

MGTLs and Dr G. Germain, Dr P. Main and Professor M. M. Woolfson for a card deck of the program LSAM.

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The Crystal Structure of Mercury(I) Orthoarsenate

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The crystal structure has been determined by single-crystal three-dimensional X-ray analysis. There are two formula units of $(\text{Hg}_2)_3(\text{AsO}_4)_2$ in the monoclinic unit cell of dimensions $a=8.73$, $b=5.08$, $c=15.64$ Å, $\beta=128.4^\circ$ and space group $P2_1/c$. The structure is built up of Hg_2^{2+} and AsO_4^{3-} ions in such a way that three of the four arsenate oxygen atoms are bonded to three different Hg–Hg pairs, thus forming puckered layers. An effectively tetrahedral coordination about the mercury atoms is established by one mercury atom from the same Hg_2^{2+} pair, one oxygen atom at 2.16 to 2.23 Å, and two other oxygen atoms at 2.42 to 2.71 Å. The Hg–Hg–O bonds deviate from collinearity by 23 to 34°. One As–O bond length is longer (1.78 Å) than the other three (mean value 1.68 Å).

Introduction

This investigation is part of a research programme to extend the data on the structural chemistry of mercury(I) compounds, because little is known even now about the coordination of the mercury atom in these compounds. There are only 15 known structures of mercury(I) compounds and about two thirds of these have been solved from three-dimensional data.

In all known structures with oxygen atoms linked to mercury, as in mercury(I) sulphate, selenate (Dorm, 1969), bromate (Dorm, 1967), *o*-phthalate (Lindh, 1967) and trifluoroacetate (Grdenić & Sikirica, 1973), linear or almost linear O–Hg–Hg–O chains or groups are found. The linear oxonium ion $\text{H}_2\text{O–Hg–Hg–OH}_2$ is present in hydrated salts, as in mercury(I) nitrate (Grdenić, 1956), hexafluorosilicate (Dorm, 1971a), and perchlorate (Johansson, 1966). If one member of the Hg–Hg pair is linked to a different ligand a linear Hg–Hg–O grouping is formed, as in 1,10-phenanthroline-mercury(I) nitrate (Elder, Halpern & Pond, 1967). In the present structure a puckered-layer structure in

which one arsenate ion links three Hg_2^{2+} pairs has been found.

Experimental

Red-brown prismatic crystals were prepared by the reaction between aqueous mercury(I) nitrate solution and arsenic acid (Guérin & Boulitrop, 1951). The system is monoclinic, $a=8.73 \pm 0.02$, $b=5.08 \pm 0.01$, $c=15.64 \pm 0.03$ Å, and $\beta=128.4 \pm 0.3^\circ$. The cell parameters were determined from oscillation and Weissenberg photographs. The systematic absence of reflexions $h0l$ for l odd and $0k0$ for k odd uniquely determined the space group as $P2_1/c$. The cell contains two formula units $(\text{Hg}_2)_3(\text{AsO}_4)_2$; the pycnometrically measured density was 9.05 g cm^{-3} and the calculated density was 9.06 g cm^{-3} . Three-dimensional intensity data ($0kl \dots 7kl$, $h0l \dots h4l$, and $hk0 \dots hk14$) were obtained on an integrating Weissenberg camera using the multiple-film technique and nickel-filtered Cu K radiation. The intensities of 1137 independent observed reflexions were estimated by means of a microdensitometer, corrected for Lorentz and polarization effects

and placed on the same relative scale. The crystal was a sphere ground to 0.18 mm in diameter and the intensities were corrected for absorption ($\mu r = 15.4$). Structure factors, all corrections and the Patterson synthesis were calculated on the University Institute of Mathematics CAE 90.40 computer, Fourier synthesis on a CDC 3300 computer, while the least-squares refinement was carried out on the UNIVAC 1106 computer of the University Computing Centre, Zagreb.

Structure determination

The mercury atoms were located from three Patterson projections obtained by means of the von Eller photo-sommateur and the positions afterwards confirmed by a three-dimensional Patterson synthesis. A three-dimensional Fourier map calculated on the basis of

the contributions of three mercury atoms located the arsenic and oxygen atoms. The structure was then refined by five cycles of the full-matrix least-squares method assuming isotropic thermal parameters for all atoms. At this stage the reliability index was $R = 0.145$. The last scale factors were adjusted and the refinement was continued for three cycles assuming anisotropic thermal motion for the mercury and arsenic atoms. The final reliability index for the observed reflexions is $R = 0.085$. The refinement was carried out with a local version of the Busing, Martin & Levy (1962) program modified to make real and imaginary anomalous dispersion corrections. The atomic scattering factors for the mercury, arsenic and oxygen atoms were those from *International Tables for X-ray Crystallography* (1962), and the necessary parameters used for anomalous dispersion were those given by Cromer &

Table 1. Observed and calculated structure factors ($\times 10$)

h	k	l	F _o	F _c	h	k	l	F _o	F _c	h	k	l	F _o	F _c	h	k	l	F _o	F _c	h	k	l	F _o	F _c
0	0	0	100	100	0	0	0	100	100	0	0	0	100	100	0	0	0	100	100	0	0	0	100	100
1	0	0	100	100	1	0	0	100	100	1	0	0	100	100	1	0	0	100	100	1	0	0	100	100
2	0	0	100	100	2	0	0	100	100	2	0	0	100	100	2	0	0	100	100	2	0	0	100	100
3	0	0	100	100	3	0	0	100	100	3	0	0	100	100	3	0	0	100	100	3	0	0	100	100
4	0	0	100	100	4	0	0	100	100	4	0	0	100	100	4	0	0	100	100	4	0	0	100	100
5	0	0	100	100	5	0	0	100	100	5	0	0	100	100	5	0	0	100	100	5	0	0	100	100
6	0	0	100	100	6	0	0	100	100	6	0	0	100	100	6	0	0	100	100	6	0	0	100	100
7	0	0	100	100	7	0	0	100	100	7	0	0	100	100	7	0	0	100	100	7	0	0	100	100
8	0	0	100	100	8	0	0	100	100	8	0	0	100	100	8	0	0	100	100	8	0	0	100	100
9	0	0	100	100	9	0	0	100	100	9	0	0	100	100	9	0	0	100	100	9	0	0	100	100
10	0	0	100	100	10	0	0	100	100	10	0	0	100	100	10	0	0	100	100	10	0	0	100	100
11	0	0	100	100	11	0	0	100	100	11	0	0	100	100	11	0	0	100	100	11	0	0	100	100
12	0	0	100	100	12	0	0	100	100	12	0	0	100	100	12	0	0	100	100	12	0	0	100	100
13	0	0	100	100	13	0	0	100	100	13	0	0	100	100	13	0	0	100	100	13	0	0	100	100
14	0	0	100	100	14	0	0	100	100	14	0	0	100	100	14	0	0	100	100	14	0	0	100	100
15	0	0	100	100	15	0	0	100	100	15	0	0	100	100	15	0	0	100	100	15	0	0	100	100
16	0	0	100	100	16	0	0	100	100	16	0	0	100	100	16	0	0	100	100	16	0	0	100	100
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25	0	0	100	100	25	0	0	100	100	25	0	0	100	100	25	0	0	100	100	25	0	0	100	100
26	0	0	100	100	26	0	0	100	100	26	0	0	100	100	26	0	0	100	100	26	0	0	100	100
27	0	0	100	100	27	0	0	100	100	27	0	0	100	100	27	0	0	100	100	27	0	0	100	100
28	0	0	100	100	28	0	0	100	100	28	0	0	100	100	28	0	0	100	100	28	0	0	100	100
29	0	0	100	100	29	0	0	100	100	29	0	0	100	100	29	0	0	100	100	29	0	0	100	100
30	0	0	100	100	30	0	0	100	100	30	0	0	100	100	30	0	0	100	100	30	0	0	100	100
31	0	0	100	100	31	0	0	100	100	31	0	0	100	100	31	0	0	100	100	31	0	0	100	100
32	0	0	100	100	32	0	0	100	100	32	0	0	100	100	32	0	0	100	100	32	0	0	100	100
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35	0	0	100	100	35	0	0	100	100	35	0	0	100	100	35	0	0	100	100	35	0	0	100	100
36	0	0	100	100	36	0	0	100	100	36	0	0	100	100	36	0	0	100	100	36	0	0	100	100
37	0	0	100	100	37	0	0	100	100	37	0	0	100	100	37	0	0	100	100	37	0	0	100	100
38	0	0	100	100	38	0	0	100	100	38	0	0	100	100	38	0	0	100	100	38	0	0	100	100
39	0	0	100	100	39	0	0	100	100	39	0	0	100	100	39	0	0	100	100	39	0	0	100	100
40	0	0	100	100	40	0	0	100	100	40	0	0	100	100	40	0	0	100	100	40	0	0	100	100
41	0	0	100	100	41	0	0	100	100	41	0	0	100	100	41	0	0	100	100	41	0	0	100	100
42	0	0	100	100	42	0	0	100	100	42	0	0	100	100	42	0	0	100	100	42	0	0	100	100
43	0	0	100	100	43	0	0	100	100	43	0	0	100	100	43	0	0	100	100	43	0	0	100	100
44	0	0	100	100	44	0	0	100	100	44	0	0	100	100	44	0	0	100	100	44	0	0	100	100
45	0	0	100	100	45	0	0	100	100	45	0	0	100	100	45	0	0	100	100	45	0	0	100	100
46	0	0	100	100	46	0	0	100	100	46	0	0	100	100	46	0	0	100	100	46	0	0	100	100
47	0	0	100	100	47	0	0	100	100	47	0	0	100	100	47	0	0	100	100	47	0	0	100	100
48	0	0	100	100	48	0	0	100	100	48	0	0	100	100	48	0	0	100	100	48	0	0	100	100
49	0	0	100	100	49	0	0	100	100	49	0	0	100	100	49	0	0	100	100	49	0	0	100	100
50	0	0	100	100	50	0	0	100	100	50	0	0	100	100	50	0	0	100	100	50	0	0	100	100

Table 2. The final heavy-atom parameters and their estimated standard deviations

Values are $\times 10^4$. The temperature factors are in the form

$$T = \exp [-(\beta_{11}h^2 + \beta_{22}k^2 + \beta_{33}l^2 + 2\beta_{12}hk + 2\beta_{13}hl + \beta_{23}kl)] .$$

	x	y	z	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Hg(1)	1274 (3)	1312 (5)	847 (2)	86 (4)	256 (10)	37 (2)	-34 (5)	26 (2)	-30 (3)
Hg(2)	1917 (3)	3500 (5)	-1255 (2)	86 (4)	272 (10)	33 (1)	37 (5)	31 (2)	29 (3)
Hg(3)	3552 (3)	6410 (5)	416 (2)	100 (4)	285 (10)	40 (2)	34 (6)	34 (2)	39 (3)
As	6852 (7)	1219 (11)	2256 (4)	63 (8)	179 (22)	26 (3)	1 (12)	20 (4)	0 (7)

Liberman (1970). Unit weight was assigned to all reflexions. Table 1 lists the observed structure amplitudes and calculated structure factors based on the atomic coordinates given in Tables 2 and 3.

Table 3. *The final oxygen-atom parameters and their estimated standard deviations*

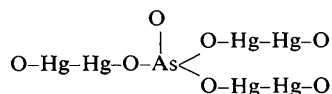
The coordinates are $\times 10^3$.

	<i>x</i>	<i>y</i>	<i>z</i>	<i>B</i> (\AA^2)
O(1)	142 (6)	0.512 (7)	0.281 (3)	2.24 (73)
O(2)	260 (6)	0.527 (8)	0.153 (3)	2.84 (74)
O(3)	534 (5)	0.513 (6)	0.374 (3)	2.18 (59)
O(4)	300 (5)	0.971 (7)	0.273 (3)	2.54 (63)

Description and discussion of the structure

The structure is built up of Hg_2^{2+} and AsO_4^{3-} ions. There are three crystallographically independent mercury atoms in general positions. One Hg_2^{2+} pair is related by the symmetry centre [Hg(1)] while the mercury atoms Hg(2) and Hg(3) are linked together in another pair. In both cases the mercury-mercury bond length is 2.535 Å, a value close to but slightly longer than those found in other mercury(I) structures solved from three-dimensional work (Dorm, 1971*a, b*).

The structure can be described as consisting of puckered layers with characteristic groupings



in which three of four arsenate oxygen atoms are bonded to three different pairs with Hg-O distances of 2.16, 2.22 and 2.23 Å respectively. The fourth arsenate oxygen is linked to three other mercury atoms from the pairs related by translation along the *b* axis so that this oxygen atom has a distorted tetrahedral coordination. These mercury-oxygen distances are 2.46, 2.48 and 2.71 Å. The characteristic groupings are also linked together along the *a* axis by the $\text{Hg}(1) \cdots \text{O}(3^v)$ (2.68 Å) and $\text{Hg}(3) \cdots \text{O}(2)$ (2.42 Å) interactions, as well as along the *c* axis by the $\text{Hg}(2^{\text{iii}}) \cdots \text{O}(1^{\text{iv}})$ (2.47 Å) interaction. Considering all these interactions the structure proves to be a three-dimensional network, and only such a close packing can explain the high density of this compound. In contrast to the majority of the known mercury(I) structures, in the present structure the O-Hg-Hg-O groups deviate significantly from linearity. The $\text{Hg}(1^{\text{i}})-\text{Hg}(1)-\text{O}(2)$, $\text{Hg}(2)-\text{Hg}(3)-\text{O}(3^{\text{ii}})$ and $\text{Hg}(3)-\text{Hg}(2)-\text{O}(1^{\text{viii}})$ bond angles are 146, 153 and 157° respectively. The coordination polyhedron about each mercury atom can be described as a distorted tetrahedron. According to Grdenić (1965) the characteristic coordination of each mercury atom is digonal with two nearest neighbours (one mercury from the same pair and one oxygen atom at 2.16 to 2.23 Å), while the effective coordination is completed by two additional oxygen atoms at 2.42 to 2.71 Å.

The structure is shown in Fig. 1 and the interatomic

distances and angles together with standard deviations (*International Tables for X-ray Crystallography*, 1959) are listed in Table 4.

Table 4. *Interatomic distances and angles and their standard deviations*

The positions are denoted as follows

Superscript	<i>x</i>	<i>y</i>	<i>z</i>
None	<i>x</i>	<i>y</i>	<i>z</i>
i	- <i>x</i>	- <i>y</i>	- <i>z</i>
ii	- <i>x</i> +1	<i>y</i> +½	½- <i>z</i>
iii	<i>x</i> , ½- <i>y</i>	½+ <i>z</i>	
iv	- <i>x</i>	<i>y</i> -½	½- <i>z</i>
v	- <i>x</i> +1	<i>y</i> -½	½- <i>z</i>
vi	<i>x</i>	1+ <i>y</i>	<i>z</i>
vii	<i>x</i>	1- <i>y</i>	<i>z</i>
viii	<i>x</i> , ½- <i>y</i>	- <i>z</i>	
ix	- <i>x</i>	- <i>y</i> +1	- <i>z</i>
x	<i>x</i> , ½- <i>y</i>	<i>z</i> -½	

(a) Coordination about the mercury atoms.

Hg(1)——Hg(1 ⁱ)	2.535 (4) Å
Hg(2)——Hg(3)	2.535 (4)
Hg(1)——O(2)	2.23 (5)
Hg(2 ⁱⁱⁱ)——O(1)	2.22 (5)
Hg(3)——O(3 ⁱⁱ)	2.16 (4)
Hg(1)·····O(3 ^v)	2.68 (4)
Hg(1 ^{vi})·····O(4)	2.48 (5)
Hg(2 ⁱⁱⁱ)·····O(4)	2.46 (4)
Hg(2 ⁱⁱⁱ)·····O(1 ^{iv})	2.47 (5)
Hg(3 ⁱⁱ)·····O(4)	2.71 (5)
Hg(3)·····O(2)	2.42 (5)

Hg(1 ⁱ)——Hg(1)——O(2)	146 (1)°
Hg(1 ⁱ)——Hg(1)·····O(4 ^{vii})	124 (1)
Hg(1 ⁱ)——Hg(1)·····O(3 ^v)	105 (1)
O(2)——Hg(1)·····O(4 ^{vii})	88 (2)
O(4 ^{vii})·····Hg(1)·····O(3 ^v)	83 (1)
O(3 ^v)·····Hg(1)——O(2)	85 (2)
Hg(3)——Hg(2)——O(1 ^{viii})	157 (1)
Hg(3)——Hg(2)·····O(1 ^{ix})	114 (1)
Hg(3)——Hg(2)·····O(4 ^x)	105 (1)
O(1 ^{viii})——Hg(2)·····O(1 ^{ix})	88 (2)
O(4 ^x)·····Hg(2)——O(1 ^{viii})	83 (1)
O(1 ^{ix})·····Hg(2)·····O(4 ^x)	87 (2)
Hg(2)——Hg(3)·····O(2)	112 (1)
Hg(2)——Hg(3)·····O(4 ^y)	117 (1)
Hg(2)——Hg(3)——O(3 ⁱⁱ)	153 (2)
O(4 ^y)·····Hg(3)·····O(2)	79 (2)
O(2)·····Hg(3)——O(3 ⁱⁱ)	90 (2)
O(3 ⁱⁱ)——Hg(3)·····O(4 ^y)	81 (1)

(b) Distances and angles within the arsenate ion.

As——O(1)	1.67 (5) Å
As——O(2)	1.72 (5)
As——O(3)	1.64 (5)
As——O(4)	1.78 (4)
O(1)·····O(2)	2.76 (7)
O(1)·····O(3)	2.76 (7)
O(1)·····O(4)	2.75 (6)
O(2)·····O(3)	2.73 (7)
O(2)·····O(4)	2.82 (6)
O(3)·····O(4)	2.84 (6)
O(1)-As-O(2)	109 (3)°
O(1)-As-O(3)	113 (2)
O(1)-As-O(4)	106 (2)
O(2)-As-O(3)	109 (2)
O(2)-As-O(4)	108 (3)
O(3)-As-O(4)	112 (2)

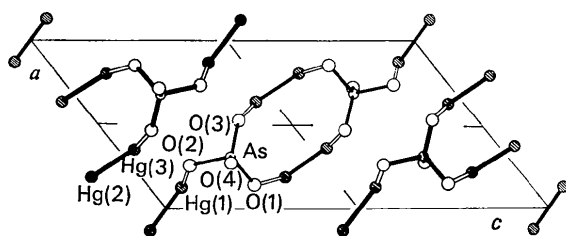


Fig. 1. The crystal structure of mercury(I) orthoarsenate projected along the *b* axis.

The dimensions within the AsO_4 tetrahedron are similar to those found in other arsenates (Poulsen & Calvo, 1967; Ferraris & Chiari, 1970; Khan, Straumanis & James, 1970; Linnros, 1970). One As–O distance is longer (1.78 Å) than the other three (average value 1.68 Å) and this oxygen [O(4)] is the one bonded to three different Hg–Hg pairs. The tetrahedral angles show smaller differences and range between 106 and 113° (mean value 109.5°).

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The Crystal and Molecular Structure of Di-(2-pyridyl)amine

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The crystal and molecular structure of the low-melting polymorph of di-(2-pyridyl)amine has been determined by three-dimensional X-ray analysis. The crystals are orthorhombic, space group *Pccn*, with $a = 18.42 \pm 0.005$, $b = 12.29 \pm 0.005$, $c = 7.69 \pm 0.007$ Å, and $Z = 8$. The structure was solved by direct methods and refined by full-matrix least-squares calculations to a final discrepancy index $R = 3.8\%$ for 565 observed reflections. Hydrogen bonds between molecules lead to the formation of dimeric units possessing twofold crystallographic symmetry and lying approximately parallel to the *ab* plane.

Introduction

The structural study of di-(2-pyridyl)amine was undertaken to obtain accurate structural parameters for this moiety since it has been widely used as a ligand in transition metal complexes. The crystal structures of three copper(II) complexes with this ligand (Johnson & Jacobson, 1971, 1973*a,b*) have been determined in this laboratory and McWhinnie (1970) has recently reviewed the ligand behavior of this compound.

The preparation of di-(2-pyridyl)amine was first

- reported by Wibaut & Dingemans (1923). Two crystalline modifications of the compound are known, one melting at 84° and a second that melts at 94°C. The structure of the low-melting polymorph was determined in this study.
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Experimental

Crystal data

Di-(2-pyridyl)amine, $\text{C}_{10}\text{H}_9\text{N}_3$, $M = 171$. Orthorhombic, *Pccn*, $a = 18.416 \pm 0.005$, $b = 12.294 \pm 0.005$,