Di-tert-butyltin(IV)–hydroxide–iodide, \( 'Bu_2Sn(OH)I \), the last missing member in the series of pure di-tert-butyltin(IV)–hydroxide–halides

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The crystal structure of di-tert-butylhydroxidoiodidotin(IV), \([Sn(C_6H_{13})_2I(OH)]\) or \( 'Bu_2Sn(OH)I \), consists of dimeric, centrosymmetric molecules exhibiting the typical structural features of diorganotin(IV)–hydroxide–halides, \( R_2Sn(OH)Hal \). Two trigonal–bipyramidally coordinated tin(IV) atoms are bridged via two hydroxyl groups, resulting in a planar, four-membered \{Sn—O\} 2 ring of rhombic shape, with acute angles at tin, obtuse angles at oxygen and two different Sn—O distances depending whether the oxygen atom adopts an axial or equatorial position at the tin(IV) atom. In contrast to its fluorine, chlorine and bromine homologues, no hydrogen bonds between the OH group and the halide atom exist, confining the intermolecular interactions to van der Waals forces.

1. Chemical context

During the hydrolysis of diorganotin(IV)-dihalides, \( R_2SnHal_2 \), to diorganotin(IV) oxides, \( R_2SnO \), various intermediates are formed. The most prominent ones are the diorganotin-hydroxide-halides, \( R_2Sn(OH)Hal \), the tetraorganodihalo-geride-distannoxanes, \( (R_2SnHal)O \), the tetraorganohydroxide-halide-distannoxanes, \( (R_2SnHal)O(R_2SnOH) \), and the tetraorganohydroxide-distannoxanes, \( (R_2SnOH)_2O \). In solution as well as in the crystalline state, all these compounds are comprised of dimeric molecules, and their compositions and structures result from a sequence of different hydrolysis, aggregation and condensation reactions.

In all three different classes of tetraorganodistannoxanes, numerous compounds have been structurally characterized. All of them exhibit a ladder-type arrangement of the Sn—O—Hal/OH framework [e.g. dihalogenides: \( R = Ph, Hal = Cl \) (Vollano et al., 1984), \( R = 'Pr, Hal = Br \) (Beckmann et al., 2002a); hydroxide-halides: \( R = Et, Hal = Cl \) (Momeni et al., 2019), \( R = Ph, Hal = Br \) (Yap et al., 2010); dihydroxides: \( R = neophyl \) (Reuter & Pawlak, 2000), \( R = trimethylsilylmethyl \) (Beckmann et al., 2002b)].

Pure hydroxide halides have been prepared and structurally characterized for \( 'Bu_2Sn(OH)Hal \) with \( Hal = F, Br \) (Puff et al., 1985), \( Hal = Cl \) (\( \alpha \)-modification: Puff et al., 1985; \( \beta \)-modification: Di Nicola et al., 2011) and for \( R = p\)-tolyl and \( Hal = Br \) (Lo & Ng, 2009). Their structures are dominated by various –OH···Hal bridges between neighbouring molecules, resulting in their chain-like arrangements. Hydroxide halides can be isolated when their hydroxyl groups are involved in hydrogen bonds to Bronstedt bases (BB). Such adducts of formula \([R_2Sn(OH)Hal]_22BB\) have been described for \( R = Ph, Hal = Cl \) with \( BB = EtOH \) (Barba et al., 2007), and \( BB = quinoline \) (Acanonca et al., 2003).
Here we present the molecular and crystal structure of the last missing member in the series of pure di-tert-butyltin hydroxide halides where Hal = I. The analogous molecule with DMSO as a hydrogen-bonded Brønsted base was formerly found as part of co-crystals with [(tBu₂Sn)₃O(OH)₂]I (Reuter & Wilberts, 2014).

2. Structural commentary

The asymmetric unit of the title compound comprises one tBu₂Sn(OH)I moiety that dimerizes to form a centrosymmetric molecule (Fig. 1). As in all other hydroxide halides, dimerization occurs via the two hydroxyl groups that act as bridges between two trigonal–bipyramidally (tbpy) coordinated Sn⁴V atoms.

The anisotropic displacement parameters as well as the small isotropic displacement parameters of the hydrogen atoms (see Refinement) indicate a negligibly small rotation of the tert-butyl groups as a whole and a small rotation of the methyl groups in particular, giving rise to very precise information on bond lengths and angles. The structural features of the tert-butyl groups are characterized by C—C bond lengths in the range 1.524 (3) to 1.533 (3) Å [mean value: 1.529 (3) Å], C—C—C angles in the range 109.5 (2) to 111.3 (2)° [mean value: 110.2 (7)°], Sn—C bond lengths between 2.187 (2) and 2.193 (2) Å [mean value: 2.190 (3) Å], and Sn—C—C angles of 107.8 (1)° to 109.6 (1)° [mean value: 108.8 (9)°]. All these values are more precise in comparison with those of the formerly determined di-tert-butyltin hydroxide halides (Puff et al., 1985; Di Nicola et al., 2011), especially as a result of low-temperature measurement and high data redundancy combined with multi-scan absorption correction, but are of the same accuracy and absolute value as those of the DMSO adduct [(tBu₂Sn)₃O(OH)₂]I (Reuter & Wilberts, 2014). These data are confirmed by a redetermination of the crystal structure of the α-modification of [(tBu₂Sn(OH)Cl)]₂ (Reuter, 2022) performed with similar experimental conditions as for the title compound and its co-crystallize. In this context, Sn—C distances are of special interest as they belong to the longest ones observed in case of Sn in a trigonal–bipyramidal coordination. In the other hydroxide halides mentioned above, the following bond lengths have been found: d(Sn—C) mean = 2.120 (8) Å for R = p-tolyl, Hal = Br; d(Sn—C) mean = 2.121 (10)/2.117 (4) Å for R = Ph, Hal = Cl, BB = quinoline/EtOH.

Figure 1
Ball-and-stick model of the dimeric, centrosymmetric molecule found in the crystal of tBu₂Sn(OH)I, with atom numbering of the asymmetric unit. With the exception of the hydrogen atoms that are shown as spheres of arbitrary radius, all other atoms are drawn as displacement ellipsoids at the 40% probability level. The black dot labelled i indicates the position of the centre of symmetry.

Figure 2
Ball-and-stick model of the trigonal–bipyramidal coordination environment of the tin atom in the dimeric molecule of tBu₂Sn(OH)I with bond lengths (Å) and angles (°) characterizing the polyhedron axes. For clarity, methyl groups of the tBu ligands are stripped down to the carbon–carbon bonds drawn as shortened sticks. Atom O1' is generated by symmetry code –x, –y + 1, –z + 1.
Within the trigonal–bipyramidal coordination of the Sn IV atom (Fig. 2), both tert-butyl groups are in equatorial (eq) positions in correspondence with the predictions of the VSEPR concept. The bond angle enclosed by the two tert-butyl groups of 126.81 (8)°/C14 is identical with the value [126.89 (9)°/C14] in the co-crystal and lies in the range 122.0 (2) to 129.3 (1)°/C14 of C—Sn—C angles found in the other hydroxide-halides. The iodine atom adopts an axial (ax) position and one of the bridging hydroxyl groups is in an equatorial, the other in an axial position. As a result of dimerization via the hydroxyl groups, the axis of the trigonal bipyramid strongly deviates from linearity [Iax—Sn—(OH)ax = 151.94 (4)°]. In addition, the Sn—I distance of 2.8734 (2) Å is only marginally shorter than in the co-crystal [Sn—I = 2.8852 (2) Å], both being significantly longer than the sum (2.78 Å) of the covalent radii (Cordero et al., 2008) of tin (1.39 Å) and iodine (1.39 Å) and much longer than the mean Sn—I distance of 2.661 (2) Å (Reuter & Pawlak, 2001) in tin(IV) iodide, SnI4, with tetrahedrally coordinated tin.

Because of the centrosymmetric nature of the dimer, the central four-membered [Sn—O]2 ring is exactly planar. Its rhombic shape (Fig. 3) is characterized by acute angles [67.02 (6)°] at tin and obtuse ones [112.98 (6)°] at oxygen. Moreover, these rings exhibit two different Sn—O distances depending on the position (ax/eq) of the oxygen atom in the trigonal–bipyramidal coordination environment of tin(IV): Sn—(OH)ax = 2.256 (1) Å versus Sn—(OH)eq = 2.063 (1) Å. All these structural features are typical. For example, for the other four-membered [Sn—O]2 rings of hydroxide halides with R = 'Bu, Hal = F, Cl, Br, the Sn—O—Sn angles range from 109.9 (2) to 112.5 (3)°, the O—Sn—O angles from 67.9 (3) to 70.1 (2)°, the Sn—(OH)eq distances from 2.012 (5) to 2.048 (10) Å, and the Sn—(OH)ax distances from 2.199 (5) to 2.257 (16) Å (Puff et al., 1985).

3. Supramolecular features

While the hydroxyl groups of the ['Bu2Sn(OH)I]2 molecules of the co-crystallize (Reuter & Wilberts, 2014) are involved in OH···O hydrogen-bonding to DMSO molecules, those of all other ['Bu2Sn(OH)Hal]2 molecules develop intermolecular O—H···Hal bonds resulting in a chain-like arrangement of the corresponding molecules in the crystal. In contrast, there are no hydrogen bonds in the crystal structure of the title compound as the voluminous iodine atoms (Fig. 4) prevent significant intermolecular OH···I interactions (Table 1). Hence, only van der Waals forces exist between the individual molecules, resulting in a layer-like arrangement (Fig. 5) with...
the Sn—I bonds perpendicular to the layer plane. These layers expand perpendicular to the [101] direction (Fig. 6).

4. Synthesis and crystallization

Yellow block-like single crystals of the title compound were obtained after several years of storage in a sample of (Bu₂Sn)₂I₂ originally prepared by the reaction of the cyclo-tetrastannane (Bu₂Sn)₄ with I₂ in toluene at elevated temperature in a molar ratio of 1:2. As other molar ratios and temperatures result in the formation of (Bu₂Sn)₄I₂ (Farrar & Skinner, 1964; Adams & Dräger, 1985; Puff et al., 1989), it seems possible that the sample was contaminated with the latter one, which reacts over the long time of storage with atmospheric moisture to give the title compound.

5. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. All hydrogen atoms were clearly identified in difference-Fourier syntheses. Those of the tert-butyl groups were refined with calculated positions (C—H = 0.98 Å) and a common Uiso(H) parameter for each of the methyl groups. The position of the H atom of the OH group was refined with a fixed O—H distance of 0.96 Å and the Uiso(H) parameter refined freely.

Acknowledgements

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References


Table 1

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Computer programs: APEX2 and SAINT (Bruker, 2009), SHELXL (Sheldrick, 2008), SHELXL (Sheldrick, 2015), DIAMOND (Brandenburg, 2006), Mercury (Macrae et al., 2020) and publCIF (Westrip, 2010).

Figure 6

Stick-model showing the arrangement of layers with respect to the unit cell; colour code as in Fig. 4.
**Di-tert-butyltin(IV)–hydroxide–iodide, ^1^Bu_2Sn(OH)I**, the last missing member in the series of pure di-tert-butyltin(IV)–hydroxide–halides

**Hans Reuter**

**Computing details**

Data collection: APEX2 (Bruker, 2009); cell refinement: SAINT (Bruker, 2009); data reduction: SAINT (Bruker, 2009); program(s) used to solve structure: SHELXS (Sheldrick, 2008); program(s) used to refine structure: SHELXL (Sheldrick, 2015); molecular graphics: DIAMOND (Brandenburg, 2006) and Mercury (Macrae et al., 2020); software used to prepare material for publication: publCIF (Westrip, 2010).

**Di-tert-butylhydroxidoiodidotin(IV)**

**Crystal data**

\[ [\text{Sn}(\text{C}_4\text{H}_9)_2\text{I}(\text{OH})] \]

\( M_r = 376.82 \)

Monoclinic, \( P2_1/n \)

\( a = 8.4903 (4) \) Å

\( b = 10.8848 (5) \) Å

\( c = 13.5107 (6) \) Å

\( \beta = 101.881 (2) ^\circ \)

\( V = 1221.85 (10) \) Å\(^3\)

\( Z = 4 \)

\( F(000) = 712 \)

\( D_\lambda = 2.048 \text{ Mg m}^{-3} \)

Mo \( K\alpha \) radiation, \( \lambda = 0.71073 \) Å

Cell parameters from 9771 reflections

\( \theta = 2.4–29.2^\circ \)

\( \mu = 4.58 \text{ mm}^{-1} \)

\( T = 100 \text{ K} \)

Block, yellow

\( 0.24 \times 0.12 \times 0.09 \text{ mm} \)

**Data collection**

Bruner APEXII CCD
diffractometer

\( \varphi \) and \( \omega \) scans

Absorption correction: multi-scan

\( \theta_{\text{max}} = 28.0^\circ, \theta_{\text{min}} = 2.6^\circ \)

\( h = -11 \rightarrow 11 \)

\( k = -14 \rightarrow 13 \)

\( l = -17 \rightarrow 17 \)

2950 independent reflections

2761 reflections with \( I > 2\sigma(I) \)

48755 measured reflections

**Refinement**

Refinement on \( F^2 \)

Least-squares matrix: full

\( R[F^2 > 2\sigma(F^2)] = 0.016 \)

\( wR(F^2) = 0.037 \)

\( S = 1.08 \)

2950 reflections

114 parameters

0 restraints

Primary atom site location: structure-invariant direct methods

Hydrogen site location: mixed

H-atom parameters constrained

\( w = 1/[\sigma(F_o^2) + (0.0075P)^2 + 1.3137P] \)

where \( P = (F_o^2 + 2F_c^2)/3 \)

\( \Delta \rho_{\text{max}} = 0.85 \text{ e Å}^{-3} \)

\( \Delta \rho_{\text{min}} = -0.49 \text{ e Å}^{-3} \)

Extinction correction: SHELXL-2014/7 (Sheldrick 2015,

\( F_c^\epsilon = kF_c[1 + 0.001x\sigma^2F_c^2/\sin(2\theta)]^{1/4} \)

Extinction coefficient: 0.00139 (11)
**Special details**

**Geometry.** All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

**Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)**

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Symmetry code: (i) −x, −y+1, −z+1.

Hydrogen-bond geometry (Å, °)

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Hydrogen-bond geometry (Å, °)