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Crystal structure of indium triiodate trihydrate, $In[IO_3]_3 \cdot 3H_2O$

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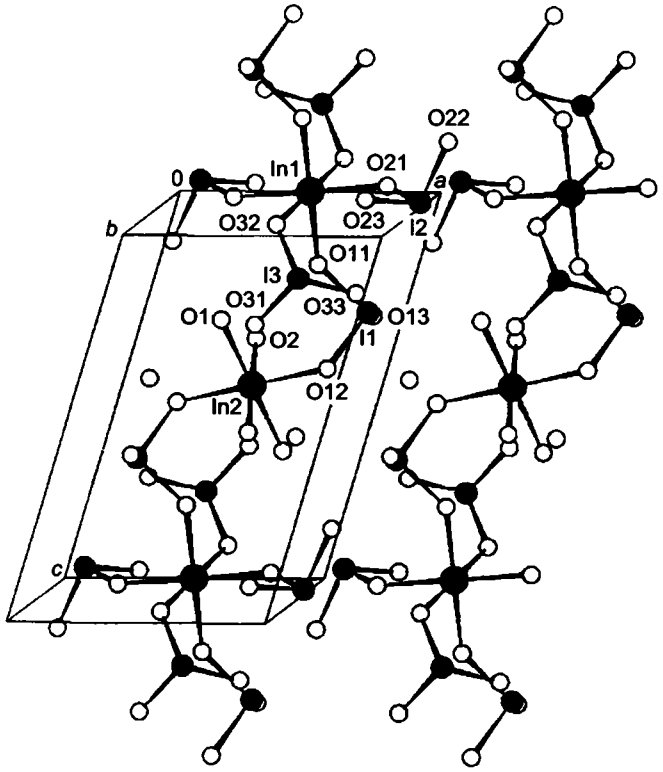

Abstract

$H_6I_3InO_{12}$, triclinic, $P\bar{1}$ (no. 2), a = 6.862(1) Å, b = 7.282(2) Å, c = 11.137(3) Å, α = 94.01(2)°, β = 107.75(1)°, γ = 93.59(1)°, V = 526.7 Å^3, Z = 2, $R_{gt}(F)$ = 0.034, $wR_{ref}(F^2)$ = 0.089, T = 293 K.

Source of material

1 mmol of $In(NO_3)_3 \cdot 5H_2O$ and 20 mmol of $NaIO_3$ were dissolved in 70 mL of 3.5 M nitric acid. The mixture was evaporated at room temperature. After 4 days, colorless rod crystals suitable for X-ray crystal structure analysis were obtained (yield 0.66 g, 95 %).

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 two indium atoms on inversion centers, three iodate groups and three water molecules. The coordination polyhedron of both indium atoms is a slightly distorted octahedron. In1 is coordinated by oxygen atoms provided from six iodate groups linked to the metal in a monodentate fashion, whereas In2 is coordinated by two oxygen atoms from iodate groups and four O1 and O2 oxygen atoms provided by water molecules. The In—O bond lengths are in the range 2.106(5) Å to 2.184(6) Å and 2.118(7) Å to 2.198(7) Å for In1 and In2, respectively, the O–In–O *cis* angles are comprised between 81.8(2)° and 98.2(2)° for In1 and between 83.7(2)° and 96.3(2)° for In2. The environment of the iodine atom in all iodate groups is formed by three I—O strong bonds (mean bond lengths: 1.808(6) Å, 1.817(6) Å and 1.803(6) Å for I1, I2 and I3, respectively) corresponding to an AX_3E configuration and three additional I···O weak bonds (distances are in the range 2.559(6) Å to 3.120(6) Å). This coordination is described as octahedral with the iodine atom displaced off center along the three-fold axis [1]. The $I(1)O_3$ group is coordinated to two indium atoms In1 and In2 in a bismonodentate fashion via O11 and O12, respectively, and the $I(2)O_3$ and $I(3)O_3$ groups are monodentate towards only one indium atom In1 via O21 and O32, respectively. The I—O bond lengths to the oxygens coordinated to indium are longer (bond lengths between 1.814(5) Å and 1.849(5) Å) than those to the oxygens non-coordinated to indium (bond lengths in the range 1.778(6) Å to 1.811(5) Å).

The crystal structure is formed by chains parallel to the [001] direction. The packing cohesion is ensured by six kinds of the nine I···O weak bonds (d(I1···O22) = 2.559(5) Å, d(I1···O12) = 3.013(5) Å, d(I2···O21) = 2.777(5) Å, d(I2···O23) = 2.610(5) Å, d(I3···O13) = 2.656(5) Å and d(I3···O23) = 3.120(5) Å). In each chain, two indium atoms are linked together via a bismonodentate $I(1)O_3$ group through O11 and O12 leading to the In···In distance equal to 5.568 Å (= c/2). The In···In inter-chain distances are 6.862 Å (= a) and 7.282 Å (= b). The solvent water molecule O3 is inserted between the chains. The three water molecules established a hydrogen bond network towards iodate oxygen atoms and also between them as evidenced by the O···O distances (2.543(6) Å to 2.979(6) Å).

Table 1. Data collection and handling.

Crystal:	colorless rod, size 0.05 × 0.07 × 0.30 mm
Wavelength:	Ag K_α radiation (0.56085 Å)
μ:	58.7 cm^{-1}
Diffractometer, scan mode:	Nonius KappaCCD, CCD
$2\theta_{max}$:	42.78°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	14815, 2415
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2158
$N(param)_{refined}$:	148
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(1)	1*d*	½	0	0	0.0140(3)	0.0121(3)	0.0144(3)	0.0003(2)	0.0049(2)	–0.0020(3)
In(2)	1*f*	½	0	½	0.0183(3)	0.0208(4)	0.0215(4)	0.0004(3)	0.0063(3)	–0.0001(3)
I(1)	2*i*	0.85033(6)	–0.05973(6)	0.31775(4)	0.0151(2)	0.0139(2)	0.0145(2)	–0.0014(2)	0.0054(2)	–0.0013(2)
I(2)	2*i*	0.97965(6)	0.25716(6)	–0.00261(4)	0.0153(2)	0.0113(2)	0.0173(2)	–0.0006(2)	0.0062(2)	–0.0013(2)
I(3)	2*i*	0.63141(7)	0.44168(6)	0.17365(4)	0.0206(2)	0.0116(2)	0.0157(2)	–0.0004(2)	0.0054(2)	–0.0021(2)
O(11)	2*i*	0.6030(8)	–0.0674(8)	0.1962(5)	0.014(2)	0.028(3)	0.019(3)	–0.002(2)	0.002(2)	–0.001(2)
O(12)	2*i*	0.7769(8)	0.0409(7)	0.4512(5)	0.020(2)	0.020(3)	0.017(2)	–0.001(2)	0.007(2)	–0.006(2)
O(13)	2*i*	0.8340(9)	–0.2937(8)	0.3525(5)	0.034(3)	0.017(3)	0.022(3)	0.001(2)	0.005(2)	0.004(2)
O(21)	2*i*	0.7976(8)	0.0511(7)	–0.0211(6)	0.018(2)	0.013(2)	0.042(3)	–0.001(2)	0.013(2)	–0.004(2)
O(22)	2*i*	1.012(1)	0.2310(8)	–0.1565(6)	0.036(3)	0.028(3)	0.022(3)	0.008(3)	0.013(2)	0.001(2)
O(23)	2*i*	0.7958(8)	0.4297(7)	–0.0294(6)	0.023(3)	0.014(2)	0.030(3)	0.003(2)	0.008(2)	–0.002(2)
O(31)	2*i*	0.514(1)	0.4170(8)	0.2956(6)	0.043(3)	0.024(3)	0.026(3)	–0.007(3)	0.017(3)	–0.005(2)
O(32)	2*i*	0.4563(8)	0.2732(7)	0.0545(5)	0.020(2)	0.015(2)	0.024(3)	0.000(2)	0.004(2)	–0.008(2)
O(33)	2*i*	0.8463(8)	0.3068(8)	0.2263(6)	0.021(3)	0.017(3)	0.034(3)	0.003(2)	0.003(2)	0.002(2)
O(1)	2*i*	0.3228(9)	0.099(1)	0.3202(6)	0.030(3)	0.039(4)	0.038(4)	0.003(3)	0.013(3)	0.006(3)
O(2)	2*i*	0.414(1)	–0.272(1)	0.4101(8)	0.054(4)	0.029(3)	0.058(5)	–0.002(3)	0.034(4)	–0.016(3)
O(3)	2*i*	0.822(1)	0.543(1)	0.5695(7)	0.041(4)	0.053(5)	0.034(4)	0.000(3)	0.002(3)	0.011(3)

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