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Redetermination of the crystal structure of tricyclohexyltin(IV) iodide, $[Sn(C_6H_{11})_3]I$

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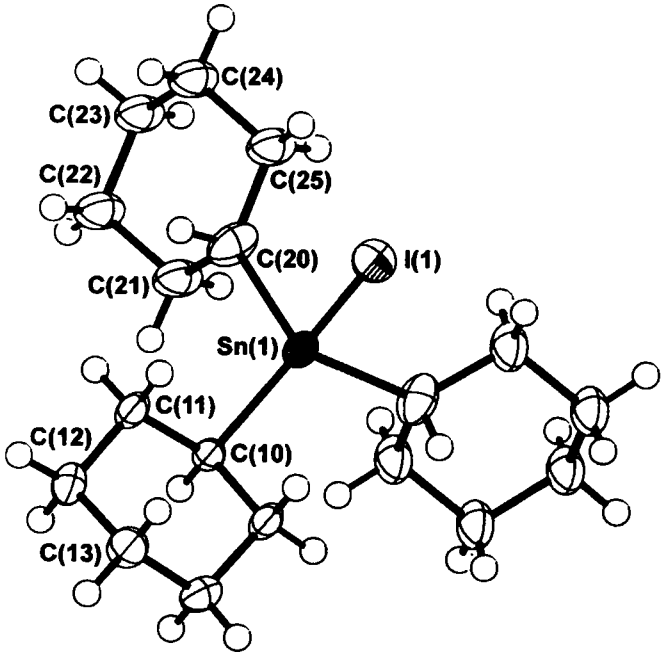

Abstract

$C_{18}H_{33}ISn$, orthorhombic, *Pnma* (no. 62), $a = 12.265(1)$ Å, $b = 16.707(2)$ Å, $c = 10.044(2)$ Å, $V = 2058.1$ Å^3, $Z = 4$, $R_{gt}(F) = 0.057$, $wR_{obs}(F^2) = 0.158$, $T = 293$ K.

Source of material

For the redetermination of the crystal structure we have synthesized the title compound in the normal two step procedure for transforming an organotin chloride into the corresponding iodide: The chloride dissolved in an organic solvent was first treated by a potassium hydroxide solution and then with hydroiodic acid. 1.99 g (2 mmol) Cy_3SnCl were dissolved in 90 ml toluene and heated together with a solution of 0.56 g (10 mmol, excess) KOH in 30 ml H_2O under reflux for 2 hours. After cooling the organic layer was separated and washed several times with distilled water. Thereafter, 2.33 g of a 55 % hydroiodic acid (HI: 0.64 g, 5 mmol, excess) were added and the mixture heated under reflux for 2 hours, again. After cooling the organic layer was separated, washed several times with distilled water and dried with Na_2SO_4. Single crystals were grown by slow evaporation of the solvent.

Experimental details

To reduce the number of parameters a common anisotropic displacement factor for all disordered carbon atoms was refined.

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Discussion

The crystal structure of tricyclohexyltin(IV)-iodide was first described in [1]. The published data, however, reveal several inconsistencies with respect to the crystal information (smaller cell volume than the corresponding bromide) as well as to the tin-iodine distance (254.4 pm) which seemed to be the length of a tin-bromine bond more than that of a tin-iodine bond normally (tin tetrahedrally coordinated) having a value of about 273 pm.

Within the crystal structure, the asymmetric unit consists of half a molecule: the tin and iodine atoms are lying on a mirror plane which also is intersecting a cyclohexyl group. One other cyclohexyl group occupies a general position showing, however, an orientational disorder, an effect often found for this group. The new crystal data are somewhat different from the earlier one, especially the cell volume now adopts a value (2058.1(5) Å^3) which represents the greater volume of the iodine atom in comparison to the bromine atom (cell volume of the isotypic Cy_3SnBr is 2004.5 Å^3 [1]) much more better. The new tin iodine distance of 272.9(2) pm as well shows a better agreement with those found in other triorganotin(IV)-iodides like Ph_3SnI (form A, 269.9 pm [2]; form B, 270.6 pm/269.2 pm [3]; (2-methoxyphenyl)$_3$SnI, 271.3 pm [4]; mesityl$_3$SnI; 275.3 pm/274.5 pm, deuterochloroform solvate [3]; 275.2 pm, 275.2 pm, toluene solvate [3]; $tmsm_3SnI$, 271.2 pm [5] and Neophyl$_3$SnI, 273.3 pm, 274.2 pm, 274.4 pm, 275.0 pm [6].

Table 1. Data collection and handling.

Crystal:	transparent, yellowish needle, size 0.23 × 0.25 × 0.58 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	27.34 cm^{-1}
Diffractometer, scan mode:	Siemens P4, θ-2θ
$2\theta_{max}$:	48°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	1370, 1056
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 792
$N(param)_{refined}$:	93
Programs:	SHELXS-97 [7], SHELXL-97 [8], SHELXTL [9]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(10)	4*c*	0.834	1/4	0.239	0.18(3)
H(111)	8*d*	1.041	0.327	0.245	0.18
H(112)	8*d*	0.929	0.372	0.237	0.18
H(121)	8*d*	0.881	0.323	0.449	0.18
H(122)	8*d*	0.990	0.372	0.459	0.18
H(131)	4*c*	1.090	1/4	0.449	0.18

Table 2. Continued.

Atom	Site	Occ.	x	y	z	U_{iso}
H(132)	4c		1.012	1/4	0.573	0.18
H(20)	8d		0.899	0.395	–0.041	0.26(4)
H(211)	8d	0.54	0.726	0.355	0.016	0.26
H(212)	8d	0.54	0.702	0.340	–0.136	0.26
H(221)	8d	0.54	0.643	0.469	–0.082	0.26
H(222)	8d	0.54	0.759	0.491	–0.025	0.26
H(231)	8d	0.54	0.714	0.461	–0.298	0.26
H(232)	8d	0.54	0.742	0.546	–0.240	0.26
H(241)	8d	0.54	0.920	0.504	–0.191	0.26
H(242)	8d	0.54	0.897	0.488	–0.343	0.26
H(251)	8d	0.54	0.867	0.352	–0.303	0.26

Table 2. Continued.

Atom	Site	Occ.	x	y	z	U_{iso}
H(252)	8d	0.54	0.981	0.375	–0.243	0.26
H(213)	8d	0.46	0.787	0.424	0.040	0.26
H(214)	8d	0.46	0.711	0.359	–0.021	0.26
H(223)	8d	0.46	0.695	0.431	–0.220	0.26
H(224)	8d	0.46	0.662	0.487	–0.102	0.26
H(233)	8d	0.46	0.834	0.549	–0.106	0.26
H(234)	8d	0.46	0.782	0.554	–0.249	0.26
H(243)	8d	0.46	0.966	0.512	–0.263	0.26
H(244)	8d	0.46	0.889	0.446	–0.322	0.26
H(253)	8d	0.46	1.015	0.384	–0.182	0.26
H(254)	8d	0.46	0.983	0.441	–0.064	0.26

Table 3. Atomic coordinates and displacement parameters (in $Å^2$).

Atom	Site	Occ.	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Sn(1)	4c		0.9243(1)	1/4	–0.0033(2)	0.1010(8)	0.0846(7)	0.145(1)	0	0.007(1)	0
I(1)	4c		1.1434(1)	1/4	–0.0512(3)	0.091(1)	0.168(2)	0.204(2)	0	0.028(1)	0
C(10)	4c		0.912(2)	1/4	0.214(2)	0.11(1)	0.08(1)	0.12(2)	0	0.02(1)	0
C(11)	8d		0.965(1)	0.3248(7)	0.272(2)	0.16(1)	0.070(8)	0.14(2)	0.003(8)	–0.01(1)	0.001(9)
C(12)	8d		0.957(2)	0.3237(9)	0.423(2)	0.19(2)	0.10(1)	0.15(2)	0.00(1)	0.00(2)	–0.03(1)
C(13)	4c		1.014(3)	1/4	0.477(3)	0.23(3)	0.13(2)	0.10(2)	0	–0.03(2)	0
C(20)	8d		0.866(1)	0.3540(9)	–0.098(2)	0.15(2)	0.13(1)	0.25(3)	0.03(1)	–0.02(2)	0.05(2)
C(21A)	8d	0.54	0.746(2)	0.371(1)	–0.074(3)	0.18(1)	0.16(1)	0.21(1)	0.034(9)	0.03(1)	0.07(1)
C(22A)	8d	0.54	0.721(2)	0.460(1)	–0.093(3)	0.18	0.16	0.21	0.034	0.03	0.07
C(23A)	8d	0.54	0.756(2)	0.489(1)	–0.231(3)	0.18	0.16	0.21	0.034	0.03	0.07
C(24A)	8d	0.54	0.877(2)	0.472(1)	–0.254(3)	0.18	0.16	0.21	0.034	0.03	0.07
C(25A)	8d	0.54	0.903(2)	0.383(1)	–0.234(2)	0.18	0.16	0.21	0.034	0.03	0.07
C(21B)	8d	0.46	0.767(2)	0.397(2)	–0.042(3)	0.18	0.16	0.21	0.034	0.03	0.07
C(22B)	8d	0.46	0.722(2)	0.459(2)	–0.142(4)	0.18	0.16	0.21	0.034	0.03	0.07
C(23B)	8d	0.46	0.811(3)	0.518(1)	–0.183(4)	0.18	0.16	0.21	0.034	0.03	0.07
C(24B)	8d	0.46	0.910(3)	0.474(2)	–0.241(4)	0.18	0.16	0.21	0.034	0.03	0.07
C(25B)	8d	0.46	0.955(1)	0.413(2)	–0.141(4)	0.18	0.16	0.21	0.034	0.03	0.07

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