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The issue is dedicated to the 70th birthday of Academician V.I. Ovcharenko

Characteristic Features of the Reactions of Triarylantimony with Hydroxybenzoic Acids. Structure of the Reaction Products

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Abstract—Tri(*meta*-tolyl)antimony and triphenylantimony react with 2,6-dihydroxybenzoic and 3,5-dinitrosalicyclic acids in the presence of hydrogen peroxide to give triarylantimony dicarboxylates (**I** and **II**·2PhH, respectively). The reaction of tris(5-bromo-2-methoxyphenyl)antimony with 2,6-dihydroxybenzoic acid gives binuclear organoantimony compound with a bridging oxygen atom (**III**·MeCN). The structures of the products were determined by X-ray diffraction (CCDC nos. 1911551, 2117678, 1970910).

Keywords: triphenylantimony, tri-*meta*-tolylantimony, tris(5-bromo-2-methoxyphenyl)antimony, 2,6-dihydroxybenzoic acid, 3,5-dinitrosalicylic acid, hydrogen peroxide, oxidative addition, X-ray diffraction

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INTRODUCTION

The synthesis of new antimony(V) derivatives is obviously a relevant task, because these derivatives are widely used in various fields of science and technology [1], for example, in the synthesis of macromolecular compounds to modify their properties [2, 3]. Antimony(V) compounds are known to exhibit catalytic [4–6], biological [7–10], and photocatalytic activities [11]. An efficient method for the synthesis of arylantimony(V) compounds is the oxidative addition of triaryl antimony derivatives, which was first proposed in [12] and studied for a large number of organic O–H acids [13–15]. However, this reaction has not been systematically studied for compounds containing several active hydrogen atoms where the reaction products may have different structures. For example, the reactions of triphenylantimony (triphenylantimony halide) with salicylic [16], 5-bromosalicyclic [17], and 2,3- and 2,6-dihydroxybenzoic acids [18] involve only the carboxyl group and give triarylantimony dicarboxylates. 3,4-Dihydroxybenzoic acid behaves as dihydroxybenzene when reacts with triphenylantimony, and the reaction gives tetranuclear macrocycle [19]. The reaction of triarylantimony dichloride with (±)-mandelic acid involves both carboxyl and hydroxyl groups and also gives a tetranuclear macrocycle [20].

As a continuation of studies of organic antimony derivatives of hydroxybenzoic acids, we carried out the reaction of tri(*meta*-tolyl)antimony and tris(5-bromo-2-methoxyphenyl)antimony with 2,6-dihydroxybenzoic acid and the reaction of triphenylantimony with 3,5-dinitrosalicylic acid and explored the structural features of the reaction products.

EXPERIMENTAL

Commercially available 2,6-dihydroxybenzoic acid and 3,5-dinitrosalicylic acid (Sigma Aldrich) were used as received. Triphenylantimony, tri(*meta*-tolyl)antimony, and tris(5-bromo-2-methoxyphenyl)antimony were synthesized by standard procedures and recrystallized from benzene and toluene. Diethyl ether, acetonitrile, toluene, benzene, heptane, and octane were dried prior to use according to standard procedures.

Synthesis of m-Tol$_3$Sb[O(O)CC$_6$H$_3$(OH)$_2$-2,6]$_2$ (I). A mixture of tri(*meta*-tolyl)antimony (0.2 g, 0.51 mmol) and 2,6-dihydroxybenzoic acid (0.16 g, 1.02 mmol) was dissolved in diethyl ether (30 mL). A 30% aqueous hydrogen peroxide solution (0.051 g) was added to the resulting solution. Recrystallization from a toluene–heptane mixture gave red crystals. The yield was 0.32 g (81%). T_{m} = 181°C.

IR (ν, cm^{-1}): 3265 s, 2360 w, 1646 s, 1602 s, 1499 w, 1474 w, 1395 w, 1354 s, 1256 s, 1219 s, 1156 s, 1117 s, 1078 m, 1030 m, 992 w, 851 w, 818 s, 773 m, 702 m, 685 m, 667 m, 609 s, 584 w, 533 w, 484 m, 428 m.

For $C_{35}H_{31}O_8Sb$		
Anal. calcd., %	C, 59.89	H, 4.42
Found, %	C, 59.81	H, 4.50

Compounds **II**·2PhH and **III**·MeCN were synthesized in a similar way.

For $Ph_3Sb[O(O)CC_6H_2(OH)$-2-$(NO_2)_2$-3,5$]_2$·2PhH (**II**), recrystallization from benzene containing some octane gave yellow crystals. The yield was 0.41 g (75%). T_m = 215°C.

IR (ν, cm^{-1}): 3097 m, 2924 m, 2858 w, 2354 m, 1650 s, 1597 s, 1539 s, 1433 s, 1362 s, 1338 s, 1260 s, 1176 s, 1087 m, 1019 m, 999 m, 939 s, 740 s, 687 m, 684 s, 669 s, 547 s, 468 s, 452 s.

For $C_{44}H_{33}N_4O_{14}Sb$		
Anal. calcd., %	C, 54.83	H, 3.43
Found, %	C, 54.81	H, 3.40

For [(5-Br-2-$MeOC_6H_3)_3SbO(O)CC_6H_3(OH)_2$-2,6$]_2$O·MeCN (**III**), recrystallization from acetonitrile gave white crystals. The yield was 0.51 g (68%). T_m = 192°C (decomp.).

IR (ν, cm^{-1}): 3091 w, 2934 w, 1644 s, 1599 s, 1476 s, 1437 m, 1375 s, 1330 m, 1282 s, 1254 s, 1220 m, 1180 w, 1155 w, 1126 m, 1093 w, 1046 m, 1013 m, 885 w, 805 s, 704 s, 620 m, 603 m, 532 s, 488 w, 471 w, 434 s.

For $C_{58}H_{49}NO_{15}Br_6Sb_2$		
Anal. calcd., %	C, 40.40	H, 2.85
Found, %	C, 40.32	H, 2.90

The IR spectra of compounds **I**, **II**·2PhH, and **III**·MeCN were measured on a Shimadzu IR Affinity-1S Fourier Transform IR spectrometer for KBr pellets.

Single crystal X-ray diffraction analysis of **I**, **II**·2PhH, and **III**·MeCN was carried out on a D8 QUEST Bruker automated four-circle diffractometer (MoK_α radiation, λ = 0.71073 Å, graphite monochromator). The data collection and editing and the refinement of unit cell parameters were performed and absorption corrections were applied using the SMART SAINT-Plus software [21]. The crystal structure solution and refinement were carried out using the SHELX/PC [22] and OLEX2 [23] software. The structure was solved by the direct method and refined by the least-squares method, first, in the isotropic and, then, in the anisotropic approximation for nonhydrogen atoms. The hydrogen atoms were placed into the geometrically calculated positions and included in the refinement by the riding model.

The crystals of compound **I** were red, prismatic, and monoclinic, $C_{35}H_{31}O_8Sb$, M = 701.35 g/mol; a = 9.014(12), b = 11.391(16), c = 30.67(4) Å, α = 90.00°, β = 92.78(5)°, γ = 90.00°, V = 3146(7) Å^3, ρ(calcd.) = 1.481 g/cm^3, Z = 2, space group $P2_1/c$. A total of 34622 reflections were measured, 3694 reflections were unique, μ = 0.928 mm^{-1}. The final residual factors were R_1 = 0.0893 and wR_2 = 0.0980 (for all reflections), R_1 = 0.0551 and wR_2 = 0.0876 (for reflections with $F^2 > 2\sigma(F^2)$).

The crystals of compound **II**·2PhH were yellow, prismatic, and triclinic, $C_{44}H_{33}N_4O_{14}Sb$, M = 963.49 g/mol; a = 10.198(10) Å, b = 11.867(10) Å, c = 18.906(15) Å, α = 75.24(3)°, β = 79.32(4)°, γ = 85.54(6)°, V = 2173(3) Å^3, ρ(calcd.) = 1.472 g/cm^3, Z = 2, space group $P\overline{1}$. A total of 57753 reflections were measured, 9112 reflections were unique, μ = 0.707 mm^{-1}. The final residual factors were R_1 = 0.0464 and wR_2 = 0.0683 (for all reflections), R_1 = 0.0301 and wR_2 = 0.0629 (for reflections with $F^2 > 2\sigma(F^2)$).

The crystals of compound **III**·MeCN were colorless, prismatic, and monoclinic, $C_{58}H_{49}NO_{15}Br_6Sb_2$, M = 1722.94 g/mol; a = 12.259(8), b = 15.501(10), c = 32.54(3) Å, α = 90.00°, β = 90.18(3)°, γ = 90.00°, V = 6184(8) Å^3, ρ(calcd.) = 1.851 g/cm^3, Z = 4, space group $P2_1/n$. A total of 119381 reflections were measured, 13659 reflections were unique, μ = 4.815 mm^{-1}. The final residual factors were: R_1 = 0.0829 and wR_2 = 0.1199 (for all reflections), R_1 = 0.0451 and wR_2 = 0.1012 (for reflections with $F^2 > 2\sigma(F^2)$).

The full set of X-ray diffraction parameters for the structures of **I**, **II**·2PhH, and **III**·MeCN are deposited with the Cambridge Crystallographic Data Centre (CCDC nos. 1911551, 2117678, and 1970910, respectively; deposit@ccdc.cam.uk).

RESULTS AND DISCUSSION

The reactions of tri(*meta*-tolyl)antimony with 2,6-dihydroxybenzoic acid and triphenylantimony with 3,5-dinitrosalicylic acid in the presence of hydrogen peroxide gave triarylantimony dicarboxylates. Only the carboxyl groups of the acids participated in the reaction, while the hydroxyl groups remained unaffected:

$$\begin{gathered} Ar_3Sb + 2Ar'C(O)OH + H_2O_2 \\ \rightarrow Ar_3Sb[O(O)CAr']_2 + 2H_2O, \\ Ar = m\text{-Tol}(\mathbf{I}),\ Ph(\mathbf{II}), \\ Ar' = C_6H_3(OH)_2\text{-}2,6\ (\mathbf{I}), \\ C_6H_2(OH)\text{-}2\text{-}(NO_2)_2\text{-}3,5\ (\mathbf{II}). \end{gathered}$$

Compound **II** was isolated after recrystallization as a solvate with two benzene molecules.

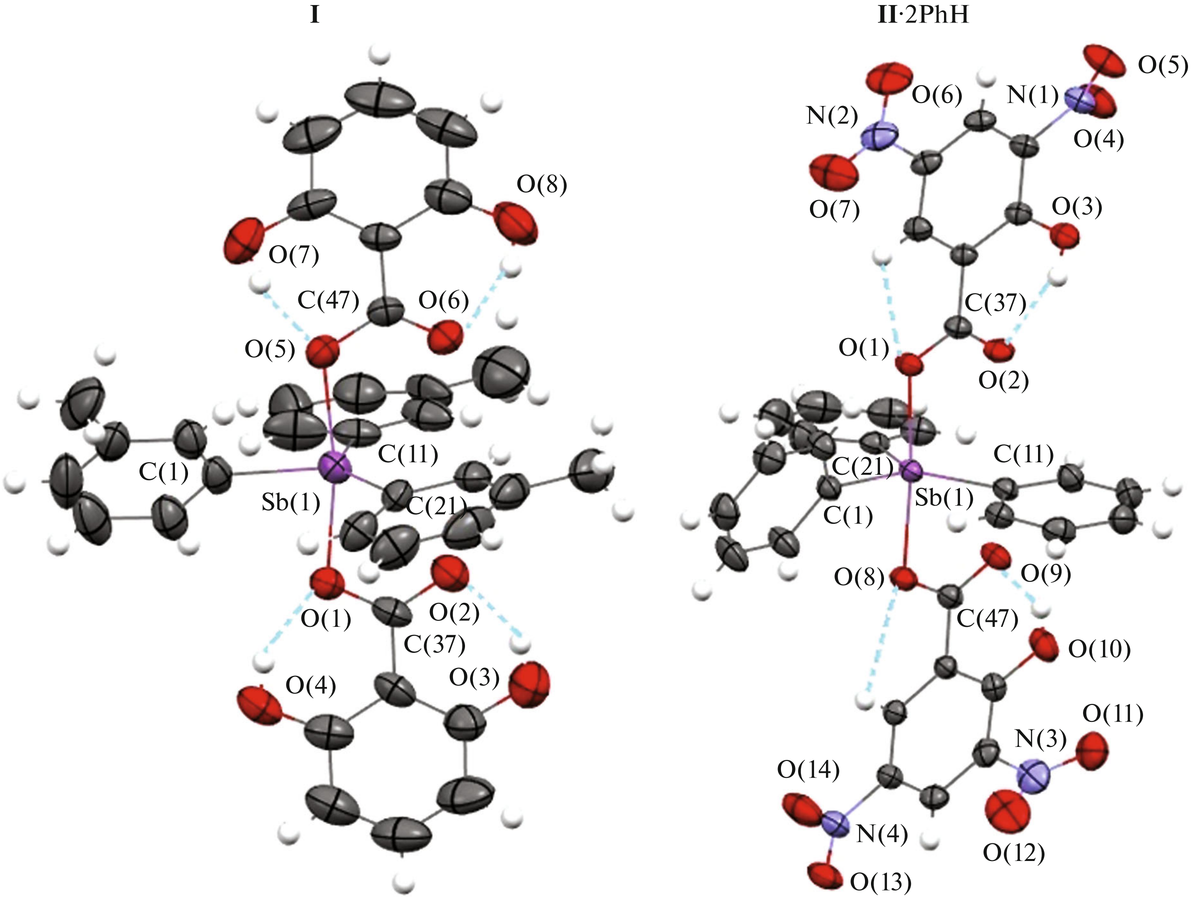
It is noteworthy that triarylantimony dicarboxylates are also formed in the reactions of triphenyl- and tri(*para*-tolyl)antimony with 2,6-dihydroxybenzoic acid [18]. The reactions of salicylic and 5-bromosalicylic acids with triphenylantimony did not affect the acid hydroxyl groups either [16, 17].

Irrespective of the molar ratio of the components, the reaction of tris(5-bromo-2-methoxyphenyl)antimony with 2,6-dihydroxybenzoic acid always gave μ_2-oxo-bis[(2,6-dihydroxybenzoato)tris(5-bromo-2-methoxyphenyl)antimony], in which antimony atoms were linked by a μ_2-bridging oxygen atom (the product was isolated as an acetonitrile solvate after recrystallization):

$$2(5\text{-Br-2-MeOC}_6\text{H}_3)_3\text{Sb} + 2\text{HO(O)CC}_6\text{H}_3(\text{OH})_2\text{-2,6} + 2\text{H}_2\text{O}_2$$
$$\rightarrow [(5\text{-Br-2-MeOC}_6\text{H}_3)_3\text{SbO(O)CC}_6\text{H}_3(\text{OH})_2\text{-2,6}]_2\text{O} + 3\text{H}_2\text{O}.$$

In the IR spectra of **I**, **II**, and **III**, the absorption bands of the C=O groups (1646, 1650, and 1644 cm^{-1}, respectively) are shifted to lower frequency relative to the IR spectra of the initial acids (1674 and 1680 cm^{-1} for 2,6-dihydroxybenzoic and 3,5-dinitrosalicylic acid, respectively).

In the molecules of **I** and **II** (Fig. 1), the antimony atoms have a slightly distorted trigonal-bipyramidal coordination, as indicated by the slight deviation of the antimony atom from the equatorial plane (0.005 and 0.015 Å, respectively) and by the sum of angles in the equatorial plane close to 360°. The OSbO axial angles are 170.6(2)° and 175.29(6)°, the Sb–C bond lengths and Sb–O distances are somewhat shorter in **I** than in **II**: 2.082(8)–2.122(8) Å (**I**), 2.115(3)–2.131(3) Å (**II**) and 2.137(5), 2.143(6) Å (**I**), 2.185(2), 2.161(2) Å (**II**) (Table 1). In the triarylantimony bis(2,6-dihydroxybenzoates) synthesized previously, the Sb–O distances are in the 2.120(3)–2.141(3) Å range [18], which is commensurable with those in **I**. The Sb–O bond lengths in the triphenylantimony disalicylates (*cis*- and *trans*-forms) are 2.100(3)–2.127(3) Å, which is markedly shorter than the corresponding distances in **II** [24]. The Sb···O distances between the antimony atom and the carbonyl oxygen atoms are 2.900(3), 3.017(4) Å (**I**) and 2.992(2), 2.994(2) Å (**II**), which attests to a pronounced asym-



I **II**·2PhH

Fig. 1. Molecular structure of compounds **I** and **II**·2PhH (benzene solvate molecules in **II** are not shown).

Table 1. Selected bond lengths and bond angles in compounds **I**, **II**·2PhH, and **III**·MeCN

Bond	*d*, Å	Angle	ω, deg
		I	
Sb(1)–C(1)	2.122(8)	C(1)Sb(1)O(1)	86.0(3)
Sb(1)–O(1)	2.137(5)	C(1)Sb(1)O(5)	84.6(3)
Sb(1)–O(5)	2.143(6)	O(1)Sb(1)O(5)	170.6(2)
Sb(1)–C(11)	2.082(8)	C(11)Sb(1)C(1)	110.1(3)
Sb(1)–C(21)	2.094(9)	C(11)Sb(1)O(1)	92.1(3)
C(37)–O(1)	1.306(9)	C(11)Sb(1)O(5)	91.9(2)
C(37)–O(1)	1.234(8)	C(11)Sb(1)C(21)	137.5(4)
C(47)–O(5)	1.318(8)	C(21)Sb(1)C(1)	112.5(4)
C(47)–O(6)	1.227(8)	C(21)Sb(1)O(1)	91.2(3)
		C(21)Sb(1)O(5)	91.6(3)
		II·2PhH	
Sb(1)–O(8)	2.185(2)	O(1)Sb(1)O(8)	175.29(6)
Sb(1)–O(1)	2.161(2)	C(21)Sb(1)O(8)	88.70(10)
Sb(1)–C(21)	2.115(3)	C(21)Sb(1)O(1)	90.94(10)
Sb(1)–C(11)	2.116(3)	C(21)Sb(1)C(11)	138.79(11)
Sb(1)–C(1)	2.131(3)	C(21)Sb(1)C(1)	112.06(12)
O(8)–C(47)	1.308(3)	C(11)Sb(1)O(8)	91.12(10)
O(9)–C(47)	1.246(3)	C(11)Sb(1)O(1)	92.24(10)
		C(11)Sb(1)C(1)	109.14(11)
		C(1)Sb(1)O(8)	88.77(10)
		C(1)Sb(1)O(1)	87.01(10)

Bond	*d*, Å	Angle	ω, deg	Angle	ω, deg
			III·MeCN		
Sb(1)–O(8)	2.263(4)	O(1)Sb(1)O(8)	177.27(15)	O(1)Sb(2)O(12)	173.77(16)
Sb(1)–O(1)	1.925(4)	O(1)Sb(1)C(21)	91.46(18)	O(1)Sb(2)C(41)	93.59(18)
Sb(1)–C(21)	2.102(5)	O(1)Sb(1)C(11)	98.80(18)	O(1)Sb(2)C(61)	89.92(19)
Sb(1)–C(11)	2.106(5)	O(1)Sb(1)C(1)	94.6(2)	O(1)Sb(2)C(51)	92.3(2)
Sb(1)–C(1)	2.101(5)	C(21)Sb(1)O(8)	86.21(16)	C(41)Sb(2)O(12)	91.51(18)
Sb(2)–O(12)	2.214(4)	C(21)Sb(1)C(11)	122.8(2)	C(61)Sb(2)O(12)	91.30(19)
Sb(2)–O(1)	1.936(4)	C(11)Sb(1)O(8)	81.31(16)	C(61)Sb(2)C(41)	113.7(2)
Sb(2)–C(41)	2.104(5)	C(1)Sb(1)O(8)	87.69(18)	C(61)Sb(2)C(51)	119.9(2)
Sb(2)–C(61)	2.100(5)	C(1)Sb(1)C(21)	113.4(2)	C(51)Sb(2)O(12)	81.81(19)
Sb(2)–C(51)	2.103(5)	C(1)Sb(1)C(11)	121.5(2)	C(51)Sb(2)C(41)	126.0(2)
		Sb(1)O(1)Sb(2)	178.1(2)		

metry of the coordination of carboxylate ligands. The lengths of C–O single and C=O double bonds in carboxyl groups are also different: 1.306(9), 1.318(8) and 1.234(8), 1.227(8) Å (**I**) and 1.308(3), 1.307(3) and 1.246(3), 1.247(3) Å (**II**). The molecules of **I** and **II** have two and one intramolecular hydrogen bonds, respectively, between the hydroxyl groups and carboxyl oxygen atoms with the following parameters: O–H, 0.82–0.91 (**I**); 0.82 Å (**II**); O···H, 1.72–1.85 (**I**); 1.81, 1.84 Å (**II**); O···O, 2.54(1)–2.58(2) (**I**); 2.545(2), 2.568(2) Å (**II**); OHO, 146°–157° (**I**); 147° (**II**).

The crystal of compound **II** contains numerous intra- and intermolecular bonds between the oxygen atoms of the nitro group and carboxyl and hydroxyl groups. Solvate molecules of benzene do not take part in the formation of intra- and intermolecular bonds.

In the molecule of **III** (Fig. 2), the Sb(1)O(1)Sb(2) angle differs from 180° and amounts to 178.1(2)°; the

III

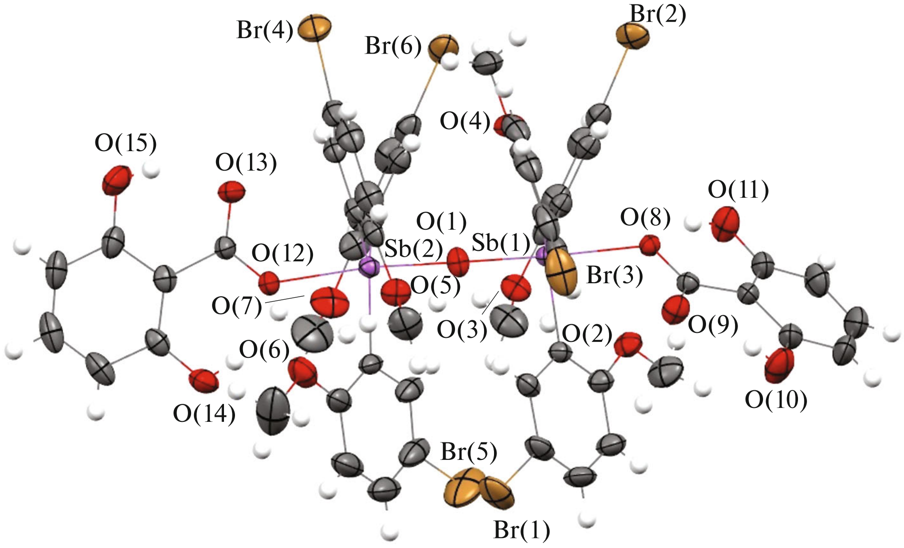


Fig. 2. Molecular structure of compound **III**·MeCN (acetonitrile solvate molecule is not shown).

CSb(1,2)C equatorial angles vary in the ranges of 113.4(2)°–122.8(2)° and 113.7(2)°–126.0(2)°; their sums differ insignificantly from the theoretical value (357.7(2)°, 359.6(2)°). The $O_tSb(1,2)O_b$ axial angles are 177.27(15)° and 173.77(16)°. The $O_tSb(1,2)C$ bond angles, amounting to 81.31(16)°–87.69(18)° and 81.81(19)°–91.51(18)°, differ somewhat from 90°. The Sb(1,2)–C distances are 2.101(5)–2.106(5) and 2.100(5)–2.104(5) Å. The $Sb(1,2)–O(1)_b$ bond lengths (1.925(4) and 1.936(4) Å) are substantially shorter than the $Sb(1,2)–O_t$ bond lengths, which are 2.263(4) and 2.214(4) Å. In **III**, there are intramolecular hydrogen bonds between free hydroxyl groups and the carboxyl oxygen atoms with the following parameters: O–H, 0.82 Å; O···H, 1.82–1.89 Å; O···O, 2.54(9)–2.57(5) Å; OHO, 138°–147°.

Thus, it was established that reactions of tri(*meta*-tolyl)antimony with 2,6-dihydroxybenzoic acid and triphenylantimony with 3,5-dinitrosalicylic acid involve only the carboxyl group and give triarylantimony dicarboxylates, in which the antimony atoms have a slightly distorted trigonal-bipyramidal coordination. The reaction of tris(5-bromo-2-methoxyphenyl)antimony with 2,6-dihydroxybenzoic acid also involves only the carboxyl group, but gives a different product: binuclear organoantimony compound with a bridging oxygen atom, in which the antimony atoms also have a distorted trigonal-bipyramidal coordination.

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CONFLICT OF INTEREST

The authors declare that they have no conflicts of interest.

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