both $|F_o|$ and $\sin\theta$. The value of S, defined by $S = [\Sigma w_i(|F_o| - |F_c|)^2/(m - n)]^{1/2}$, where m and n are the numbers of observations and parameters varied, was 1·31. A final difference map did not indicate any further atoms. The form factors were those of Doyle & Turner (1968) for neutral Hg, Cl and O. Anomalous dispersion corrections for Hg and Cl (Cromer & Liberman, 1970) were included. Final values of positional and thermal parameters are given in Table 2,* selected interatomic distances and angles in Table 3.

Description and discussion

The asymmetric part of the unit cell contains six Hg atoms, all of point symmetry 1. Of these, Hg(1), Hg(2), Hg(3) and Hg(6) are two-coordinated. Hg(1), Hg(2) and Hg(3) are each bonded to two O atoms in a nearly linear mode with Hg–O between 2·03 (2) and 2·10 (2) Å and O–Hg–O angles between 167·7 (8) and 175·1 (7)°. Hg(6) also coordinates two atoms approximately linearly, an O atom at 2·10 (2) and a Cl atom at 2·36 (1) Å, the angle O–Hg–Cl being 158·9 (5)°. The next nearest neighbour to Hg(2), Hg(3) and Hg(6) is an O atom at a distance ≥2·59 Å, to Hg(1) a Cl atom at 2·94 (1) Å. The short Hg–O distances (2·03–2·10 Å) are in good agreement with corresponding distances for linear coordination of Hg in, for example, HgO(orth.) [Hg–O 2·04 (3), 2·07 (3) Å, O–Hg–O 179·5 (1·1)°; Aurivillius, 1964*b*]. The distance Hg–Cl can be compared with, for example, 2·29 (2) Å for Hg–2Cl in $HgCl_2$ (Akishin, Spiridonov & Khodchenkov, 1959), 2·30 (2) Å for Hg–Cl in Hg_3OCl_4 (Aurivillius, 1964*a*),

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 32952 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Table 2. *Fractional atomic coordinates*

$P2_1/c$; all parameters are in 4(*e*). Isotropic thermal parameters are in the form $\exp[-8\pi^2 U(\sin\theta/\lambda)^2]$. Standard deviations are given in parentheses. All values have been multiplied by 10^4.

	x	*y*	*z*	
Hg(1)	9261 (1)	521 (1)	7231 (1)	
Hg(2)	1927 (1)	176 (1)	5758 (1)	
Hg(3)	3901 (1)	1073 (1)	8699 (1)	
Hg(4)	2808 (1)	991 (1)	1160 (1)	
Hg(5)	9260 (1)	1744 (1)	35 (1)	
Hg(6)	5423 (1)	1928 (1)	6276 (1)	
	x	*y*	*z*	*U* (Å²)
Cl(1)	3607 (7)	340 (8)	3747 (6)	217 (14)
Cl(2)	1244 (7)	2609 (8)	3885 (7)	222 (14)
Cl(3)	6409 (7)	2350 (8)	8763 (7)	245 (15)
Cl(4)	8144 (7)	1273 (8)	1879 (7)	230 (14)
O(1)	9414 (17)	1064 (20)	5549 (16)	112 (35)
O(2)	3322 (20)	1498 (23)	6825 (18)	198 (42)
O(3)	4480 (16)	1127 (19)	613 (15)	96 (34)
O(4)	1027 (18)	145 (21)	1141 (17)	141 (36)

Table 3. *Selected interatomic distances* (Å) *and angles* (°) *with standard deviations in parentheses*

The prime denotes atoms symmetry-related to those within one single chain (Fig. 2).

Within the endless bands

Distances		Angles O–Hg–O	
Hg(1)–O(1)	2·068 (18)	O(1)–Hg(1)–O(4)	175·1 (7)
Hg(1)–O(4)	2·099 (18)		
Hg(2)–O(2)	2·028 (21)	O(1)–Hg(2)–O(2)	171·4 (8)
Hg(2)–O(1)	2·076 (18)		
Hg(2)–O(1′)	2·781 (18)		
Hg(3)–O(2)	2·089 (20)	O(2)–Hg(3)–O(3)	167·7 (8)
Hg(3)–O(3)	2·097 (17)		
Hg(3)–O(3′)	2·648 (17)		
Hg(4)–O(4)	2·078 (19)	O(3)–Hg(4)–O(4)	154·9 (7)
Hg(4)–O(3)	2·108 (17)	O(2′)–Hg(4)–O(4)	118·6 (7)
Hg(4)–O(2′)	2·469 (22)	O(2′)–Hg(4)–O(3)	84·7 (7)
Hg(5)–O(1)	2·119 (18)	O(1)–Hg(5)–O(4)	159·0 (7)
Hg(5)–O(4)	2·184 (19)	O(1)–Hg(5)–O(4′)	117·7 (7)
Hg(5)–O(4′)	2·436 (19)	O(4)–Hg(5)–O(4′)	76·3 (7)
Hg(6)–O(3)	2·100 (18)	O(3)–Hg(6)–Cl(1)	158·9 (5)
Hg(6)–O(2′)	2·586 (20)		
Hg(6)–Cl(1)	2·364 (8)	Angles Hg–O–Hg	
Hg–Hg	≥3·339 (2)	Hg(1)–O(1)–Hg(2)	107·4 (8)
O–O	≥2·861 (36)	Hg(1)–O(1)–Hg(5)	118·4 (8)
Cl(1)–O	≥3·387 (17)	Hg(2)–O(1)–Hg(5)	111·0 (8)
		Hg(2)–O(2)–Hg(3)	115·5 (1·0)
		Hg(3)–O(3)–Hg(4)	108·9 (8)
		Hg(3)–O(3)–Hg(6)	110·5 (8)
		Hg(4)–O(3)–Hg(6)	107·8 (8)
		Hg(4)–O(4)–Hg(1)	120·7 (9)
		Hg(1)–O(4)–Hg(5)	106·7 (8)
		Hg(5)–O(4)–Hg(4)	104·9 (8)

To atoms outside the bands

Shortest distances

Hg(4)–Cl(2)	2·934 (7)	Hg(5)–Cl(4)	2·805 (7)
Hg(6)–Cl(3)	2·753 (8)	Cl(1)–Cl(2)	3·364 (7)

and 2·383 (1) Å for Hg–2Cl in $HgCl_2.2KCl.H_2O$ (Aurivillius & Stålhandske, 1976).

Hg(4) and Hg(5) are also bonded to two O atoms at short distances, 2·08 (2)–2·18 (2) Å (Table 3), but the next nearest neighbours are in each case an O atom at a distance which may imply a (2 + 1)-coordination, the longer distances being 2·47 (2) and 2·44 (2) Å. The Hg–O distances, of 2·4–2·5 Å, are in the same range as, for example, the long Hg–O distance 2·324 (14) Å reported for a (1 + 2)-coordination in $Hg_3O_2Cl_2$ (Aurivillius & Stålhandske, 1974) and the Hg–O distances 2·32 (1)–2·38 (1) Å found for octahedral Hg–O coordination in $\{Hg[(CH_3)_2SO]_6\}(ClO_4)_2$ (Sandström & Persson, 1977). Outside the inner coordination spheres of Hg(4) and Hg(5), in each case three Cl atoms are located at distances indicating that the bonding is probably ionic [Hg–Cl 2·805 (7)–3·221 (8) Å]. The best way to describe the arrangements around Hg(4) and Hg(5) is to assume a (2 + 1)-coordination.

Hg(4) and its closest neighbours, the three O atoms, lie nearly in the same plane. The deviation of the Hg atom from the plane through the O atoms is 0·15 Å. The values of the angles O–Hg–O (Table 3) deviate considerably from the ideal value, 120°, for sp^2 hybridization. Hg(5) is located 0·91 Å from the corresponding plane through its three O atoms. For the coordination Hg–3O in $Hg_3O_2Cl_2$ the distance between Hg and the plane through the O atoms is 0·29 Å. The deviations of the angles O–Hg–O from 120° are also in this case considerable.

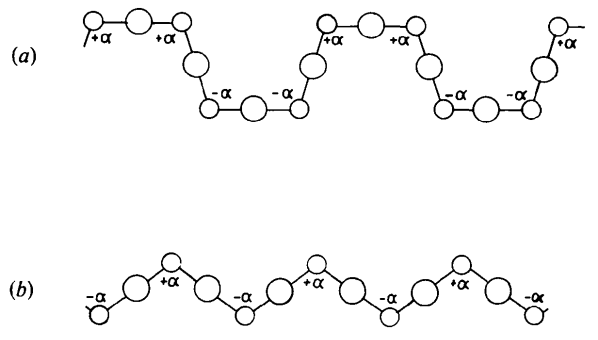


Fig. 1. Infinite chains –O–Hg–O–. (*a*) An endless planar chain, denoted a + + – – chain with respect to the signs of α, the angle Hg–O–Hg. (*b*) An endless planar chain, in the same way denoted a + – chain.

The crystal structure may be discussed in different ways. Fundamental building elements of the structure are infinite –O–Hg–O– chains of the type found in $Hg(OHg)_4Br_2$ (Fig. 1*a*) (Aurivillius, 1965, 1968). If only the short Hg–O and Hg–Cl contacts are taken into account – *i.e.* all Hg atoms, Hg(1)–Hg(6), are assumed to be two-coordinated – the present single chains are linked to double chains, in which 12-membered $(Hg–O)_6$ rings can be observed (Fig. 2); if the next nearest contacts Hg–O are also considered the double chains are connected in pairs to form endless bands.

The current infinite + + – – –O–Hg–O– chain (Fig. 1*a*) is of a different geometry from the endless zigzag + – chain (Fig. 1*b*) in HgO(orth.) (Aurivillius, 1956) and in many other Hg^{II} oxide salts. Four symmetry-related chains of the + + – – type run through the unit cell of $Hg_6O_4Cl_4$ approximately parallel to [001]. The chains comprise Hg(1)–Hg(4) and all the O atoms. Two such chains, related to each other as x,y,z and $x, \frac{1}{2}-y, \frac{1}{2}+z$, are linked by the atoms Hg(5), which are bonded to O(1) and O(4), one from each chain (Fig. 2). The repetition unit of one chain has thus two bonds O(1)–Hg(5)–O(4) to its nearest chain. Hg(6) is bonded to O(3) of the chain and to Cl(1) at short distances, forming a finite 'side-chain' –Hg–Cl. Basic building elements of the structure of $Hg_6O_4Cl_4$ are thus endless double chains of the general formula $[Hg_2(OHg)_4Cl^{3+}]_n$ (Fig. 2). The single chains forming them are more puckered in this structure than in $Hg(OHg)_4Br_2$.

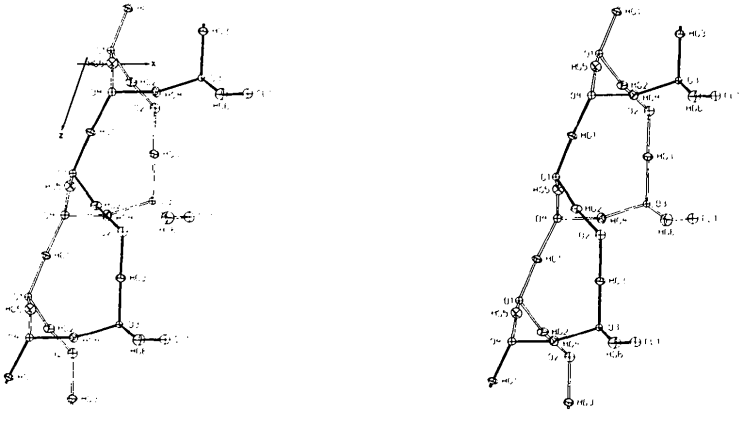


Fig. 2. A stereodrawing of part of the structure of $Hg_6O_4Cl_4$ showing a double chain $[Hg_2(OHg)_4Cl^{3+}]_n$. The bonds of one single chain are filled and of the other open.

If the longer Hg—O contacts are also considered, *i.e.* Hg(4) and Hg(5) are assumed to be (2 + 1)-coordinated, the single chains have in addition to the linking through Hg(5) another contact, Hg(4)—O(2), of 2·47 (2) Å (Fig. 3). A weaker support between them also exists through the contact Hg(6)—O(2) of 2·59 (2) Å. Furthermore, the endless double chains $[Hg_2(OHg)_4Cl^{3+}]_n$ are linked in pairs through the contacts Hg(5)—O(4) of 2·44 (2) Å, forming bands that are infinite along [001] (Fig. 3). Between the bands the shortest distance is 2·90 (1) Å [Hg(4)—Cl(1)].

The distances from Cl(2), Cl(3) and Cl(4) to Hg are ≥2·75 Å indicating that the bonds are predominantly ionic. The Cl^- ions are situated in cavities in the structure (Fig. 3). The best way to write the formula of the compound is $[Hg_2(OHg)_4Cl]Cl_3$. The structure is similar to that of $[Hg(OHg)_4]Br_2$. The infinite —O—Hg—O— chains (Fig. 1*a*) of the Br compound comprise four of the five Hg atoms of the structure and all the O atoms. The chains are linked four by four by the remaining Hg atom which is bonded to four O atoms tetrahedrally at 2·24 (3) Å, forming $[Hg(OHg)_4^{2+}]_n$ sheets. The layers are endless along [010] and [001] and have a thickness of 3·6 Å along [100]. The shortest Hg—O contact between the sheets is 2·78 (3) Å.

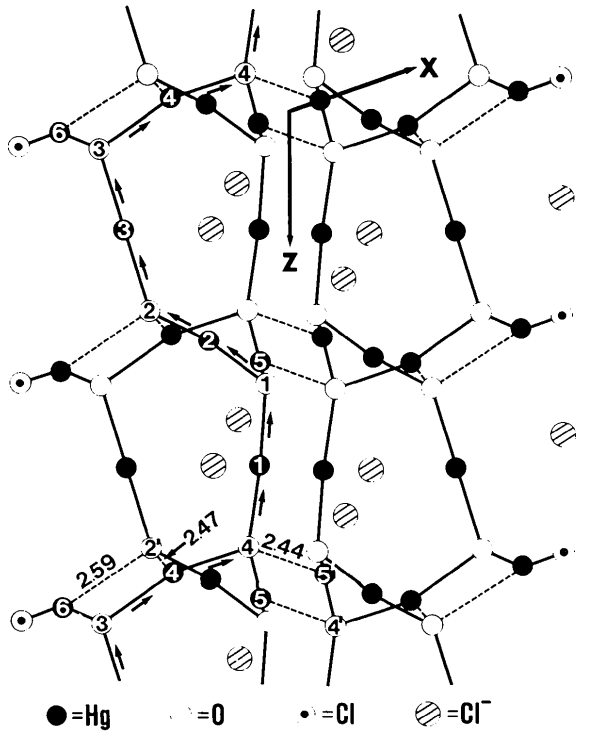
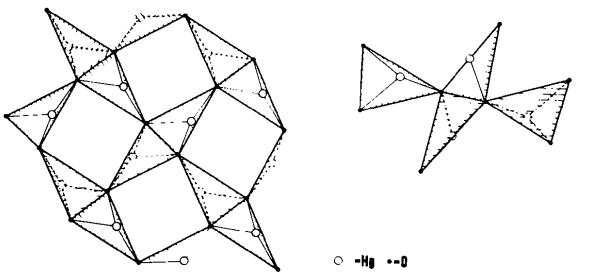


Fig. 3. A projection of the structure of $Hg_6O_4Cl_4$ along [010]. The atoms of one single chain and the linking Hg atom are numbered according to Table 3. Some symmetry-related atoms in adjacent chains are also marked. Short Hg—O and Hg—Cl bonds are drawn with full lines and long Hg—O contacts by dotted lines. The direction of one single chain is marked by arrows.



(*a*) (*b*)

Fig. 4. (*a*) The structure of $Hg_3O_2Cl_2$. A projection of part of the structure on the *yz* plane showing the linked flat HgO_3 pyramids. (*b*) The structure of $Hg_6O_4Cl_4$. Group of linked flat HgO_3 pyramids.

In Hg_3OCl_4, $Hg_3O_2Cl_2$, $Hg_5O_4Cl_2$ and $Hg_6O_4Cl_4$ the Cl atoms have to some extent different states. In $Hg_3O_2Cl_2$ and $Hg_5O_4Cl_2$ they are all bonded by ionic forces to the networks $[Hg_3O_2^{2+}]_n$ and $[Hg_5O_4^{2+}]_n$ respectively. In $Hg_6O_4Cl_4$ 25% and, in Hg_3OCl_4, 75% of the Cl atoms are bonded covalently to the Hg atoms forming $[Hg_6O_4Cl^{3+}]_n$ bands and $OHg_3Cl_3^+$ ions. When $Hg_6O_4Cl_4$ is heated the bands lose their finite 'side chains' —Hg—Cl, which with Cl^- ions form $HgCl_2$, and transform to $Hg_5O_4Cl_2$.

The similarities of the structures of $Hg_6O_4Cl_4$ and $Hg_3O_2Cl_2$ are not very obvious. A feature in common is the presence of flat HgO_3 pyramids, each with a Hg atom at its apex. In $Hg_3O_2Cl_2$, sheets built up of such pyramids are linked by additional two-coordinated Hg atoms to a three-dimensional network $[Hg_3O_2^{2+}]_n$ (Fig. 4*a*). In $Hg_6O_4Cl_4$, groups of similar flat pyramids exist, with Hg(4) and Hg(5) at their apices. The polyhedra of Hg(5) have an edge in common and share two basal corners with those of Hg(4) (Fig. 4*b*).

On the stability relations of $Hg_3O_2Cl_2$ and $Hg_6O_4Cl_4$

To study the stability relationships of $Hg_6O_4Cl_4$ and $Hg_3O_2Cl_2$, samples were shaken in 0·05 *M* HCl for one week at 25·0 °C. The saturated solutions were analysed for Hg by atomic absorption spectroscopy and the solid residues examined by X-ray powder diffractograms which showed that they were pure phases. As the solubility (mol l^{-1}) of $Hg_3O_2Cl_2$ is somewhat more than twice that of $Hg_6O_4Cl_4$, $\Delta G < 0$ for the reaction $2Hg_3O_2Cl_2(s) \rightarrow Hg_6O_4Cl_4(s)$. $Hg_6O_4Cl_4$ is thus the stable phase at this temperature. When the experiments were repeated for a long time the X-ray diffractograms of the residues in the case of $Hg_3O_2Cl_2$ also showed weak lines of $Hg_6O_4Cl_4$.

The volume per unit $Hg_3O_2Cl_2$ is 2% larger for $Hg_6O_4Cl_4$ than for $Hg_3O_2Cl_2$. Samples of $Hg_6O_4Cl_4$

sealed in gold tubes were therefore exposed to a pressure of 20 kbar at 25 °C for one day. No lines of $Hg_3O_2Cl_2$ were found in the X-ray powder diffractograms of the samples after the experiment.

When heated in air at 200 °C for about 70 h, $Hg_3O_2Cl_2$ and $Hg_6O_4Cl_4$ were both transformed to $Hg_5O_4Cl_2$, the structure of which has a clear resemblance to that of $Hg_6O_4Cl_4$.

$Hg_3O_2Cl_2$ and $Hg_6O_4Cl_4$ were also investigated *in vacuo* by differential thermal analysis with a heating rate of 5° min^{-1} in the temperature range 20–300 °C. The DTA curves for $Hg_3O_2Cl_2$ and $Hg_6O_4Cl_4$ show at respectively 213–260 °C and 239–268 °C endothermic changes which are not reversible. Powder photographs indicated that the residues after the DTA measurements up to 270 °C consisted of $Hg_5O_4Cl_2$. After further heating to 300 °C the diffractograms of the samples gave only lines of orthorhombic HgO. The DTA measurements were performed at the Arrhenius Laboratory, Stockholm University.

When $Hg_3O_2Cl_2$ was heated *in vacuo* at about 200 °C for one week it was transformed to $Hg_6O_4Cl_4$ according to powder photograms. After further heating at 230 °C for three days the sample only showed lines of $Hg_5O_4Cl_2$ and $HgCl_2$.

To summarize, at temperatures of about 200 °C $Hg_6O_4Cl_4$ and, at about 230 °C, $Hg_5O_4Cl_2$ are the stable phases. The formulae for the changes *in vacuo* are $2Hg_3O_2Cl_2(s) \rightarrow Hg_6O_4Cl_4(s) \rightarrow Hg_5O_4Cl_2(s) + HgCl_2(s)$; $Hg_5O_4Cl_2(s) \rightarrow 4HgO(s) + HgCl_2(s)$.

The authors thank Professor Bengt Aurivillius for many valuable discussions and Dr Mats Nygren for the DTA analyses. This investigation forms part of a research programme on compounds of heavy metals, financially supported by the Swedish Natural Science Research Council.

Note added in proof:—An independent structure investigation of this compound of about the same accuracy was recently published by Neuman, Petersen & Lo (1976). Their description based on distorted OHg_4 tetrahedra is, however, quite different from that given by us.

References

Akishin, P. A., Spiridonov, V. P. & Khodchenkov, A. N. (1959). *Zh. Fiz. Khim.* **33**, 20–24.

Arctowski, H. (1895). *Z. Anorg. Chem.* **9**, 178–189.

Aurivillius, K. (1954). *Acta Chem. Scand.* **8**, 523.

Aurivillius, K. (1956). *Acta Chem. Scand.* **10**, 852–866.

Aurivillius, K. (1964*a*). *Ark. Kemi*, **22**, 517–523, 537–550.

Aurivillius, K. (1964*b*). *Acta Chem. Scand.* **18**, 1305–1306.

Aurivillius, K. (1965). *Ark. Kemi*, **23**, 469–473.

Aurivillius, K. (1968). *Ark. Kemi*, **28**, 279–288.

Aurivillius, K. (1971). Abstr. 14th Meet. Scandinavian Chemists, Umeå, Sweden, p. 148 (in Swedish).

Aurivillius, K. & Stålhandske, C. (1974). *Acta Cryst.* B**30**, 1907–1910.

Aurivillius, K. & Stålhandske, C. (1976). *Acta Chem. Scand.* A**30**, 735–739.

Cromer, D. T. & Liberman, D. (1970). *J. Chem. Phys.* **53**, 1891–1898.

Doyle, P. A. & Turner, P. S. (1968). *Acta Cryst.* A**24**, 390–397.

Neuman, M. A., Petersen, D. R. & Lo, G. Y. S. (1976). *J. Cryst. Mol. Struct.* **6**, 177–203.

Sandström, M. & Persson, I. (1977). *Acta Chem. Scand.* To be published.

Weiss, A., Nagorsen, G. & Weiss, A. (1954). *Z. Naturforsch. Teil B*, **9**, 81.