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Crystal structure of bismuth triiodate dihydrate, $Bi[IO_3]_3 \cdot 2H_2O$

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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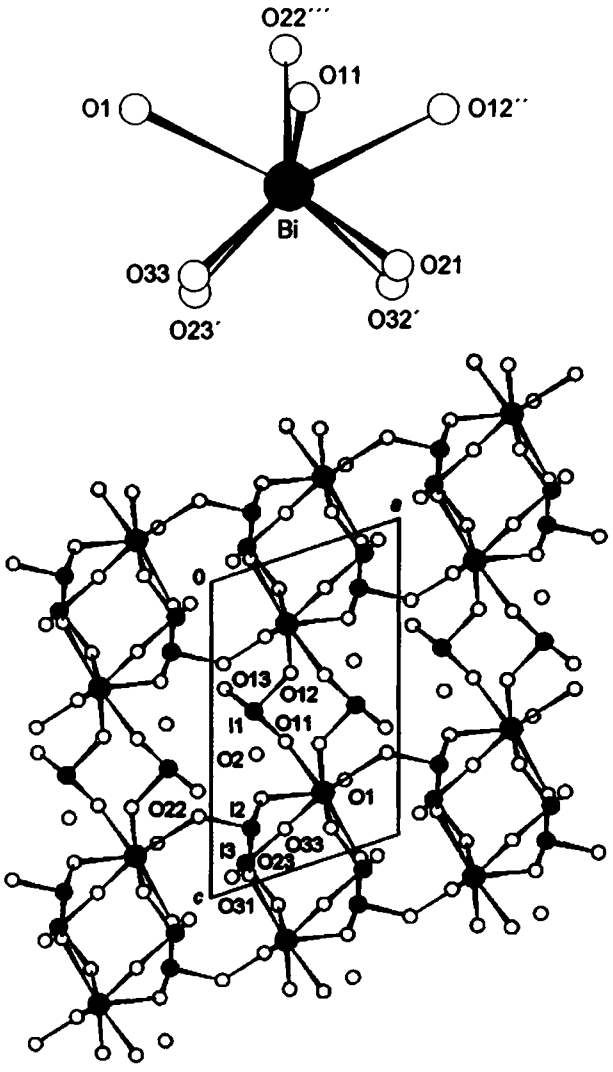

Abstract

$BiH_4I_3O_{11}$, triclinic, $P\bar{1}$ (no. 2), a = 7.056(2) Å, b = 7.337(1) Å, c = 10.740(1) Å, α = 95.06(1)°, β = 106.16(1)°, γ = 109.56(1)°, V = 493.0 Å^3, Z = 2, $R_{gt}(F)$ = 0.035, $wR_{ref}(F^2)$ = 0.090, T = 293 K.

Source of material

1 mmol of bismuth nitrate pentahydrate and 4 mmol of potassium or sodium iodate were dissolved in 50 mL of 7 M nitric acid. The mixture was evaporated at 323 K. After few hours, tiny, transparent and colorless platelets suitable for X-ray crystal structure analysis were obtained. They were filtered, washed with distilled water and dried in a furnace (yield 0.5 g, 65 %).

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

Experimental details

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

Discussion

The title compound is isostructural with $Y[IO_3]_3 \cdot 2H_2O$ [1] and $Ln[IO_3]_3 \cdot 2H_2O$ (Ln = Eu, Gd, Dy, Er, Tm, Yb) [2]. The asymmetric unit is made up of one bismuth atom, three iodate groups and two water molecules. Bismuth atom is coordinated by eight oxygen atoms provided from seven iodate groups (two $I(1)O_3$, three $I(2)O_3$ and two $I(3)O_3$ groups) and one O1 water molecule (figure, top). The polyhedron can be described as a bicapped trigonal prism whose trigonal faces are defined by the O11–O21–O33 and O22''–O23'–O32' oxygen atoms. The dihedral angle between these faces is equal to 15°. The two capping atoms are O1 (d(Bi—O1) = 2.497(8) Å) and O12'' (d(Bi—O12'') = 2.460(7) Å). The Bi—O bond lengths are in the range 2.301(7) Å to 2.553(7) Å and the O–Bi–O angles are comprised between 67.1(2)° and 151.9(2)°. The environment of iodine is formed by three I—O strong bonds (mean bond lengths are: 1.808(7) Å, 1.808(7) Å and 1.804(7) Å, respectively) corresponding to an AX_3E configuration of the iodate anion. Three additional I···O weak bonds are presented on I2 and I3 and four on I1 (distances are in the range 2.644(7) Å to 3.069(7) Å) as already observed on $Bi(IO_3)_3$ [3]. The I—O bond lengths are shorter for oxygens being non-coordinated to the metal atoms (distances in the range 1.776(8) Å to 1.801(7) Å) than for coordinated oxygens (distances in the range 1.801(7) Å to 1.831(7) Å). All iodate groups do not show the same coordination scheme towards bismuth atoms: the $I(1)O_3$ and $I(3)O_3$ groups coordinate two cations in a bismonodentate way via O11 and O12 and O32 and O33, respectively, whereas the $I(2)O_3$ group bridges three cations in a trismonodentate fashion.

The crystal structure is made up of layers extended in the (010) plane and piling up along the [010] direction with an interplane distance equal to the b parameter. In a layer, a bismuth atom possesses five bismuth neighbors linked 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 a Bi···Bi distance equal to 5.354 Å (figure, bottom). Two others are linked via two O22–I2–O23 or two O11–I1–O12 bridges forming isolated eight-membered rings and leading to the Bi···Bi distances equal to 5.876 Å and 6.266 Å, respectively. Finally, the last two neighbors are interlinked by a chain Bi–O21–I2–O22–Bi leading to the Bi···Bi distances equal to 7.056 Å (= a). The packing cohesion is assumed by three kinds of I···O weak bonds (d(I2···O31) = 2.652(7) Å, d(I3···O13) = 2.939(7) Å, d(I3···O31) = 2.746(7) Å). The O2 oxygen atom provided by the solvation water molecule bridges I1 and I2 iodine atoms with bond lengths equal to 3.064(7) Å and 2.799(7) Å, respectively. Both water molecules establish an hydrogen bond network with iodate oxygen atoms evidenced by the O···O distances (2.721(7) Å to 2.924(7) Å).

Table 1. Data collection and handling.

Crystal:	colorless platelet, size 0.12 × 0.23 × 0.25 mm
Wavelength:	Ag K_α radiation (0.56085 Å)
μ:	147.96 cm^{-1}
Diffractometer, scan mode:	Nonius KappaCCD, ω/φ
$2\theta_{max}$:	42.78°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	3838, 2249
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1907
$N(param)_{refined}$:	136
Programs:	SIR92 [4], SHELXL-97 [5], GRETEP [6]

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}
Bi	2*i*	0.59772(5)	0.40837(5)	0.78533(3)	0.0151(2)	0.0161(2)	0.0107(2)	0.0069(1)	0.0050(1)	0.0030(1)
I(1)	2*i*	0.2276(1)	0.27924(9)	0.45458(6)	0.0144(3)	0.0149(3)	0.0099(3)	0.0058(2)	0.0043(2)	0.0024(2)
I(2)	2*i*	0.21461(9)	0.63299(9)	0.82289(6)	0.0118(3)	0.0119(3)	0.0119(3)	0.0053(2)	0.0043(2)	0.0042(2)
I(3)	2*i*	0.18806(9)	0.14034(8)	0.92610(6)	0.0125(3)	0.0117(3)	0.0110(3)	0.0033(2)	0.0034(2)	0.0031(2)
O(11)	2*i*	0.396(1)	0.200(1)	0.5835(7)	0.023(4)	0.015(3)	0.014(3)	0.007(3)	0.004(3)	0.007(3)
O(12)	2*i*	0.423(1)	0.369(1)	0.3722(7)	0.025(4)	0.025(4)	0.016(3)	0.014(3)	0.015(3)	0.014(3)
O(13)	2*i*	0.072(1)	0.043(1)	0.3510(8)	0.026(4)	0.019(4)	0.022(4)	0.006(3)	0.003(3)	–0.002(3)
O(21)	2*i*	0.270(1)	0.439(1)	0.7391(7)	0.013(3)	0.014(3)	0.024(4)	0.005(3)	0.010(3)	–0.002(3)
O(22)	2*i*	–0.064(1)	0.547(1)	0.7286(7)	0.011(3)	0.022(3)	0.019(3)	0.009(3)	0.004(3)	0.007(3)
O(23)	2*i*	0.205(1)	0.528(1)	0.9670(7)	0.028(4)	0.019(3)	0.013(3)	0.006(3)	0.004(3)	0.007(3)
O(31)	2*i*	0.115(1)	–0.109(1)	0.9535(8)	0.028(4)	0.014(3)	0.023(4)	0.006(3)	0.015(3)	0.008(3)
O(32)	2*i*	0.360(1)	0.263(1)	1.0931(7)	0.021(4)	0.027(4)	0.013(3)	0.011(3)	0.005(3)	–0.003(3)
O(33)	2*i*	0.396(1)	0.140(1)	0.8592(8)	0.017(4)	0.019(4)	0.028(4)	0.007(3)	0.014(3)	0.012(3)
O(1)	2*i*	0.714(1)	0.124(1)	0.7681(8)	0.032(5)	0.032(4)	0.024(4)	0.018(4)	0.013(3)	0.010(3)
O(2)	2*i*	0.241(1)	–0.216(1)	0.5952(9)	0.033(5)	0.026(4)	0.041(5)	0.014(4)	0.021(4)	0.012(4)

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