

A Second Modification of Mercury(I) Orthoarsenate(V): Preparation and Crystal Structure of β -(Hg₂)₃(AsO₄)₂

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Light-yellow crystals of a second modification of mercury(I) orthoarsenate(V), β -(Hg₂)₃(AsO₄)₂, were obtained during a diffusion-controlled precipitation reaction starting from diluted solutions of Hg₂(NO₃)₂, Hg(NO₃)₂, and H₃AsO₄. The crystal structure was determined from a single crystal X-ray diffraction data set. The previously reported crystal structure of synthetic *chursinite*, α -(Hg₂)₃(AsO₄)₂, has also been re-investigated and refined with better accuracy and precision. Both phases crystallize in space group type $P2_1/c$ (no. 14) [α - (β -): $Z = 2$ (4), $a = 8.7915(6)$ (10.2034(18)), $b = 5.0699(4)$ (8.5875(14)), $c = 15.6839(10)$ (13.5172(19)) Å, $\beta = 128.761(1)$ (101.016(3))°, 1661 (3528) structure factors, 74 (146) parameters, $RF^2 > 2\sigma(F^2) = 0.0195$ (0.0492)] and are made up from Hg₂²⁺ dumbbells and AsO₄³⁻ tetrahedra as the main building units, but the topologies of the structures are quite different.

Key words: Mercury, Arsenate, Crystal Structure, Polymorphism

Introduction

In one of the older handbooks on mercury and its compounds within the system Hg-As-O-(H₂O) several phases with different oxidation states for both Hg and As have been reported [1]. In the meantime for many phases with As in oxidation state +V the corresponding structures were solved and refined but for the mentioned arsenites with As in oxidation state +III no structural information is available in the literature. Up to now the crystal structures of the mercury(I) compounds (Hg₂)₃(AsO₄)₂ [2], Hg₂(H₂AsO₄)₂ [3], (Hg₂)₂As₂O₇ [4] and (Hg₂)As₂O₆ [3], of the mercury(II) compounds Hg₃(AsO₄)₂ [5] and HgAs₂O₆ [3, 6], and of the phase (Hg₃)₃(AsO₄)₄ [7] have been described. In the latter compound triangular Hg₃⁴⁺ cluster cations with mercury in the formal oxidation state +1.33 are present. In addition to these compounds, the crystal structure of the hydrogenarsenate(V) (Hg₂)Hg(HAsO₄)₂ with two unique Hg₂²⁺ and Hg²⁺ cations was presented at the poster session of the 15th congress of the IUCR, Bordeaux, France 1990. However, in the book of abstracts [8] besides a very general description of the structure only space group and lattice parameters of this compound are given. To my knowledge neither information concerning prepa-

ration and atomic coordinates nor a proper structure description have since been published. In order to provide details of the preparation conditions and of the structure of this compound, crystal growth experiments for a subsequent structure determination were started. During this project single crystals of a new modification of mercury(I) arsenate(V), hereafter referred to as β -(Hg₂)₃(AsO₄)₂, were obtained. (Hg₂)₃(AsO₄)₂ occurs also in nature as the mineral *chursinite* [9]. Due to the existence of a new modification of the compound described in the present article and due to isotypism with the corresponding mercury(I) phosphate, α -(Hg₂)₃(PO₄)₂ [10], synthetic *chursinite* is referred to as α -(Hg₂)₃(AsO₄)₂. The crystal structure of β -(Hg₂)₃(AsO₄)₂ adopts a new structure type and is not isotypic with the second modification of mercury(I) phosphate, β -(Hg₂)₃(PO₄)₂ [10]. Along with a re-investigation of the previously reported crystal structure of synthetic *chursinite*, α -(Hg₂)₃(AsO₄)₂, the new crystal structure of the β -modification is described in this article.

Experimental Section

Preparation: Crystals of α - and β -(Hg₂)₃(AsO₄)₂ were grown during a diffusion-controlled precipitation reaction

Compound	α -(Hg_2) $_3(\text{AsO}_4)_2$ (synthetic <i>chursinite</i>)	β -(Hg_2) $_3(\text{AsO}_4)_2$
Diffractometer	– Siemens SMART CCD detector –	
Radiation; wavelength λ [Å]	– Mo- $\text{K}\alpha$; 0.71073 –	
Temperature [°C]	– 22(2) –	
Formula weight [g·mol $^{-1}$]	– 1481.38 –	
Space group	– $P2_1/c$ (no. 14) –	
Crystal dimensions [mm 3]	0.015 \times 0.02 \times 0.40	0.03 \times 0.03 \times 0.19
Crystal description	orange brown needle	light-yellow bar
Formula units Z	2	4
Lattice parameters		
a [Å]	8.7915(6)	10.2034(18)
b [Å]	5.0699(4)	8.5875(14)
c [Å]	15.6839(10)	13.5172(19)
β [°]	128.761(1)	101.016(3)
Volume [Å 3]	545.10(7)	1162.6(3)
V/Z [Å 3]	272.56	290.66
μ [mm $^{-1}$]	90.221	84.605
X-ray density [g·cm $^{-3}$]	9.025	8.464
Range $\theta_{\min} - \theta_{\max}$	2.74 \rightarrow 30.49	2.03 \rightarrow 30.51
Range $h; k; l$	–12 \rightarrow 12; –7 \rightarrow 7; –22 \rightarrow 22	–14 \rightarrow 14; –12 \rightarrow 12; –19 \rightarrow 19
Structure solution and refinement	– SHELXL97 [12] –	
Measured reflections	8004	17514
Independent reflections	1661	3528
Observed reflections [$I > 2\sigma(I)$]	1449	3016
R_i	0.0500	0.0636
Absorption correction	numerical; 8 faces HABITUS [11]	numerical; 10 faces HABITUS [11]
Coef. of Transmission $T_{\min}; T_{\max}$	0.0188; 0.3793	0.0316; 0.2304
Number of parameters	74	146
Extinction coefficient (SHELXL97)	0.00233(10)	0.00008(3)
Difference electron density [e·Å $^{-3}$] with distance to atom [Å]	$\Delta\rho_{\max} = 2.25$ [0.72; Hg2]; $\Delta\rho_{\min} = -2.05$ [0.81; As]	$\Delta\rho_{\max} = 5.92$ [0.72; Hg1]; $\Delta\rho_{\min} = -3.17$ [0.64; Hg1]
$R[F^2 > 2\sigma(F^2)]; wR(F^2 \text{ all})$	0.0195; 0.0451	0.0492; 0.1002
Goof	0.989	1.144
CSD-number	413886	413887

Table 1. Details of data collections, structure solutions and refinements.

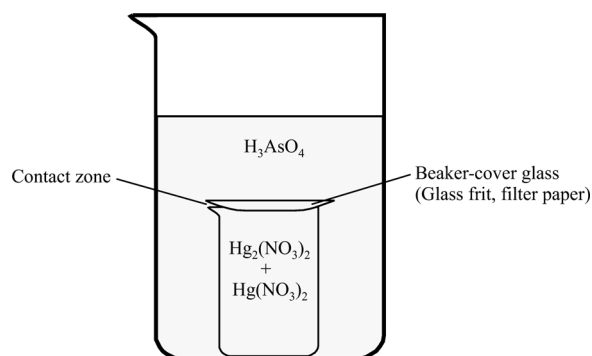


Fig. 1. Schematic experimental setup for the crystallisation of the title compounds.

aimed at the crystallisation of the mixed-valent compound $(\text{Hg}_2)\text{Hg}(\text{HAsO}_4)_2$ [8]. A 100 ml beaker containing diluted and slightly acidified equimolar solutions of $\text{Hg}_2(\text{NO}_3)_2$ and $\text{Hg}(\text{NO}_3)_2$ was placed in a 1500 ml beaker. The small beaker was filled up to the maximum level to avoid bubbles remaining in the solution. Then a beaker-cover glass was placed on

top of the small beaker so that only a small area around the beakers' muzzle was uncovered. Instead of the cover glass, filter paper or glass frits might be used for this purpose. Then 1000 ml demineralized water was carefully charged into the larger beaker and a diluted solution of arsenic acid was added into the large beaker. A schematic view of this experimental setup is illustrated in Fig. 1. This device was then placed in a dark room and set aside for one week. After the expiration of this term, crystals of good quality and different in habit and colour had formed both at the bottom of the large beaker and at the bottom of the small beaker. Large orange-brown needles of α -(Hg_2) $_3(\text{AsO}_4)_2$ up to 2 mm in length had mainly formed in the large beaker whereas light yellow bars of β -(Hg_2) $_3(\text{AsO}_4)_2$ up to 0.5 mm in length had mainly formed in the small beaker. Besides both modifications of mercurous orthoarsenate, honey coloured crystals of the mercuric orthoarsenate, $\text{Hg}_3(\text{AsO}_4)_2$, with mainly plate-like habit had crystallized in both beakers. No crystals of $(\text{Hg}_2)\text{Hg}(\text{HAsO}_4)_2$ had formed during these reactions.

Single crystal diffraction intensities of the crystals were collected in the ω -scan technique with 0.3° rotation width

Table 2. Atomic coordinates^a and equivalent isotropic displacement parameters [\AA^2].

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{eq} ^b
α -(Hg ₂) ₃ (AsO ₄) ₂				
Hg1	0.12750(4)	0.13295(4)	0.084184(18)	0.02584(8)
Hg2	0.19184(3)	0.35060(4)	-0.125619(17)	0.02244(7)
Hg3	0.35442(4)	0.64265(4)	0.040961(19)	0.02761(8)
As	0.68393(7)	0.12408(9)	0.22568(4)	0.01214(11)
O1	0.1444(6)	0.5090(7)	0.2833(3)	0.0187(8)
O2	0.2602(6)	0.5146(7)	0.1558(3)	0.0216(8)
O3	0.5426(6)	0.5285(7)	0.3831(3)	0.0218(8)
O4	0.3050(6)	0.9571(7)	0.2705(3)	0.0188(8)
β -(Hg ₂) ₃ (AsO ₄) ₂				
Hg1	0.05750(6)	0.24046(8)	0.46028(5)	0.03113(16)
Hg2	0.08852(7)	0.56645(9)	0.16471(5)	0.03628(18)
Hg3	0.18844(7)	0.10835(10)	0.19654(5)	0.0403(2)
Hg4	0.50860(8)	0.27309(7)	0.40374(5)	0.03481(18)
Hg5	0.51548(6)	0.05432(7)	0.14184(5)	0.02705(15)
Hg6	0.72709(6)	0.41388(7)	0.16637(5)	0.02601(15)
As1	0.16262(13)	0.58457(17)	0.40734(10)	0.0174(3)
As2	0.36658(12)	0.40455(15)	0.09670(10)	0.0132(2)
O1	0.0226(9)	0.6921(13)	0.3795(7)	0.020(2)
O2	0.1665(11)	0.0352(13)	0.0042(7)	0.025(2)
O3	0.1783(14)	0.4717(16)	0.3070(8)	0.041(3)
O4	0.2172(8)	0.3309(11)	0.1117(7)	0.0142(18)
O5	0.3736(11)	0.0957(12)	0.4731(8)	0.025(2)
O6	0.3759(11)	0.5818(11)	0.1489(8)	0.021(2)
O7	0.4925(9)	0.2984(12)	0.1651(8)	0.022(2)
O8	0.7091(9)	0.2117(13)	0.0674(9)	0.030(3)

^a All atoms are located on the general position $4e$; ^b $U_{eq} = (1/3)\sum_j U_{ij} a_i^* a_j^* \cdot a_j$.

Table 3. Selected interatomic distances [\AA], angles [$^\circ$] and bond valence sums (BVS) [v.u.].

α -(Hg ₂) ₃ (AsO ₄) ₂			β -(Hg ₂) ₃ (AsO ₄) ₂		
Hg1	O2	2.174(4)	Hg1	O2	2.247(10)
Hg1	O4	2.462(3)	Hg1	O4	2.439(9)
Hg1	Hg1	2.5305(4)	Hg1	Hg2	2.5189(9)
Hg1	O3	2.663(4)	Hg1	O1	2.524(10)
Hg2	O1	2.189(4)	Hg2	O3	2.127(11)
Hg2	O1	2.492(4)	Hg2	Hg1	2.5189(9)
Hg2	Hg3	2.5269(3)	Hg2	O4	2.586(9)
Hg2	O4	2.573(4)	Hg2	O6	2.983(10)
Hg3	O3	2.172(4)	Hg3	O4	2.276(9)
Hg3	O2	2.494(4)	Hg3	O1	2.316(9)
Hg3	Hg2	2.5269(3)	Hg3	Hg6	2.5209(9)
Hg3	O4	2.740(4)	Hg3	O2	2.643(9)

and 30 s exposure time per frame using a SMART three-circle diffractometer (Siemens) equipped with a CCD camera. Four independent sets of 600 frames were recorded thus scanning the whole reciprocal sphere. The measured intensities were corrected for Lorentz and polarization effects, and due to the high linear absorption coefficients absorption corrections were applied using the program HABITUS [11]. The crystal structure of β -(Hg₂)₃(AsO₄)₂ was solved by direct methods and refined with the SHELX97 program package [12]. Crystal data of this new compound were standard-

Table 3 (continued).

α -(Hg ₂) ₃ (AsO ₄) ₂			β -(Hg ₂) ₃ (AsO ₄) ₂		
\bar{d} (Hg-Hg)			2.5287	Hg4 O6 2.217(9)	
\bar{d} (Hg-O)			2.44	Hg4 O5 2.364(10)	
				Hg4 Hg5 2.4929(9)	
				Hg4 O8 2.714(11)	
				Hg5 O7 2.139(10)	
				Hg5 Hg4 2.4929(9)	
				Hg5 O8 2.739(12)	
				Hg5 O6 2.844(10)	
				Hg6 O8 2.178(10)	
				Hg6 O5 2.511(11)	
				Hg6 Hg3 2.5209(9)	
				Hg6 O7 2.588(10)	
				\bar{d} (Hg-Hg)	
				\bar{d} (Hg-O)	
				2.5109	
				2.47	
As	O3	1.690(4)	As1	O2	1.659(10)
As	O4	1.690(4)	As1	O1	1.682(9)
As	O2	1.692(4)	As1	O8	1.689(11)
As	O1	1.704(4)	As1	O3	1.699(12)
\bar{d} (As-O)			1.694	As2 O6 1.672(9)	
				As2 O5 1.686(10)	
				As2 O7 1.697(10)	
				As2 O4 1.698(8)	
				\bar{d} (As-O)	
				1.685	
O2	Hg1	Hg1	148.39(11)	O2	Hg1 Hg2 152.3(3)
O1	Hg2	Hg3	156.34(10)	O3	Hg2 Hg1 158.5(3)
O3	Hg3	Hg2	150.09(10)	O4	Hg3 Hg6 150.2(2)
				O6 Hg4 Hg5 132.2(2)	
				O7 Hg5 Hg4 167.7(3)	
				O8 Hg6 Hg3 163.0(3)	
O3	As	O2	111.3(2)	O2	As1 O1 114.1(6)
O4	As	O2	108.56(17)	O2	As1 O8 109.9(5)
O3	As	O1	111.30(18)	O1	As1 O8 106.4(5)
O4	As	O1	108.09(18)	O2	As1 O3 106.5(6)
O2	As	O1	108.41(19)	O1	As1 O3 109.9(5)
				O8 As1 O3 110.1(7)	
				O6 As2 O5 114.2(5)	
				O6 As2 O7 106.4(5)	
				O5 As2 O7 111.6(5)	
				O6 As2 O4 105.6(5)	
				O5 As2 O4 109.1(5)	
				O7 As2 O4 109.8(5)	
BVS: Hg1 1.06, Hg2 1.07, Hg3			BVS: Hg1 1.07, Hg2 1.05, Hg3		
1.05, As 4.87, O1 1.81, O2 1.87,			1.08, Hg4 1.13, Hg5 1.08, Hg6		
O3 1.80, O4 1.69.			1.08, As1 5.04, As2 4.94, O1		
			1.74, O2 1.84, O3 1.71, O4		
			1.95, O5 1.69, O6 1.82, O7 1.85		
			O8 1.88.		

ized with the program STRUCTURE-TIDY [13]. The crystal structure of α -(Hg₂)₃(AsO₄)₂ was refined in the same cell setting in $P2_1/c$ and with the atomic parameters as starting values as given in the original reference [2]. For both refinements, the displacement parameters of all atoms were refined anisotropically, and the final difference Fourier maps did not indicate any additional atomic sites. The highest difference peaks were located close to the mercury positions. Further details of the data collections and refinements are

summarized in Table 1, atomic parameters and isotropic displacement parameters are given in Table 2, and selected distances, angles as well as bond-valence sums (BVS) for the individual atoms are listed in Table 3. Additional crystallographic information on the structures is available from the Fachinformationszentrum Karlsruhe, D-76344 Eggenstein-Leopoldshafen, Germany, email: crysdata@fiz-karlsruhe.de, by quoting the literature citation, the name of the author and the depository numbers listed at the end of Table 1. Drawings of structural details were produced using the program ATOMS [14].

Results and Discussion

Preparation: Attempts to prepare the mixed-valent phase $(\text{Hg}_2)\text{Hg}(\text{HAsO}_4)_2$ [8] have failed in this project. When slightly acidified equimolar solutions of $(\text{Hg}_2)(\text{NO}_3)_2$ and $\text{Hg}(\text{NO}_3)_2$ were reacted with diluted H_3AsO_4 at reflux temperatures for several minutes, canary coloured microcrystalline $(\text{Hg}_3)_3(\text{AsO}_4)_4$ was obtained as a single phase product. Reactions under hydrothermal conditions (180 °C, 5d) with equimolar quantities of $(\text{Hg}_2)(\text{NO}_3)_2$, $\text{Hg}(\text{NO}_3)_2$ and H_3AsO_4 as aqueous solutions yielded α -(Hg_2) $_3(\text{AsO}_4)_2$ as the only reaction product, whereas hydrothermal reactions with Na_2HAsO_4 solution led to disproportionation of the employed Hg_2^{2+} and resulted in elementary Hg and $\text{Hg}_3(\text{AsO}_4)_2$ as the crystalline phase. The diffusion-controlled precipitation reaction described in the experimental section finally yielded a mixture of the phases α -(Hg_2) $_3(\text{AsO}_4)_2$, β -(Hg_2) $_3(\text{AsO}_4)_2$ and $\text{Hg}_3(\text{AsO}_4)_2$. It was not possible to prepare β -(Hg_2) $_3(\text{AsO}_4)_2$ as a single microcrystalline phase by fast precipitation of a $(\text{Hg}_2)(\text{NO}_3)_2$ solution with solutions of different concentrations of Na_2HAsO_4 or H_3AsO_4 , or by adding the solutions in reverse order. All experiments gave α -(Hg_2) $_3(\text{AsO}_4)_2$ as the only reaction product. This was likewise the only phase obtained during similar diffusion-controlled precipitation reactions as described in the experimental section but without addition of a $\text{Hg}(\text{NO}_3)_2$ solution.

Structure: The structure of α -(Hg_2) $_3(\text{AsO}_4)_2$ was solved and refined nearly 30 years ago from integrated Weissenberg data [2]. Although the main features of the crystal structure have been correctly described, some unusual bond lengths have been reported, especially for the AsO_4 tetrahedron. The As-O distances determined in the previous study vary from 1.67(5) to 1.789(4) Å, but there is no structural reason for such a large range. Especially the As-O distance of 1.79 Å

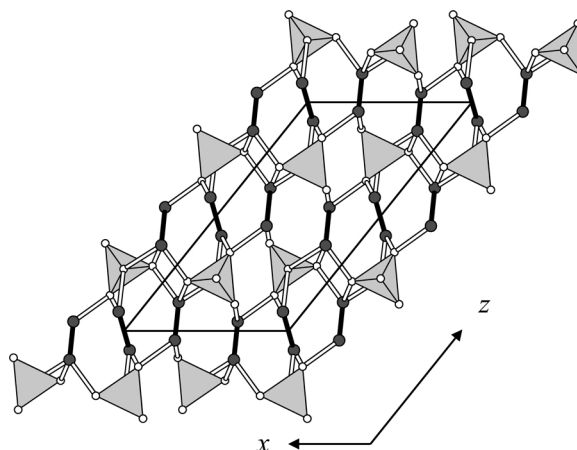


Fig. 2. α -(Hg_2) $_3(\text{AsO}_4)_2$. Projection of the crystal structure along [010]. Hg atoms are given as dark-grey spheres, AsO_4 tetrahedra as light-grey polyhedra. Only Hg-O bonds < 2.6 Å are plotted.

is too long for a tetrahedral AsO_4 group but is rather typical for a six-coordinate As atom in an AsO_6 octahedron. Therefore a re-determination of this structure with a modern single crystal diffractometer appeared appropriate. The newly determined interatomic distances and angles indeed show a much higher precision and accuracy compared to the previous results. Since a detailed structure description of α -(Hg_2) $_3(\text{AsO}_4)_2$ has already been given in the original study [2] as well as for the isotopic phosphate α -(Hg_2) $_3(\text{PO}_4)_2$ [4], only the main structural features are reported here.

The structure of the α -modification is made up from Hg_2^{2+} dumbbells and AsO_4^{3-} tetrahedra forming puckered layers parallel to (001) which are piled up along [001] (Fig. 2). Via additional As-O and Hg-O interactions parallel to the [010] direction, a densely-packed three-dimensional network is achieved. The Hg-Hg distances of the two independent Hg_2^{2+} dumbbells with a mean of 2.5287 Å are nearly identical to those observed for the isotopic phosphate but are slightly longer than the average of 2.514 Å calculated for more than 70 different Hg_2^{2+} dumbbells in various oxo compounds. Under consideration that a significant Hg-O interaction is realized for distances < 3.0 Å, each Hg atom is surrounded by three O atoms with a mean Hg-O distance of 2.44 Å. In contrast to many other mercurous oxo compounds, the angles between the tightly bonded O atoms and the Hg_2^{2+} dumbbells with a mean of ca. 152° deviate considerably from linearity. The AsO_4^{3-} group is only

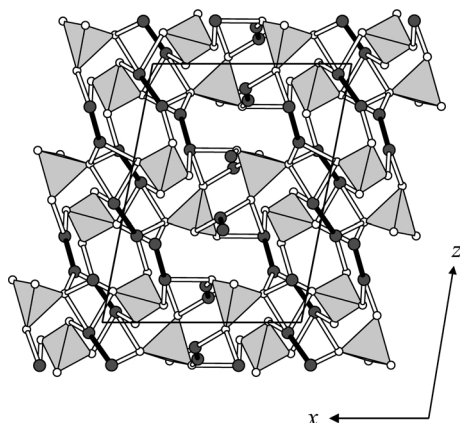


Fig. 3. β -(Hg₂)₃(AsO₄)₂. Projection of the crystal structure along [010]. Hg atoms are given as dark-grey spheres, AsO₄ tetrahedra as light-grey polyhedra. Only Hg-O bonds < 2.6 Å are plotted.

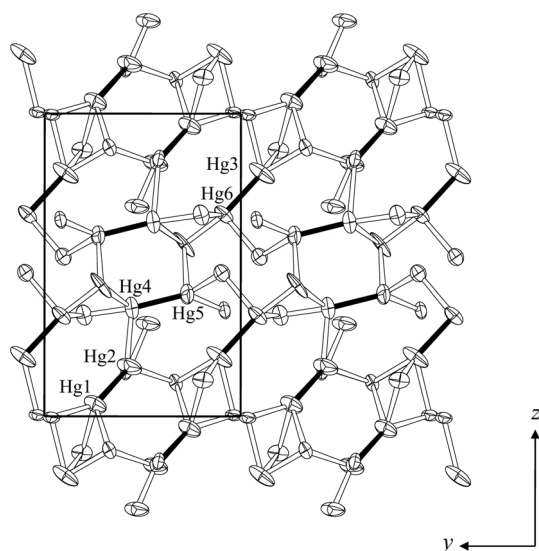


Fig. 4. β -(Hg₂)₃(AsO₄)₂. Part of the Hg-O layer spreading parallel to [101] in projection along [100] drawn with displacement parameters at the 90% probability level. Hg-O bonds are given for distances < 3.0 Å.

slightly distorted from the geometry of an ideal tetrahedron. The As-O distances and \langle O-As-O \rangle angles range from 1.690(4) to 1.704(4) Å and 108.4(2) to 111.3(2)°, respectively.

Like in the α -modification, the structure of β -(Hg₂)₃(AsO₄)₂ can be described in terms of puckered layers of Hg₂²⁺ dumbbells and AsO₄³⁻ tetrahedra as the main building units forming a three-dimensional network. In the β -phase, the cationic layers spread parallel to [101] and are inter-connected *via* the anionic

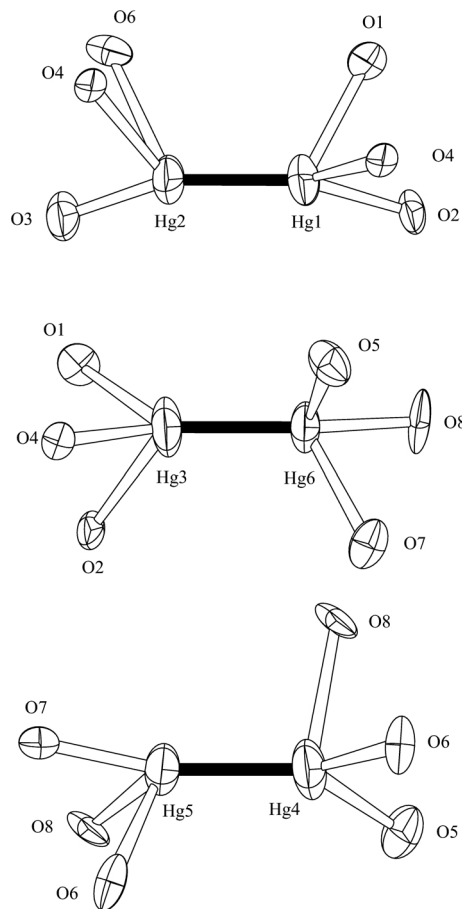


Fig. 5. β -(Hg₂)₃(AsO₄)₂. The three unique Hg₂²⁺ dumbbells with surrounding O atoms drawn with anisotropic displacement parameters at the 90% probability level.

AsO₄ entities (Fig. 3, 4). For all atoms in both modifications the bond valence sum (BVS) calculations [15] using the parameters of *Breese* and *O'Keeffe* [16] are comparable and are within the range of the expected values (Table 3). Although the mean Hg-Hg, As-O and Hg-O distances of both modifications are nearly the same, the α -form shows a density 6.2% greater than the β -form owing to the slightly larger voids present in the structure of the new modification.

The structure of β -(Hg₂)₃(AsO₄)₂ contains three unique Hg₂²⁺ dumbbells comprising six crystallographically different Hg atoms. The individual Hg-Hg distances of 2.5189(9), (Hg1-Hg2), 2.5209(9) (Hg3-Hg6) and 2.4929(9) Å (Hg4-Hg5) are characteristic for this group and scatter only slightly around the aforementioned mean of 2.514 Å for various mercurous oxo compounds. The oxygen coordination around the

Hg₂²⁺ dumbbells is asymmetrical (Fig. 5). Like in the α -modification, each Hg atom is surrounded by three oxygen atoms with distances ranging from 2.127(11) for the shortest to 2.984(10) Å for the longest Hg-O bonds with an overall mean Hg-O distance of 2.47 Å. Just like for α -(Hg₂)₃(AsO₄)₂, the \langle Hg-Hg-O \rangle angles in the β -modification ranging from 132 to 168° are far from linearity.

The two crystallographically independent AsO₄ tetrahedra in β -(Hg₂)₃(AsO₄)₂ show similar As-O distances typical for this group with nearly identical

means of 1.682 (As1) and 1.688 Å (As2). Compared to the AsO₄ group in α -(Hg₂)₃(AsO₄)₂, both anions in the β -form show a slightly greater distortion.

The coordination numbers of the O atoms in the new modification range from 2 to 4. O3 has CN = 2, is very tightly bonded to Hg and shows a long bond to an As atom. The atoms O1, O2, O5 and O7 have CN = 3 and are bonded to one As atom and two Hg atoms at short and medium distances. O4, O6 and O8 show CN = 4 with a bond to As atoms and three bonds to different Hg atoms.

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