Preparation, Structure, and Properties of Mercury Layer and Chain Compounds*

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In this paper is reviewed the preparation, structure, and electrical properties of salts of the polyatomic mercury cations Hg_3^{2+} and Hg_4^{2+}, the infinite chain compounds $Hg_{3-\delta}MF_6$ (M = As, Sb, Nb, and Ta), and the layer compounds Hg_3NbF_6 and Hg_3TaF_6. © 1985 Academic Press, Inc.

Introduction

Two examples of a novel type of mercury compound having the nonstoichiometric compositions $Hg_{2.82}AsF_6$ and $Hg_{2.90}SbF_6$ were first reported in 1973. They were prepared by the reaction of mercury with a solution of AsF_5 or SbF_5 in liquid SO_2. The two compounds have a shiny golden metallic appearance and exhibit an anisotropic metallic conductivity. Neutron and X-ray diffraction studies have shown that these compounds contain two nonintersecting mutually perpendicular chains of mercury atoms in a tetragonal lattice of MF_6^- ions. Because of their unusual structures and anisotropic metallic conductivity these compounds have been of considerable interest to solid-state chemists and physicists, and extensive studies have been made on them. It was not until quite recently, however, that any further examples of this type of

compound were prepared. In this paper we describe the preparation and properties of the new compounds $Hg_{2.88}NbF_6$ and $Hg_{2.88}TaF_6$ which have structures and properties very similar to compounds $Hg_{2.82}AsF_6$ and $Hg_{2.90}SbF_6$. Because of some uncertainty concerning the composition of these compounds, which we refer to later, they are usually given the general formula $Hg_{3-\delta}MF_6$. We also describe the two stoichiometric compounds Hg_3NbF_6 and Hg_3TaF_6 which are formed from the golden crystals of $Hg_{3-\delta}NbF_6$ and $Hg_{3-\delta}TaF_6$. The compounds Hg_3NbF_6 and Hg_3TaF_6 form soft, silver-colored, thin, plate-like crystals. They have a structure completely different from the chain compounds in that they contain sheets of mercury atoms separated by layers of NbF_6^- or TaF_6^- octahedra.

In addition to these compounds containing infinite chains or sheets of mercury atoms the same reactions lead to the formation of the polymercury cations Hg_3^{2+} and Hg_4^{2+}. We first describe these polymercury cations and then proceed to a discussion of the chain and layer mercury compounds.

Finite Polymercury Cations

Covalent metal–metal bonds have become a fashionable and popular area of investigation in inorganic chemistry. Many examples have been found in finite clusters and in infinite structures. The first example of such a bond to be recognized—the mercury–mercury bond—had been known for a very long time in the mercurous ion Hg_2^{2+} and in several covalent mercury(I) compounds such as mercury(I) chloride, Cl–Hg–Hg–Cl. However, it was not until 1971 that longer chains of mercury atoms were first made (1–4). At that time it was shown that the oxidation of mercury with AsF_5 and SbF_5 in solution in SO_2 produced the ions Hg_3^{2+} and Hg_4^{2+}. These were isolated as the crystalline AsF_6^- and $Sb_2F_{11}^-$ salts. Typical reactions may be represented by the equations

$$3Hg + 3AsF_5 \rightarrow Hg_3(AsF_6)_2 + AsF_3$$

$$2Hg + 5SbF_5 \rightarrow Hg_3(Sb_2F_{11})_2 + SbF_3$$

$$4Hg + 3AsF_5 \rightarrow Hg_4(AsF_6)_2 + AsF_3$$

Although many attempts were made to prepare other related salts by this method none were successful. This appears to be mainly due to the fact that no other pentafluorides were found that are capable of oxidizing mercury at room temperature in SO_2. Recently, however, we have found that the

TABLE I

Hg_3^{2+} SALTS

	d(Hg–Hg) (Å)	ν(Hg–Hg) (cm^{-1})	Reference
$Hg_3(AsF_6)_2$	2.55	140	2
$Hg_3(Sb_2F_{11})_2$	—	133	2
$Hg_3(AlCl_4)_2$	2.56	123	a
$Hg_3(NbF_6)_2$	—	125	5
$Hg_3(Ta_2F_{11})_2$	—	128	5
$Hg_3(NbCl_6)_2$	—	130	5

^{a} Torsi, G., Fung, K. W., Begun, G. H., and Manantov, G., Inorg. Chem. 10, 2285 (1975).

TABLE II

Hg_4^{2+} SALTS

|	d(Hg–Hg) (Å)	ν(Hg–Hg) (cm^{-1})	Reference
$Hg_4(AsF_6)_2$	2.59, 2.62	80, 104	3
$Hg_4(Sb_2F_{11})_2$	—	116	5
$Hg_4(Ta_2F_{11})_2$	—	120	8

reaction of mercury with various Hg(II) hexafluorides, $Hg(MF_6)_2$, is a more useful and more general method of preparing Hg_3^{2+} and Hg_4^{2+} salts (5). For example, $Hg_3(NbF_6)_2$ has been prepared by the reaction
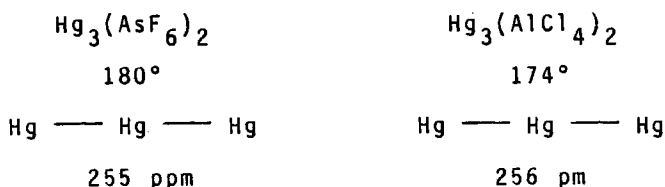
$$2Hg + Hg(NbF_6)_2 \rightarrow Hg_3(NbF_6)_2$$
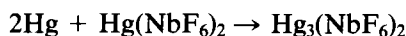
and $Hg_3(Ta_2F_{11})_2$, by the reaction

$$2Hg + Hg(Ta_2F_{11})_2 \rightarrow Hg_3(Ta_2F_{11})_2$$
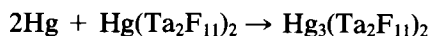
The appropriate $Hg(MF_6)_2$ salt or $Hg(M_2F_{11})_2$ salt can be prepared by the reaction of HgF_2 with the pentafluoride MF_5 in solution in SO_2. In some cases the reaction can be carried out in one step, that is, by the reaction of the appropriate amounts of HgF_2, MF_5, and Hg in SO_2. For example, $Hg_4(Ta_2F_{11})_2$ can be prepared by the reaction

$$HgF_2 + 4TaF_5 + 3Hg \rightarrow Hg_4(Ta_2F_{11})_2$$
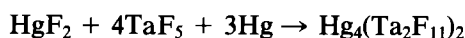
As we shall discuss, these reactions are more complex than indicated by these simple equations. In the preparation of an Hg_4^{2+} salt a mixture of soluble Hg_3^{2+} and Hg_4^{2+} salts is normally obtained, together with a small amount of the insoluble metallic compound of composition $Hg_{3-\delta}MF_6$. When the solution is crystallized a mixture of a yellow Hg_3^{2+} salt and a deep red–black Hg_4^{2+} salt is obtained and the crystals must be separated by handpicking. The known Hg_3^{2+} and Hg_4^{2+} salts are listed in Tables I and II. The MF_6^- salts, with the exception of the AsF_6^- salts, are generally insoluble in SO_2 whereas the $M_2F_{11}^-$ salts are generally very

$$Hg_3(AsF_6)_2$$

$$180°$$

$$Hg \longrightarrow Hg \longrightarrow Hg$$

$$255\ ppm$$

$$Hg_3(AlCl_4)_2$$

$$174°$$

$$Hg \longrightarrow Hg \longrightarrow Hg$$

$$256\ pm$$

FIG. 1. The structure of Hg_3^{2+}.

soluble. All these salts disproportionate very rapidly in the presence of water to give the element and Hg_2^{2+} salts.
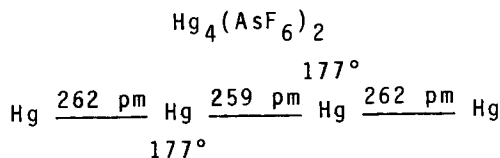
Structures of Hg_3^{2+} and Hg_4^{2+}

Determination of the crystal structures of $Hg_3(AsF_6)_2$ and $Hg_3(AlCl_4)_2$ has shown that the Hg_3^{2+} ion has a linear, or very nearly linear, structure with two Hg–Hg bonds of equal length (Fig. 1) (6, 7).

The structure of the Hg_4^{2+} ion has been determined in $Hg_4(AsF_6)_2$ (3) and Hg_4 $(Ta_2F_{11})_2$ (8). It has an almost linear structure. The terminal Hg–Hg bonds are slightly longer than the central Hg–Hg bond and both bonds are slightly longer than in Hg_3^{2+} (Fig. 2). Although the structure can be approximately described as consisting of independent Hg_4^{2+} and AsF_6^- ions, the distances between two adjacent Hg_4^{2+} ions (2.99 Å) is close to the shortest Hg–Hg distance in metallic mercury. Thus the Hg_4^{2+} ions may be considered to be associated together in long zigzag chains. No evidence has been obtained for soluble compounds containing chains of more than four mercury atoms.

Infinite Mercury Chain Compounds

In the reaction of mercury with AsF_5, SbF_5, or $Hg(MF_6)_2$ golden crystals with a shiny metallic appearance appear on the

$$Hg_4(AsF_6)_2$$

$$177°$$

$$Hg \xrightarrow{262\ pm} Hg \xrightarrow{259\ pm} Hg \xrightarrow{262\ pm} Hg$$

$$177°$$

FIG. 2. The structure of Hg_4^{2+}.

surface of the liquid mercury within 10 to 15 min. Some time later the SO_2 solution becomes yellow or orange–red in color as Hg_3^{2+} and Hg_4^{2+} are formed. In the presence of sufficient oxidizing agent the golden crystals are completely converted to a soluble Hg_3^{2+} salt but with a less oxidizing agent the golden crystals which are completely insoluble in SO_2 remain at the end of the reaction and can be isolated.

Room-Temperature Structure

X-Ray precession photographs of the golden crystals isolated from the reaction of mercury with AsF_5 showed unusual sheets of intensity perpendicular to the a and b axes of a tetragonal crystal (Fig. 3) (9). These arise from chains of mercury atoms running in two mutually perpendicular directions through tunnels in a lattice of AsF_6^- octahedra (Fig. 4). The mercury–mercury distance in the chains of 2.67 Å is not commensurate with the tetragonal AsF_6^- lattice for which $a = b = 7.54$ Å. Thus the compound has the nonstoichiometric formula $Hg_{2.92}AsF_6$. The structure has been confirmed by a detailed neutron diffraction study (10). The reaction of SbF_5 with mercury resulted in the analogous compound $Hg_{2.90}SbF_6$, and very recently from the reaction of $Hg(MF_6)_2$ with mercury we have obtained the new compounds $Hg_{2.88}NbF_6$ and $Hg_{2.88}TaF_6$ (11). All three compounds have the same golden metallic appearance of $Hg_{2.82}AsF_6$; X-ray determination of their structures has shown that they have essentially the same structure as the AsF_6^- compound (Table III) (12, 13). The lattice dimensions of the four compounds differ
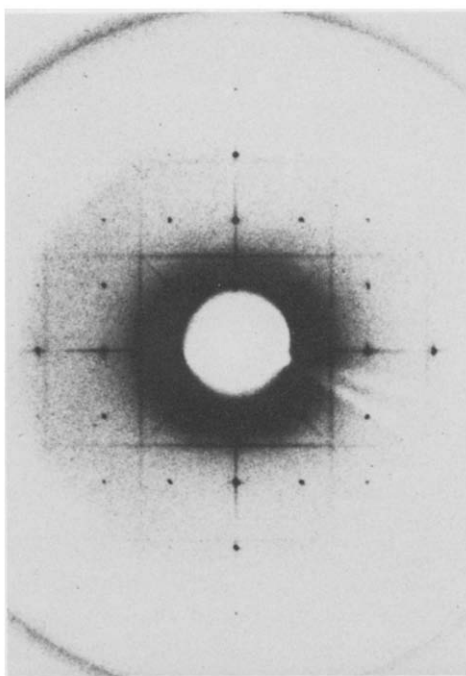
FIG. 3. X-Ray precession photograph of the $(hk0)$ plane of Hg_3SbF_6. The lattice of Bragg peaks is associated with the SbF_6^- lattice, the lines with the Hg chains.

FIG. 4. The structure of the chain compounds Hg_3MF_6 (M = As, Sb, Nb, Ta). The octahedra represent the MF_6^- anions and the circles the Hg atoms.

slightly because the anions are slightly different in size, and since the Hg–Hg distance is 2.67 Å in all three compounds, their compositions differ slightly from that of the AsF_6^- compound.

The lack of structure in the diffuse sheets of diffracted intensity at room temperature shows that there is no correlation b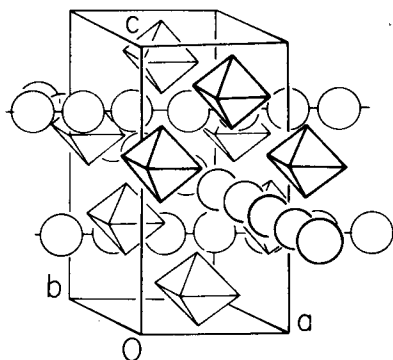etween the positions of the Hg atoms in one chain and those in another chain—in other words there is complete disorder between the chains. The chains can be regarded as one-dimensional liquids which can move freely in the tunnels in which they are accommodated. Each mercury atom has a charge of approximately $+\frac{1}{3}$. There is ionic bonding between the chains and the MF_6^- ions and metallic bonding in the chains.

An interesting feature of all four structures is that the chains are not exactly straight, but they have a small undulation with a periodicity equal to the lattice spacing of the host lattice. The undulation is such as slightly to increase the contact distance between the chains where they cross so that this distance becomes 3.22–3.24 Å in all four compounds. The maximum displacement of each chain from the straight

TABLE III

STRUCTURAL DATA FOR $Hg_{3-\delta}MF_6$ SALTS

	$Hg_{3-\delta}AsF_6$	$Hg_{3-\delta}SbF_6$	$Hg_{3-\delta}NbF_6$	$Hg_{3-\delta}TaF_6$
Space group	$I4_1/amd$	$I4_1/amd$	$I4_1/amd$	$I4_1/amd$
$a = b$ (Å)	7.534	7.711	7.692	7.711
c (Å)	12.395	12.641	12.679	12.714
V_{cell} (Å³)	703.6	751.6	750.2	756.0
δ	0.178(6)	0.10(2)	0.119	0.116
Hg–Hg (Å)	2.670(5)	2.66(2)	2.670(3)	2.674(4)

configuration is largest for the AsF_6^- compound (0.07 Å) and the smallest for the NbF_6^- and TaF_6^- compounds (0.026 Å).

Low-Temperature Structures

Because of the incommensurability between the Hg–Hg distance in the chains and the dimensions of the MF_6^- lattice, it is impossible for the chains to order relative to the host lattice; the Hg atoms necessarily occupy different positions in each unit cell. It is possible, however, for the chains to order relative to each other.

We have seen that there is very little, if any, ordering between the chains at room temperature, but as the temperature is decreased the sheets of diffuse intensity split up into broad spots, indicating the onset of some short-range ordering between the chains (14, 15). These spots become somewhat sharper and increase in intensity with decreasing temperature, indicating an increased ordering of the mercury atoms in parallel chains (Fig. 5).

Below 120 K the intensities of the broad peaks diminish rapidly and they are replaced by a new set of Bragg reflections (Fig. 5). These arise because interactions between the chains cause them to form two ordered lattices which share a common di-
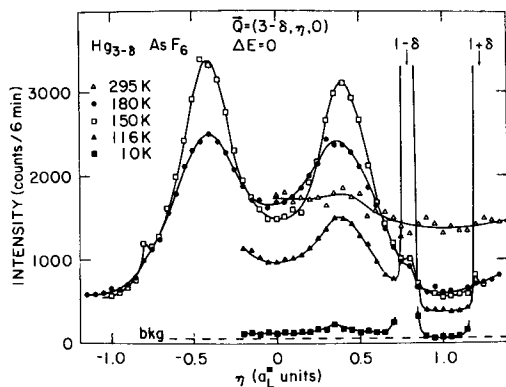
TABLE IV

TEMPERATURE DEPENDENCE OF THE STRUCTURE OF $Hg_{3-\delta}AsF_6$ (14)

	293 K	0 K
$a = b$ (Å)	7.534(7)	7.44
c (Å)	12.395(8)	12.248
V (Å)^3	703.6	678.0
Hg–Hg (Å)	2.670(5)	2.670(5)
δ (Å)	0.178(6)	0.210(4) (80 K)

rection. The mercury atoms in the [100] chains are arranged on a body-centered monoclinic lattice and the mercury atoms in the [010] chains lie on a similar lattice but with a and b interchanged. The mercury atom lattices are still, however, incommensurate with the AsF_6^- lattice. Thus, the fully ordered phase consists of three interpenetrating lattices—the tetragonal host lattice and the two monoclinic mercury chain lattices.

Composition

A particularly interesting feature of these compounds is that although the MF_6^- lattice contracts as expected with decreasing temperature the mercury–mercury distance remains constant. This would appear to imply that the composition of these compounds changes with temperature—the percentage of mercury decreasing slightly (Table IV). Presumably mercury is eliminated from the structure with decreasing temperature. It has been found in DTA experiments (16) that if a crystal is cooled below a certain threshold temperature and then warmed to room temperature an endotherm at 235 K is observed, corresponding to the melting of solid mercury. The threshold temperature varied somewhat from sample to sample but was normally in the range 200–210 K. Thus it appears that on cooling a crystal below 200–210 K some solid mercury separates form the crystal and that on warming this mercury melts and is reabsorbed by the

FIG. 5. Neutron diffraction scans at various temperatures along the line (3-δ, η, 0) in the plane of the diffuse scattering (from Ref. (14)).

TABLE V

DENSITY OF $Hg_{3-\delta}AsF_6$ (18)

Observed	7.07 g cm⁻³
Calculated for:	
$Hg_{2.82}AsF_6$	7.16 g cm⁻³
$Hg_3AsF_6(Hg_{2.82}AsF_6 + 0.18$ Hg)	7.49 g cm⁻³
$Hg_{2.82}(AsF_6)_{0.94}$	7.05 g cm⁻³

crystal. The cooling–warming cycle can be repeated several times with the same results.

Another problem with respect to the composition is that chemical analysis leads to a composition much closer to the stoichiometric value Hg_3AsF_6 than that obtained from the crystal structure (17, 18). It appears that, either the crystal contains additional mercury, or it has a deficiency of anions. The latter explanation is more consistent with the observed density of the crystals (Table V). The measured density is 7.07 g cm⁻³, which compares with a calculated value of 7.16 g cm⁻³ for $Hg_{2.82}AsF_6$ and 7.49 g cm⁻³ for Hg_3AsF_6, equivalent to $Hg_{2.82}AsF_6 + 0.18$ Hg in interstitial sites. However, if there are 6% anion vacancies corresponding to the composition $Hg_{2.82}(AsF_6)_{0.94}$ the calculated density is 7.05 g cm⁻³, which is in good agreement with the experimental value.

Resistivity

The resistivity in the *ab* plane of $Hg_{3-\delta}AsF_6$ and $Hg_{3-\delta}SbF_6$ is 1.0 × 10⁻⁴ ohm cm and 1.1 × 10⁻⁴, respectively, at room temperature. The resistivities decrease with decreasing temperature down to 4.2 K, with a resistivity ratio, between room temperature and 4.2 K, of 500 for $Hg_{3-\delta}AsF_6$ and 300 for $Hg_{3-\delta}SbF_6$ (Fig. 6) (19, 20). Thus, these compounds are metallic over the whole temperature range and there is no evidence of a Peierls metal–insulator transition which is predicted for a one-dimensional metal. This is presumably due to the fact that the chains are not inde-

pendent but are coupled together. Indeed, the fact that there is some conductivity in the *c* direction shows that there is some interaction between perpendicular chains where they cross each other. The ratio of the resistivities at room temperature in the *ab* plane and along the *c* axis is 105 for $Hg_{3-\delta}AsF_6$ and 40 for $Hg_{3-\delta}SbF_6$.

Superconductivity

It has been reported that $Hg_{3-\delta}AsF_6$ and $Hg_{3-\delta}SbF_6$ are superconducting at low temperature (20, 21). However, the critical field H_c (0) at zero temperature and the critical temperature T_c at zero field are 0.0401 T and 4.14 K, respectively. These are very close to the corresponding values for elemental mercury of 0.412 T and 4.152 K. The very small differences may be due to the fact that the mercury is not in the free bulk state but exists either in a very thin layer on the surface of the crystal, or is present in cracks or dislocations in the crystal, or in AsF_6^- vacancies. In experiments on the magnetic susceptibility of
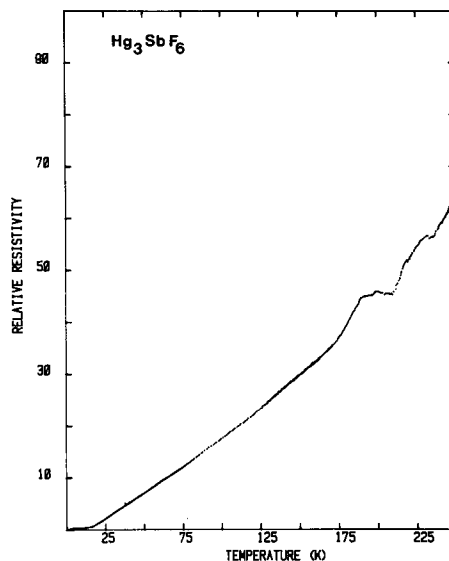

FIG. 6. The variation of the relative resistivity of Hg_3SbF_6 with temperature.

$Hg_{3-\delta}SbF_6$ it was found that there is a transition near 4.1 K with an amplitude that depends on the rate of cooling (21). Presumably, this is due to the fact that mercury is formed in an amount that depends on the rate of cooling.

Infinite Mercury Sheet Compounds

The recent preparation of the golden chain compounds $Hg_{3-\delta}NbF_6$ and $Hg_{3-\delta}TaF_6$ led to the unexpected discovery of an entirely new type of mercury compound containing sheets of mercury atoms. If the golden crystals of $Hg_{3-\delta}NbF_6$ and $Hg_{3-\delta}TaF_6$ are not removed from the reaction mixture, which contains unreacted mercury and the Hg_3^{2+} and Hg_4^{2+} ions in solution, they are transformed within a few hours to thin flexible silvery plates that resemble aluminum foil (11). If the reaction is carried out at −35°C, no golden crystals are observed and the silver crystals appear to be formed directly. These crystal have a diffraction pattern completely different from the golden crystals. Precession photographs show hexagonal symmetry (Fig. 7).

The principal features of the X-ray diffraction pattern can be explained on the basis of the space group $P\bar{3}1m$ (Table VI and
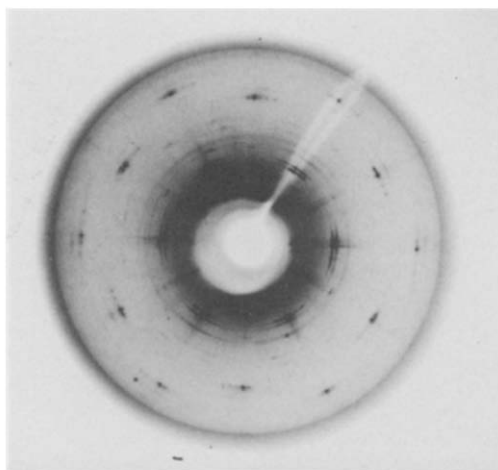
TABLE VI

PROPOSED MODEL FOR $Hg_{3-\delta}NbF_6$

Hg_3NbF_6, $M_r = 808.67$,
trigonal, space group $P\bar{3}1m$,
$Z = 1$, $a = 5.02(1)$, $c = 7.68(7)$ Å,
$D_x = 8.0(1)$ mg m^{-3}

Atomic coordinates

Atom	Site	x	y	z
Hg(1)	1b	0	0	$\frac{1}{2}$
Hg(2)	2d	$\frac{1}{3}$	$\frac{2}{3}$	$\frac{1}{2}$
Nb	1a[a]	0	0	0
F	6K	0.309[b]	0	0.143[b]

[a] In some samples the Nb atom is disordered over the three sites 2a and 2c ($\frac{1}{3}$, $\frac{2}{3}$, 0; $\frac{2}{3}$, $\frac{1}{3}$, 0).

[b] Calculated assuming an undistorted NbF_6 octahedron with Nb–F = 1.90 Å.

Fig. 8) in which layers of close-packed NbF_6^- octahedra are separated by sheets of mercury atoms arranged on a hexagonal net. Each mercury atom has six mercury neighbors at 2.90 Å within the sheet and three fluorine neighbors from each of the adjacent NbF_6^- layers at distances of 3.2 Å. The mercury and fluorine atoms form a cubic close-packed lattice, with niobium atoms occupying one-third of the octahedral holes between fluorine atoms. In some samples it appears that the Nb atoms randomly occupy different positions in different layers. The mercury–mercury distance in

FIG. 7. X-Ray precession photography of Hg_3NbF_6.

FIG. 8. The structure of Hg_3NbF_6.

these sheets is larger than within the chains, in which each mercury atom is bonded to only two others, but is smaller than in elemental mercury, in which each mercury atom has 10 or 12 neighbors.

Unlike the golden crystals from which they are formed the silver crystals have the exact stoichiometric composition Hg_3MF_6 that is determined by the close-packed structure.

Resistivity

The resistivity of Hg_3NbF_6 and Hg_3TaF_6 as a function of temperature is shown in Figs. 9 and 10 (22). The relationship is linear from room temperature down to 25 K, below which the resistivity varies as T^3 and T^5. This is quite similar to the behavior of common metals such as copper and aluminum, but is considerably different from that of the chain compounds for which there is no linear temperature dependence.

Superconductivity

A superconducting transition at 7 K was reported for these compounds on the basis
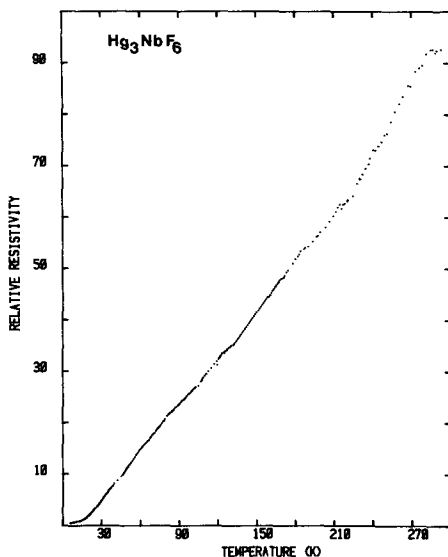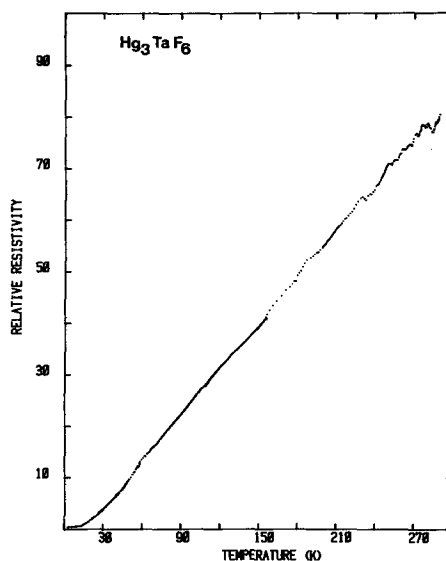

FIG. 10. The variation of the relative resistivity of Hg_3TaF_6 with temperature.

of magnetic susceptibility measurements (23). However, subsequent resistivity measurements (23) have provided no evidence for such a superconducting transition, and the susceptibility measurements must be regarded as erroneous.

Interconversion of the Chain and Sheet Compounds

If the silver crystals are heated to 120°C they rapidly transform to the golden crystals (11). This transformation can easily be observed visually and was confirmed by X-ray powder photographs. The transformation does not appear to be reversible in the absence of liquid SO_2, because the golden crystals can be cooled to room temperature and below without reverting to the silver form. However, if the golden crystals are cooled below room temperature in the presence of liquid SO_2, or are allowed to stand at room temperature in the presence of liquid SO_2, they are transformed back to the silver form. The role played by liquid SO_2 in this transformation is not clear since both

FIG. 9. The variation of the relative resistivity of Hg_3NbF_6 with temperature.

the golden and silver forms are completely insoluble in SO_2. Perhaps the transformation proceeds via the formation of small amounts of H_3^{2+} and Hg_4^{2+}.

It appears that the silver form is the thermodynamically stable form of Hg_3NbF_6 and Hg_3TaF_6 but no silver form of either $Hg_{3-\delta}AsF_6$ or $Hg_{3-\delta}SbF_6$ has yet been obtained. The fact that the golden and silver forms of Hg_3NbF_6 and Hg_3TaF_6 are so readily interconverted—despite that fact that they apparently have different compositions and that mercury is not observed to be formed in the transformation of the silver to the golden form—again raises the question of the composition of the golden form. This facile interconversion lends some support to the suggestion that the Hg : M ratio is, in fact, 3 : 1 and that the apparent nonstoichiometry indicated by the X-ray crystal structure is compensated by an appropriate number of anion deficiencies.

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