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# A STUDY OF CRYSTAL STRUCTURES OF TETRA(*p*-TOLYL)ANTIMONY 2,4,6-TRICHLOROPHENOXIDE AND 3,4,5-TRIFLUOROBENZOATE

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Tetra(*p*-tolyl)antimony 2,4,6-trichlorophenoxide *p*-$Tol_4SbOC_6H_2Cl_3$-2,4,6 (**1**) and tetra(*p*-tolyl)antimony 3,4,5-trifluorobenzoate *p*-$Tol_4SbOC(O)C_6H_2F_3$-3,4,5 (**2**) are synthesized by the interaction of penta(*p*-tolyl)antimony with equimolar amounts of 2,4,6-trichlorophenol and 3,4,5-trifluorobenzoic acid in a benzene solution. Compounds **1** and **2** are also obtained by ligand redistribution reactions between penta(*p*-tolyl)antimony and symmetric antimony derivatives *p*-$Tol_3SbX_2$. The crystal structures and structural features of compounds **1** and **2**, in which antimony atoms have a trigonal bipyramidal coordination of substituents with axially located oxygen-containing ligands, are determined by single crystal X-ray diffraction.

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**Keywords:** tetra(*p*-tolyl)antimony 2,4,6-trichlorophenoxide, tetra(*p*-tolyl)antimony 3,4,5-trifluorobenzoate, synthesis, crystal and molecular structures.

## INTRODUCTION

Pentavalent antimony compounds with the general formula $Ar_4SbX$ (where X is a ligand bonded to the antimony atom via a heteroatom) have been structurally characterized sufficiently well [1-23]. Tetraphenylantimony carboxylates and aroxides are most well studied in the wide range of these derivatives [24-37]. These compounds are synthesized by substitution reactions between acids (phenols) and pentaphenylantimony [24, 25, 27, 28] or tetraphenylantimony halide in the presence of a hydrogen halide acceptor [29-36]. A method based on reactions of tetraphenylantimony halide with acid salts has been known, too [37]. Synthesis by the ligand redistribution reaction between pentaphenylantimony and a derivative with the symmetric structure $Ph_3SbX_2$ is less popular [26]. Note that data on the synthesis and structure of tetra(*p*-tolyl)antimony derivatives over the last five years were given only in [38, 39].

This work is devoted to the synthesis and crystal structure studies of tetra(*p*-tolyl)antimony 2,4,6-trichlorophenoxide *p*-$Tol_4SbOC_6H_2Cl_3$-2,4,6 (**1**) and tetra(*p*-tolyl)antimony 3,4,5-trifluorobenzoate *p*-$Tol_4SbOC(O)C_6H_2F_3$-3,4,5 (**2**).

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## EXPERIMENTAL

**General synthesis procedure.** The starting reagent (penta(*p*-tolyl)antimony) was obtained by the procedure described in [40]. In the work, we used 2,4,6-trichlorophenol and 3,4,5-trifluorobenzoic acid (Alfa Aesar). In the first case, compound **1** was synthesized by the interaction of 0.346 mmol of penta(*p*-tolyl)antimony with 0.346 mmol of 2,4,6-trichlorophenol in 2 mL of benzene in a sealed ampoule. After heating the ampoule for 1 h at 80 °C and subsequent slow cooling colorless crystals of **1** were isolated from the reaction mixture (yield 91%, $T_{\mathrm{melt}}$ 188 °C). In the second case, **1** was obtained from 0.346 mmol of penta(*p*-tolyl)antimony and 0.346 mmol of *tris*(*p*-tolyl)antimony *bis*(2,4,6-trichlorophenoxide) in 2 mL of benzene by the similar procedure (yield 90%, $T_{\mathrm{melt}}$ 188 °C).

The IR spectrum was measured on a Shimadzu IRAffinity-1S IR-Fourier spectrometer in KBr pellets in the range 4000-400 $\mathrm{cm}^{-1}$. The C, H elemental analysis was conducted on a Carlo-Erba 1106 instrument.

Found for **1** (%): C 59.73, H 4.51; calculated for $C_{34}H_{30}OCl_3Sb$ (%): C 59.81, H 4.44.

IR spectrum (ν, $\mathrm{cm}^{-1}$): 3111, 3059, 3024, 3012, 2974, 2954, 2918, 2864, 1639, 1591, 1560, 1527, 1492, 1452, 1440, 1419, 1388, 1307, 1298, 1273, 1247, 1230, 1211, 1188, 1178, 1116, 1062, 1037, 1012, 972, 947, 869, 854, 796, 740, 700, 678, 634, 582, 572, 489, 426, 403.

**TABLE 1.** Crystallographic Data and Conditions of the Diffraction Experiment for **1** and **2**

| Parameter | **1** | **2** |
|---|---|---|
| Stoichiometric formula | $C_{34}H_{30}OCl_3Sb$ | $C_{35}H_{30}O_2F_3Sb$ |
| *M* | 682.68 | 661.34 |
| Crystal system | Monoclinic | Triclinic |
| Space group | $P2_1/n$ | $P\overline{1}$ |
| *a*, *b*, *c*, Å | 11.787(8), 17.357(13),15.897(14) | 10.276(8), 11.619(10), 14.120(9) |
| α, β, γ, deg | 90.00, 103.15(4), 90.00 | 75.95(4), 88.93(4), 70.97(4) |
| *V*, $Å^3$ | 3167(4) | 1543(2) |
| *Z* | 4 | 2 |
| $\rho_{\mathrm{calc}}$, $g/cm^3$ | 1.432 | 1.424 |
| μ, $\mathrm{mm}^{-1}$ | 1.149 | 0.941 |
| *F*(000) | 1376.0 | 668.0 |
| Crystal dimensions, mm | 0.26×0.25×0.07 | 0.38×0.36×0.25 |
| 2θ range of data collection, deg | from 5.88 to 54.3 | from 6.56 to 71.74 |
| Index range | $-15 \le h \le 15$,<br>$-22 \le k \le 22$,<br>$-20 \le l \le 20$ | $-16 \le h \le 16$,<br>$-19 \le k \le 19$,<br>$-23 \le l \le 23$ |
| Number of measured / unique reflections | 41724 / 6980<br>[$R_{\mathrm{int}}$ = 0.0484] | 109386 / 14259<br>[$R_{\mathrm{int}}$ = 0.0444] |
| Data collection completeness over θ = 25.00°,% | 0.994 | 0.985 |
| Refinement method | Full-matrix LSM on $F^2$ | Full-matrix LSM on $F^2$ |
| Number of reflections / restraints / parameters | 6980 / 0 / 356 | 14259 / 0 / 374 |
| *S*-factor on $F^2$ | 1.028 | 1.043 |
| *R*-factor [$I > 2\sigma(I)$] | $R_1$ = 0.0302,<br>$wR_2$ = 0.0629 | $R_1$ = 0.0446,<br>$wR_2$ = 0.0838 |
| *R*-factor (all data) | $R_1$ = 0.0500,<br>$wR_2$ = 0.0702 | $R_1$ = 0.0835,<br>$wR_2$ = 0.0954 |
| Residual electron density (min and max), e/$Å^3$ | –0.57 and 0.39 | –0.55 and 0.64 |

Compound **2** was obtained similarly. Yield 90%, $T_{melt}$ 182 °C.

Found for **2** (%): C 63.49, H 4.63; calculated for $C_{35}H_{30}O_2F_3Sb$ (%): C 63.56, H 4.58.

IR spectrum (ν, cm$^{-1}$): 3074, 3034, 3016, 2922, 2866, 1637, 1616, 1593, 1564, 1521, 1492, 1433, 1394, 1355, 1296, 1273, 1226, 1211, 1190, 1178, 1116, 1093, 1064, 1043, 1016, 970, 952, 894, 846, 831, 798, 783, 756, 732, 711, 700, 638, 582, 574, 551, 489, 480, 445.

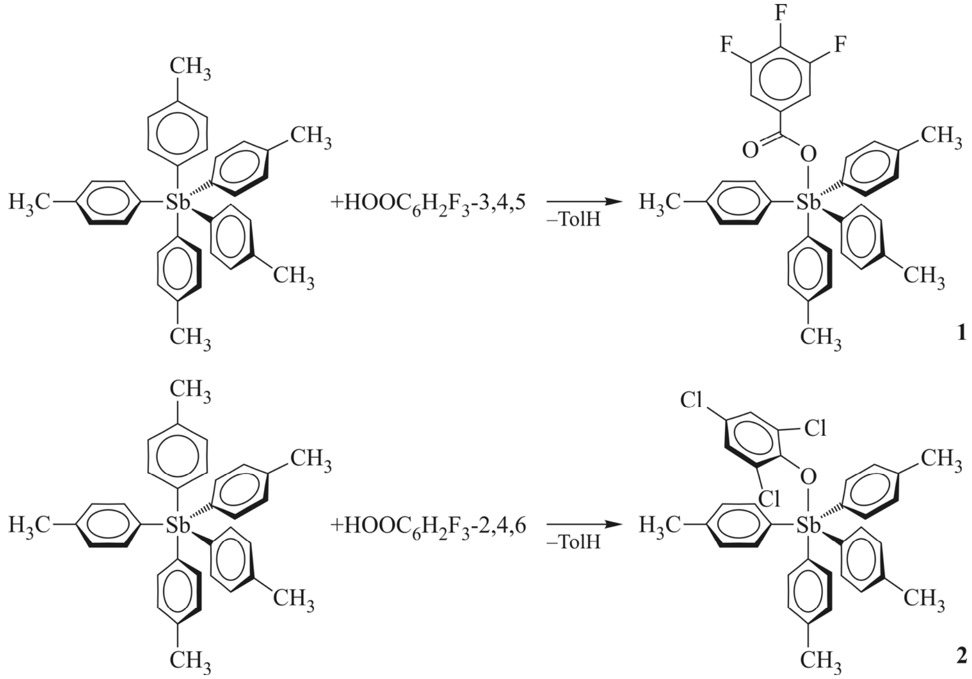
X-ray experiments were carried out on a D8 Quest Bruker diffractometer (Mo$K_\alpha$ radiation, λ = 0.71073 Å, graphite monochromator) at 293(2) K. Data collection and editing, refinement of unit cell parameters, and absorption correction were performed using SMART and SAINT-*Plus* programs [41]. All structure calculations and refinement were carried out using the SHELXL/PC [42] and Olex2 [43] programs. The structures were solved by a direct method and refined by the least squares technique in the anisotropic approximation for non-hydrogen atoms. Positions of hydrogen atoms were calculated geometrically and included in the refinement in the riding model. Full tables of atomic coordinates, bond lengths and bond angles have been deposited with the Cambridge Crystallography Data Center (Nos. 1957182 (**1**), 1978214 (**2**); deposit@ccdc.cam.ac.uk; http://www.ccdc.cam.ac.uk). Main crystallographic data and refinement results for structures **1** and **2** are summarized in Table 1; selected bond lengths and bond angles are listed in Table 2.

**TABLE 2.** Selected Bond Lengths and Bond Angles in the Structures of **1** and **2**

| Bond | *d*, Å | Angle | ω, deg |
|---|---|---|---|
| | | **1** | |
| Sb1–O1 | 2.266(2) | C1–Sb1–O1 | 83.29(10) |
| Sb1–C1 | 2.121(3) | C1–Sb1–C31 | 111.10(10) |
| Sb1–C11 | 2.118(3) | C1–Sb1–C21 | 95.10(11) |
| Sb1–C31 | 2.125(3) | C11–Sb1–O1 | 83.35(8) |
| Sb1–C21 | 2.173(3) | C11–Sb1–C1 | 128.33(10) |
| O1–C41 | 1.316(3) | C11–Sb1–C31 | 117.17(11) |
| Cl3–C46 | 1.737(3) | C11–Sb1–C21 | 96.49(10) |
| Cl2–C44 | 1.751(3) | C31–Sb1–O1 | 85.28(10) |
| Cl1–C42 | 1.727(3) | C31–Sb1–C21 | 96.77(11) |
| C4–C7 | 1.521(5) | C21–Sb1–O1 | 177.76(8) |
| C14–C17 | 1.528(5) | C41–O1–Sb1 | 130.46(15) |
| | | **2** | |
| Sb1–O1 | 2.294(2) | C1–Sb1–O1 | 85.32(8) |
| Sb1–C1 | 2.123(3) | C1–Sb1–C41 | 92.68(9) |
| Sb1–C21 | 2.118(2) | C21–Sb1–O1 | 84.32(9) |
| Sb1–C41 | 2.157(2) | C21–Sb1–C1 | 126.86(8) |
| Sb1–C11 | 2.114(2) | C21–Sb1–C41 | 96.18(9) |
| O1–C37 | 1.293(2) | C41–Sb1–O1 | 177.80(6) |
| O2–C37 | 1.218(3) | C11–Sb1–O1 | 82.57(7) |
| C35–F3 | 1.344(3) | C11–Sb1–C1 | 116.48(9) |
| C33–F1 | 1.347(4) | C11–Sb1–C21 | 113.53(8) |
| F2–C34 | 1.345(3) | C11–Sb1–C41 | 99.17(8) |
| C4–C7 | 1.533(4) | C37–O1–Sb1 | 122.17(13) |
| C24–C27 | 1.518(3) | O2–C37–O1 | 126.40(18) |

## RESULTS AND DISCUSSION

Compounds **1** and **2** were synthesized by the interaction of penta(*p*-tolyl)antimony with phenol or acid in benzene (Scheme 1)



**Scheme 1.** Synthesis of compounds **1** and **2**.

Derivatives of **1** and **2** were also obtained by the ligand redistribution reaction between penta(*p*-tolyl)antimony and a derivative with the symmetric structure *p*-$Tol_3SbX_2$ in a benzene solution

$$p\text{-}Tol_5Sb + p\text{-}Tol_3SbX_2 \rightarrow 2\ p\text{-}Tol_4SbX$$

$$X = OC_6H_2Cl_3\text{-}2,4,6\ (\mathbf{1}),\ \ OC(O)C_6H_2F_3\text{-}3,4,5\ (\mathbf{2}).$$

Both syntheses are characterized by a high yield and the purity of the target product.

To study the structures of the compounds IR spectroscopy and single crystal X-ray diffraction (XRD) were employed.

The IR spectra of compounds **1** and **2** exhibit an intense absorption band at 489 $cm^{-1}$ that characterizes vibrations of Sb–C bonds. An absorption band at 1298 $cm^{-1}$ corresponds to stretching vibrations of C–O bonds in the IR spectrum of compound **1**. The 740 $cm^{-1}$ band that is located in a higher frequency range compared to the similar band in the IR spectrum of 2,4,6-trichlorophenol (732 $cm^{-1}$) may be assigned to vibrations of C–Cl bonds. An intense absorption band at 1637 $cm^{-1}$ characterizes the carbonyl group in the spectrum of compound **2**. Absorption bands in the range 3111-3012 $cm^{-1}$ belong to vibrations of $C_{Ar}$–H bonds in aryl, phenoxide, and carboxylate ligands. The bands of stretching vibrations of methyl groups of *p*-tolyl substituents are also observed: asymmetric vibrations at 2918 $cm^{-1}$ (**1**), 2922 $cm^{-1}$ (**2**), symmetric vibrations at 2864 $cm^{-1}$ (**1**), 2866 $cm^{-1}$ (**2**) [44].

According to the data of the single crystal XRD analysis, in the molecules of **1** and **2** the antimony atoms have a distorted trigonal bipyramidal coordination with axially located aroxide and carboxylate ligands respectively (Figs. 1, 2).

The CSbO axial angles are 177.76(8)° (**1**), 177.80(6)° (**2**); the sums of CSbC angles in the equatorial plane are 356.6(10)° (**1**), 356.87(8)° (**2**). The values of $O_{ax}SbC_{eq}$ angles are less than 90° while those of $C_{ax}SbC_{eq}$ are larger than 90°. Antimony atoms are out of the $[C_3]$ equatorial plane towards the axial carbon atom by 0.225 Å (**1**) and 0.216 Å (**2**). All bond angles at antimony atoms in the molecules of **1** and **2** have similar values. The Sb–$C_{eq}$ bond lengths in the molecules are also

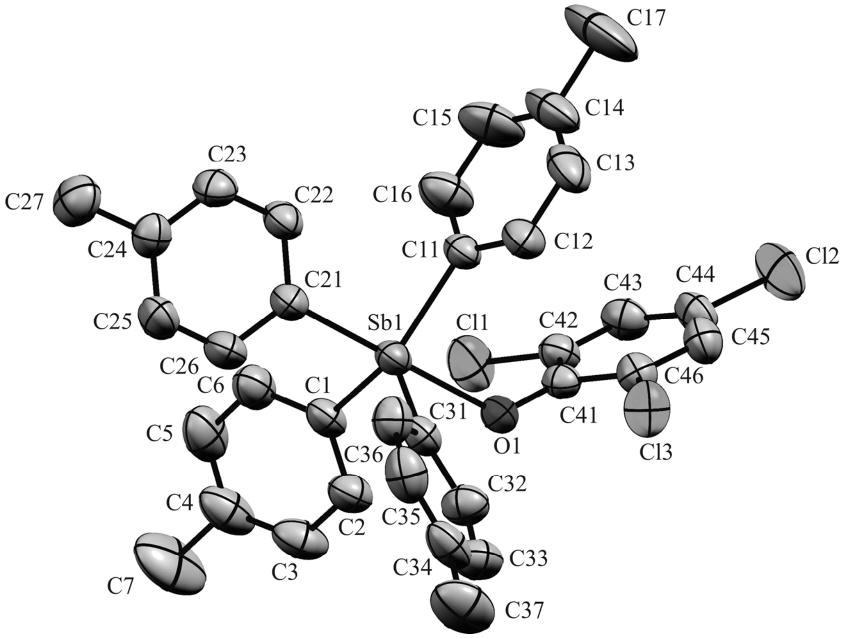


**Fig. 1.** General view of the molecule of compound **1** (hydrogen atoms are omitted).

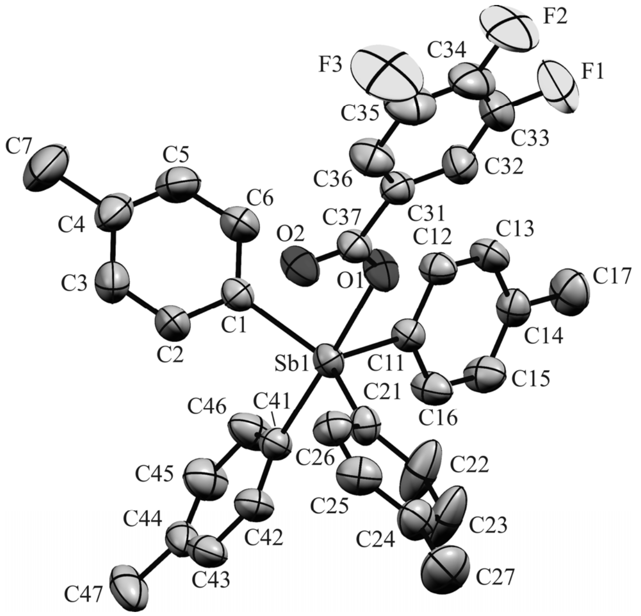


**Fig. 2.** General view of the molecule of compound **2** (hydrogen atoms are omitted).

approximately the same (2.121(3) Å, 2.118(3) Å, 2.125(3) Å (**1**) and 2.123(3) Å, 2.118(2) Å, 2.114(2) Å (**2**)). A distinction is observed in Sb–C$_{ax}$ distances, which is 0.016 Å larger in **1** than **2**. On the contrary, the Sb–O distance is 0.028 Å larger in the molecule of **2** than that in **1**. An increase in Sb–O distances in comparison with the sum of covalent radii of antimony and oxygen (2.07 Å), which is observed in the molecules of tetraarylantimony aroxides and carboxylates [24-37], indicates the coordination character of this bond [38, 39]. The increase in the Sb–O distance in **2** can be explained by a lower basicity of the carboxylate anion in comparison with the basicity of the aroxide anion, and hence, its lower coordination ability.

A feature is revealed in the arrangement of equatorial aryl groups in the molecule of **1**. Thus, two aryl rings ([C1–C6] and [C31–C36]) in the structure are turned about the equatorial Sb–C bonds so that the angles between their planes and

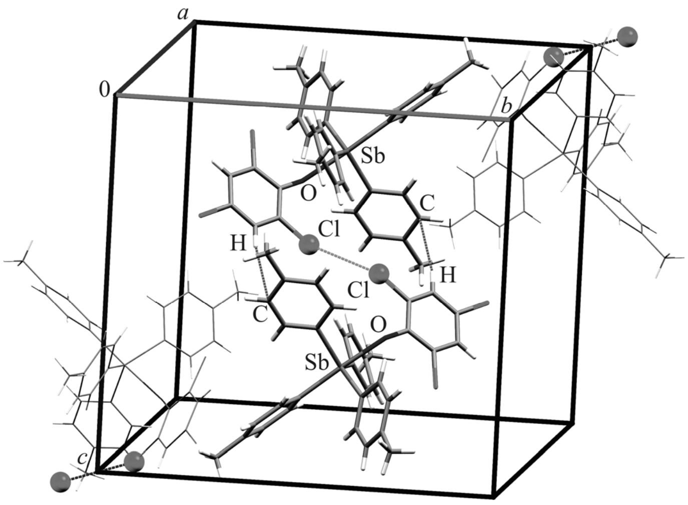


**Fig. 3.** Scheme of the molecular interaction of one crystallographic cell of compound **1**.

the equatorial [C1C11C31] plane are 57.27° and 68.94° respectively, whereas a similar angle with the plane of the third aromatic ring [C11–C16] is only 13.38°. This ring is located opposite to the C41–C46 aromatic ring plane of the aroxy group, which determines the interaction of their π systems. However, the ideal geometry of this π–π stacking interaction is distorted: the interplanar angle is 38.72°, the distance between the centers of π systems of the aroxide and *p*-tolyl ligands is 4.01 Å. Similar interactions are also observed in other tetraarylantimony aroxides [24, 25, 36, 38].

A characteristic feature of tetraarylantimony carboxylates is the presence of the intramolecular contact of the antimony atom with the carbonyl oxygen atom [26-37, 39]. In the structure of compound **2**, the Sb···O=C distance is 3.377(3) Å, which is smaller than the sum of van der Waals radii of antimony and oxygen atoms (3.70 Å) [45]. This interaction causes an increase in one of the equatorial C(1)SbC(21) angles (126.86(8)°) located from the side of the intramolecular contact.

The structures of the crystals of compounds **1** and **2** are organized by weak halogen–hydrogen hydrogen bonds of Cl2···H–C22 (2.94 Å) (**1**) and F2···H–C17 (2.47 Å) (**2**) types and C–H π-interactions.

The crystal of compound **1** also contains intermolecular Cl···Cl contacts between two molecules of one crystallographic cell ($Z = 4$) (Fig. 3). The two types of dimers formed alternate along the $[ab\overline{c}]$ parallelepiped diagonal.

The Cl···Cl distances are shorter than the sum of van der Waals radii of chlorine atoms (3.60 Å) and are 3.32 Å, which evidences the occurrence of halogen···halogen interactions. All C–Cl···Cl angles at chlorine atoms are the same (146.92(9)°) because these interactions are observed only between symmetrically equivalent chlorine atoms bound by the center of inversion. The halogen···halogen interaction corresponds to type I characterized by similar or equal values of angles around halogen atoms [46].

Thus, in this work on substitution and ligand redistribution reactions, tetra(*p*-tolyl)antimony 2,4,6-trichlorophenoxide and 3,4,5-trifluorobenzoate are obtained with a high yield and structurally characterized. The coordination polyhedron of the antimony atom in both compounds is a distorted trigonal bipyramid with an axially located oxygen-containing ligand. In the crystal of tetra(*p*-tolyl)antimony 2,4,6-trichlorophenoxide there are not only weak hydrogen bonds but also short halogen···halogen contacts.

## CONFLICT OF INTERESTS

The authors declare that they have no conflict of interests.

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