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Synthesis and Structure of Tri(*p*-tolyl)antimony Dichloride

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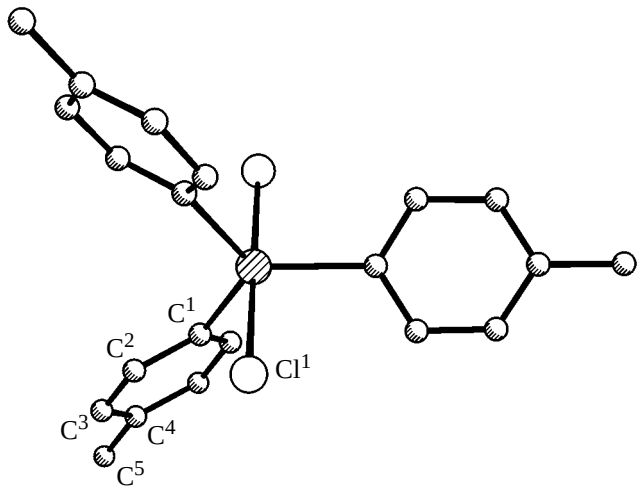
Abstract — Tri(*p*-tolyl)antimony dichloride was prepared by oxidation of tri(*p*-tolyl)stibine with chlorine or copper(II) chloride. As found by X-ray diffraction analysis, the antimony atom in tri(*p*-tolyl)antimony dichloride has the configuration of trigonal bipyramid with axially located chlorine atoms. The Sb–Cl and Sb–C distances are 2.476(2) and 2.104(7) Å, and the CSbC and ClSbCl angles are 120 and 180°, respectively.

The structure of phenyl derivatives of pentavalent antinmony of the general formula Ph_3SbX_2, where X is an electronegative substituent, has been described in [1, 2]. The majority of compounds of this type feature trigonal-bipyramidal coordination of the central atom with the electronegative ligands in the axial positions. In triphenylantimony dichloride, the hydrogen and carbon atoms of one of the phenyl groups come close to chlorine atoms inspite of the developing steric hindrances [3]. An analogous contact is observed in tri(*m*-tolyl)antimony dichloride, where the distance between the equatorial carbon atom of one of the phenyl groups and the chlorine atom (3.188 Å, [4]) is also shorter than the sum of the van der Waals radii of C and Cl (3.25 Å [5]).

To find out the reasons for nonbonded C···Cl interactions in compounds of this type, we have studied the structure of tri(*p*-tolyl)antimony dichloride (**I**). Earlier for this compound crystal lattice parameters were only reported [6]. The general view of the molecule is presented in the figure. The coordinates of non-hydrogen atoms are listed in Table 1, and the principal interatomic distances and bond angles, in Table 2.

According to X-ray diffraction data, the antimony atom in compound **I** has trigonal-bipyramidal coordination with axial tolyl groups and axial chlorine atoms. The molecular symmetry is D_3. Contrary to tri(*m*-tolyl)antimony dichloride, where the Cl–Sb–Cl angle is 177.2°, in tri(*p*-tolyl)antimony dichloride the location of these atoms is linear (the corresponding angle is 180°). The antimony atom lies strictly in the equatorial plane. All the phenyl ring planes form the same angles with the equatorial plane (37.9°) and with each other (64.2°). The Sb–Cl distances are equal to each other [2.476(2) Å], and the same is true of the Sb–C distances [2.104(7) Å]. Note that the antimony atom in tri(*m*-tolyl)antimony dichloride has a distorted trigonal-bipyramidal coordination. The corresponding interatomic distances are 2.4617, 2.4793, and 2.101, 2.112, and 2.116 Å.



General view of the molecule of compound **I**.

Hence, *para*-methyl substituents in the aryl rings favor increased molecular symmetry of triarylantimony dichloride. Because of the molecular symmetry

Table 1. Atomic coordinates ($\times 10^{-4}$ Å) and equivalent isotropic thermal parameters ($B \times 10^3$, Å^2) in the molecule of compound **I**

Atom	*x*	*y*	*z*	*B*
Sb	1250	1250	1250	61(1)
Cl^1	127(1)	127(1)	127(1)	83(1)
C^1	81(4)	1250	2419(4)	62(2)
C^2	–346(5)	324(4)	2774(5)	79(1)
C^3	–1098(5)	336(4)	3552(5)	81(2)
C^4	–1476(4)	1250	3976(4)	67(2)
C^5	–2297(5)	1250	4797(5)	95(3)

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Table 2. Interatomic distances (*d*) and bond angles (ω) in the molecule of compound **I**

Bond	*d*, Å	Angle	ω, deg
$Sb–C^{1\#1}$	2.104(7)	$C^{1\#1}–Sb–C^{1}$	120.0
$Sb–C^{1}$	2.104(7)	$C^{1\#1}–Sb–C^{1\#2}$	120.0
$Sb–C^{1\#2}$	2.104(7)	$C^{1}–Sb–C^{1\#2}$	120.0
$Sb–Cl^{1\#3}$	2.476(2)	$C^{1\#1}–Sb–Cl^{1\#3}$	90.0
$Sb–Cl^{1}$	2.476(2)	$C^{1}–Sb–Cl^{1\#3}$	90.0
$C^{1}–C^{2\#4}$	1.374(7)	$C^{1\#2}–Sb–Cl^{1\#3}$	90.0
$C^{1}–C^{2}$	1.374(7)	$C^{1\#1}–Sb–Cl^{1}$	90.0
$C^{3}–C^{4}$	1.371(6)	$C^{1}–Sb–Cl^{1}$	90.0
$C^{4}–C^{3\#4}$	1.371(6)	$C^{1\#2}–Sb–Cl^{1}$	90.0
$C^{4}–C^{5}$	1.478(11)	$Cl^{1\#3}–Sb–Cl^{1}$	180.0

of compound **I**, no intermolecular contacts between the carbon atoms of the p-tolyl rings and the chlorine atoms are observed.

EXPERIMENTAL

Single crystal X-ray diffraction analysis of tri-(*p*-tolyl)antimony dichloride (I). $C_{21}H_{21}Cl_2Sb$. The unit cell parameters and the intensities of 1153 unique reflections with $I > \sigma(I)$ were measured on a CAD-4 automatic diffractometer (λMoK_α radiation, λ 0.71073 Å, Nb filter, 2θ/θ scanning). Cubic crystals; at 20°C, α 12.729(1), *V* 2062.4(3) $Å^3$, space group $P4_332$, *Z* 6, and d_{calc} 1.501 g/cm^3. The structure was solved by the heavy atom method and refined anisotropically (isotropically for hydrogen atoms) to *R* 0.023 and R_W 0.065. All calculations were carried out using the SHELXL-97 program package [7].

Tri(*p*-tolyl)antimony dichloride (I). To a solution of 1.00 g of tri(*p*-tolyl)stibine in 10 ml of acetone, a solution of 0.77 g of copper dichloride in 10 ml of ethanol was added. The reaction mixture was kept at room temperature for 12 h, the solvent was removed, and the residue was crystallized from toluene to obtain 1.15 g (95%) of compound **I**, mp 155°C. Published data [8]: mp 155–156°.

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