

The crystal structure of tedhadleyite, $\text{Hg}^{2+}\text{Hg}_{10}^{1+}\text{O}_4\text{I}_2(\text{Cl},\text{Br})_2$, from the Clear Creek Claim, San Benito County, California

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ABSTRACT

The crystal structure of tedhadleyite, ideally $\text{Hg}^{2+}\text{Hg}_{10}^{1+}\text{O}_4\text{I}_2(\text{Cl},\text{Br})_2$, triclinic, $A\bar{1}$, a 7.0147(5), b 11.8508(7), c 12.5985(8) Å, α 115.583(5), β 82.575(2), γ 100.619(2)°, V 927.0(2) Å³, $Z = 2$, was solved by direct methods and refined to an R_1 index of 4.5% for 2677 unique reflections. There are six symmetrically distinct Hg sites in tedhadleyite: Hg(1) is occupied by Hg^{2+} and Hg(2–6) are occupied by Hg^+ that forms three $[\text{Hg}-\text{Hg}]^{2+}$ dimers with Hg–Hg separations between 2.527 and 2.556 Å. These $[\text{Hg}-\text{Hg}]^{2+}$ dimers have strong covalent bonds to O atoms, forming pseudo-linear O–Hg–Hg–O arrangements, and weak bonds to halogen and O atoms at high angles to the dimer axis. The [O–Hg–Hg–O] groups share anions to form four-membered square rings of composition $[\text{Hg}_8\text{O}_4]$ that link along [100] via [O–Hg–Hg–O] groups and along [001] via [O–Hg–O] groups, forming rectangular rings of composition $[\text{Hg}_{14}\text{O}_8]$. The rings form a corrugated layer that interweaves with a symmetrically related layer whereby the [O–Hg(6)–Hg(6)–O] linking groups of one layer pass through the centres of the square $[\text{Hg}_8\text{O}_4]$ rings of the other layer to form $[\text{Hg}_{11}\text{O}_4]$ complex slabs parallel to (010) that link through Hg–I and Hg–Br,Cl bonds.

KEYWORDS: tedhadleyite, crystal structure, mercury mineral, San Benito County, California.

Introduction

TEDHADLEYITE, ideally $\text{Hg}^{2+}\text{Hg}_{10}^{1+}\text{O}_4\text{I}_2(\text{Cl},\text{Br})_2$, is a recently described supergene mineral from a small prospect pit near the long-abandoned Clear Creek mercury mine, New Idria district, San Benito County, California (Roberts *et al.*, 2002). It is associated with native mercury, calomel and traces of cinnabar, eglestonite and montroydite in a host rock composed mainly of quartz and magnesite. Tedhadleyite occurs as a spheroidal mass that is partly hollow, suggesting that it formed as an *in situ* replacement of native mercury during a period of high activity of I (with lower Cl and Br) in the fluid or vapour phase (Roberts *et al.*, 2002). As part of our general interest in the crystal chemistry of mercury minerals (Hawthorne *et al.*, 1994; Roberts *et al.*,

2002, 2003*a,b*, 2005; Cooper and Hawthorne, 2003), we report here the crystal-structure determination of tedhadleyite.

X-ray data collection and structure refinement

A thin fragment of tedhadleyite was cut from the ~200 µm round grain used for the precession study done as part of the new-mineral description (Roberts *et al.*, 2002) and mounted on a Bruker four-circle diffractometer equipped with a 1K CCD detector at a crystal-to-detector distance of 4 cm. In excess of a sphere of intensity data was collected to 60°2θ using a frame width of 0.2° and an exposure time per frame of 90 s. The data were then integrated using a triclinic primitive cell and Lorentz, polarization and background corrections were applied. The structure was solved and refined using the *SHELXTL* system of programs (Bruker, 1997), and then the cell was reoriented to an A-centred setting so that the main structural features coincided with the crystallographic axes.

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TABLE 1. Miscellaneous information for tedhadleyite.

a (Å)	7.0147(5)	Crystal size (mm)	$0.02 \times 0.08 \times 0.09$
b	11.8508(7)	Radiation/Mono	Mo- $K\alpha$ /Graphite
c	12.5985(8)	No. of reflections	7918
α (°)	115.583(5)	No. in Ewald sphere	4784
β	82.575(2)	No. unique reflections	2677
γ	100.619(2)	R_{merge} %	3.5
V (Å ³)	927.0(2)	R_1 %	4.5
Space group	$A\bar{1}$	wR_2 %	12.6
Z	2	Goodness of Fit (GoF)	1.064
μ (mm ⁻¹)	95.9		
$D_{\text{calc.}}$ (g/cm ³)	9.43		

The final unit-cell parameters (Table 1) are based on least-squares refinement of 4367 reflections ($I > 10\sigma I$). An empirical absorption correction was applied using the *SADABS* program, and 7918 reflections were averaged to give 4784 reflections within the Ewald sphere. In the space group $A\bar{1}$, there are 2677 unique data with merging in Laue group $\bar{1}$ of 3.5%. The structure was solved by direct methods in $P\bar{1}$, transformed to $A\bar{1}$ (100/011/0 $\bar{1}$ 1) and refined by least-squares against F^2 to $R_1 = 4.5\%$ with anisotropic-displacement parameters for all atoms. Atom positions and displacement parameters are listed

in Table 2, and selected interatomic distances and angles are given in Table 3.

Description of the structure

There are six symmetrically distinct *Hg* sites in tedhadleyite (Table 2). The *Hg*(1) site lies on a centre of symmetry and is coordinated by two O atoms at a distance of 2.046 Å, two I atoms at 3.255 Å, and two *X* anions (where the *X* site is occupied by both Cl and Br) at a distance of 3.479 Å, in a geometry that is characteristic of divalent Hg. There are five *Hg* sites, *Hg*(2–6), at

TABLE 3. Selected bond distances (Å) and angles Hg–Hg– ϕ (°) ($\phi = O, I, X$) in tedhadleyite.

<i>Hg</i> (1)			<i>Hg</i> (4)– <i>Hg</i> (5)	2.5271(9)	
<i>Hg</i> (1)–O(1),a	2.046(12)	$\times 2$	– <i>Hg</i> (4)–O(2)	2.173(10)	164.5(3)
<i>Hg</i> (1)– <i>X</i> (1)b,c	3.479(3)	$\times 2$	– <i>Hg</i> (5)–O(1)h	2.175(11)	164.9(3)
<i>Hg</i> (1)–I(1)d,e	3.2548(11)	$\times 2$	– <i>Hg</i> (4)–O(2)g	2.538(11)	106.5(2)
			– <i>Hg</i> (4)– <i>X</i> (1)g	3.257(3)	119.00(7)
<i>Hg</i> (2)– <i>Hg</i> (3)	2.5308(9)		– <i>Hg</i> (4)– <i>X</i> (1)	3.500(3)	110.69(7)
– <i>Hg</i> (2)–O(2)	2.131(10)	172.8(3)	– <i>Hg</i> (5)– <i>X</i> (1)i	3.265(3)	90.95(8)
– <i>Hg</i> (3)–O(1)f	2.134(10)	174.0(3)	– <i>Hg</i> (5)–I(1)g	3.3766(14)	94.51(4)
– <i>Hg</i> (2)– <i>X</i> (1)	2.979(3)	99.92(6)	– <i>Hg</i> (5)–I(1)h	3.3771(16)	106.44(3)
– <i>Hg</i> (2)–I(1)	3.2555(13)	97.62(3)	– <i>Hg</i> (5)–I(1)j	3.4281(15)	107.90(4)
– <i>Hg</i> (3)–O(1)c	2.641(12)	93.4(2)	<i>Hg</i> (6)– <i>Hg</i> (6)	2.5564(14)	
– <i>Hg</i> (3)– <i>X</i> (1)d	3.379(4)	104.03(6)			
– <i>Hg</i> (3)–I(1)c	3.4914(13)	109.62(3)	– <i>Hg</i> (6)–O(2)	2.133(11)	167.1(3)
– <i>Hg</i> (3)–I(1)f	3.6676(14)	107.58(3)	– <i>Hg</i> (6)– <i>X</i> (1)g	3.095(3)	116.42(7)
			– <i>Hg</i> (6)– <i>X</i> (1)l	3.471(4)	91.95(6)

a: \bar{x} , $\bar{y}+1$, \bar{z} ; b: $x-1$, $y-1/2$, $z-1/2$; c: $\bar{x}+1$, $\bar{y}+3/2$, $\bar{z}+1/2$; d: x , $y-1/2$, $z-1/2$; e: \bar{x} , $\bar{y}+3/2$, $\bar{z}+1/2$; f: $\bar{x}+1$, y , z ; g: $\bar{x}+1$, $\bar{y}+3/2$, $\bar{z}+3/2$; h: $\bar{x}+1$, $\bar{y}+1$, $\bar{z}+1$; i: $\bar{x}+2$, $\bar{y}+3/2$, $\bar{x}+3/2$; j: x , $y-1/2$, $z+1/2$; k: \bar{x} , $\bar{y}+1$, $\bar{z}+1$; l: $x-1$, y , z .

TABLE 2. Final atomic coordinates and displacement parameters (\AA^2) for tedhadleyite.

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> ₁₁	<i>U</i> ₂₂	<i>U</i> ₃₃	<i>U</i> ₂₃	<i>U</i> ₁₃	<i>U</i> ₁₂	<i>U</i> _{eq}
Hg(1)	0	½	0	0.0273(4)	0.0256(4)	0.0166(4)	0.0058(3)	−0.0021(3)	0.0086(3)	0.0235(2)
Hg(2)	0.63055(9)	0.67337(7)	0.47594(5)	0.0293(3)	0.0272(3)	0.0209(3)	0.0101(2)	0.0047(2)	0.0058(3)	0.02616(17)
Hg(3)	0.87144(10)	0.64468(7)	0.30156(5)	0.0330(3)	0.0293(4)	0.0228(3)	0.0116(3)	0.0072(2)	0.0059(3)	0.02875(17)
Hg(4)	0.54443(10)	0.60364(7)	0.72377(6)	0.0384(4)	0.0249(3)	0.0268(6)	0.0098(3)	−0.0081(3)	0.0109(3)	0.02892(18)
Hg(5)	0.70164(12)	0.47617(9)	0.79600(7)	0.0467(4)	0.0436(5)	0.0336(4)	0.0168(3)	−0.0086(3)	0.0222(4)	0.0383(2)
Hg(6)	0.14552(10)	0.57424(8)	0.55608(7)	0.0255(3)	0.0344(4)	0.0392(4)	0.0143(3)	−0.0141(3)	−0.0034(3)	0.03332(19)
O(1)	0.0950(16)	0.6166(12)	0.1643(10)	0.018(5)	0.029(6)	0.027(5)	0.019(5)	0.003(4)	0.008(5)	0.022(2)
O(2)	0.4250(16)	0.6725(11)	0.6158(9)	0.027(6)	0.016(5)	0.019(5)	0.010(4)	−0.001(4)	0.001(4)	0.020(2)
I(1)	0.34154(17)	0.82199(12)	0.41700(9)	0.0320(6)	0.0329(6)	0.0242(5)	0.0055(4)	−0.0040(4)	0.0145(5)	0.0304(3)
X(1)	0.8910(5)	0.8307(3)	0.6725(3)	0.0379(18)	0.043(2)	0.0266(15)	0.0065(13)	−0.0008(11)	−0.0100(13)	0.0411(11)

* refined occupancy: 0.405(9) Br + 0.595 Cl.

which the constituent Hg forms three $[\text{Hg}-\text{Hg}]^{2+}$ dimers: $\text{Hg}(2)-\text{Hg}(3)$, $\text{Hg}(4)-\text{Hg}(5)$ and $\text{Hg}(6)-\text{Hg}(6)$ (Fig. 1), with Hg–Hg separations of between 2.527 and 2.556 Å. These values are similar to those observed in other $[\text{Hg}-\text{Hg}]^{2+}$ -bearing minerals: e.g. 2.543 Å in shakhovite (Tillmanns *et al.*, 1982); 2.53 Å in magnolite (Grice, 1989); 2.522 and 2.524 Å in edgarbailleyite (Angel *et al.*, 1990); 2.526 Å in watersite (Groat *et al.*, 1995); 2.536 and 2.544 Å in hanawaltite (Grice, 1999); 2.502–2.565 Å in poyarkovite (Vasil'ev *et al.*, 1999); 2.533–2.592 Å in vasil'yevite (Cooper and Hawthorne, 2003). These $[\text{Hg}-\text{Hg}]^{2+}$ dimers have strong covalent bonds to O atoms at each end of the dimer, with Hg–O distances in the range 2.131–2.175 Å, and form pseudo-linear O–Hg–Hg–O arrangements with Hg–Hg–O angles in the range 164–174°. Moreover, these terminal Hg–O bonds are the same lengths in each $[\text{Hg}-\text{Hg}]^{2+}$ dimer: 2.131 and 2.134 Å for $\text{Hg}(2)-\text{Hg}(3)$, 2.173 and 2.175 Å for $\text{Hg}(4)-\text{Hg}(5)$, 2.133 and 2.133 Å for $\text{Hg}(6)-\text{Hg}(6)$. These pseudo-linear O–Hg–Hg–O groups are typical of structures with Hg^{2+} cations that bond directly to form $[\text{Hg}_2]^{2+}$ dimers. The $\text{Hg}(2)-\text{Hg}(3)$ and $\text{Hg}(4)-\text{Hg}(5)$ dimers each link to one other O atom at a longer distance (2.641 and 2.538 Å, respectively) and at a high angle (93.4 and 106.5°) to the dimer axis, whereas the $\text{Hg}(6)-\text{Hg}(6)$ dimer does not link to such an O atom. The $\text{Hg}(2)-\text{Hg}(3)$ and $\text{Hg}(4)-\text{Hg}(5)$ dimers each link to three I atoms at distances in the range 3.256–3.668 Å, and all at high angles (94.5–109.6°) to the dimer axis, whereas the $\text{Hg}(6)-\text{Hg}(6)$ dimer does not link to I atoms. All three dimers link to anions of the mixed-anion site X(1) that is occupied by Br and Cl. These distances fall in the range 2.979–3.500 Å, and all at high angles (91.0–119.0°) to the dimer axis. The crystal chemistry of these minerals and similar synthetics has been reviewed by Pervukhina *et al.* (1999*a,b*) and Borisov *et al.* (1999).

Connectivity

In the tedhadleyite crystal structure, strongly bonded $[\text{O}-\text{Hg}-\text{O}]$ groups join at their anion vertices to form a four-membered square ring of composition $[\text{Hg}_8\text{O}_4]$ (Fig. 2). These $[\text{Hg}_8\text{O}_4]$ rings link together along $[100]$ via $[\text{O}-\text{Hg}-\text{Hg}-\text{O}]$ groups and along $[001]$ via

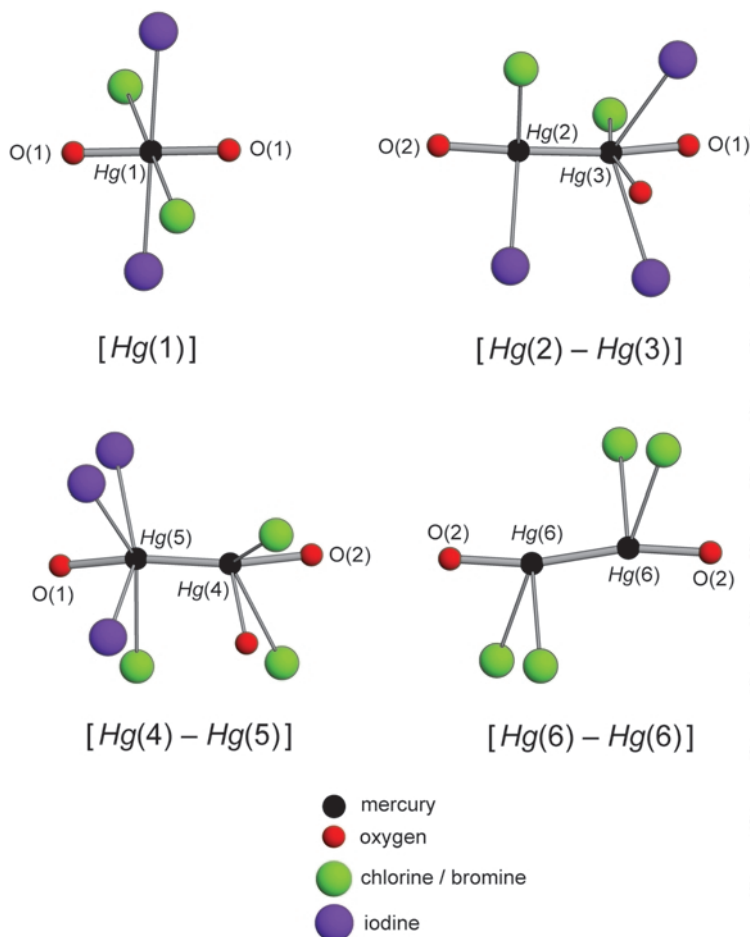


FIG. 1. The coordination of the six Hg -sites in tedhadleyite; thick lines – strong axial bonds; thin lines – weak meridional bonds.

TABLE 4. Mixed Hg^+/Hg^{2+} minerals.

Formula	$\frac{Hg^{2+}}{Hg^{2+}+Hg^+}$	Ref.
Deanesmithite	$Hg_2^+Hg_3^{2+}Cr^{6+}O_5S_2$	[1]
Terlinguaite	$Hg^+Hg^{2+}OCl$	[2]
Aurivilliusite	$Hg^+Hg^{2+}OI$	[3]
CCUK-15	$Hg_{10}^+Hg_3^{2+}O_6I_2 (Cl,Br)_2$	[4]
Wattersite	$Hg_4^+Hg_2^{2+}Cr^{6+}O_6$	[5]
Hanawaltite	$Hg_6^+Hg^{2+}O_3Cl_2$	[6]
Tedhadleyite	$Hg_{10}^+Hg^{2+}O_4I_2(Cl,Br)_2$	[7]

References: [1] Roberts *et al.* (1993); [2] Palache *et al.* (1951); [3] Roberts *et al.* (2004); [4] Dunning *et al.* (2005); [5] Roberts *et al.* (1991); [6] Roberts *et al.* (1996); [7] Roberts *et al.* (2002).

CRYSTAL STRUCTURE OF TEDHADLEYITE

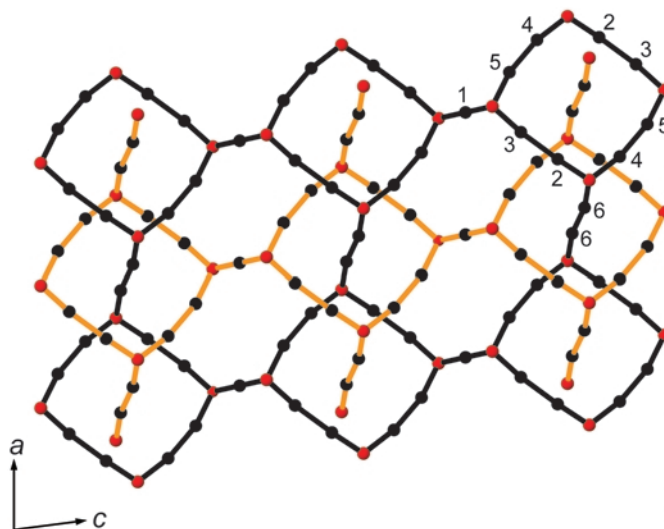


FIG. 2. The crystal structure of tedhadleyite projected down a direction 15° from [010]. Legend as in Fig. 1, with strong bonding between [O–Hg–Hg–O] and [O–Hg–O] groups shown as thick black lines indicating bonds belonging to one net; thick orange lines indicate bonds belonging to a symmetrically equivalent net. Hg atoms are labelled.

[O–Hg–O] groups and, in the process, generate larger distorted rectangular rings of composition $[\text{Hg}_{14}\text{O}_8]$. The interconnected rings form a corrugated layer that interweaves with a symmetrically related layer whereby the [O–Hg(6)–Hg(6)–O] linking groups of one layer pass through the centres of the square $[\text{Hg}_8\text{O}_4]$ rings of the other layer to form an interwoven $[\text{Hg}_{11}\text{O}_4]$ complex slab parallel to (010). Inter-slab connectivity is provided by bonds to the halogen sites which are midway between adjacent slabs (Fig. 3).

Other $\text{Hg}^+/\text{Hg}^{2+}$ compounds

All known Hg minerals that contain both Hg^+ and Hg^{2+} are listed in Table 4. Tedhadleyite contains the smallest fraction of Hg^{2+} relative to total Hg content of all these minerals. All the minerals listed in Table 4 occur at the Clear Creek locality in extremely small amounts and their occurrences suggest diverse micro-environments that can be separated by as little as a few millimetres (Dunning *et al.*, 2005). A review of the mixed $\text{Hg}^+/\text{Hg}^{2+}$ compounds on the ICSD showed that the lowest $\text{Hg}^{2+}/(\text{Hg}^{2+}+\text{Hg}^+)$ ratio for a synthetic inorganic

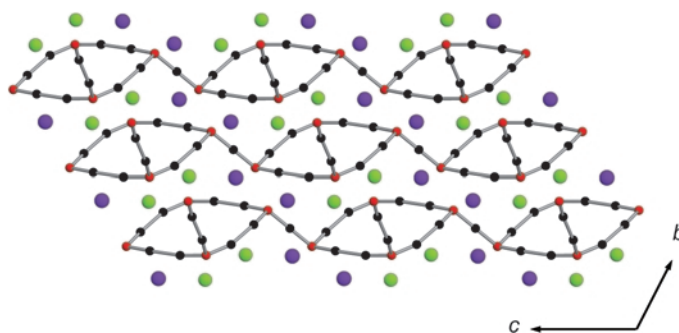


FIG. 3. The crystal structure of tedhadleyite projected down [100]. Legend as in Fig. 1; weak meridional bonds not shown.

compound is 0.20, which occurs in the two polymorphs of $\text{Hg}^{2+}(\text{Hg}_2^+\text{O})_2(\text{Re}^{7+}\text{O}_4)_2$ (Picard *et al.*, 1982; Schriewer-Poettgen and Jeitschko, 1995). Both of these structures are based on layered $[\text{Hg}-\text{O}-]$ ring complexes, similar to the structural connectivity in tedhadleyite.

Related structures

Several other structures involve anion sharing of $[\text{O}-\text{Hg}-\text{O}]$ and $[\text{O}-\text{Hg}-\text{Hg}-\text{O}]$ groups to form

discrete layers of various $[\text{Hg}-\text{O}-]$ ring topologies. In watersite ($\text{Hg}_4^+\text{Hg}^{2+}\text{Cr}^{6+}\text{O}_6$), rings of composition $[\text{Hg}_{10}\text{O}_6]$ are formed by linkage of four $[\text{O}-\text{Hg}-\text{Hg}-\text{O}]$ groups and two $[\text{O}-\text{Hg}-\text{O}]$ groups (Groat *et al.*, 1995) (Fig. 4a). The same ring topology occurs in one of the polymorphs of $\text{Hg}^{2+}(\text{Hg}_2^+\text{O})_2(\text{Re}^{7+}\text{O}_4)_2$ (Picard *et al.*, 1982). Three inorganic compounds $[\text{Hg}^+\text{Hg}^{2+}\text{OI}]$ (aurivilliusite), Stalhandske *et al.* (1985); $\text{Hg}^+\text{Hg}^{2+}\text{O}(\text{NO}_3)$, Kamenar *et al.* (1986); $\text{Hg}^+\text{Hg}^{2+}\text{O}(\text{Re}^{7+}\text{O}_4)$, Schriewer-Poettgen and

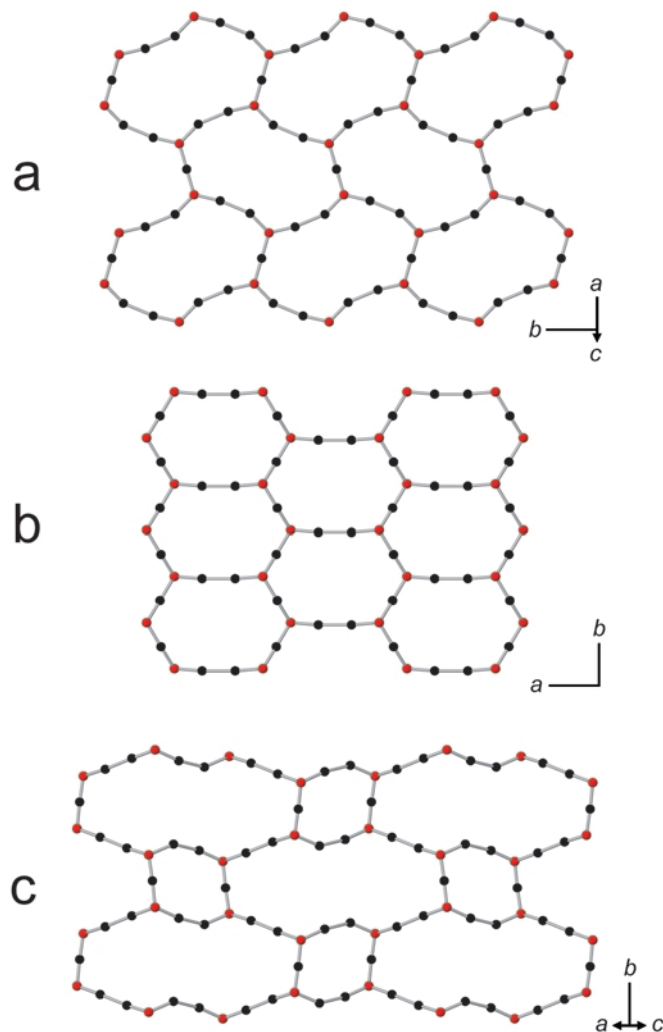


FIG. 4. The strongly bonded connectivity of $[\text{O}-\text{Hg}-\text{Hg}-\text{O}]$ and $[\text{O}-\text{Hg}-\text{O}]$ groups in: (a) watersite projected onto (001) (Groat *et al.*, 1995); (b) $\text{Hg}^+\text{Hg}^{2+}\text{OI}$ projected onto (00 $\bar{1}$) (Stalhandske *et al.*, 1985); (c) $\text{Hg}^{2+}(\text{Hg}_2^+\text{O})_2(\text{Re}^{7+}\text{O}_4)_2$ projected onto (10 $\bar{2}$) (Schriewer-Poettgen and Jeitschko, 1995). Legend as in Fig. 1.

CRYSTAL STRUCTURE OF TEDHADLEYITE

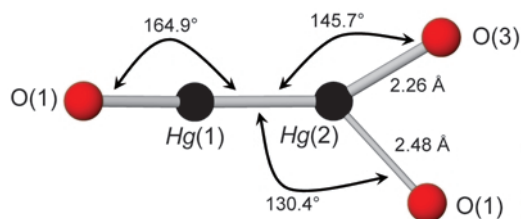


FIG. 5. Strong $Hg(2)-O(3)$ and intermediate strength $Hg(2)-O(1)$ bonds associated with the $[Hg(1)-Hg(2)]$ dimer in $Hg^{2+}(Hg_2^+O)_2(Re^{7+}O_4)_2$ (Schriewer-Poettgen and Jeritschko, 1995).

Jeitschko (1994)] contain topologically identical rings of composition $[Hg_8O_6]$ formed by linkage of two $[O-Hg-Hg-O]$ groups and four $[O-Hg-O]$ groups (Fig. 4b). The other polymorph of $Hg^{2+}(Hg_2^+O)_2(Re^{7+}O_4)_2$ (Schriewer-Poettgen and Jeitschko, 1995) contains rings of composition $[Hg_{14}O_8]$ (Fig. 4c) (resembling those in tedhadleyite), formed by linkage of six $[O-Hg-Hg-O]$ groups and two $[O-Hg-O]$ groups. Additionally, there are smaller rings of composition $[Hg_6O_4]$ formed by linkage of two $[O-Hg-Hg-O]$ and two $[O-Hg-O]$ groups. It should be noted that the designation of $Hg^{2+}(Hg_2^+O)_2(Re^{7+}O_4)_2$ as a layer structure is somewhat arbitrary; if one considers the $[O(1)-Hg(1)-Hg(2)-O(2)]$ group to be splayed and includes the O(1) and O(3) anions at the $Hg(2)$ end of the $[Hg-Hg]$ dimer, then strong $-Hg-O-$ bonding occurs over a three-dimensional network (Fig. 5).

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