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Synthesis and Structures of Tris(3-fluorophenyl)antimony Dicarboxylates $(3\text{-}FC_6H_4)_3Sb[OC(O)R]_2$ ($R = CH_2C_6H_4F$-3, $C_6H_3F_2$-2,3, and C_6F_5)

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Abstract—Tris(3-fluorophenyl)antimony dicarboxylates $(3\text{-}FC_6H_4)_3Sb[OC(O)R]_2$ ($R = CH_2C_6H_4F$-3 (**I**), $C_6H_3F_2$-2,3 (**II**), and C_6F_5 (**III**)) are synthesized via oxidative addition from tris(3-fluorophenyl)antimony, carboxylic acid, and *tert*-butyl hydroperoxide in a diethyl ether solution. The structures of the compounds are characterized by IR spectroscopy, ^{1}H, ^{13}C{^{1}H}, and ^{19}F{^{1}H} NMR spectroscopy, elemental analysis, and single-crystal X-ray diffraction (CIF files CCDC nos. 2055807 (**I**), 2055816 (**II**), and 2055817 (**III**)). The crystals of complexes **I**–**III** contain the trigonal bipyramidal molecules with the axially arranged carboxylate ligands.

Keywords: tris(3-fluorophenyl)antimony dicarboxylates, synthesis, structure

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INTRODUCTION

A sufficiently broad series of triarylantimony dicarboxylates was synthesized and studied [1]. Interest in compounds of this type is associated with high possibilities of their application. Some of the indicated derivatives manifest anticancer activity [2–4] and are used for the treatment of leushmaniasis [5–8]. The chemistry of antimony-containing polymers is actively developed. There are published data on the introduction of metallic antimony in the colloidal state into polymethyl methacrylate and polyacrylic acid [9, 10]. The organoantimony derivatives be incorporated as substituents in both the main and side chain of macromolecules [11, 12]. Acrylates, methacrylates, vinyl benzoates, and other antimony derivatives capable of polymerizing have already been used for the synthesis of metal-containing polymers based on methyl methacrylate, styrene, vinyl acetate, and acrylic acid manifesting fungicidal and biocidal activity [13, 14]. Additives of these compounds to polymeric materials improve their thermal stability [15] and X-ray radiation absorption [16]. Note that the properties of triarylantimony dicarboxylates depend on the nature and type of the ligands at the metal atom [1] and, therefore, the synthesis of the dicarboxylates with heteroatoms in the aryl substituents is an urgent task. Triarylantimony dicarboxylates with halogen atoms in the aryl ligands are known [1, 17–20], and among them a few tris(3-fluorophenyl)antimony dicarboxylates can be emphasized [19, 20].

Continuing the studies of the pentavalent antimony compounds, we synthesized tris(3-fluorophenyl)antimony dicarboxylates $[(3\text{-}FC_6H_4)_3Sb][OC(O)R]_2$ ($R = CH_2C_6H_4F$-3 (**I**), $C_6H_3F_2$-2,3 (**II**), and C_6F_5 (**III**)) and studied their structures.

EXPERIMENTAL

Commercial reagents (Alfa Aesar) were used for the syntheses.

Synthesis of bis(3-fluorophenylacetato)tris(3-fluorophenyl)antimony (I). A mixture of tris(3-fluorophenyl)antimony (203 mg, 0.5 mmol), 3-fluorophenylacetic acid (154 mg, 1.0 mmol), and a 70% solution (64 mg) of *tert*-butyl hydroperoxide in diethyl ether (20 mL) was stirred for 1 h. After 18 h the formed crystals were filtered off and dried in air. The yield was 290 mg (81%), $T_m = 99°C$.

For $C_{34}H_{24}F_5O_4Sb$		
Anal. calcd., %	C, 57.22	H, 3.37
Found, %	C, 57.18	H, 3.51

IR (ν, cm^{-1}): 3088, 3065, 1659, 1616, 1585,1522, 1489, 1472, 1450, 1425, 1310, 1287, 1265, 1217, 1163, 1140, 1088, 1053, 997, 962, 924, 893, 870, 853, 783, 766, 721, 679, 658, 633, 584, 555, 538, 523, 486, 440. ^{1}H NMR ($CDCl_3$; δ, ppm) 7.58–7.49 (m, 6H), 7.39 (td, J = 8.0, 5.4 Hz, 3H), 7.25–7.14 (m, 5H), 6.90 (td, J = 8.5, 2.6 Hz, 2H), 6.80 (d, J = 7.6 Hz, 2H), 6.69 (dd, J = 9.8 Hz, 2.0 Hz, 2H), 3.43 (s, 2H). ^{13}C{^{1}H} NMR ($CDCl_3$; δ, ppm): 175.33, 162.77 (d, J = 245.8 Hz), 162.71 (d, J = 251.4 Hz), 139.15 (d, J = 5.5 Hz), 137.10 (d, J = 8.3 Hz), 130.80 (d, J = 7.4 Hz), 129.92 (d, J = 7.9 Hz), 129.31 (d, J = 3.3 Hz), 124.88 (d, J = 2.2 Hz), 120.95 (d, J = 22.8 Hz), 118.79 (d, J = 20.8 Hz), 116.06 (d, J = 21.7 Hz), 113.77 (d, J = 20.8 Hz), 42.39. ^{19}F NMR ($CDCl_3$; δ, ppm): –109.36 m, –113.40 m.

Compounds **II** and **III** were synthesized similarly.

Bis(2,3-difluorobenzoato)tris(3-fluorophenyl)antimony (**II**). The yield was 83%, T_{m} = 183°C.

For $C_{32}H_{18}F_7O_4Sb$

Anal. calcd., %	C, 53.24	H, 2.50
Found, %	C, 53.20	H, 2.58

IR (ν, cm^{-1}): 3102, 3073, 1634, 1587, 1487, 1474, 1425, 1348, 1273, 1215, 1184, 1163, 1151, 1088, 1067, 999, 959, 899, 878, 853, 835, 789, 773, 762, 675, 660, 635, 544, 511, 492, 459, 440. ^{1}H NMR (600 MHz; chloroform-d; δ, ppm) 7.85–7.81 (m, 3H), 7.80 (d, J = 7.8 Hz, 3H), 7.48–7.37 (m, 5H), 7.21–7.10 (m, 5H), 6.95 (tdd, J = 8.0, 4.6, 1.4 Hz, 2H). ^{13}C{^{1}H} NMR (151 MHz; chloroform-d; δ, ppm): 167.43 (t, J = 3.6 Hz), 162.94 (d, J = 251.8 Hz), 151.16 (dd, J = 248.1, 13.5 Hz), 150.49 (dd, J = 260.2, 14.0 Hz), 138.84 (d, J = 5.4 Hz), 131.20 (d, J = 7.5 Hz), 129.58 (d, J = 3.3 Hz), 127.27 (d, J = 3.3 Hz), 124.49–123.36 (m), 122.61 (d, J = 6.6 Hz), 121.28 (d, J = 23.0 Hz), 120.99 (d, J = 17.5 Hz), 119.16 (d, J = 20.7 Hz). ^{19}F NMR (565 MHz; chloroform-d; δ, ppm): –108.93 to –109.03 m, –135.74 (dt, J = 20.6, 6.3 Hz), –137.21 (ddd, J = 21.2, 9.7, 4.1 Hz).

Bis(pentafluorobenzoato)tris(3-fluorophenyl)antimony (**III**). The yield was 79%, T_{m} = 131°C.

For $C_{32}H_{12}O_4F_{13}Sb$

Anal. calcd., %	C, 46.32	H, 1.45
Found, %	C, 46.24	H, 1.48

IR (ν, cm^{-1}): 3102, 3074, 1699, 1682, 1653, 1591, 1522, 1499, 1476, 1427, 1333, 1252, 1217,1167, 1105, 1090, 1059, 995, 926, 870, 856, 822, 787, 748, 698, 677, 660, 623, 582, 523, 492, 440. ^{1}H NMR (chloroform-d; δ, ppm): 7.79–7.70 (m, 6H), 7.49 (td, J = 8.0, 5.4 Hz, 3H), 7.21 (ddd, J = 8.3, 2.5, 1.0 Hz, 3H). ^{13}C{^{1}H} NMR (151 MHz; chloroform-d; δ, ppm): 163.07 (d, J = 252.9 Hz), 162.18, 145.16 (dtd, J = 255.2, 7.6, 3.9 Hz), 142.57 (dm, J = 259.7 Hz), 137.72 (dddd, J = 253.0, 17.4, 12.8, 5.5 Hz), 136.56 (d, J = 5.9 Hz), 131.63 (d, J = 7.2 Hz), 129.52 (d, J = 3.9 Hz), 128.47, 121.13 (d, J = 23.7 Hz), 119.86 (d, J = 20.8 Hz), 110.18 (td, J = 17.0, 3.9 Hz). ^{19}F{^{1}H} NMR ($CDCl_3$; δ, ppm): –104.82 to –114.20 m, –139.75 to 139.81 m, –150.43 (t, J = 20.6 Hz), –160.72 (td, J = 20.2, 6.1 Hz).

IR spectra were recorded on a Shimadzu IR Affinity-1S FT-IR spectrometer in KBr pellets in a range of 4000–400 cm^{-1}. ^{1}H (600 MHz), ^{13}C (151 MHz), and ^{19}F (565 MHz) NMR spectra were recorded for solutions of the compounds in $CDCl_3$ on a Bruker AVANCE NEO 600 MHz NMR spectrometer equipped with a Prodigy Cryoprobe accessory. Signals of the solvent served as the internal standard for ^{1}H (7.26 ppm) and ^{13}C (77.16 ppm) NMR spectra, and $CFCl_3$ was used as the external standard for ^{19}F NMR spectra.

Elemental analyses to C and H were carried out on a Carlo Erba CHNS-O EA 1108 elemental analyzer. Melting points were measured on a Netzsch 449C Jupiter synchronous thermoanalyzer.

X-ray diffraction (XRD). The structures of single crystals of compounds **I**–**III** were studied on a Bruker D8 QUEST diffractometer (MoK_α radiation, λ = 0.71073 Å, graphite monochromator). Data were collected and edited, unit cell parameters were refined, and an absorption correction was applied using the SMART and SAINT-Plus programs [21]. All calculations were performed using the SHELXTL/PC [22] and OLEX2 [23] programs. The structures were solved by a direct method and refined by least squares in the anisotropic approximation for non-hydrogen atoms. The parameters of the crystals, data collection, and refinement details for compounds **I**–**III** are given in Table 1.

The full tables of atomic coordinates, bond lengths, and bond angles were deposited with the Cambridge Crystallographic Data Centre (CIF files CCDC nos. 2055807 (**I**), 2055816 (**II**), and 2055817 (**III**); deposit@ccdc.cam.ac.uk or http://www.ccdc.cam.ac.uk).

RESULTS AND DISCUSSION

It is known that the oxidation of triarylantimony by *tert*-butyl hydroperoxide in the presence of carboxylic acids affords triarylantimony dicarboxylates of the general formula $Ar_3Sb[OC(O)R)]_2$ [24–27]. We found that the reactions of tris(3-fluorophenyl)antimony with such carboxylic acids as 3-fluorophenylacetic, 2,3-difluorobenzoic, and pentafluorobenzoic acids in the presence of *tert*-butyl hydroperoxide (molar ratio 1 : 2 : 1) occurred in ether to form tris(3-fluorophenyl)antimony dicarboxylates isolated in the yield up to 83%.

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

Parameter	Value		
	I	**II**	**III**
FW	713.28	721.21	829.17
Crystal system	Monoclinic	Triclinic	Monoclinic
Space group	$C2/c$	$P\bar{1}$	$C2/c$
a, Å	18.813(12)	8.751(7)	26.308(15)
b, Å	9.851(6)	11.126(8)	13.078(10)
c, Å	16.259(11)	15.367(16)	19.015(11)
α, deg	90	99.40(3)	90
β, deg	97.05(3)	95.22(3)	111.121(14)
γ, deg	90	93.21(2)	90
V, Å^3	2990(3)	1466(2)	6103(7)
Z	4	2	8
ρ_{calc}, g/cm^3	1.584	1.634	1.805
μ, mm^{-1}	0.992	1.022	1.022
F(000)	1424.0	712.0	3232.0
Crystal size, mm	0.48 × 0.24 × 0.2	0.65 × 0.46 × 0.45	0.48 × 0.36 × 0.17
2θ, deg	6.256–56.994	5.77–57	5.88–59.26
Ranges of reflection indices	$-25 \le h \le 25$, $-13 \le k \le 13$, $-21 \le l \le 21$	$-11 \le h \le 11$, $-14 \le k \le 14$, $-20 \le l \le 20$	$-36 \le h \le 33$, $-18 \le k \le 18$, $-26 \le l \le 26$
All reflections	37362	60514	138680
Independent reflections (R_{int})	3783 (0.0275)	7436 (0.0327)	8583 (0.0381)
Number of refined parameters	210	401	451
GOOF	1.099	1.157	1.050
R factors for $F^2 > 2\sigma(F^2)$	$R_1 = 0.0293$, $wR_2 = 0.0882$	$R_1 = 0.0367$, $wR_2 = 0.0872$	$R_1 = 0.0307$, $wR_2 = 0.0737$
R factors for all reflections	$R_1 = 0.0306$, $wR_2 = 0.0903$	$R_1 = 0.0416$, $wR_2 = 0.0902$	$R_1 = 0.0458$, $wR_2 = 0.0838$
Residual electron density (max/min), e/Å^{-3}	1.71/–0.63	1.77/–0.99	0.74/–0.69

$$(3\text{-}FC_6H_4)_3Sb + 2HOC(O)R + t\text{-}BuOOH$$
$$\rightarrow (3\text{-}FC_6H_4)_3Sb[OC(O)R)]_2 + t\text{-}BuOH + H_2O,$$
$$R = CH_2C_6H_4F\text{-}3\ (\mathbf{I}),\ \ C_6H_3F_2\text{-}2,3\ (\mathbf{II}),\ \ C_6F_5\ (\mathbf{III}).$$

According to the XRD data, in compounds **I–III** the Sb atoms have a distorted trigonal bipyramidal coordination with the oxygen atoms of the carboxylate ligands in the axial positions and with the aryl substituents in the equatorial plane (Figs. 1–3).

The axial OSbO angles are 175.96(9)°, 174.63(8)°, and 171.32(7)° in compounds **I**, **II**, and **III**, respectively. The Sb–C bond lengths in compounds **I–III** vary in a range of 2.094(2)–2.123(3) Å, and the Sb–O distances (2.099(2)–2.128(2) Å) are somewhat lower than the sum of covalent radii of antimony and oxygen atoms (2.14 Å [28]). The sums of the CSbC angles in the equatorial plane of molecules **I–III** are 360°. The planar aryl rings in the structures are turned around the Sb–C bonds in such a way that intra- and intermolecular contacts would be reduced to minimum. Molecules **I–III** contain intramolecular contacts Sb···O(=C) that change in a range of 2.862(9)–3.381(3) Å. The dihedral angles between the planes of the carboxyl groups in compounds **I** and **II** are 13.93° and 2.62°, and the carboxylate ligands have a *cis* orientation relative to the SbC_3 fragment. This is characteristic of the most part of triorganylantimony dicarboxylates [29]. Dihedral angles arranged in such a way that the Sb···O(=C) intramolecular contacts would be formed inside one equatorial angle, the value of which increases to 138.30(14)° and 144.20(12)°, respectively, with a decrease in two other equatorial angles. In molecules **III**, the dihedral angle between

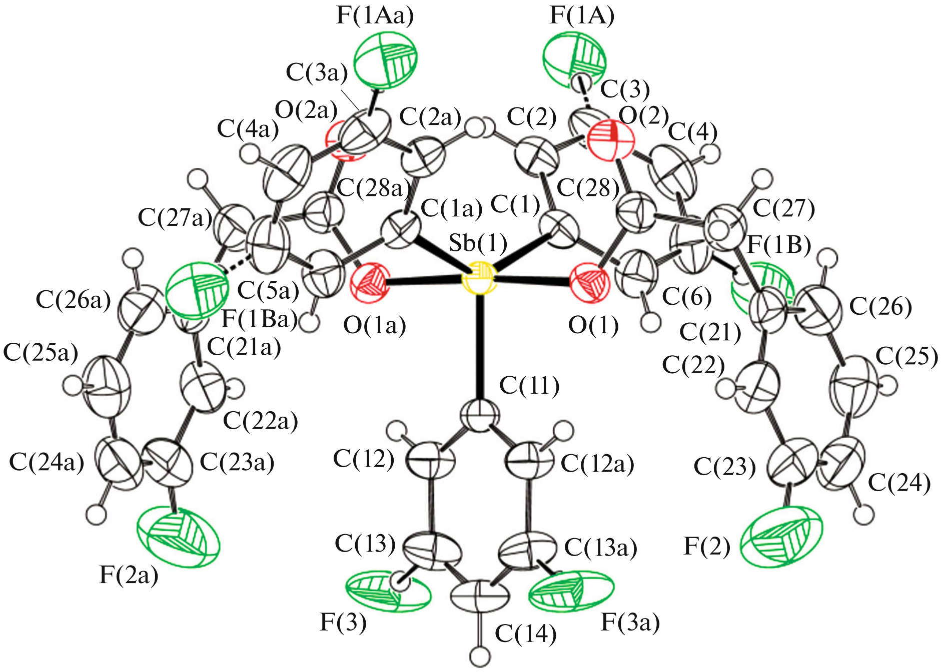

Fig. 1. Structure of compound **I**.

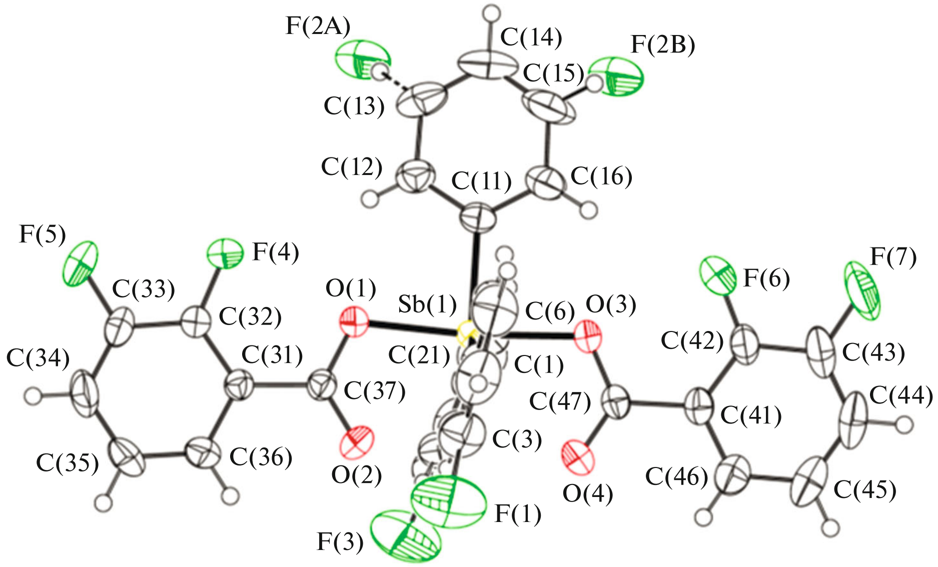

Fig. 2. Structure of compound **II**.

the planes of the carboxyl groups is unusually high (76.27°) due to which the carbonyl oxygen atoms are opposite to different equatorial angles and the CSbC angles (111.99(9)°, 117.56(10)°, 130.37(9)°) vary in the range usual for compounds of the general formula Ar_3SbX_2 (120° ± 10° [30]).

It can be concluded from a comparative analysis of the XRD data for tris(3-fluorophenyl)antimony dicarboxylates (Table 2) that the strengthening of intramolecular contacts in the $(3\text{-}FC_6H_4)_3Sb[OC(O)R]_2$ molecules leads to an increase in one of the equatorial angles (from the side of the intramolecular contacts) and, on the contrary, the differences in the equatorial angles are less appreciable at the lowest intramolecular contacts.

The IR spectra of compounds **I**–**III** exhibit an intense absorption band of stretching vibrations of the SbC_3 fragment at ~440 cm^{-1}. The intense bands at

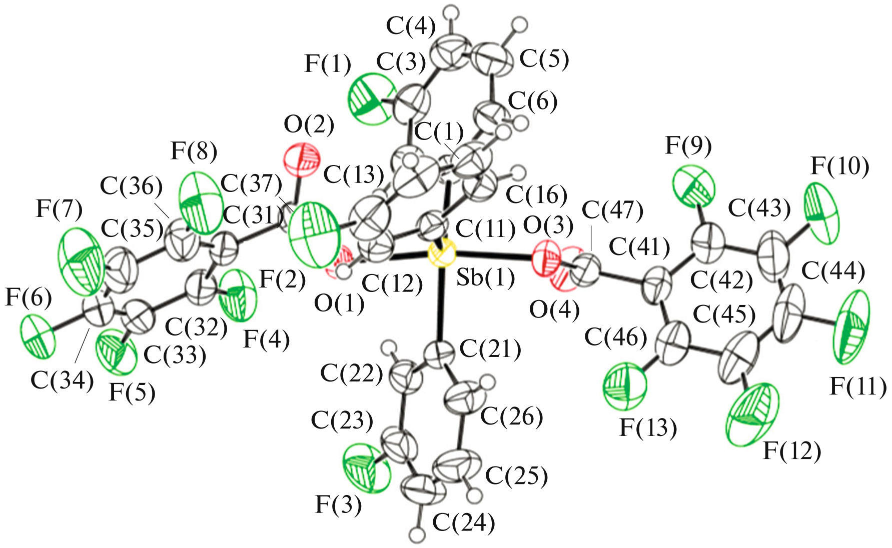

Fig. 3. Structure of compound **III**.

1659 (**I**), 1634 (**II**), and 1682 cm^{-1} (**III**) originate from stretching vibrations of the carbonyl C=O groups. The absorption bands at 1585, 1471, and 1425 cm^{-1} (**I**); 1587, 1473, and 1425 cm^{-1} (**II**); and 1591, 1476, and 1427 cm^{-1} (**III**) characterize stretching vibrations of the carbon skeleton of the aryl rings. The absorption bands of medium intensity at 3088 (**I**), 3102 (**II**), and 3102 (**III**) cm^{-1} correspond to stretching vibrations of the C_{Ar}–H bonds, and the intense bands at 766, 762, and 748 cm^{-1} correspond to out-of-plane bending vibrations of the same bonds. The IR spectra of compounds **I**, **II**, and **III** contain absorption bands of the C–F stretching vibrations at 1217, 1215, and 1252 cm^{-1}. Since the molecules of the complexes are symmetric, the signals in all ^{1}H, ^{13}C, and ^{19}F NMR spectra are isochronous. The fluorine atoms induce an additional signal splitting in the ^{1}H NMR spectra and characteristic splitting of a series of signals in the ^{13}C NMR spectra with the appearance of typical C–F spin-spin coupling constants.

Thus, different numbers of electronegative fluorine atoms in the organic radical of carboxylic acid exert no effect on the scheme of the oxidative addition involving tris(3-fluorophenyl)antimony. The geometric characteristics of the synthesized tris(3-fluorophenyl)antimony dicarboxylates (Sb–C and Sb–O bond lengths, intramolecular Sb···O contacts, and bond angles) are close to each other for the same coordination polyhedron and coordination number of the antimony atom.

Table 2. Selected geometric parameters of $(3\text{-}FC_6H_4)_3Sb[OC(O)R]_2$ molecules

R	Bond, Å			Angle, deg	
	Sb···O=C (average)	Sb–O (average)	Sb–C (average)	OSbO	CSbC (max.)
CH=CHPh	2.601	2.142	2.116	175.81	152.12 [19]
CH_2Br	2.872	2.119	2.105	175.92	143.09 [19]
C_6F_4H-2	2.878	2.131	2.114	175.72	142.47 [20]
$C_6H_3F_2$-2,3 (**II**)	2.906	2.120	2.122	174.63	144.20
$C_6H_3(NO_2)_2$-3,5	2.931	2.127	2.111	174.70	141.08 [19]
$CH_2C_6H_4F$-3 (**I**)	3.122	2.099	2.117	175.96	138.30
C_6F_5 (**III**)	3.246	2.115	2.098	171.32	130.37

CONFLICT OF INTEREST

The authors declare that they have no conflicts of interest.

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