Crystal structure of indium triiodate dihydrate, $In[IO_3]_3 \cdot 2H_2O$

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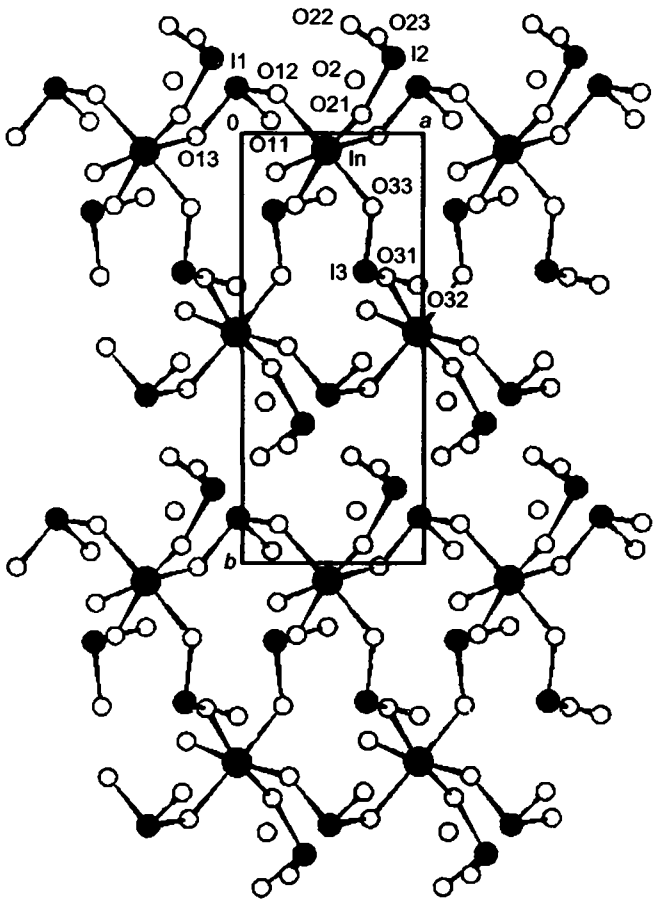

Abstract

$H_4I_3InO_{11}$, orthorhombic, $P2_12_12_1$ (no. 19), a = 5.038(2) Å, b = 12.154(2) Å, c = 16.041(3) Å, V = 982.2 Å^3, Z = 4, $R_{gt}(F)$ = 0.022, $wR_{ref}(F^2)$ = 0.042, T = 293 K.

Source of material

0.1 mmol of indium nitrate pentahydrate and 2 mmol of potassium iodate were dissolved in 40 mL of 14 M nitric acid. The mixture was evaporated at room temperature. After 18 days, tiny, transparent and colorless platelets suitable for X-ray crystal structure analysis were obtained.

Experimental details

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

Discussion

The asymmetric unit of the title compound is made up of one indium atom, three iodate groups and two water molecules. The coordination polyhedron of the indium is a slightly oxygenated distorted octahedron provided from five iodate groups (two $I(1)O_3$ and two $I(3)O_3$ bismonodentate groups and one $I(2)O_3$ monodentate group) and one water molecule. The In—O bond lengths are in the range 2.078(5) Å to 2.159(5) Å, the O–In–O *cis* angles are comprised between 80.3(2)° and 99.3(2)° and the O–In–O *trans* angles between 162.1(2)° and 174.5(2)°. The environment of the iodine atom in all iodate groups is formed by three I—O strong bonds (mean bond lengths are: 1.807(5) Å, 1.803(5) Å and 1.806(5) Å, respectively) corresponding to an AX_3E configuration and three I···O additional weak bonds (distances are in the range 2.545(5) Å to 3.252(5) Å). This coordination is described as octahedral with the iodine atom displaced off center along the ternary axis [1]. The I—O bond lengths are shorter for oxygens being non-coordinated to metal atoms (bond lengths in the range 1.772(5) Å to 1.803(5) Å) than for coordinated oxygens (bond lengths in the range 1.808(5) Å to 1.838(5) Å).

The crystal structure is formed by ribbons parallel to the [100] direction. In a ribbon, indium atoms are deduced by 2_1 axes parallel to the [100] direction leading to an In···In distance equal to 6.169 Å through the $I(3)O_3$ bridging bismonodentate iodate. The shortest In···In intermetallic distance in the ribbon corresponds to the a parameter (5.038 Å) via the bismonodentate $I(1)O_3$ iodate group. The packing cohesion is assumed by three kinds of I···O weak bonds (d(I1···O23) = 2.572(5) Å, d(I2···O32) = 2.667(5) Å and d(I3···O22) = 2.975(5) Å). Oxygen atoms involved in these weak bonds are not coordinated to cation. A solvation water molecule (O2) is inserted between ribbons. Both water molecules establish an hydrogen bond network towards iodate oxygen atoms as evidenced by the O···O distances (2.676(5) Å to 2.968(5) Å).

Table 1. Data collection and handling.

Crystal:	colorless platelet, size 0.14 × 0.22 × 0.30 mm
Wavelength:	Ag K_α radiation (0.56085 Å)
μ:	62.89 cm^{-1}
Diffractometer, scan mode:	Nonius KappaCCD, CCD
$2\theta_{max}$:	42.8°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	12064, 2264
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2043
$N(param)_{refined}$:	136
Programs:	SIR92 [2], SHELXL-97 [3], GRETEP [4]

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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}
In	4*a*	0.4703(1)	0.03965(4)	0.92648(3)	0.0090(3)	0.0103(2)	0.0114(2)	0.0000(2)	−0.0004(2)	−0.0000(2)
I(1)	4*a*	−0.02211(9)	−0.10646(3)	1.04193(3)	0.0088(2)	0.0084(2)	0.0110(2)	−0.0001(2)	−0.0005(2)	0.0009(2)
I(2)	4*a*	0.84071(9)	−0.17389(4)	0.82150(3)	0.0124(2)	0.0116(2)	0.0107(2)	−0.0018(2)	0.0015(2)	−0.0014(2)
I(3)	4*a*	0.68288(9)	0.32071(4)	0.89055(3)	0.0117(2)	0.0102(2)	0.0106(2)	−0.0008(2)	0.0003(2)	−0.0016(2)
O(11)	4*a*	0.166(1)	−0.0298(4)	1.1159(3)	0.016(3)	0.011(2)	0.021(3)	−0.001(2)	−0.010(2)	−0.003(2)
O(12)	4*a*	0.202(1)	−0.0915(4)	0.9536(3)	0.015(2)	0.014(2)	0.015(2)	−0.003(2)	0.005(2)	0.002(2)
O(13)	4*a*	−0.244(1)	0.0065(4)	1.0166(3)	0.015(3)	0.014(2)	0.018(3)	0.009(2)	−0.008(2)	−0.002(2)
O(21)	4*a*	0.666(1)	−0.0432(4)	0.8244(3)	0.023(3)	0.014(2)	0.018(3)	0.003(2)	−0.001(3)	−0.004(2)
O(22)	4*a*	0.604(1)	−0.2506(4)	0.7624(3)	0.019(3)	0.019(2)	0.016(3)	−0.007(2)	−0.005(2)	−0.005(2)
O(23)	4*a*	0.756(1)	−0.2216(4)	0.9245(3)	0.020(3)	0.021(2)	0.012(2)	−0.008(2)	0.004(2)	−0.002(2)
O(31)	4*a*	0.800(1)	0.3348(4)	0.9966(3)	0.025(3)	0.013(2)	0.009(2)	−0.003(3)	−0.003(2)	0.003(2)
O(32)	4*a*	0.972(1)	0.3534(4)	0.8326(3)	0.014(3)	0.021(3)	0.024(3)	−0.004(2)	0.007(3)	−0.001(2)
O(33)	4*a*	0.7235(9)	0.1708(4)	0.8832(3)	0.010(2)	0.011(2)	0.024(3)	−0.001(2)	0.003(2)	−0.001(2)
O(1)	4*a*	0.192(1)	0.0892(4)	0.8324(3)	0.011(2)	0.025(3)	0.014(3)	−0.001(2)	−0.003(2)	0.009(2)
O(2)	4*a*	0.631(1)	−0.1229(5)	1.1745(3)	0.021(3)	0.032(3)	0.018(3)	0.002(2)	0.003(2)	0.005(2)

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