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Crystal structure of ytterbium triiodate dihydrate, $Yb[IO_3]_3 \cdot 2H_2O$

D. Phanon* and I. Gautier-Luneau

Université Joseph Fourier, Laboratoire de Cristallographie, CNRS, 25 avenue des Martyrs, BP166, 38042 Grenoble Cedex 9, France

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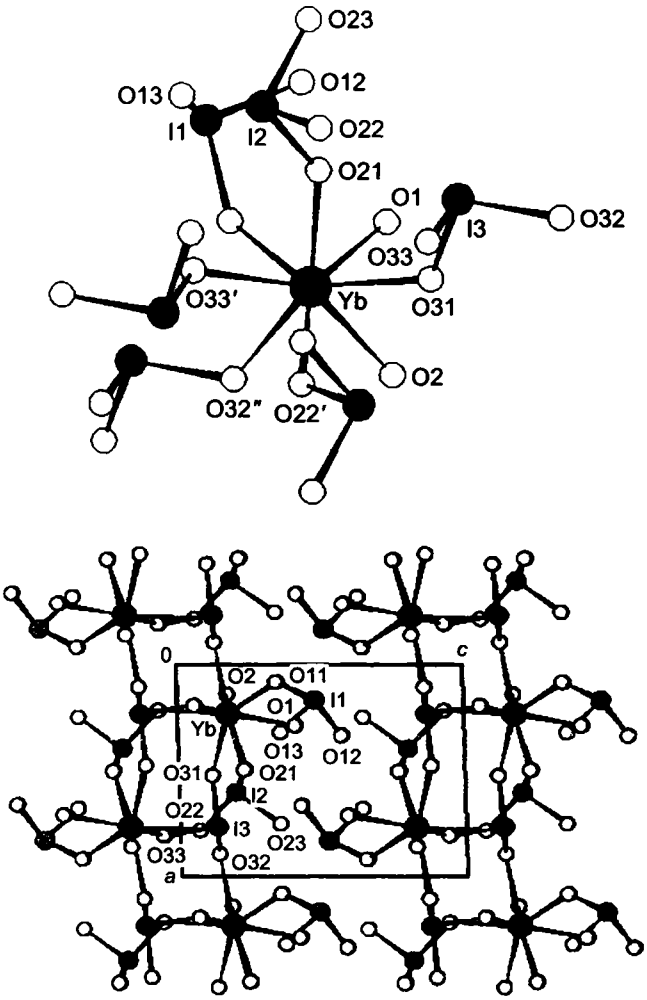

Abstract

$H_4I_3O_{11}Yb$, triclinic, $P\bar{1}$ (no. 2), a = 7.268(1) Å, b = 7.441(1) Å, c = 9.319(1) Å, α = 79.54(1)°, β = 85.18(1)°, γ = 71.90(1)°, V = 470.9 Å^3, Z = 2, $R_{gt}(F)$ = 0.028, $wR_{ref}(F^2)$ = 0.070, T = 293 K.

Source of material

0.25 mmol of ytterbium sulphate and 1.5 mmol of iodic acid were added in 10 mL of water under stirring. The white precipitate was put into a Teflon-lined steel autoclave. After sealed, the autoclave was heated to 473 K in oven and held 24 hours at this temperature. Then the autoclave was rapidly cooled to room temperature and immediately opened. The colorless solution contained colorless block crystals suitable for X-ray crystal structure analysis (yield 0.25 g, 70 %).

Experimental details

Hydrogen atoms from water molecules could not be located by Fourier difference maps.

Discussion

$Yb(IO_3)_3 \cdot 2H_2O$ is isostructural with $Y(IO_3)_3 \cdot 2H_2O$ [1] and $Lu(IO_3)_3 \cdot 2H_2O$ [2]. This isomorphism of the Yb, Y and Lu compounds is consistent with the ionic radii of the cation (Yb, 0.99 Å; Y, 1.02 Å; Lu, 0.98 Å [3]). The present compound is a polymorph of the reported ytterbium iodate hydrate [4] whom structure consists of layers with only one water molecule coordinated.

The asymmetric unit of the title structure is made up of one ytterbium atom, three iodate groups and two water molecules. The ytterbium atom is coordinated by eight oxygen atoms provided from two water molecules in *cis* position and six iodate anions, achieving a square antiprism (figure, top). The mean deviations from a calculated mean plane of the two square faces (O1-O2-O11-O32" and O21-O22'-O31-O33') are equal to 0.02 Å and 0.09 Å, respectively. The dihedral angle between these two faces is equal to 2.76°. Yb—O bond lengths are in the range 2.258(5) Å to 2.449(5) Å and the O–Yb–O angles are comprised between 70.7(2)° and 148.2(2)°. The environment of iodine is formed by three I—O strong bonds (mean bond lengths are: 1.805(5) Å, 1.799(5) Å and 1.807(5) Å) corresponding to an AX_3E configuration of the iodate anion and three additional I···O weak bonds (distances are in the range 2.532(5) Å to 3.293(5) Å). This coordination is described as octahedral with the iodine atom displaced off center along the ternary axis [5]. Each iodate group shows a different coordination scheme towards ytterbium atoms: the $I(1)O_3$ group coordinates only one cation in a monodentate way via O11, the $I(2)O_3$ group coordinates two cations in a bridging bismonodentate fashion via O21 and O22 whereas $I(3)O_3$ bridges three cations in a trismonodentate fashion. The I—O bond lengths are shorter for oxygens being non-coordinated to metal (bond lengths in the range 1.770(5) Å to 1.798(5) Å) than for coordinated oxygens (bond lengths in the range 1.799(5) Å to 1.828(5) Å).

The crystal structure is constituted of double chains extending along the a axis (figure, bottom). In a double chain, an ytterbium atom is linked to four ytterbium neighbors through bridging iodate. The closer is linked via two $I(2)O_3$ and two $I(3)O_3$ groups, through an inversion center, leading to six eight membered-rings and to an Yb···Yb distance equal to 5.128 Å. The second distance is to 6.083 Å through two (O33-I3-O32) bridges leading to the formation of an eight-membered ring. Finally, the two last are related through a chain Yb-O31-I3-O32-Yb leading to Yb···Yb distances of 7.268 Å (= a). The packing cohesion is assumed by five kinds of I···O weak bonds with distances between 2.555(5) Å and 3.293(5) Å. Both water molecules establish an hydrogen bond network with iodate oxygen atoms and also between them as evidenced by the O···O distances (2.710(5) Å to 2.957(5) Å).

* Correspondence author (e-mail: delphine.phanon@grenoble.cnrs.fr)

Table 1. Data collection and handling.

Crystal:	colorless block, size 0.18 × 0.18 × 0.25 mm
Wavelength:	Ag K_α radiation (0.56085 Å)
μ:	105.64 cm^{-1}
Diffractometer, scan mode:	Nonius KappaCCD, ω/φ
$2\theta_{max}$:	42.8°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	7810, 2159
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1913
$N(param)_{refined}$:	136
Programs:	SIR92 [6], SHELXL-97 [7], GRETEP [8]

Table 2. 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}
Yb	2*i*	0.23187(4)	0.65689(4)	0.17535(3)	0.0074(2)	0.0090(2)	0.0083(2)	−0.0030(1)	−0.0005(1)	−0.0019(1)
I(1)	2*i*	0.16621(6)	0.25383(7)	0.48079(5)	0.0083(2)	0.0118(2)	0.0088(2)	−0.0038(2)	−0.0008(2)	−0.0002(2)
I(2)	2*i*	0.60250(7)	0.15957(7)	0.19702(5)	0.0122(2)	0.0097(2)	0.0095(2)	−0.0052(2)	−0.0010(2)	−0.0002(2)
I(3)	2*i*	0.76716(6)	0.60095(7)	0.13237(5)	0.0095(2)	0.0095(2)	0.0105(2)	−0.0048(2)	−0.0037(2)	−0.0008(2)
O(11)	2*i*	0.0817(7)	0.4735(7)	0.3486(6)	0.012(2)	0.011(2)	0.010(2)	−0.002(2)	−0.002(2)	0.004(2)
O(12)	2*i*	0.3348(8)	0.3199(8)	0.5737(6)	0.013(2)	0.020(3)	0.016(3)	−0.007(2)	−0.003(2)	−0.003(2)
O(13)	2*i*	0.3199(8)	0.1063(8)	0.3585(6)	0.017(3)	0.014(3)	0.014(3)	−0.006(2)	0.000(2)	−0.004(2)
O(21)	2*i*	0.4963(8)	0.4022(8)	0.2305(7)	0.010(2)	0.006(2)	0.040(4)	−0.001(2)	−0.004(2)	−0.005(2)
O(22)	2*i*	0.7837(8)	0.2232(8)	0.0678(6)	0.018(3)	0.023(3)	0.014(3)	−0.014(2)	−0.003(2)	0.000(2)
O(23)	2*i*	0.7515(8)	0.0703(8)	0.3507(6)	0.019(3)	0.019(3)	0.011(3)	−0.004(2)	−0.005(2)	0.003(2)
O(31)	2*i*	0.5229(7)	0.7589(8)	0.1174(6)	0.006(2)	0.016(3)	0.026(3)	−0.001(2)	−0.006(2)	0.004(2)
O(32)	2*i*	0.8935(7)	0.7743(8)	0.1358(6)	0.008(2)	0.016(3)	0.018(3)	−0.008(2)	−0.001(2)	−0.004(2)
O(33)	2*i*	0.8067(9)	0.5811(8)	−0.0596(6)	0.038(3)	0.017(3)	0.008(3)	−0.012(3)	−0.001(2)	−0.005(2)
O(1)	2*i*	0.2871(8)	0.6968(9)	0.4099(6)	0.020(3)	0.026(3)	0.016(3)	−0.012(2)	−0.005(2)	−0.007(2)
O(2)	2*i*	0.1399(8)	0.9778(8)	0.1748(7)	0.020(3)	0.012(3)	0.027(3)	−0.001(2)	−0.009(2)	−0.005(2)

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