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Synthesis and Structures of Tri(*meta*-tolyl)antimony Derivatives $(3\text{-}MeC_6H_4)_3Sb[OC(O)R]_2$ (R = $CH_2C_6H_4F$-3, CH=CHPh, C≡CPh) and $[(3\text{-}MeC_6H_4)_3SbOSO_2CF_3]_2O$

V. V. Sharutin[a], * and O. K. Sharutina[a]

[a] *South Ural State University (National Research University), Chelyabinsk, 454080 Russia*

**e-mail: vvsharutin@rambler.ru*

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Abstract—The reactions of tri(*meta*-tolyl)antimony with 3-fluorophenylchloroacetic, cinnamic, and phenylpropiolic acids in the presence of *tert*-butyl hydroperoxide (molar ratio 1 : 2 : 1) in diethyl ether afford tri(*meta*-tolyl)antimony dicarboxylates $(3\text{-}MeC_6H_4)_3Sb[OC(O)CH_2C_6H_4F\text{-}3]_2$ (**I**), $(3\text{-}MeC_6H_4)_3Sb[OC(O)CH{=}CHPh]_2$ (**II**), and $(3\text{-}MeC_6H_4)_3Sb[OC(O)C{\equiv}CPh]_2$ (**III**), respectively. Under similar conditions, the reaction of tri(*meta*-tolyl)antimony with trifluoromethanesulfonic acid gives μ-oxobis[tri(*meta*-tolyl)(trifluoromethanesulfonato)antimony] $[(3\text{-}MeC_6H_4)_3SbOSO_2CF_3]_2O$ (**IV**). The Sb atoms in compounds **I**–**IV** have the coordination of a distorted trigonal bipyramid with the electronegative ligands in the axial positions.

Keywords: bis(3-fluorophenyl acetate), dicinnamate, bis(phenyl propiolate), tri(*meta*-tolyl)antimony, μ-oxobis[tri(*meta*-tolyl)(trifluoromethanesulfonato)antimony], synthesis, diffraction studies

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INTRODUCTION

It is known that triarylantimony dicarboxylates can exert antitumor, antileishmanial, and antibacterial effects and possess the electrochemical, photoluminescence, and photocatalytic properties [1–16]. Triphenyl- and *p*-tolylantimony dicarboxylates have been studied in most detail to the present time [17]. The *ortho*- and *meta*-tolyl antimony derivatives are known to a lower extent [18, 19]. New derivatives of tri(*meta*-tolyl)antimony were synthesized in this work, and their structural features were revealed.

EXPERIMENTAL

3-Fluorophenylacetic, cinnamic, phenylpropiolic, and trifluoromethanesulfonic acids and *tert*-butyl hydroperoxide (Alfa Aesar) were used. Tri(*meta*-tolyl)antimony was synthesized using a described procedure [17]. Prior to the synthesis, the solvents (reagent grade) were dried over calcium chloride and distilled.

Bis(3-fluorophenylacetato)tri(*meta*-tolyl)antimony $(3\text{-}MeC_6H_4)_3Sb[OC(O)CH_2C_6H_4F\text{-}3]_2$ (**I**) was synthesized using a described procedure [20]. The yield of the colorless crystals was 91%, T_m = 63°C. IR (ν, cm^{-1}): 3084 w, 3055 w, 1662 vs, 1614 m, 1591 s, 1487 s, 1471 m, 1448 m, 1292 vs, 1276 m, 1265 w, 1244 s, 1139 m, 1099 m, 1074 w, 1045 w, 991 m, 964 s, 924 s, 889 w, 868 m, 829 w, 783 s, 769 vs, 721 s, 686 s, 638 m, 580 m, 549 w, 520 m, 509 w, 472 w, 439 w, 424 s.

For $C_{38}H_{35}O_4F_2Sb$		
Anal. calcd., %	C, 63.78	H, 4.90
Found, %	C, 63.65	H, 4.98

Compounds **II**–**IV** were synthesized using a similar procedure.

Compound $(3\text{-}MeC_6H_4)_3Sb[OC(O)CH{=}CHPh]_2 \cdot PhH$ (**II**·PhH): colorless transparent crystals, 84% yield, T_m = 140°C. IR (ν, cm^{-1}): 3055 m, 3024 m, 2914 w, 1641 vs, 1587 vs, 1477 s, 1449 s, 1337 vs, 1288 s, 1215 w, 1202 m, 1101 s, 1070 w, 1036 w, 988 m, 974 s, 885 w, 872 m, 850 w, 773 vs, 739 s, 716 w, 683 vs, 590 s, 548 m, 507 m, 486 m, 426 s, 410 w. The crystals of **II**·PhH suitable for X-ray diffraction (XRD) were obtained by the recrystallization of compound **II** from a benzene–octane (3 : 1 vol/vol) mixture.

For $C_{45}H_{41}O_4Sb$		
Anal. calcd., %	C, 70.31	H, 5.34
Found, %	C, 70.22	H, 5.47

Compound $(3\text{-}MeC_6H_4)_3Sb[OC(O)C{\equiv}CPh]_2$ (**III**): colorless transparent crystals, 88% yield, T_{decomp} = 152°C. IR (ν, cm^{-1}): 3051 w, 2924 w, 2254 w, 2210 s,

1625 vs, 1614 s, 1595 s, 1491 s, 1473 s, 1445 s, 1398 w, 1319/1313 vs, 1215 vs, 1175 m, 1123 w, 1099 m, 1072 w, 1026 w, 991 m, 939 m, 920 w, 885 w, 829 w, 779 s, 767/759 vs, 687 s, 615 s, 534 m, 511 m, 426 s.

For $C_{39}H_{31}O_4Sb$		
Anal. calcd., %	C, 68.32	H, 4.53
Found, %	C, 68.26	H, 4.65

Compound $[(3\text{-}MeC_6H_4)_3SbOSO_2CF_3]_2O$ (**IV**): colorless transparent crystals, 38% yield, T_{decomp} = 110°C. IR (ν, cm^{-1}): 3051 w, 2924 m, 2854 w, 1591 m, 1570 w, 1473 s, 1450 w, 1408 w, 1383 w, 1328 vs, 1253 m, 1234 vs, 1199 vs, 1174/1166 s, 1097 m, 1034 s, 1005 vs, 875 w, 827 w, 770 vs, 760 s, 685 s, 633 vs, 579 m, 516 s, 503 w, 424 s.

For $C_{44}H_{42}O_7F_6S_2Sb_2$		
Anal. calcd., %	C, 47.83	H, 3.80
Found, %	C, 47.69	H, 3.95

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

XRD of the crystals of compounds **I**, **II**·PhH, **III**, and **IV** was carried out on a D8 QUEST automated four-circle diffractometer (Bruker) (MoK_α radiation, λ = 0.71073 Å, graphite monochromator). The data were collected and edited, the unit cell parameters were refined, and an absorption correction was applied using the SMART and SAINT-Plus programs [21]. All calculations on structure determination and refinement were performed using the SHELXL/PC [22] and OLEX2 [23] programs. The structures of compounds **I**–**IV** were determined by a direct method and refined by least squares in the anisotropic approximation for nonhydrogen atoms. The main crystallographic data and structure refinement results for compounds **I**, **II**·PhH, **III**, and **IV** are given in Table 1. Selected bond lengths and bond angles are listed in Table 2.

The full tables of the atomic coordinates, bond lengths, and bond angles were deposited with the Cambridge Crystallographic Data Centre (CIF file CCDC nos. 2049792 (**I**), 2050081 (**II**·PhH), 2049694 (**III**), and 2050999 (**IV**)); deposit@ccdc.cam.ac.uk or http://www.ccdc.cam.ac.uk/data_request/cif).

RESULTS AND DISCUSSION

It is known that the oxidation of triarylantimony with hydrogen peroxide or *tert*-butyl hydroperoxide in the presence of carboxylic acids results in the synthesis of triarylantimony dicarboxylates of the general formula $Ar_3Sb[OC(O)R]_2$ [16–20].

We found that the reactions of tri(*meta*-tolyl)antimony with 3-fluorophenylchloroacetic, cinnamic, and phenylpropiolic acids in the presence of *tert*-butyl hydroperoxide in a molar ratio of 1 : 2 : 1 in diethyl ether proceeded via the oxidative addition reaction to form tri(*meta*-tolyl)antimony dicarboxylates according to the reaction

$$(3\text{-}MeC_6H_4)_3Sb + 2HOC(O)R + t\text{-}BuOOH \rightarrow (3\text{-}MeC_6H_4)_3Sb[OC(O)R]_2 + t\text{-}BuOH + H_2O, \quad \textbf{(III)}$$

$$R = CH_2C_6H_4F\text{-}3\ (\textbf{I}),\ CH=CHPh\ (\textbf{II}),\ C\equiv CPh.$$

A similar reaction with trifluoromethanesulfonic acid afforded μ-oxobis[tri(*meta*-tolyl)(trifluoromethanesulfonato)antimony] (**IV**) in a yield of 38%.

$$2(3\text{-}MeC_6H_4)_3Sb + 2HOSO_2CF_3 + 2t\text{-}BuOOH \rightarrow [(3\text{-}MeC_6H_4)_3SbOSO_2CF_3]_2O + 2t\text{-}BuOH + H_2O. \quad \textbf{(IV)}$$

A change in the molar ratio of the starting reactants (1 : 1 : 1 instead of 1 : 2 : 1) was found to be accompanied by an increase in the yield of the target product to 80%. Evidently, the formation of the antimony compound of the bridged type can be explained by steric hindrances caused by the bulky trifluoromethanesulfonate substituents at the metal atom.

Compounds **I**–**IV** are colorless crystalline substances resistant to air moisture and oxygen, having distinct melting temperatures, and highly soluble in aromatic hydrocarbons and polar organic solvents.

The IR spectra of compounds **I**, **II**, **III**, and **IV** exhibit intense bands at 424, 426, 426, and 424 (Sb–C); 1487, 1477, 1491, and 1474 (Ar); 2924, 2914, 2924, and 2924 (H–C_{Alk}); and 3055, 3055, 3051, and 3051 cm^{-1} (H–C_{Ar}), respectively. The range of stretching vibrations of carbonyl groups in the spectra of compounds **I**, **II**, and **III** contains bands at 1663, 1641, and 1626 cm^{-1}, respectively. The IR spectra of

Table 1. Crystallographic data and experimental and structure refinement parameters for compounds **I**, **II**·PhH, **III**, and **IV**

Parameter	Value			
	I	**II**·PhH	**III**	**IV**
FW	715.41	767.53	685.39	1104.40
Crystal system	Monoclinic	Triclinic	Triclinic	Monoclinic
Space group	$C2/c$	$P\bar{1}$	$P\bar{1}$	$P2_1/c$
a, Å	19.728(19)	9.129(3)	10.593(17)	12.624(8)
b, Å	10.271(7)	10.762(5)	12.75(3)	14.937(9)
c, Å	16.106(11)	20.376(7)	13.56(3)	24.837(15)
α, deg	90.00	100.25(2)	65.29(10)	90.00
β, deg	96.22(3)	98.031(13)	73.95(7)	90.23(2)
γ, deg	90.00	94.178(19)	81.20(7)	90.00
V, Å^3	3244(4)	1940.7(12)	1598(6)	4683(5)
Z	4	2	2	4
ρ_{calc}, g/cm^3	1.465	1.313	1.425	1.566
μ, mm^{-1}	0.901	0.752	0.903	1.313
F(000)	1456.0	788.0	696.0	2200.0
Crystal size, mm	0.64 × 0.3 × 0.15	0.38 × 0.32 × 0.18	0.48 × 0.29 × 0.09	0.45 × 0.23 × 0.08
Range of data collection over 2θ, deg	6.22–55.84	5.62–54.3	5.2–58.9	5.7–49.5
Ranges of reflection indices	$-25 \le h \le 25$, $-13 \le k \le 13$, $-21 \le l \le 20$	$-11 \le h \le 11$, $-13 \le k \le 13$, $-26 \le l \le 26$	$-14 \le h \le 13$, $-16 \le k \le 16$, $-18 \le l \le 18$	$-14 \le h \le 14$, $-17 \le k \le 17$, $-29 \le l \le 29$
Measured reflections	20531	47743	32614	100440
Independent reflections (R_{int})	3866 (0.0302)	8578 (0.0268)	6971 (0.0629)	7981 (0.0576)
Reflections with $I > 2\sigma(I)$	3080	7939	5605	6334
Refinement variables	207	454	400	556
GOOF	1.082	1.091	1.341	1.043
R factors for $F^2 > 2\sigma(F^2)$	$R_1 = 0.0471$, $wR_2 = 0.1248$	$R_1 = 0.0226$, $wR_2 = 0.0541$	$R_1 = 0.0945$, $wR_2 = 0.2654$	$R_1 = 0.0386$, $wR_2 = 0.0937$
R factors for all reflections	$R_1 = 0.0615$, $wR_2 = 0.1382$	$R_1 = 0.0260$, $wR_2 = 0.0561$	$R_1 = 0.1314$, $wR_2 = 0.3108$	$R_1 = 0.0555$, $wR_2 = 0.1054$
Residual electron density (max/min), e/Å^3	1.16/−1.74	0.27/−0.52	2.18/−2.50	1.91/−0.87

compounds **III** and **IV** exhibit bands (2210 and 1234 cm^{-1}) that can be attributed to vibrations of the C≡C and S=O groups, respectively.

According to the XRD data, the Sb atoms in the molecules of compounds **I**–**III** have the coordination of a distorted trigonal bipyramid with the oxygen atoms of the carboxyl ligands in the axial positions (Figs. 1–3).

In the V-shaped binuclear molecule of compound **IV** (SbOSb angle 138.84(18)°), the metal atoms have the trigonal bipyramidal environment with the alkanesulfonate ligand and bridging oxygen atom in the axial positions (Fig. 4).

The sums of the CSbC bond angles in the equatorial plane are 359.99° (**I**); 359.97° (**II**); 359.9° (**III**); and 357.7°, 356.55° (**IV**). The Sb atoms in compound **I** lie on the C_3 equatorial plane and deviate from it by 0.02 Å to the O(1) atom in **II**; by 0.027 Å to the O(3) atom in **III**; and by 0.188, 0.226 Å to the bridging O(1) atom in **IV**. The OSbC angles differ from the theoretical values by at most 4°: 87.84(7)°–91.76(15)° (**I**), 87.66(6)°–91.69(6)° (**II**), and 88.7(3)°–92.7(3)° (**III**). Similar deviations in compound **IV** are significantly higher (80.52(17)°–98.42(17)°). The OSbO axial angles (175.68(14)° in **I**; 175.84(4)° in **II**; 177.73(19)° in **III**; and 178.48(14)°, 174.17(13)° in **IV**) somewhat differ from an ideal value of 180°. The Sb–C bond

Table 2. Selected bond lengths and bond angles in the structures of compounds **I**, **II**·PhH, **III**, and **IV***

Bond	*d*, Å	Angle	ω, deg
		I	
Sb(1)−O(1)	2.107(4)	O(1)Sb(1)O(1)$^{\#}$	175.68(14)
Sb(1)−O(1)$^{\#}$	2.107(4)	O(1)Sb(1)C(1)	91.76(15)
Sb(1)−C(1)	2.112(3)	O(1)Sb(1)C(1)$^{\#}$	89.80(14)
Sb(1)−C(1)$^{\#}$	2.112(3)	O(1)1Sb(1)C(1)	89.79(14)
Sb(1)−C(11)	2.119(5)	O(1)Sb(1)C(11)	87.84(7)
O(1)−C(28)	1.303(5)	C(1)1Sb(1)C(1)	137.87(19)
O(2)−C(28)	1.209(6)	C(1)Sb(1)C(11)	111.06(9)
C(23)−F(1)	1.331(8)	C(1)1Sb(1)C(11)	111.06(9)
Sb(1)···O(1)	3.169(5)		
		II·PhH	
Sb(1)−C(1)	2.1249(18)	C(1)Sb(1)O(1)	87.66(6)
Sb(1)−O(1)	2.1389(12)	C(11)Sb(1)C(1)	107.20(7)
Sb(1)−C(11)	2.1112(17)	C(11)Sb(1)O(1)	91.69(6)
Sb(1)−O(3)	2.1137(12)	C(11)Sb(1)O(3)	89.60(6)
Sb(1)−C(21)	2.1116(18)	C(11)Sb(1)C(21)	146.87(8)
O(1)−C(39)	1.311(2)	O(3)Sb(1)C(1)	88.18(6)
O(2)−C(39)	1.229(2)	O(3)Sb(1)O(1)	175.84(4)
O(3)−C(49)	1.307(2)	C(21)Sb(1)C(1)	105.90(8)
O(4)−C(49)	1.229(2)	C(21)Sb(1)O(1)	91.07(6)
Sb(1)···O(2)	2.790(1)	C(21)Sb(1)O(3)	90.01(6)
Sb(1)···O(4)	2.879(1)	C(39)O(1)Sb(1)	107.96(10)
		III	
Sb(1)−C(1)	2.112(9)	C(1)Sb(1)O(3)	90.0(3)
Sb(1)−C(21)	2.099(8)	C(1)Sb(1)O(1)	89.9(3)
Sb(1)−C(11)	2.095(10)	C(21)Sb(1)C(1)	108.2(3)
Sb(1)−O(3)	2.129(7)	C(21)Sb(1)C(11)	108.5(4)
Sb(1)−O(1)	2.120(7)	C(21)Sb(1)O(3)	89.0(3)
O(3)−C(49)	1.309(10)	C(21)Sb(1)O(1)	88.9(3)
O(1)−C(39)	1.275(10)	C(11)Sb(1)C(1)	143.2(3)
O(2)−C(39)	1.232(11)	C(11)Sb(1)O(3)	88.7(3)
O(4)−C(49)	1.227(10)	C(11)Sb(1)O(1)	92.7(3)
Sb(1)···O(2)	2.990(9)	O(1)Sb(1)O(3)	177.73(19)
Sb(1)···O(4)	2.872(9)		
		IV	
Sb(1)−O(1)	1.946(3)	O(1)Sb(1)O(5)	178.48(14)
Sb(1)−O(5)	2.368(3)	O(1)Sb(1)C(21)	92.46(16)
Sb(1)−C(21)	2.094(5)	O(1)Sb(1)C(11)	98.42(17)
Sb(1)−C(1)	2.116(5)	C(21)Sb(1)O(5)	87.05(16)
Sb(1)−C(11)	2.099(5)	C(21)Sb(1)C(1)	119.8(2)
Sb(2)−O(1)	1.950(3)	C(21)Sb(1)C(11)	115.8(2)
Sb(2)−O(2)	2.396(3)	C(1)Sb(1)O(5)	87.01(18)
Sb(2)−C(31)	2.091(5)	C(11)Sb(1)O(5)	80.52(17)
Sb(2)−C(51)	2.108(5)	C(11)Sb(1)C(1)	122.1(2)

Table 2. (Contd.)

Bond	*d*, Å	Angle	ω, deg
Sb(2)–C(41)	2.084(5)	O(1)Sb(2)O(2)	174.17(13)
S(1)–O(2)	1.435(4)	Sb(1)O(1) Sb(2)	138.84(18)
S(1)–O(3)	1.508(6)	O(1)Sb(2)C(51)	95.01(17)
S(1)–O(4)	1.379(5)	O(1)Sb(2)C(41)	100.12(16)
S(1)–C(38)	1.772(8)	C(31)Sb(2)O(2)	81.97(16)
S(2)–O(5)	1.450(4)	C(31)Sb(2)C(51)	120.76(18)
S(2)–O(6)	1.428(5)	C(41)Sb(2)O(2)	85.31(15)
S(2)–O(7)	1.422(4)	C(41)Sb(2)C(31)	117.83(18)
S(2)–C(8)	1.808(10)	C(41)Sb(2)C(51)	117.96(19)
F(2)–C(38)	1.395(12)	O(2)S(1)O(3)	110.4(3)

* Symmetry transforms: # $-1-x, y, 3/2-z$ (**I**).

lengths have close values: 2.107(4)–2.119(5) Å in **I**, 2.1112(17)–2.1249(18) Å in **II**, 2.095(10)–2.112(9) Å in **III**, and 2.091(5)–2.116(5) Å in **IV**. The Sb–O distances (2.107(4) Å in **I**; 2.1137(12), 2.1389(12) Å in **II**; and 2.120(7), 2.129(7) Å in **III**) somewhat exceed the sum of covalent radii of antimony and oxygen atoms (2.05 Å [20]), and the Sb–O(1) bonds (1.946(3), 1.950(3) Å) in **IV** are considerably shorter than the terminal Sb–O bonds (2.368(3), 2.396(3) Å) like in similar binuclear compounds with the linear structure of the central fragment [24–26].

In compounds **I**–**III**, the carboxylate ligands have a pronounced anisobidentate character of binding. The intramolecular Sb···O(=C) distances are 3.169(5) Å in **I**; 2.790(1), 2.879(1) Å in **II**; and 2.872(9), 2.990(9) Å in **III**, which is lower than the sum of the van der Waals radii of antimony and oxygen atoms (3.58 Å [27]) and approaches similar values observed in triphenylantimony dicarboxylates bearing the electron-withdrawing substituents in the carboxylate ligands (e.g., 3.055 Å in $Ph_3Sb[OC(O)C_6F_5)]_2$ [28]). In the carboxylate groups of the molecules of

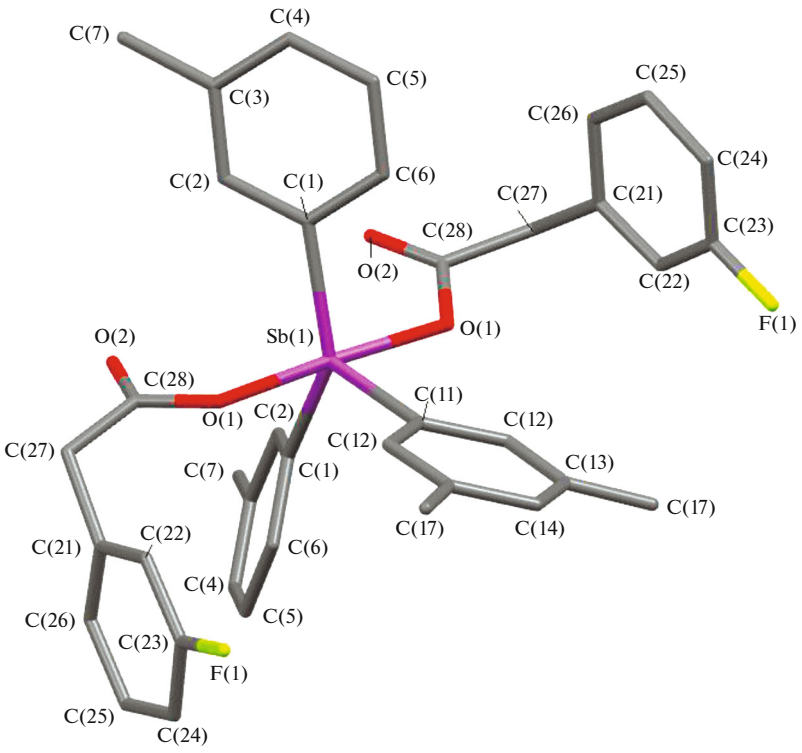

Fig. 1. Structure of the molecule of compound **I** (hydrogen atoms are omitted).

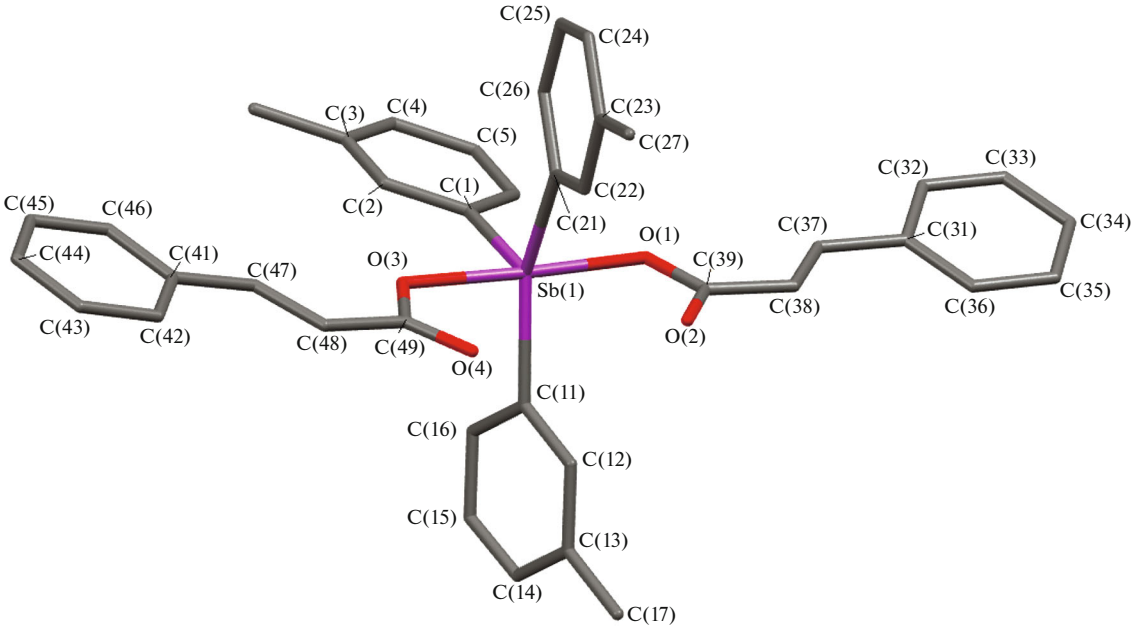

Fig. 2. Structure of the molecule of compound **II** in the crystals of compound **II**·PhH (hydrogen atoms and solvate benzene molecule are omitted).

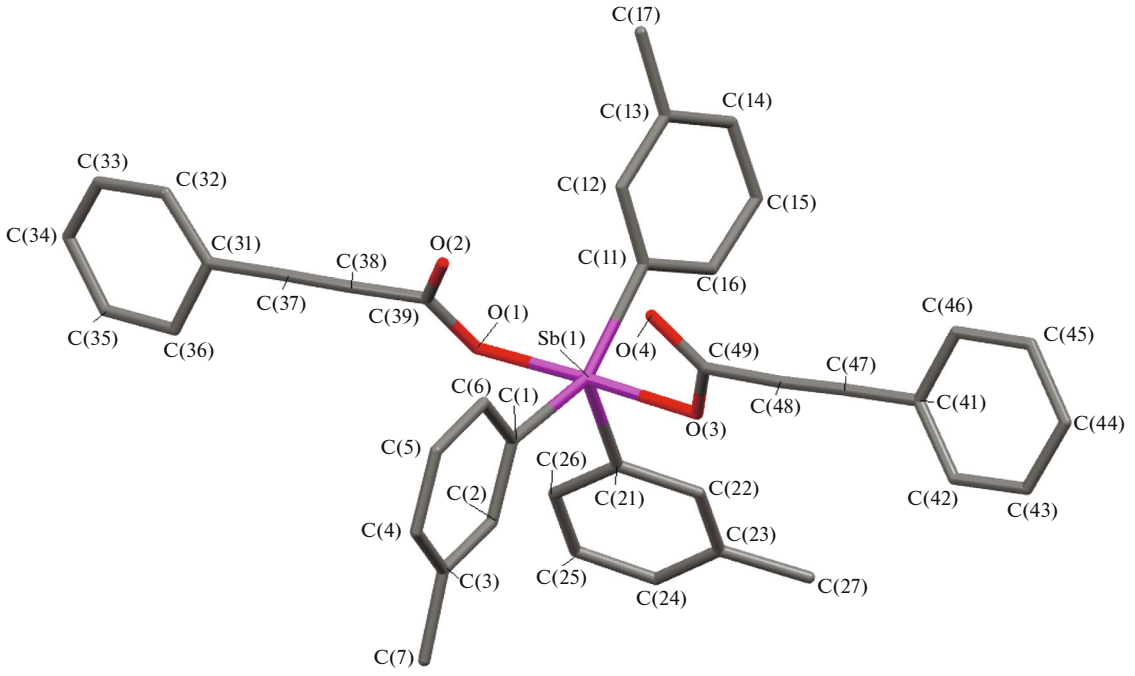

Fig. 3. Structure of the molecule of compound **III** (hydrogen atoms are omitted).

compounds **I**–**III**, the lengths of the ordinary and double carbon–oxygen bonds differ: 1.303(5) and 1.209(6) Å (**I**); 1.311(2), 1.307(2) and 1.229(2), 1.229(2) Å (**II**); and 1.275(10), 1.309(10) and 1.232(11), 1.227(10) Å (**III**). The S–O ordinary bonds (1.508(6), 1.450(4) Å) are longer than the S=O double bonds (1.379(5)–1.435(4) Å) in the sulfonate group of the molecule of compound **IV**.

The carboxylate ligands in the molecules of compounds **I**, **II**, and **III** are arranged relative to the SbC_3 fragment in such a way that the Sb···O(=C) intramolecular contacts are formed inside one equatorial

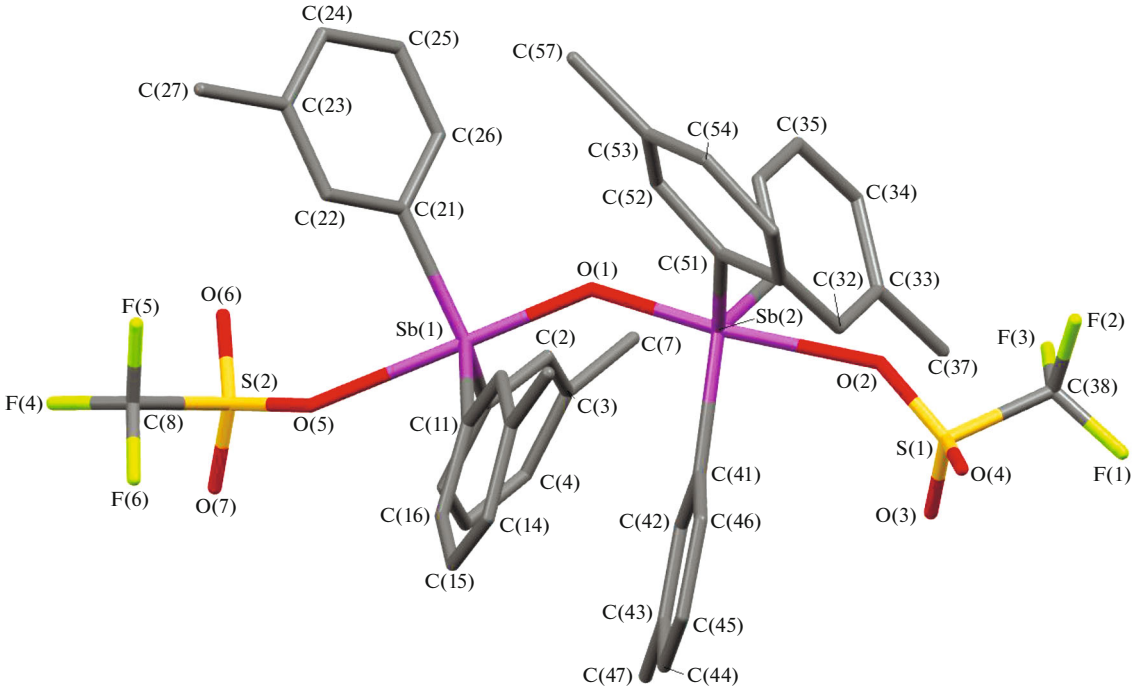

Fig. 4. Structure of the molecule of compound **IV** (hydrogen atoms are omitted).

angle, whose values increase to 137.87(19)°, 146.87(8)°, and 143.2(3)°, respectively. There is a relationship between the strength of the Sb···O contact and value of the angle. No intramolecular contacts are observed in the crystal of compound **IV**.

The structural organization of the crystals of compounds **I**–**IV** is due to the following multiple intermolecular hydrogen bonds: H···O 2.68 Å, H···F 2.57 Å in **I**; H···O 2.71 Å, H···C 2.82, 2.85 Å in **II**; H···C 2.82, 2.85 Å in **III**; and H···O 2.38–2.67 Å in **IV**.

Thus, the characteristic nonvalent interactions of the carbonyl oxygen atoms with the central metal atom are observed in compounds **I**–**III** synthesized from tri(*meta*-tolyl)antimony, carboxylic acid, and *tert*-butyl hydroperoxide in diethyl ether (molar ratio 1 : 2 : 1). The presence of the electron-withdrawing fluorine atoms in the carboxylate ligands of compound **I** induces the weakening of the Sb···O(=C) intramolecular interactions in compound **I** compared to compounds **II** and **III**. The introduction of tri(*meta*-tolyl)antimony with trifluoromethanesulfonic acid in the presence of *tert*-butyl hydroperoxide in the oxidative addition reaction results only in the formation of the bridged antimony compound with anomalously short bonds between the bridging oxygen atom and antimony atoms regardless of the ratio of the starting reactants.

CONFLICT OF INTEREST

The authors declare that they have no conflicts of interest.

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