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Specific Features of the Oxidative Addition of Triarylantimony to Dihydroxybenzoic Acids

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Abstract—Tris(2-methoxyphenyl)antimony and tris(3-fluorophenyl)antimony react with 2,3-dihydroxybenzoic and 3,4-dihydroxybenzoic acids in the presence of *tert*-butyl hydroperoxide to form carboxycatecholato-*O*,*O*'-triarylantimony. Under similar conditions, tris(4-fluorophenyl)antimony reacts with 2,3-dihydroxybenzoic acid to form tris(4-fluorophenyl)antimony dicarboxylate. Structural features of the reaction products are determined by X-ray diffraction (XRD) (CIF files CCDC nos. 2126358 (**I**), 2124252 (**II**·H_2O·Et_2O), 2121839 (**III**·0.5C_6H_6), and 2131083 (**IV**·2.5C_6H_6).

Keywords: tris(2-methoxyphenyl)antimony, tris(3-fluorophenyl)antimony, *tris*(4-fluorophenyl)antimony, 2,3-dihydroxybenzoic acid, 3,4-dihydroxybenzoic acid, *tert*-butyl hydroperoxide, oxidative addition, XRD

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INTRODUCTION

The oxidative method for the synthesis of aryl derivatives of antimony(V) has been studied in rather detail and is known as a resultative and simple approach to the preparation of compounds Ar_3SbX_2 and $(Ar_3SbX)_2O$, where X is the residue of alcohol [1], phenol [2, 3], oxime [4, 5], and carboxylic [6–9] and sulfonic [10, 11] acids.

Although the oxidative synthetic method is widely popular, reactions with organic compounds bearing several functional groups were not systematically studied. For instance, several works are devoted to the oxidative addition of triarylantimony to bifunctional compounds. When using diatomic alcohols [12] and dihydroxybenzenes [13], the structure of the product was found to depend on the arrangement of functional groups relative to each other. In the reaction with triphenylantimony (hydrogen peroxide as the oxidant), phthalic acid reacts as a monobasic acid to form triphenylantimony dicarboxylate [14]. However, under similar conditions, bifunctional oxime, 2,2'-methylenedi(cyclopentan-1-one) dioxime, reacts with triphenylantimony to give the macrocyclic binuclear complex in which the dioxime residue is the bridging ligand [15]. An example for the synthesis of coordination polymers from triphenylantimony dibromide and 3-formyl-substituted catecholates in the presence of triethylamine is also known [16].

The number of publications describing reactions using the compounds bearing functional groups with different mobilities of hydrogen atoms is still lower. Presently available publications indicate that dihydroxybenzoic acids, for example, in oxidative addition reactions, can behave as both acids [17] and dihydroxybenzenes [18].

Continuing the studies of the organoantimony derivatives of dihydroxybenzoic acids, we carried out the reactions of tris(2-methoxyphenyl)antimony, tris(3-fluorophenyl)antimony, and tris(4-fluorophenyl)antimony with 2,3- and 3,4-dihydroxybenzoic acids in the presence of *tert*-butyl hydroperoxide and determined structural features of the following reaction products: 3-carboxycatecholato-*O*,*O*'-tris(2-methoxyphenyl)antimony (**I**), 3-carboxycatecholato-*O*,*O*'-tris(3-fluorophenyl)antimony solvate (**II**·H_2O·Et_2O), 4-carboxycatecholato-*O*,*O*'-tris(2-methoxyphenyl)antimony solvate (**III**·0.5C_6H_6), and bis(2,3-dihydroxybenzoato)tris(4-fluorophenyl)antimony solvate (**IV**·2.5C_6H_6).

EXPERIMENTAL

Dihydroxybenzoic acids (Alfa Aesar) were used. The triarylantimony compounds were synthesized by a described procedure [19]. Prior to synthesis, solvents (reagent grade) were dehydrated over calcium chloride and distilled.

Synthesis of 3-carboxycatecholato-*O*,*O*'-tris(2-methoxyphenyl)antimony (I). A 70% aqueous solution of *tert*-butyl hydroperoxide (0.028 g) was added to a solution of tris(2-methoxyphenyl)antimony solvate with benzene (0.10 g, 0.22 mmol) [20] and 2,3-dihydroxybenzoic acid (0.034 g, 0.22 mmol) in diethyl ether (20 mL). The mixture was stored at 20°C for 24 h, and the formation of colorless crystals was observed. The yield was 0.112 g (86%). $T_m = 155°C$. IR (ν, cm^{-1}): 3528 w, 3009 w, 2968 w, 2943 w, 2839 w, 2795 w, 1726 vs, 1576 s, 1474 s, 1271 s, 1238 vs, 1221 vs, 1163 s, 1123 m, 1055 m, 1016 s, 970 m, 897 w, 849 m, 787 m, 754 vs, 699 w, 633 m, 569 w, 478 m, 436 m.

For $C_{28}H_{25}O_7Sb$

Anal. calcd., %	C, 56.45	H, 4.20
Found, %	C, 56.39	H, 4.31

Compounds **II**·H_2O·Et_2O, **III**·$0.5C_6H_6$, and **IV**·$2.5C_6H_6$ were synthesized similarly.

3-Carboxycatecholato*O*,*O*'-tris(3-fluorophenyl)antimony solvate with water and diethyl ether (**II**·H_2O·Et_2O): colorless crystals, yield 0.148 g (93%), $T_m = 94°C$. IR (ν, cm^{-1}): 3566 w, 3499 w, 2978 w, 2881 w, 1699 vs, 1576 m, 1506 w, 1474 vs, 1452 m, 1412 m, 1296 m, 1256 s, 1213 vs, 1161 w, 1086 w, 999 w, 974 w, 849 m, 787 s, 748 s, 685 m, 654 w, 631 w, 550 w, 442 m.

For $C_{29}H_{28}F_3O_6Sb$

Anal. calcd., %	C, 53.48	H, 4.34
Found, %	C, 53.36	H, 4.42

4-Carboxycatecholato-*O*,*O*'-tris(2-methoxyphenyl)antimony solvate with benzene (**III**·$0.5C_6H_6$): colorless crystals, yield after recrystallization from benzene 0.135 g (96%). $T_m = 164°C$. IR (ν, cm^{-1}): 3566 w, 3061 w, 2967 w, 2938 w, 2833 w, 1668 vs, 1578 vs, 1497 s, 1472 s, 1431 s, 1347 m, 1273 vs, 1240 vs, 1179 w, 1161 w, 1117 m, 1055 m, 1015 s, 943 m, 882 w, 820 s, 789 w, 754 s, 684 w, 637 m, 569 w, 482 w, 438 m.

For $C_{31}H_{28}O_7Sb$

Anal. calcd., %	C, 54.91	H, 4.61
Found, %	C, 54.78	H, 4.69

Bis(2,3-dihydroxybenzoato)tris(4-fluorophenyl)antimony solvate with benzene (**IV**·$2.5C_6H_6$): colorless crystals, yield after recrystallization from benzene 0.172 g (78%). $T_m = 92°C$. IR (ν, cm^{-1}): 3545 w, 3496 w, 3418 w, 3088 w, 3071 w, 3034 w, 1634 s, 1584 vs, 1491 vs, 1460 s, 1395 m, 1323 s, 1267 vs, 1234 vs, 1165 vs, 1059 s, 1011 m, 961 m, 823 vs, 791 s, 758 s, 725 w, 677 s, 640 m, 584 w, 507 s, 474 m, 417 m.

For $C_{47}H_{37}F_3O_8Sb$

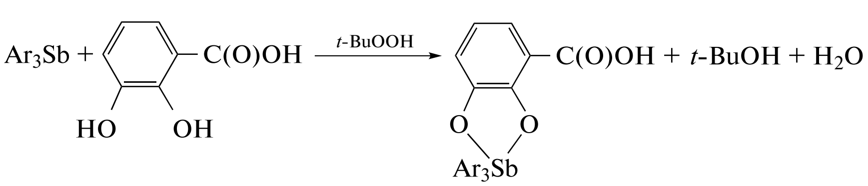
Anal. calcd., %	C, 62.13	H, 4.11
Found, %	C, 61.99	H, 4.19

IR spectra were recorded on a Shimadzu FT-IR spectrometer in KBr pellets. Melting points were measured on a Netzsch 449C Jupiter synchronous thermoanalyzer. Elemental analysis was carried out on a Euro EA3028-HT analyzer. XRD was conducted on a D8 QUEST diffractometer (Bruker) at 293 K. Data were collected and processed, unit cell parameters were determined and refined, an absorption correction was applied, and structures were solved and refined using the known programs [21–23]. The structures of compounds **I**, **II**·H_2O·Et_2O, **III**·$0.5C_6H_6$, and **IV**·$2.5C_6H_6$ (hereinafter, **I**–**IV**) were solved by a direct method and refined by least squares in the anisotropic approximation for non-hydrogen atoms. The main crystallographic data and structure refinement results for compounds **I**–**IV** are given in Table 1. Selected bond lengths and bond angles are listed 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. 2126358 (**I**), 2124252 (**II**), 2121839 (**III**), and 2131083 (**IV**); http://www.ccdc.cam.ac.uk).

RESULTS AND DISCUSSION

It was found that 2,3- and 3,4-dihydroxybenzoic acids reacted with tris(2-methoxyphenyl)antimony and tris(3-fluorophenyl)antimony in the presence of *tert*-butyl hydroperoxide in a diethyl ether solution as 1,2-dihydroxybenzenes, and the carboxyl group in the molecule remained inert. The reaction products are the compounds with the antimony-containing cycles with the bidentate chelating ligands: $(2\text{-}MeOC_6H_4)_3Sb[O,O'\text{-}C_6H_3(COOH\text{-}3)]$ (**I**), $(3\text{-}FC_6H_4)_3Sb[O,O'\text{-}C_6H_3(COOH\text{-}3)]$ (**II**), and $(2\text{-}MeOC_6H_4)_3Sb[O,O'\text{-}C_6H_3(COOH\text{-}4)]$ (**III**).

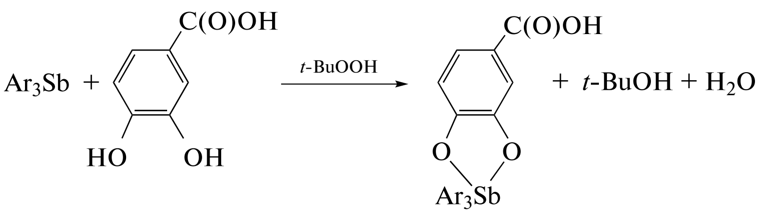


Ar = (2-MeO)C_6H_4 (**I**), 3-FC_6H_4 (**II**)

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

Parameter	Value			
	I	**II**$\cdot H_2O\cdot Et_2O$	**III**$\cdot 0.5C_6H_6$	**IV**$\cdot 2.5C_6H_6$
FW	595.23	651.26	634.28	908.51
Crystal system	Monoclinic	Monoclinic	Monoclinic	Triclinic
Space group	$P2_1/c$	$P2_1/n$	$P2_1/n$	$P\bar{1}$
a, Å	11.750(4)	10.086(18)	8.759(6)	12.952(13)
b, Å	13.167(5)	23.56(3)	22.700(12)	12.999(16)
c, Å	16.946(8)	11.94(2)	14.899(10)	14.994(14)
α, deg	90.00	90.00	90.00	73.01(4)
β, deg	92.415(19)	95.60(9)	100.78(4)	71.47(3)
γ, deg	90.00	90.00	90.00	65.26(4)
V, Å^3	2619.3(18)	2823(9)	2910(3)	2136(4)
Z	4	4	4	2
ρ_{calc}, g/cm^3	1.509	1.532	1.448	1.412
μ, mm^{-1}	1.097	1.037	0.993	0.711
$F(000)$	1200.0	1312.0	1284.0	922.0
Crystal size, mm	0.41 × 0.32 × 0.18	0.26 × 0.19 × 0.08	0.5 × 0.22 × 0.1	0.29 × 0.16 × 0.14
Data collection range over 2θ, deg	5.72–63.04	6.12–56.52	5.85–54.294	5.754–57.732
Ranges of reflection indices	$-17 \le h \le 17$, $-19 \le k \le 19$, $-24 \le l \le 24$	$-13 \le h \le 13$, $-31 \le k \le 31$, $-15 \le l \le 15$	$-11 \le h \le 11$, $-29 \le k \le 29$, $-19 \le l \le 19$	$-17 \le h \le 17$, $-17 \le k \le 17$, $-19 \le l \le 20$
Measured reflections	57535	64637	42609	69526
Independent reflections (R_{int})	8801 (0.0298)	6956 (0.0874)	6409 (0.0331)	10519 (0.0471)
Reflections with $I > 2\sigma(I)$	5987	4983	5599	8269
Refinement variables	322	360	372	552
GOOF	1.074	1.067	1.149	1.049
R factors for $F^2 > 2\sigma(F^2)$	$R_1 = 0.0335$, $wR_2 = 0.0659$	$R_1 = 0.0465$, $wR_2 = 0.0942$	$R_1 = 0.0335$, $wR_2 = 0.0907$	$R_1 = 0.0401$, $wR_2 = 0.1071$
R factors for all reflections	$R_1 = 0.0588$, $wR_2 = 0.0751$	$R_1 = 0.0791$, $wR_2 = 0.1047$	$R_1 = 0.0409$, $wR_2 = 0.0944$	$R_1 = 0.0599$, $wR_2 = 0.1190$
Residual electron density (max/min), e/Å^3	0.91/–1.05	0.64/–0.82	0.79/–0.56	1.20/–0.66

Compound **II** crystallized from the reaction mixture as solvate **II**$\cdot H_2O\cdot Et_2O$. To obtain single crystals suitable for XRD studies, complex **III** was recrystallized from benzene, and crystals of solvate **III**$\cdot 0.5C_6H_6$ were precipitated from the solution.



Ar = (2-MeO)C_6H_4 (**III**)

Table 2. Selected bond lengths and bond angles in compounds **I–IV**

Bond	d, Å	Angle	ω, deg	Angle	ω, deg
I					
Sb(1)–O(4)	2.0101(18)	O(5)Sb(1)C(1)	158.06(8)	O(4)Sb(1)C(21)	103.93(9)
Sb(1)–O(5)	2.1161(18)	O(5)Sb(1)C(11)	83.94(8)	C(1)Sb(1)C(11)	98.21(9)
Sb(1)–C(1)	2.131(2)	O(5)Sb(1)C(21)	94.57(8)	C(1)Sb(1)C(21)	102.80(9)
Sb(1)–C(11)	2.118(3)	O(4)Sb(1)C(1)	85.00(8)	C(11)Sb(1)C(21)	120.82(10)
Sb(1)–C(21)	2.103(2)	O(4)Sb(1)C(11)	132.75(9)	O(4)Sb(1)O(5)	77.82(7)
II$\cdot H_2O\cdot Et_2O$					
Sb(1)–O(1)	2.031(3)	O(2)Sb(1)C(11)	163.82(13)	C(21)Sb(1)C(1)	102.49(16)
Sb(1)–O(2)	2.119(4)	O(5)Sb(1)C(1)	169.68(13)	O(1)Sb(1)O(2)	78.84(16)
Sb(1)–C(1)	2.153(6)	O(1)Sb(1)C(21)	158.28(14)	O(2)Sb(1)O(5)	81.54(16)
Sb(1)–C(11)	2.146(4)	O(1)Sb(1)C(1)	94.62(16)	O(2)Sb(1)C(1)	89.56(17)
Sb(1)–C(21)	2.139(4)	O(1)Sb(1)C(11)	87.52(17)	O(2)Sb(1)C(21)	87.91(16)
Sb(1)–O(5)	2.283(5)	C(1)Sb(1)C(11)	100.27(18)	O(5)Sb(1)C(11)	87.31(18)
O(3)–C(37)	1.224(5)	C(21)Sb(1)C(11)	102.28(18)	O(5)Sb(1)C(21)	82.50(14)
III$\cdot 0.5C_6H_6$					
Sb(1)–O(4)	2.096(2)	O(4)Sb(1)C(21)	158.44(11)	O(4)Sb(1)C(11)	83.17(11)
Sb(1)–O(5)	2.030(3)	O(5)Sb(1)C(1)	111.67(13)	C(1)Sb(1)C(11)	107.63(13)
Sb(1)–C(1)	2.099(3)	O(5)Sb(1)C(11)	136.54(12)	C(1)Sb(1)C(21)	107.38(14)
Sb(1)–C(11)	2.123(3)	O(5)Sb(1)C(21)	85.94(11)	C(11)Sb(1)C(21)	99.57(14)
Sb(1)–C(21)	2.139(3)	O(4)Sb(1)C(1)	91.89(12)	O(4)Sb(1)O(5)	77.80(8)
IV$\cdot 2.5C_6H_6$					
Bond	d, Å	Angle	ω, deg	Angle	ω, deg
Sb(1)–O(1)	2.104(3)	O(1)Sb(1)O(5)	170.28(8)	O(1)Sb(1)C(11)	89.94(13)
Sb(1)–O(5)	2.096(3)	C(1)Sb(1)C(11)	113.85(14)	O(1)Sb(1)C(21)	96.07(12)
Sb(1)–C(1)	2.158(3)	C(1)Sb(1)C(21)	111.19(14)	C(1)Sb(1)O(5)	87.66(11)
Sb(1)–C(11)	2.118(4)	C(21)Sb(1)C(11)	134.95(12)	C(11)Sb(1)O(5)	90.42(13)
Sb(1)–C(21)	2.131(3)	O(1)Sb(1)C(1)	83.32(11)	C(21)Sb(1)O(5)	90.55(12)
Sb(1)···O(2)	2.997(5)	C(37)O(1)Sb(1)	114.58(19)	C(22)C(21)Sb(1)	125.0(2)
Sb(1)···O(6)	2.314(6)	C(47)O(5)Sb(1)	118.21(18)	C(26)C(21)Sb(1)	115.1(3)

Compounds **I–III** isolated in a high yield (86–96%) are colorless crystals with distinct melting points.

Under similar conditions, 2,3-dihydroxybenzoic acid reacts with tris(4-fluorophenyl)antimony at the carboxyl group to form tris(4-fluorophenyl)antimony 2,3-dihydroxybenzoate $(4\text{-}FC_6H_4)_3Sb[OC(O)C_6H_3(OH)_2\text{-}2,3]_2$ (**IV**), whose recrystallization from benzene gives solvate **IV**$\cdot 2.5C_6H_6$.

$$(4\text{-}FC_6H_4)_3Sb + 2HOC(O)C_6H_3(OH)_2\text{-}2,3 \xrightarrow{t\text{-BuOOH}} (4\text{-}FC_6H_4)_3Sb[OC(O)C_6H_3(OH)_2\text{-}2,3]_2 + H_2O$$

(IV)

Note that 3,4-dihydroxybenzoic acid also behaves as dihydroxybenzene in the reaction with triphenylan-

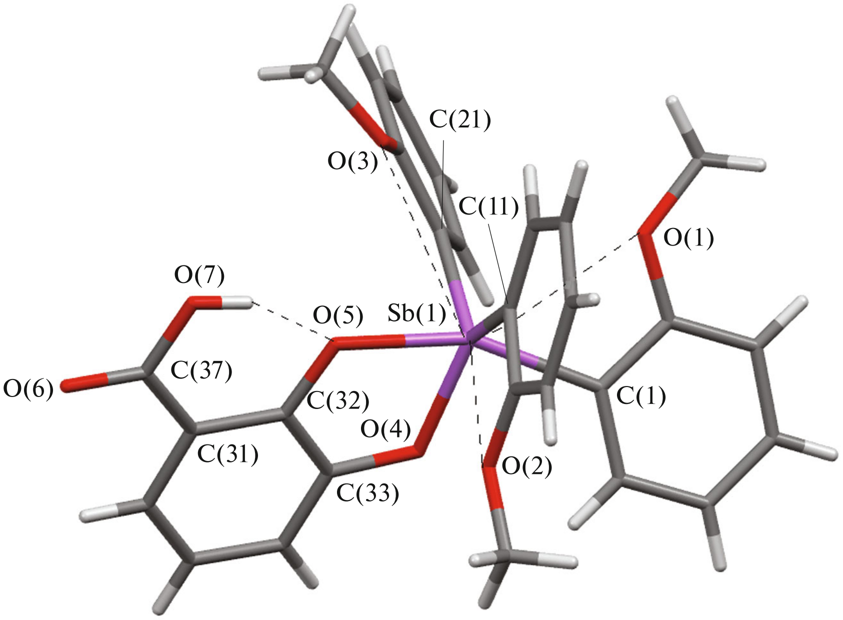


Fig. 1. Structure of compound **I**.

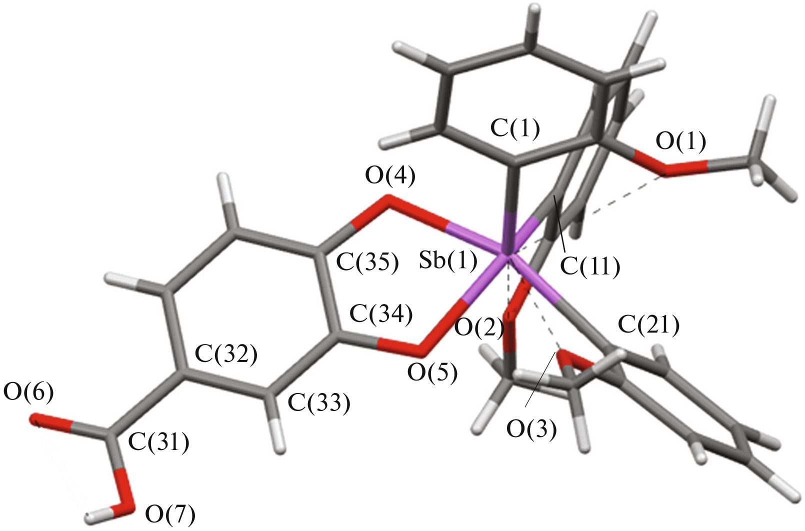


Fig. 2. Structure of solvate **III** with benzene (solvate benzene molecule is omitted).

timony as found previously [18]. The reactions of 2,3-dihydroxybenzoic acid with triphenyl- and tri(*para*-tolyl)antimony in the presence of hydrogen peroxide afford triarylantimony dicarboxylates [17].

The IR spectra of compounds **I**–**IV** exhibit bands at ~450 (Sb–C), ~1250 (O–C), ~1470 (Ar), and ~3050 ($H-C_{Ar}$) cm^{-1} along with the absorption bands of the carbonyl groups (1720, 1700, 1668, and 1634 cm^{-1}, respectively). In the last case, the indicated band is most shifted to the low-frequency range compared to the IR spectrum of the initial acid, which is explained by the elongation of the C=O bonds in triarylantimony dicarboxylates [24].

The bands at 3528, 3566, and 3566 cm^{-1} in the IR spectra of compounds **I**, **II**, and **III**, respectively, correspond to vibrations of the free hydroxyl of the carboxyl group. The broad band at 3499 cm^{-1} in the IR spectrum of compound **II** confirms the presence of the solvate water molecule [25, 26]. The presence of free hydroxy groups in compound **IV** is indicated by the bands at ~3500 cm^{-1} in the IR spectrum.

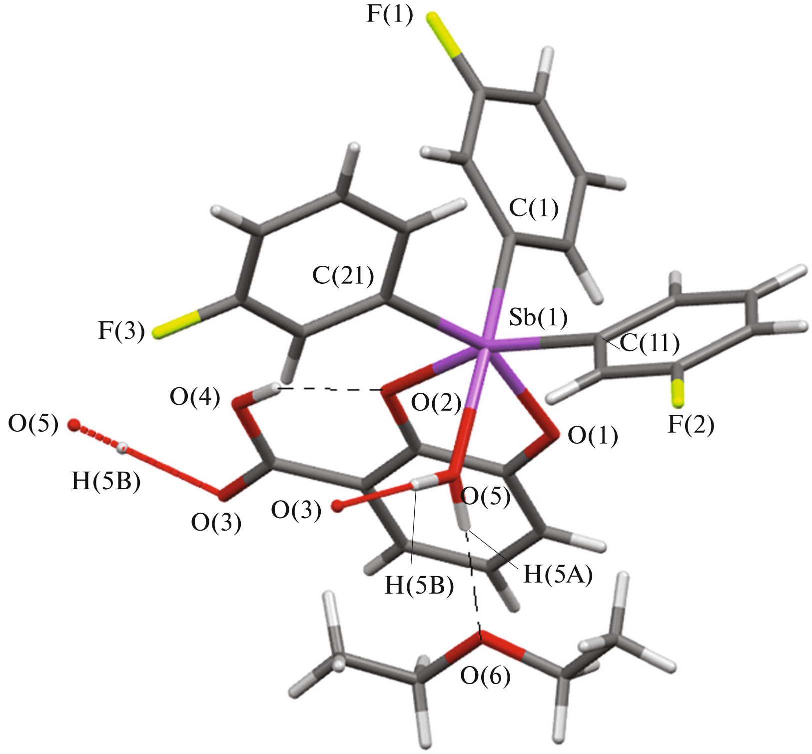


Fig. 3. Structure of solvate **II** with diethyl ether and water.

According to the XRD data, the antimony atoms have the coordination of a strongly trigonal bipyramid with a high contribution of the square pyramid coordination in the molecules of compounds **I** and **III** bearing the five-membered metallocycles (Figs. 1, 2).

The coordination distortion is indicated by the axial angles C(1)SbO(5) 158.06(8)° (**I**) and C(21)SbO(4) 158.44(11)° (**III**), which strongly differ from the theoretical value, as well as the values of equatorial angles: C(11)SbC(21) 120.82(10)°, C(11)SbO(4) 132.75(9)°, C(21)SbO(4) 103.93(9)° (sum 357.5(9)° in **I**) and C(1)SbC(11) 107.63(13)°, C(11)SbO(5) 136.54(12)°, C(1)SbO(5) 111.67(13)° (sum 355.84(13)° in **II**). The five-membered metallocycles [SbO_2C_2] have a slight inflection along the O–O diagonal (dihedral angles between the corresponding angles are 2.80° and 0.61° in compounds **I** and **III**, respectively). The Sb–C bonds vary in the closest ranges (2.103(2)–2.131(2) Å in **I** and 2.099(3)–2.139(3) Å in **III**), and the maximum values belong to the axial bonds (Sb–C(1) and Sb–C(21) in **I** and **III**, respectively). The Sb–O bond lengths in the metallocycles somewhat differ from each other (2.0101(18), 2.1161(18) Å in **I** and (2.030(3)–2.096(2) Å in **III**) and are comparable with analogous distances in the antimony(V) catecholate complexes [27–33].

Molecule **II** differs from molecules **I** and **III** by the environment of the central atom, whose coordination sphere contains the water molecule, building up the coordination polyhedron to the octahedron (Fig. 3).

The C(1)SbO(5), C(11)SbO(2), and C(21)SbO(1) angles are 169.68(13)°, 163.82(13)°, and 158.28(14)°; and other angles at the antimony atom vary in a range of 78.60°–102.49(16)°. The Sb–C bonds (2.139(4)–2.153(6) Å) are noticeably longer than those in compounds **I** and **III**, which can be explained by the electronegative fluorine atom in the aromatic ring and an increase in the coordination number of the central atom. The Sb–O(1,2) bonds in the metallocycle are 2.031(3) and 2.119(4) Å, whereas the coordination bond with the water molecule Sb–O(5) is 2.283(5) Å. It should be mentioned that the catecholate complex with the water molecule in the coordination sphere of antimony was described [34]. In this complex, the coordination Sb···O bond is substantially longer than that in compound **II** and is equal to 2.593(2) Å.

In the carboxyl groups of molecules **I**–**III**, the ordinary C–O and double C=O bonds differ substantially in length (1.210(4) and 1.338(4) Å, 1.224(5) and 1.324(6) Å, 1.234(5) and 1.302(5) Å), as it is observed in carboxylic acids.

As should be expected, intramolecular hydrogen bonds between the free hydroxy group of the acid and the oxygen atom of the chelate cycle are observed in molecules **I** and **II**: O(7)–H(7)···O(5) and O(4)–H(4)···O(2), respectively (O(7)···O(5) and

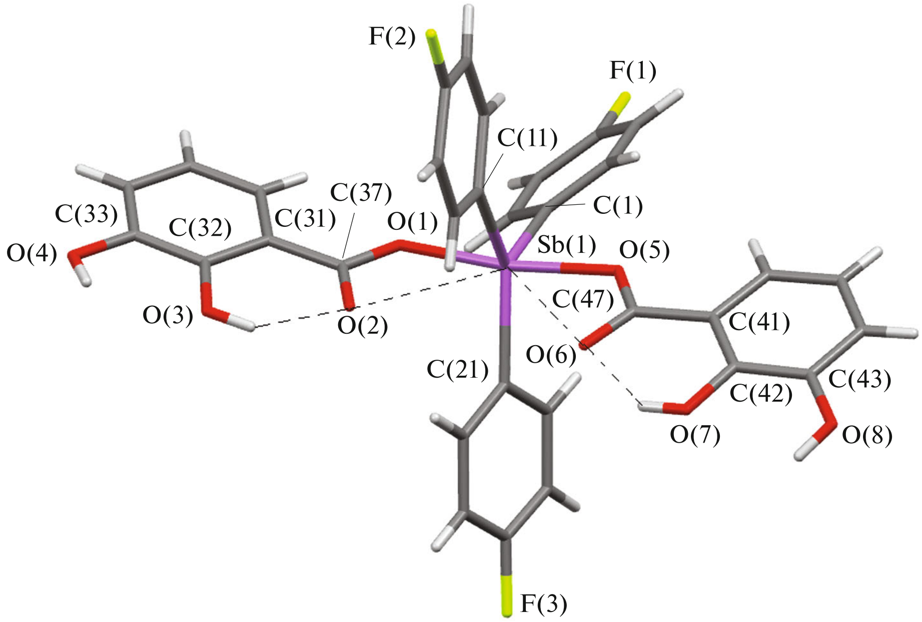


Fig. 4. Structure of solvate **IV** with benzene (solvate benzene molecules are omitted).

O(4)··O(2) distances are 2.570(3) and 2.588(5) Å). In addition, the coordinated water molecule in compound **II** forms hydrogen bonds with the adjacent molecule through the carbonyl oxygen atom (distance O(5)···O(3) 2.683(5) Å) and with the solvate diethyl ether molecule (distance O(5)···O(6) 2.645(7) Å). Dimers are formed via the intermolecular hydrogen bonds O(7)–H(7)···O(6) between the carboxyl groups in the crystal of compound **III**, and the O(7)···O(6) distance is 2.639(5) Å.

Short Sb···OMe distances (2.808(2)–3.152(2) and 2.933(3)–3.082(3) Å at the sum of van der Waals radii of the atoms equal to 3.58 Å [35]) between the metal atom and oxygen atoms of the methoxy groups in the *ortho*-positions of the aryl rings in molecules **I** and **III** should be noted.

In molecule **IV**, the antimony atom has a slightly distorted trigonal bipyramidal coordination, which is indicated by a small shift of the antimony atom from the equatorial plane (0.011 Å) and the sum of angles in the equatorial plane close to 360° (359.04(14)°) (Fig. 4).

The OSbO axial angle is 170.28(8)°. The Sb–C(1,11,21) bonds (2.118(4), 2.131(3), 2.158(3) Å) are longer than Sb–O(1,5) (2.104(3), 2.096(3) Å), which is not characteristic, as a rule, of triarylantimony dicarboxylates [36–41] and is probably related to the electronegative fluorine atom in the *para*-position of the aryl rings as in [42]. It should be noted for comparison that the Sb–C and Sb–O distances lie in ranges of 2.097(6)–2.107(4) and 2.108(3)–2.145(4) Å in the earlier synthesized triphenyl- and tri(*para*-tolyl)antimony bis(2,3-dihydroxybenzoates), respectively [17].

As in molecules of other triarylantimony dicarboxylates, compound **IV** contains short contacts of the central atom with the carbonyl oxygen atoms (Sb···O(2,6) 2.997(3), 3.140(4) Å) leading to the opening of the C(11)Sb(21) equatorial angle from the side of the contacts to 134.95(12)°, which is accompanied by a decrease in two other angles (111.19(14)°, 113.85(14)°).

The ordinary C–O and double C=O bonds in the carboxyl group are different. For instance, in one of the carboxyl groups, the bond in the carbonyl group (C(47)–O(6) 1.253(4) Å) is significantly shorter than the C(47)–O(5) bond: 1.332(4) Å. In another carboxyl group, the C(37)–O(1) and C(37)–O(2) bonds are nearly equalized (1.297(4) and 1.287(4) Å, respectively), which correlates with the shorter intramolecular distance Sb···O. The C(37)–O(2) bond elongation is also favored by the intramolecular hydrogen bond with the hydroxy group O(3)–H(3)···O(2) having the following parameters: distances O(3)–H(3) 0.82, O(3)···O(2) 2.536(3), and H(3)···O(2) 1.83 Å; angle O(3)H(3)O(2) 143°. The second intramolecular hydrogen bond O(7)–H(7)···O(6) is weaker (distances O(7)–H(7) 0.82, O(7)···O(6) 2.610(4), and H(7)···O(6) 1.92 Å; angle O(7)H(7)O(6) 141°).

Thus, the structures of the reaction products of the oxidative addition of triaryl compounds of antimony with 2,3- and 3,4-dihydroxybenzoic acids are determined by both the arrangement of the hydroxy groups in the benzene ring of the acid and the nature of the aryl groups at the antimony atom. In the presence of the oxidant, 2,3- and 3,4-dihydroxybenzoic acids react with tris(2-methoxyphenyl)- and tris(3-fluorophenyl)antimony at the hydroxy groups as 1,2-dihydroxybenzene to form the cyclic product with the free carboxyl group in the benzene ring. At the same time, for the reaction with tris(4-fluorophenyl)antimony under similar conditions, the hydroxy groups of 2,3-dihydroxybenzoic acid remain inert and the reaction proceeds at the carboxyl group to form tris(4-fluorophenyl)antimony 2,3-dihydroxybenzoate.

CONFLICT OF INTEREST

The authors declare that they have no conflicts of interest.

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