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Triphenyl- and Tris(*para*-tolyl)antimony Dicarboxylates: Synthesis and Structures

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Abstract—The reactions of tris(*para*-tolyl)antimony $(4\text{-MeC}_6H_4)_3Sb$ with chloroacetic acid and of triphenylantimony Ph_3Sb with 3,4,5-trifluorobenzoic and 2,3-difluorobenzoic acids in the presence of *tert*-butyl hydroperoxide afford triarylantimony dicarboxylates $Ar_3Sb[OC(O)R]_2$, where Ar = *p*-Tol, R = CH_2Cl (**I**), Ar = Ph, $C_6H_2F_3$-3,4,5 (**II**), and $C_6H_3F_2$-2,3 (**III**). According to the X-ray diffraction data (CIF files CCDC nos. 1883320 (**I**), 1979844 (**II**), and 1980334 (**III**)), the Sb atoms in compounds **I**–**III** have the coordination of a trigonal bipyramid. The OSbO axial angles are 174.47(6)° (**I**), 173.98(7)° (**II**), and 175.98(6)° (**III**). The Sb–O and Sb–C bond lengths are 2.145(2), 2.143(2), and 2.104(2)–2.109(2) Å in compound **I**; 2.121(2), 2.121(2) and 2.098(2)–2.112(3) Å in compound **II**; and 2.1302(18), 2.1132(18), and 2.109(3)–2.117(3) Å in compound **III**. The intramolecular Sb···O distances with the carbonyl oxygen atom (3.054(3) and 3.080(3) Å (**I**), 3.033 Å (**II**), and 2.959(3) and 3.054(2) Å (**III**)) are less than the sum of the van der Waals radii of Sb and O. The structural organization of the compounds is due to hydrogen bonds of the O···H and F···H types, CH···π and F···π interactions, and stacking effect.

Keywords: triarylantimony, triarylantimony dicarboxylate, oxidative addition reaction, X-ray diffraction analysis

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INTRODUCTION

It is known that the biological activity of the organic antimony compounds depends on the type of ligands at the metal atom and the nature of substituents in the ligands [1, 2]. Triarylantimony dicarboxylates containing fluorine atoms in the aryl ligands are found to be more reactive [3, 4].

It can be assumed that the introduction of fluorine atoms in the carboxylate ligands would enhance their biological activity and, hence, the synthesis of similar antimony compounds is very promising.

A series of triarylantimony dicarboxylates synthesized via the oxidative addition reaction from triarylantimony and carboxylic acids HX in the presence of peroxide [5–12] or via the substitution reaction is known to the present time [3, 4, 13, 14]. The synthesis and specific features of the structures of triphenylantimony dicarboxylates with a high concentration of fluorine atoms in the caboxylate ligands were reported [9, 15–18].

Continuing the studies of specific features of the synthesis and structures of the pentavalent antimony compounds, we synthesized tris(*para*-tolyl)antimony dicarboxylate $p\text{-Tol}_3Sb[OC(O)CH_2Cl]_2$ (**I**) and triphenylantimony dicarboxylates $Ph_3Sb[OC(O)R]_2$ (R = $C_6H_2F_3$-3,4,5 (**II**) and $C_6H_3F_2$-2,3 (**III**)) with halogen atoms (chlorine, fluorine) in the carboxylate ligands and studied their molecular and crystal structures.

EXPERIMENTAL

Synthesis of tris(*para*-tolyl)antimony bis(chloroacetate) (I). A 70% aqueous solution of *tert*-butyl hydroperoxide (0.032 g, 0.25 mmol) was added to a solution of tris(*para*-tolyl)antimony (0.100 g, 0.25 mmol) and chloroacetic acid (0.047 g, 0.50 mmol) in diethyl ether (20 mL). The mixture was kept at 20°C for 24 h observing the formation of colorless crystals. The yield of compound **I** was 179 mg (99%), T_{decomp} = 148°C.

IR (ν, cm^{-1}): 3035, 3018, 2954, 2920, 2866, 1683, 1654, 1591, 1492, 1444, 1398, 1328, 1317, 1255, 1219, 1197, 1192, 1165, 1118, 1068, 1014, 933, 837, 798, 698, 677, 586, 514, 486, 443.

For $C_{25}H_{25}O_4Cl_2Sb$

Anal. calcd., %	C, 51.58	H, 4.34
Found, %	C, 51.45	H, 4.43

Compounds **II** and **III** were synthesized using a similar procedure.

Triphenylantimony bis(3,4,5-trifluorobenzoate) **(II)** was synthesized as colorless crystals in a yield of 99%, $T_m = 236°C$.

IR (ν, cm^{-1}): 3145, 3080, 3066, 2991, 1971, 1886, 1822, 1778, 1662, 1622, 1595, 1577, 1552, 1525, 1481, 1436, 1369, 1355, 1336, 1286, 1267, 1232, 1195, 1188, 1161, 1097, 1082, 1070, 1047, 1022, 997, 960, 914, 889, 875, 844, 804, 777, 740, 709, 690, 669, 657, 644, 615, 561, 522, 474, 457, 449.

For $C_{32}H_{19}O_4F_6Sb$

Anal. calcd., %	C, 54.65	H, 2.73
Found, %	C, 54.53	H, 2.80

Triphenylantimony bis(2,3-difluorobenzoate) **(III)** was synthesized as colorless crystals in a yield of 99%, $T_m = 194°C$.

IR (ν, cm^{-1}): 3093, 3072, 3045, 1967, 1894, 1828, 1778, 1728, 1645, 1589, 1573, 1481, 1436, 1408, 1348, 1328, 1273, 1224, 1199, 1184, 1151, 1099, 1064, 1020, 997, 954, 918, 854, 835, 785, 758, 738, 690, 634, 513, 493, 453.

For $C_{32}H_{21}O_4F_4Sb$

Anal. calcd., %	C, 57.60	H, 3.17
Found, %	C, 57.54	H, 3.23

The IR spectra of complexes **I**–**III** were recorded on a Shimadzu IR Affinity-1S IR spectrometer in a range of 4000–400 cm^{-1} (KBr pellets).

X-ray diffraction analysis (XRD) of the crystals of complexes **I**–**III** was carried out on a D8 Quest diffractometer (Bruker) (MoK_α radiation, λ = 0.71073 Å, graphite monochromator) at 293 K. Data were collected and edited, unit cell parameters were refined, and an absorption correction was applied using the SMART and SAINT-Plus programs [19]. All calculations on structure determination and refinement were performed using the SHELXL/PC [20] and OLEX2 [21] programs. The structures were determined by a direct method and refined by least squares in the anisotropic approximation for non-hydrogen atoms. The positions of hydrogen atoms were determined geometrically by the riding model. The crystallographic data and structure refinement results are presented in Table 1. Selected bond lengths and bond angles are given in Table 2.

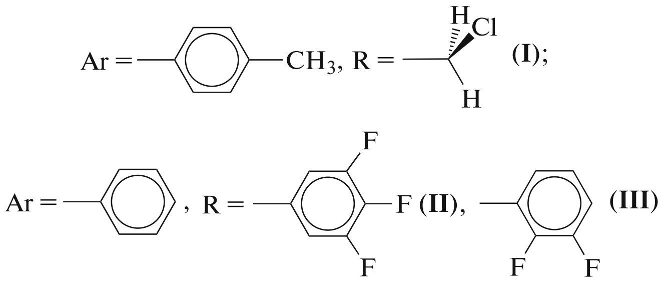
The full tables of atomic coordinates, bond lengths, and bond angles were deposited with the Cambridge Crystallographic Data Centre (CIF files CCDC nos. 1883320 (**I**), 1979844 (**II**), and 1980334 (**III**); deposit@ccdc.cam.ac.uk; http://www.ccdc.cam.ac.uk).

RESULTS AND DISCUSSION

It is known that the oxidation of triphenylantimony by *tert*-butyl hydroperoxide in the presence of carboxylic acids affords triphenylantimony dicarboxylates of the general formula $Ph_3Sb[OC(O)R)]_2$ [4].

We found that the reactions of tris(*para*-tolyl)antimony with chloroacetic acid and of triphenylantimony with 3,4,5-trifluoro- and 2,3-difluorobenzoic acids in the presence of *tert*-butyl hydroperoxide (molar ratio 1 : 2 : 1) occur in diethyl ether to form triarylantimony dicarboxylates isolated in the yield up to 99%.

$$Ar_3Sb + 2HOC(O)R + t\text{-}BuOOH \rightarrow Ar_3Sb[OC(O)R]_2 + H_2O + t\text{-}BuOH$$



After diethyl ether was evaporated, stable in air colorless crystals of the target compounds were obtained.

The IR spectra of compounds **I**–**III** exhibit an intense absorption band of stretching vibrations of the SbC_3 fragment at 486, 457, and 453 cm^{-1}, respectively. The presence of the carbonyl group is characterized by the high-intensity absorption band of stretching vibrations of the C=O bond at 1683 (**I**), 1662 (**II**), and 1645 (**III**) cm^{-1}. The absorption bands at 1591, 1492, and 1444 cm^{-1} (**I**); 1595, 1481, and 1436 cm^{-1} (**II**); and 1589, 1481, and 1436 cm^{-1} (**III**) characterize stretching vibrations of the carbon skeletal of the benzene rings. The medium-intensity absorption band at 3035 (**I**), 3066 (**II**), and 3072 (**III**) cm^{-1} corresponds to stretching vibrations of the C_{Ar}–H bond, whereas the highly intense bands at 798 and 698 (**I**), 740 and 690 (**II**), and 738 and 690 (**III**) cm^{-1} correspond to out-of-plane bending vibrations of the same bonds. The IR

Table 1. Crystallographic data and experimental and structure refinement parameters for compounds **I–III**

Parameter	**I**	**II**	**III**
FW	582.10	703.22	667.24
Crystal system	Triclinic	Monoclinic	Triclinic
T, K	293.15	293.15	293.15
Space group	$P\bar{1}$	$I2/a$	$P\bar{1}$
a, Å	10.273(6)	11.525(7)	9.113(5)
b, Å	11.681(8)	22.814(13)	11.043(6)
c, Å	12.876(6)	11.965(8)	14.770(12)
α, deg	106.891(19)	90	69.74(3)
β, deg	104.18(2)	114.48(5)	86.98(3)
γ, deg	108.55(3)	90	83.203(18)
V, Å^3	1301.2(14)	2863(3)	1384.5(16)
Z	2	4	2
ρ_{calc}, g/cm^3	1.486	1.631	1.601
μ, mm^{-1}	1.293	1.039	1.060
F(000)	584.0	1392.0	664.0
Crystal size, mm	0.32 × 0.14 × 0.1	0.36 × 0.24 × 0.17	0.36 × 0.33 × 0.19
2θ, deg	5.866–56.998	6.534–56.988	5.666–57
Ranges of reflection indices	$-13 \le h \le 13$, $-15 \le k \le 15$, $-17 \le l \le 17$	$-15 \le h \le 15$, $-30 \le k \le 30$, $-16 \le l \le 15$	$-12 \le h \le 12$, $-14 \le k \le 14$, $-19 \le l \le 19$
Total number of reflections	77253	54936	59367
Independent reflections (R_{int})	6582 (0.0365)	3636 (0.0313)	7001 (0.0280)
Reflections with $I > 2\sigma(I)$	5933	3352	6369
Number of refined parameters	293	197	377
GOOF	1.032	1.074	1.084
R factors for $F^2 > 2\sigma(F^2)$	$R_1 = 0.0271$, $wR_2 = 0.0695$	$R_1 = 0.0228$, $wR_2 = 0.0564$	$R_1 = 0.0273$, $wR_2 = 0.0647$
R factors for all reflections	$R_1 = 0.0323$ $wR_2 = 0.0722$	$R_1 = 0.0265$ $wR_2 = 0.0583$	$R_1 = 0.0328$ $wR_2 = 0.0697$
Residual electron density (max/min), e/Å^3	0.76/–1.06	0.39/–0.34	0.88/–0.41

spectrum of compound **I** contains absorption bands of stretching vibrations of the methyl groups, whose asymmetric vibrations lie at 2920 cm^{-1} and symmetric vibrations are observed at 2866 cm^{-1} [22, 23].

According to the XRD data, the Sb atoms in compounds **I–III** lie in the equatorial plane and have a distorted trigonal bipyramidal coordination with the oxygen atoms of the carboxylate ligands in the axial positions (Fig. 1). The molecules of compound **II** are symmetric and contain the symmetry axis of the second order lying along the Sb(1)–C(7) bond. The fluorine atoms of one carboxylate ligand of compound **III** are disordered over two positions. The refined ratio of contributions of the positions to the disordered fragment is 0.91/0.09.

The sums of angles in the equatorial CSbC plane in the molecules of triarylantimony dicarboxylates are 360°. The antimony atom shifts from the equatorial plane [C_3] only in the structures of compounds **I** and **III** by 0.002 and 0.028 Å, respectively, and the antimony atom in the structure of compound **II** lies in this plane. The equatorial ligands in the structures of compounds **I–III** are unfolded around the Sb–C bonds in such a way that the intra- and intermolecular contacts would be reduced to minimum. The dihedral angles between the planes of the benzene rings of the aryl ligands and equatorial plane are 9.30° C(1)–C(6), 87.46° C(11)–C(16), and 4.88° C(21)–C(26) (**I**); 19.98° C(1)–C(6), 19.98° C(1')–C(6'), and 80.29° C(7)–C(8') (**II**); and 87.14° C(1)–C(6), 15.87° C(11)–C(16), and 12.90° C(21)–C(26) (**III**). Note

Table 2. Selected bond lengths and bond angles in the structures of compounds **I**–**III***

Bond	Length, Å	Angle	ω, deg
I			
Sb(1)–O(1)	2.145(2)	O(3)Sb(1)O(1)	174.47(6)
Sb(1)–O(3)	2.143(2)	C(1)Sb(1)O(1)	90.52(9)
Sb(1)–C(1)	2.109(2)	C(1)Sb(1)O(3)	89.55(9)
Sb(1)–C(11)	2.109(2)	C(11)Sb(1)O(1)	86.99(8)
Sb(1)–C(21)	2.104(2)	C(11)Sb(1)O(3)	87.90(8)
O(1)–C(8)	1.264(3)	C(11)Sb(1)C(1)	113.26(9)
O(2)–C(8)	1.220(3)	C(21)Sb(1)O(1)	91.59(9)
O(3)–C(18)	1.264(3)	C(21)Sb(1)O(3)	92.22(9)
O(4)–C(18)	1.215(3)	C(21)Sb(1)C(1)	135.67(9)
Sb(1)···O(2)	3.054(3)	C(21)Sb(1)C(11)	111.07(9)
Sb(1)···O(4)	3.080(3)	C(8)O(1)Sb(1)	115.73(16)
II			
Sb(1)–O(1)	2.121(2)	O(1)Sb(1)O(1)^1	173.98(7)
Sb(1)–O(1)^1	2.121(2)	C(1)^{1}Sb(1)O(1)	91.73(8)
Sb(1)–C(1)	2.098(2)	C(1)Sb(1)O(1)^1	91.73(8)
Sb(1)–C(1)^1	2.098(2)	C(1)^{1}Sb(1)O(1)^1	90.53(8)
Sb(1)–C(7)	2.112(3)	C(1)Sb(1)O(1)	90.53(8)
O(1)–C(17)	1.305(2)	C(1)^{1}Sb(1)C(1)	135.90(12)
O(2)–C(17)	1.213(3)	C(1)^{1}Sb(1)C(7)	112.05(6)
F(2)–C(14)	1.336(3)	C(1)Sb(1)C(7)	112.05(6)
F(1)–C(13)	1.343(4)	C(7)Sb(1)O(1)	86.99(3)
Sb(1)···O(2)	3.033	C(7)Sb(1)O(1)^1	86.99(3)
III			
Sb(1)–O(1)	2.1302(18)	O(3)Sb(1)O(1)	175.98(6)
Sb(1)–O(3)	2.1132(18)	O(3)Sb(1)C(1)	88.16(8)
Sb(1)–C(1)	2.117(3)	C(1)Sb(1)O(1)	88.24(8)
Sb(1)–C(11)	2.111(3)	C(11)Sb(1)O(1)	87.94(8)
Sb(1)–C(21)	2.109(3)	C(11)Sb(1)O(3)	91.73(8)
O(1)–C(37)	1.300(3)	C(11)Sb(1)C(1)	111.70(10)
O(2)–C(37)	1.221(3)	C(21)Sb(1)O(1)	91.26(8)
O(3)–C(47)	1.301(3)	C(21)Sb(1)O(3)	91.52(8)
O(4)–C(47)	1.222(3)	C(21)Sb(1)C(1)	107.40(10)
Sb(1)···O(2)	2.959(3)	C(21)Sb(1)C(11)	140.85(9)
Sb(1)···O(4)	3.054(2)	C(37)O(1)Sb(1)	113.19(13)

* Symmetry transforms: ^{1} $1/2 - x, y, -z$ (**II**).

that in all structures one of the rings arranged in front of the largest equatorial angle is nearly perpendicular to the equatorial plane. The OSbO axial angles in compounds **I**–**III** are 174.47(6)°, 173.98(7)°, and 175.98(6)°, respectively.

The ranges of Sb–C bond length changing are 2.104(2)–2.109(2) (average 2.107(2)) (**I**), 2.098(2)–2.112(3) (2.103(2)) (**II**), and 2.109(3)–2.117(3) Å (2.112(3) Å) (**III**). The Sb–O distances are 2.145(2) and 2.143(2) (**I**), 2.121(2) and 2.121(2) (**II**), and 2.1302(18) and 2.1132(18) Å (**III**). The Sb–C bonds are shorter than the Sb–O distances, which is characteristic of the earlier structurally characterized triarylantimony dicarboxylates [19]. As in other triarylantimony dicarboxylates, the interaction of the central atom with the oxygen atoms of the carbonyl groups takes place in compounds **I**–**III**. The Sb···O(=C) distances are 3.054(3) and 3.080(3) (**I**), 3.033 (**II**), and 2.959(3) and 3.054(2) Å (**III**). It should be mentioned that similar distances in triarylantimony dicarboxylates containing fluorine atoms in the aryl groups are significantly shorter [3, 4, 9–11], which is due to the influence of the electronic effects of the substituent in the aryl ligand. The Sb···O distances in the molecules of compounds **I**–**III** are comparable with similar distances in other structurally characterized triarylantimony dicarboxylates bearing acid residues containing electronegative groups [15–18, 24–26]. The shortening of these distances is observed in the series from compound **I** to **III** due to the enhancement of the –*I* effect in the acidic residues.

The carboxylate ligands in compounds **I**–**III** exhibit the anisobidentate properties, and the degree of asymmetry of ligand coordination, which can be expressed by the ratio of the Sb···O(=C) distances to the Sb–O bond lengths, differs and is equal to 1.424 and 1.437 (**I**), 1.430 (**II**), and 1.389 and 1.445 Å (**III**).

The carboxyl groups have the *cis* conformation relative to the equatorial plane [C_3] and lie in approximately the same plane (the dihedral angles between the planes of the carboxyl groups are 6.85°, 8.17°, and 1.57° in compounds **I**–**III**, respectively). This arrangement of the carboxyl groups leads to an increase in one equatorial CSbC angle from the side of the Sb···O(=C) contacts (135.67(9)° (**I**), 135.90(12)° (**II**), and 140.85(9)° (**III**)), and two other equatorial angles decrease. It should be mentioned that the larger equatorial angle corresponds to the short Sb···O(=C) distances.

The formation of the three-dimensional network in the crystals of compounds **I** and **II** is caused by weak hydrogen bonds of the O···H type involving the carbonyl oxygen atom (2.64, 2.58, and 2.57 (**I**); 2.48 Å (**II**)) and CH···π interactions. The stacking effect between the aromatic rings of the carboxylate ligands (the distance between the planes of the benzene rings is 3.44 Å) is also observed in compound **II**. The CH···π and F···π interactions, intermolecular F···H contacts (2.58, 2.52, and 2.65 Å), and the partial interaction of the π systems of the aromatic rings of the carboxylate ligands are observed in the crystal of compound **III**. The shortest distance between the planes of these π systems is 3.387(4) Å (C(32)···C(42)).

Thus, the reactions of tris(*para*-tolyl)antimony with chloroacetic acid and of triphenylantimony with 3,4,5-trifluoro- and 2,3-difluorobenzoic acids in the presence of *tert*-butyl hydroperoxide afford the corresponding triarylantimony dicarboxylates. According to the XRD data, the coordination polyhedron of the antimony atom is a trigonal bipyramid with the oxygen atoms of the carboxylate ligands in the axial positions. The presence of the intramolecular contacts of the antimony atom with the carbonyl oxygen atom increases one of the equatorial angles. The structural

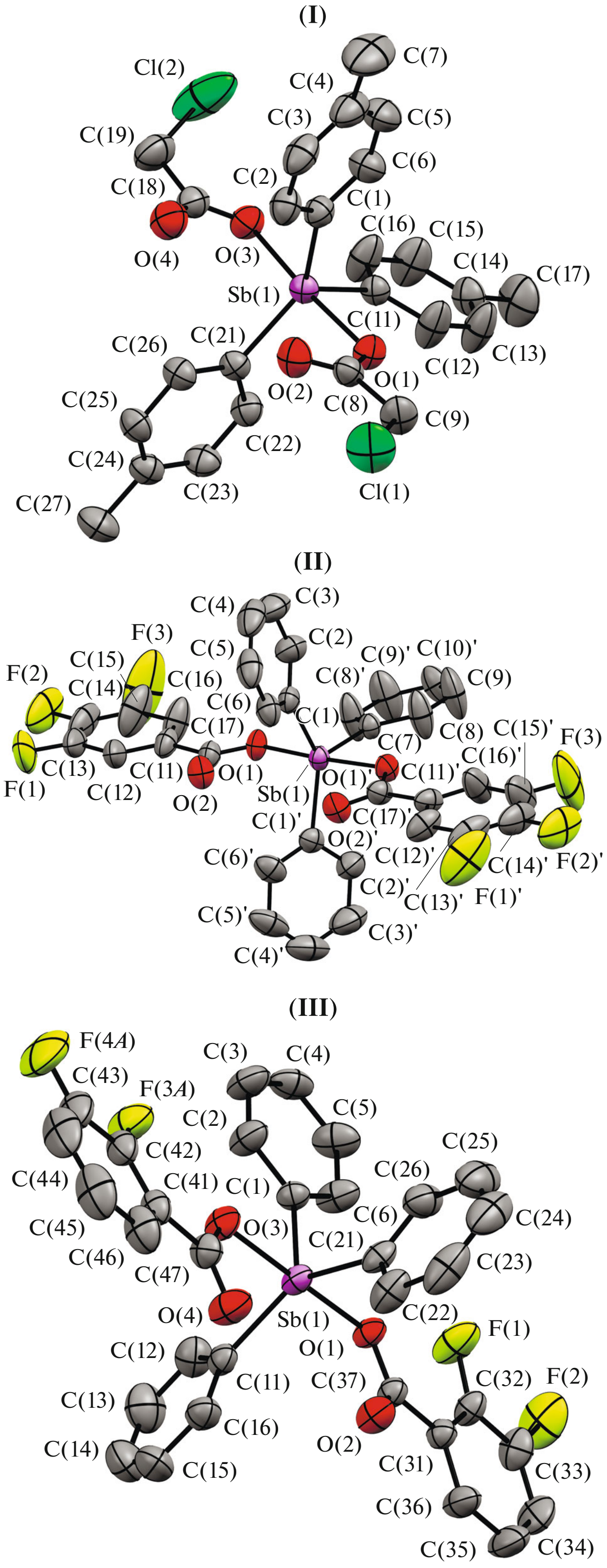


Fig. 1. Structures of compounds **I**–**III** (for compound **III**, disordered F(3*B*) and F(4*B*) are omitted).

organization of the compounds is due to both hydrogen bonds of the O···H (in **I** and **II**) and F···H (in **III**) types and also by the CH···π and F···π interactions (in **III**) and stacking effect (in **II**).

CONFLICT OF INTEREST

The authors declare that they have no conflicts of interest.

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