Crystal structure of trimercury(II) dihydrogenhexaoxoiodate(VII), $Hg_3(H_2IO_6)_2$

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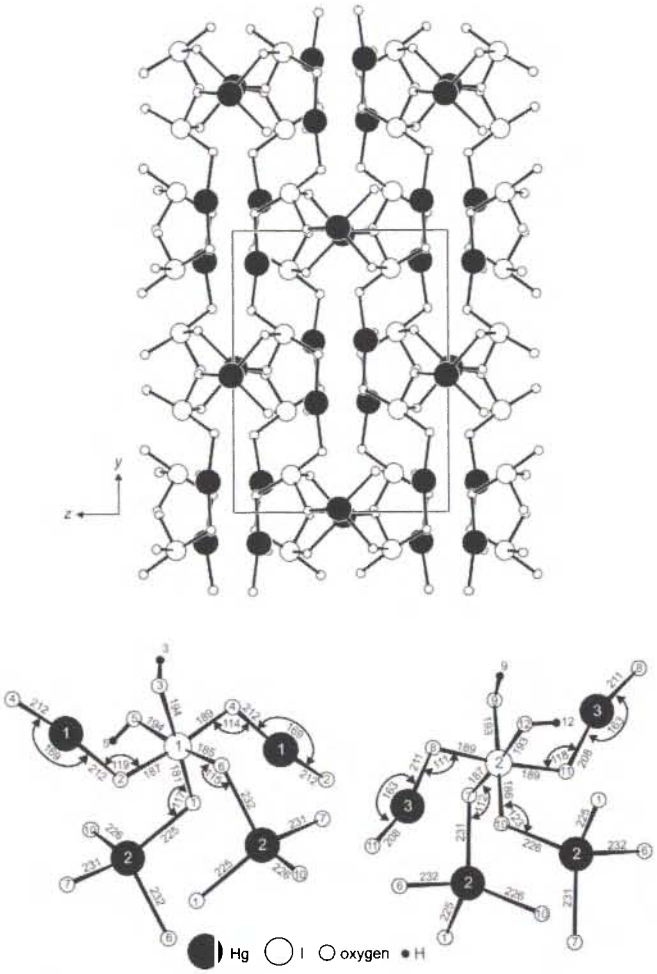

Abstract

$H_4Hg_3I_2O_{12}$, monoclinic, $P12_1/c1$ (No. 14), $a = 8.5429(7)$ Å, $b = 12.2051(8)$ Å, $c = 9.3549(8)$ Å, $\beta = 90.884(7)°$, $V = 975.3$ Å^3, $Z = 4$, $R_{gt}(F) = 0.038$, $wR_{ref}(F^2) = 0.079$, $T = 293$ K.

Source of material

$Hg_3(H_2IO_6)_2$ was obtained by precipitation reacting (aqueous) solutions of 10.7 g of $NaIO_4$ (50 mmol) in 25 ml 10 m HNO_3 and 8.1 g (25 mmol) of $Hg(NO_3)_2(H_2O)$ in 25 ml 10 m HNO_3 at 333 K. The sulfur-yellow precipitate of $Hg_3(H_2IO_6)_2$ was washed and dried at 333 K. An energy dispersive x-ray flourescence analysis in a scanning electron microscope showed no impurity elements heavier than sodium.

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Experimental details

The positional parameters have been standardized using the program STRUCTURE TIDY [2]. The positions of the hydrogen atoms were located by difference Fourier syntheses and fitted by least-squares refinement.

Discussion

The periodate $Hg_3(H_2IO_6)_2$ has been prepared already by Siebert and Fuckert [1]. The crystal structure is reported here. The two different iodine atoms have octahedral oxygen coordination with I—O distances ranging from 181.1(10) pm to 194.1(9) pm for I1, and from 185.6(9) pm to 192.7(10) pm for I2 with average distances of 188.3 pm and 189.3 pm, respectively. Two of the three different mercury atoms are almost inearly coordinated by two oxygen atoms with Hg—O distances of 211.9(9) pm and 212.0(9) pm for Hg1, and 208.3(10) pm and 210.7(9) pm for Hg3. The coordination spheres of these two mercury atoms are augmented by six oxygen atoms each with considerably longer Hg—O distances ranging from 266.9(10) pm to 283.9(10) pm for Hg1 and from 267.6(8) pm to 280.0(9) pm for Hg3. The Hg2 atom has tetrahedral oxygen coordination with Hg—O distances between 225.2(10) pm and 232.3(9) pm and an average of 228.6 pm. In this structure the $(OH)_2IO_4$ octahedra are condensed with nearly linear O–Hg–O bridges to infinite chains extending along the *b* axis. These chains are linked in the other two translation directions via mercury atoms, which obtain tetrahedral oxygen coordination. This arrangement is shown in the upper part of the figure, where the OH groups are omitted for clarity. In the lower part of this figure the near-neighbor environments are shown.

Table 1. Data collection and handling.

Crystal:	sulfur-yellow, monoclinic, size 0.02 × 0.02 × 0.03 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	535.03 cm^{-1}
Diffractometer, scan mode:	Enraf-Nonius CAD4, ω/2θ
$2\theta_{max}$:	69.92°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	6623, 3807
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2029
$N(param)_{refined}$:	107
Programs:	STRUCTURE TIDY [2], SHELX-97 [3], DIAMOND [4]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
O(1)	4*e*	0.047(1)	0.0065(8)	0.157(1)	0.018(2)
O(2)	4*e*	0.068(1)	0.2178(8)	0.0868(9)	0.014(2)
O(3)	4*e*	0.079(1)	0.7716(9)	0.179(1)	0.020(2)
H(3)	4*e*	0.05(2)	0.76(1)	0.11(2)	0.03
O(4)	4*e*	0.101(1)	0.5625(8)	0.1046(9)	0.013(2)
O(5)	4*e*	0.175(1)	0.1452(9)	0.346(1)	0.018(2)
H(5)	4*e*	0.28(2)	0.14(1)	0.32(2)	0.027
O(6)	4*e*	0.2133(9)	0.6311(7)	0.3526(9)	0.012(2)

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
O(7)	4*e*	0.2855(9)	0.3528(7)	0.3398(9)	0.010(2)
O(8)	4*e*	0.398(1)	0.4363(8)	0.0983(9)	0.011(2)
O(9)	4*e*	0.430(1)	0.2259(8)	0.161(1)	0.017(2)
H(9)	4*e*	0.50(2)	0.22(2)	0.11(2)	0.025
O(10)	4*e*	0.530(1)	0.4915(8)	0.3498(9)	0.014(2)
O(11)	4*e*	0.576(1)	0.2755(8)	0.405(1)	0.015(2)
O(12)	4*e*	0.677(1)	0.3630(9)	0.154(1)	0.017(2)
H(12)	4*e*	0.71(2)	0.36(1)	0.21(2)	0.025

Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Hg(1)	4*e*	0.09328(6)	0.38917(4)	0.11612(6)	0.0213(3)	0.0076(2)	0.0195(2)	0.0019(2)	0.0031(2)	0.0010(2)
Hg(2)	4*e*	0.25345(6)	0.01318(5)	0.01070(6)	0.0158(2)	0.0312(3)	0.0164(2)	0.0012(2)	0.0027(2)	0.0009(2)
Hg(3)	4*e*	0.59364(6)	0.10725(4)	0.37132(6)	0.0219(3)	0.0064(2)	0.0212(3)	0.0020(2)	0.0049(2)	0.0002(2)
I(1)	4*e*	0.01796(9)	0.63435(6)	0.26543(8)	0.0098(3)	0.0057(4)	0.0111(3)	0.0008(3)	0.0017(3)	0.0016(3)
I(2)	4*e*	0.48277(8)	0.36072(7)	0.25745(8)	0.0086(3)	0.0050(3)	0.0116(3)	0.0001(3)	0.0021(3)	0.0008(3)

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