

SYNTHESIS AND STRUCTURE OF μ_2 -OXO-BIS [(PENTAFLUOROPROPIONATO)-TRIS(5-BROMO-2-METHOXYPHENYL)ANTIMONY]

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The synthesis of μ_2 -oxo-bis[(pentafluoropropionato)tris(5-bromo-2-methoxyphenyl)antimony] (**1**) has been carried out by the oxidative addition reaction of tris(5-bromo-2-methoxyphenyl)antimony with pentafluoropropionic acid in the presence of hydrogen peroxide at an equimolar ratio of reactants. The compound is characterized by IR spectroscopy and X-ray diffraction analysis. According to X-ray diffraction data, there are two types of crystallographically independent molecules with slightly different geometrical parameters in the crystal. The antimony atoms in each molecule are linked by the bridging oxygen atom (the SbOSb angle is 167.6(7)°). The trigonal bipyramidal coordination of antimony atoms is distorted; the axial OSbO angles are 175.9(4)°, 174.5(4)° for Sb(1) and Sb(2) atoms, respectively. The sums of the valence CSbC angles in the equatorial planes are 359.6(6)° and 356.4(6)°. The mean values of the Sb(1)–C and Sb(2)–C bond lengths are 2.12(2) Å, the Sb–O(μ_2) distances are 1.89(1), 1.98(1) Å. The carboxylate ligands in each molecule are monodentate. The distances between the antimony atoms and the corresponding terminal oxygen atoms are 2.22(1), 2.39(1) Å, the Sb···O=C distances are 3.38(2), 3.71(1) Å. The d(Sb···O=C)/d(Sb–O) ratios, which can be used to estimate the asymmetry of the ligand related to metal atom coordination, are 1.67 and 1.41. In crystal **1** there are such contacts as Sb···OMe (2.88(1)–3.16(1), 3.01(1)–3.20(1) Å), hydrogen bonds involving halogen atoms H···F (2.16, 2.50, 2.58 Å), H···Br (2.92, 2.96, 3.04 Å) and carbonyl oxygen atoms H···O (2.43, 2.59 Å), as well as halogen···halogen F···F interactions (2.70, 2.71 Å).

Keywords: tris(5-bromo-2-methoxyphenyl)antimony, pentafluoropropionic acid, oxidative addition, structure, X-ray diffraction analysis, IR spectroscopy.

Introduction

It is known that triarylantimony complexes with carboxylate ligands are biologically active compounds, having antibacterial [1, 2], antileishmanial [2–5], photoluminescent [6] properties; they act as photocatalysts [7] and reagents in organic synthesis [8]. Various triarylantimony dicarboxylates Ar_3SbX_2 were obtained by substitution [1–3, 5–8] and oxidative addition reactions, with the molar ratio of triaryl antimony and carboxylic acid 1:2 [4, 9–11]. Synthesis of binuclear triarylantimony carboxylates with monodentate ligands has been described in a few papers only [12–17]. It is obvious that such compounds have not been studied enough, and a further study of the oxidative addition reactions of triarylantimony with carboxylic acids with an equimolar ratio of reagents, as well as the structure determination of the products obtained is of interest.

The present work is related to the study of the interaction of tris(5-bromo-2-methoxyphenyl)antimony with pentafluoropropionic acid in the presence of hydrogen peroxide at 1:1:1 molar ratio of the reactants and the structure determination of the reaction product.

Experimental

Synthesis of μ_2 -oxo-bis[(pentafluoropropionato)tris(5-bromo-2-methoxyphenyl)antimony] (**1**)

Tris(5-bromo-2-methoxyphenyl)antimony (0.2 g, 0.29 mmol) and pentafluoropropionic acid (0.048 g, 0.29 mmol) were dissolved in 10 ml of diethyl ether, then 30 % aqueous solution of hydrogen peroxide (0.033 g, 0.29 mmol) was added. The mixture was kept for 24 h at 20 °C. 0.221 g (89 %) of colorless crystals of **1** with MP 203 °C was obtained.

IR spectrum, ν , cm^{-1} : 1719, 1701, 1576, 1476, 1439, 1376, 1314, 1283, 1254, 1207, 1169, 1151, 1092, 1053, 1024, 881, 864, 808, 727, 708, 676, 619, 538, 521, 440, 424.

Found, %: C 33.85, H 2.16. For $C_{38}H_{31}Br_3N_4O_9Sb$ calculated, %: C 33.88, H 2.13.

Химия элементоорганических соединений

IR spectra of compound **1** were recorded on a Shimadzu IRAffinity-1S FTIR-spectrometer; samples were prepared by pelleting with KBr (absorption region 4000–400 cm⁻¹).

X-ray diffraction analysis of crystalline substance **1** was performed on a Bruker *D8 QUEST* automatic four-circle diffractometer (Mo K α -emission, λ 0.71073 Å, graphite monochromator).

Data collection and editing, unit-cell parameters refinement, and correction for absorption were carried out in *SMART* and *SAINT-Plus* software [18]. All calculations aimed at solving and refining the structure of compound **1** were performed in *SHELXL/PC* software [19]. Structure **1** was determined by direct methods and refined with the least squares method in the anisotropic approximation for non-hydrogen atoms. Selected bond lengths and bond angles of **1** are summarized in Table 1.

Crystal Data for C₉₆H₇₂O₂₂F₂₀Br₁₂Sb₄ ($M = 3403.46$ g/mol): triclinic, space group $P\bar{1}$, a 13.156(7) Å, b 17.932(11) Å, c 27.102(12) Å, α 90.05(3)°, β 90.02(2)°, γ 96.75(3)°, V 6349(6) Å³, Z 2, μ_{Mo} 4.705 mm⁻¹, D_{calc} 1.780 g/cm³, 130469 reflections measured, 25008 unique reflections (R_{int} 0.1675), the number of refinement variables 1399, *GOOF* 1.016, R factors for $F^2 > 2\sigma(F^2)$: R_1 0.1012, wR_2 0.2748, R factors for all reflections R_1 0.1820, wR_2 0.3267.

The full tables of atomic coordinates, bond lengths, and bond angles were deposited with the Cambridge Crystallographic Data Centre (CCDC 1912122 for compound **1**; deposit@ccdc.cam.ac.uk; http://www.ccdc.cam.ac.uk).

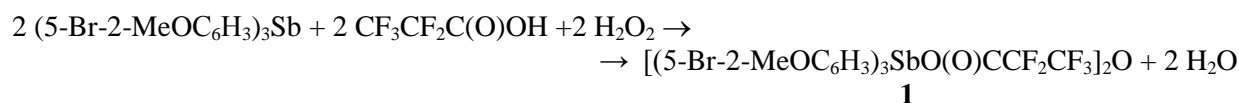
Table 1

Selected bond lengths and bond angles in structure 1

<i>a</i>					
Bond	d , Å	Angle	ω , deg	Angle	ω , deg
Sb(1)–C(1)	2.12(1)	Sb(1)O(6)Sb(2)	167.6(7)	O(1)Sb(1)C(1)	89.6(5)
Sb(1)–C(11)	2.08(2)	O(6)Sb(1)O(1)	175.9(4)	O(1)Sb(1)C(11)	101.2(5)
Sb(1)–C(21)	2.17(1)	O(6)Sb(2)O(10)	174.5(4)	O(10)Sb(2)C(31)	98.4(5)
Sb(2)–C(31)	2.11(2)	C(1)Sb(1)C(11)	117.0(6)	O(10)Sb(2)C(41)	73.8(5)
Sb(2)–C(41)	2.15(1)	C(11)Sb(1)C(21)	118.2(6)	O(10)Sb(2)C(51)	78.4(5)
Sb(2)–C(51)	2.11(2)	C(1)Sb(1)C(21)	124.4(6)	O(6)Sb(1)C(1)	87.2(5)
Sb(1)–O(1)	2.22(1)	C(31)Sb(2)C(51)	126.1(6)	O(6)Sb(1)C(11)	82.5(6)
Sb(1)–O(6)	1.98(1)	C(41)Sb(2)C(31)	113.8(6)	O(6)Sb(1)C(21)	105.4(5)
Sb(2)–O(10)	2.39(1)	C(41)Sb(2)C(51)	116.5(6)	O(6)Sb(2)C(41)	101.7(5)
Sb(2)–O(6)	1.89(1)	O(1)Sb(1)C(21)	74.5(5)	O(6)Sb(2)C(51)	101.2(5)
<i>b</i>					
Sb(3)–C(61)	2.08(2)	Sb(3)O(17)Sb(4)	167.8(7)	O(12)Sb(3)C(71)	89.2(5)
Sb(3)–C(71)	2.13(1)	O(12)Sb(3)O(17)	175.9(4)	O(12)Sb(3)C(81)	74.8(5)
Sb(3)–C(81)	2.16(1)	O(17)Sb(4)O(21)	175.2(4)	O(21)Sb(4)C(91)	98.1(5)
Sb(4)–C(91)	2.11(1)	C(61)Sb(3)C(71)	115.7(6)	O(21)Sb(4)C(101)	78.4(5)
Sb(4)–C(101)	2.12(2)	C(61)Sb(3)C(81)	119.8(6)	O(21)Sb(4)C(111)	73.4(5)
Sb(4)–C(111)	2.16(2)	C(71)Sb(3)C(81)	124.1(5)	O(17)Sb(3)C(61)	82.6(6)
Sb(3)–O(12)	2.21(1)	C(91)Sb(4)C(101)	125.2(6)	O(17)Sb(3)C(71)	87.6(5)
Sb(3)–O(17)	1.98(1)	C(91)Sb(4)C(111)	114.1(6)	O(17)Sb(3)C(81)	104.9(5)
Sb(4)–O(21)	2.38(1)	C(101)Sb(4)C(111)	116.7(6)	O(17)Sb(4)C(91)	85.8(6)
Sb(4)–O(17)	1.89(1)	O(12)Sb(3)C(61)	101.1(6)	O(17)Sb(4)C(111)	102.5(5)

Results and Discussion

It has been found that the oxidative addition reaction of *tris*(5-bromo-2-methoxyphenyl)antimony with pentafluoropropionic acid in the presence of hydrogen peroxide at 1:1:1 molar ratio with the formation of binuclear organic antimony compound **1** with μ_2 -bridging oxygen atom:



Compound **1** is a crystalline substance, highly soluble in aromatic and aliphatic hydrocarbons, resistant to moisture and air oxygen.

Structure **1** has been determined by X-ray diffraction analysis and confirmed by IR spectroscopy.

In the IR spectrum of compound **1** there are bands at 521 cm^{-1} , characterizing the Sb–C vibrations [20], and at 440 cm^{-1} due to the Sb–O vibrations [21]. The intense absorption band at 1719 cm^{-1} characterizes the carbonyl group C=O stretching vibrations, the band at 1375 cm^{-1} does it for the C–F bonds. The absorption bands at 1169 cm^{-1} and 1283 cm^{-1} correspond to vibrations of the C_{Ar}–Br and C_{Ar}–OMe bonds, respectively.

According to X-ray diffraction data, in crystal **1** there are two types of crystallographically independent molecules **a** and **b**, the geometric parameters of which are equal within the error limits, therefore, in the following, we discuss the structural data of molecule **1 a**. The antimony atoms in **1** have a distorted trigonal-bipyramidal coordination with oxygen atoms in axial positions (Fig. 1). The antimony atoms in the binuclear molecule are linked by the bridging oxygen atom, the Sb(1)O(6)Sb(2) bond angle is $167.6(7)^\circ$. The sums of the CSb(1)C and CSb(2)C valence angles in the equatorial planes are $359.6(6)^\circ$ and $356.4(6)^\circ$, respectively, while the values of the individual angles differ from the theoretical 120° by no more than $6.2(6)^\circ$. The Sb(1,2) atoms deviate from the corresponding equatorial planes by 0.071 and 0.233 Å to the direction of the bridging oxygen atom. The axial OSb(1,2)O angles are $175.9(4)^\circ$, $174.5(4)^\circ$. The $O_{\text{term}}\text{Sb}(1,2)\text{C}$ angles vary within the ranges $74.5(5)^\circ$ – $101.2(5)^\circ$, $73.8(5)^\circ$ – $98.4(5)^\circ$, $O(\mu_2)\text{Sb}(1,2)\text{C}$ – $82.5(6)^\circ$ – $105.4(5)^\circ$, $86.3(6)^\circ$ – $101.7(5)^\circ$.

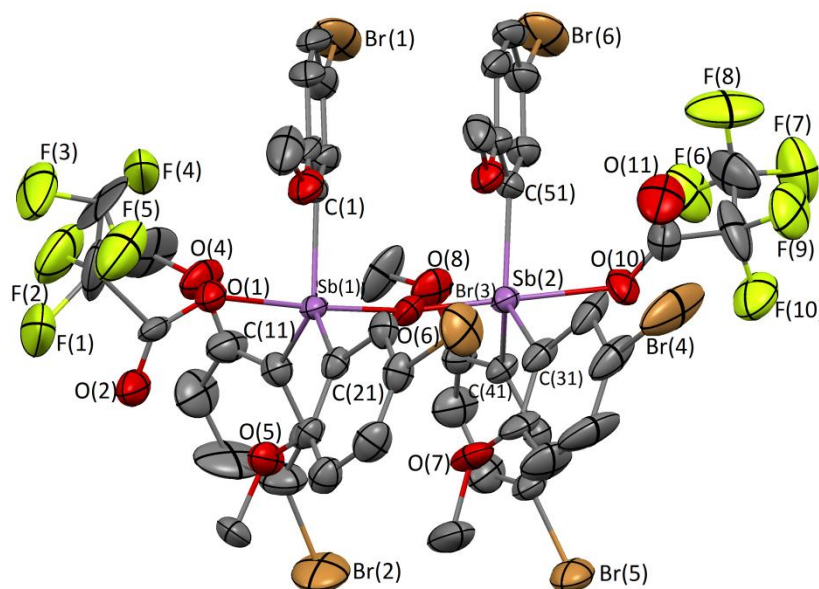


Fig. 1. Structure **1a** showing thermal ellipsoids at 30% probability. Hydrogen atoms have been omitted for clarity

The average values of the Sb(1)–C and Sb(2)–C bond lengths are $2.12(2)\text{ \AA}$. The Sb(1,2)– $O(\mu_2)$ distances ($1.98(1)$, $1.89(1)\text{ \AA}$) are less than Sb(1,2)– O_{term} ($2.22(1)$, $2.39(1)\text{ \AA}$) and less than the sum of covalent radii of the antimony and oxygen atoms 2.07 \AA [22].

Carboxylate ligands show anisobidentate properties, coordinated by a carbonyl oxygen atom to the antimony atom, the Sb(1,2)···O=C distances are $3.71(1)$, $3.38(2)\text{ \AA}$. The coordination asymmetry of ligands can be estimated by the $d(\text{Sb}\cdots\text{O}=\text{C})/d(\text{Sb}-\text{O}_{\text{term}})$ ratio, which is equal to 1.67 and 1.41.

In the molecule there are contacts Sb(1,2)···OMe, the corresponding distances equal $2.88(1)$ – $3.16(1)$, $3.01(1)$ – $3.20(1)\text{ \AA}$.

Molecules **a** and **b** in crystal **1** are linked by intermolecular contacts $\text{F}\cdots\text{H}_{\text{Ar}}$ (2.50 , 2.58 \AA), $\text{F}\cdots\text{H}_{\text{Me}}$ (2.16 \AA). The structural organization of **1** is due to hydrogen bonds, involving oxygen atoms of carboxylate ligands $\text{H}_{\text{Ar}}\cdots\text{O}(\text{C})$ (2.43 , 2.59 \AA) and bromine atoms $\text{H}_{\text{Me}}\cdots\text{Br}$ (2.92 , 2.96 , 3.04 \AA), as well as $\text{F}\cdots\text{F}$ contacts (2.70 , 2.71 \AA) of the first type (according to the classification given in [23]), $\theta_1, \text{CF}(7)\text{F}(3)$ ($124(2)^\circ$) \approx $\theta_2, \text{CF}(3)\text{F}(7)$ ($125(4)^\circ$), $\theta_1, \text{CF}(12)\text{F}(17)$ ($116(4)^\circ$) \approx $\theta_2, \text{CF}(17)\text{F}(12)$ ($117(2)^\circ$) (Fig. 2).

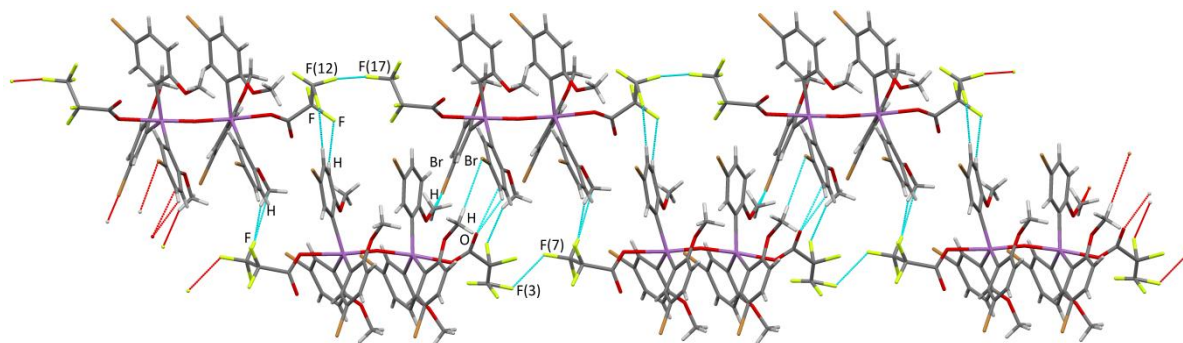


Fig. 2. The intermolecular interactions in 1

Conclusion

The oxidative addition reaction of *tris*(5-bromo-2-methoxyphenyl)antimony with pentafluoropropionic acid at 1:1 molar ratio in the presence of hydrogen peroxide leads to formation of the binuclear compound with a bridging oxygen atom, the structural organization of which is due to hydrogen bonds and contacts with halogen atoms.

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СИНТЕЗ И СТРОЕНИЕ μ_2 -ОКСО- БИС[(ПЕНТАФТОРПРОПИОНАТО)ТРИС(5-БРОМ-2- МЕТОКСИФЕНИЛ)СУРЬМЫ]

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По реакции окислительного присоединения трис(5-бром-2-метоксифенил)сурьмы с пентафторпропионовой кислотой в присутствии пероксида водорода при эквимольном соотношении реагентов синтезирована μ_2 -оксо-бис[(пентафторпропионато)трис(5-бром-2-

метоксифенил)сурьма] (**1**). Соединение охарактеризовано методами ИК-спектроскопии и рентгеноструктурного анализа. Согласно данным рентгеноструктурного анализа, в кристалле находятся два типа кристаллографически независимых молекул, геометрические параметры которых незначительно отличаются. Атомы сурьмы в молекуле связаны мостиковым атомом кислорода (угол SbOSb составляет 167,6(7)°). Тригонально-бипирамидальная координация атомов сурьмы искажена, аксиальные углы OSbO равны 175,9(4)°, 174,5(4)° для атомов Sb(1) и Sb(2) соответственно. Суммы валентных углов CSbC в экваториальных плоскостях составляют 359,6(6)° и 356,4(6)°. Средние значения длин связей Sb(1)–C и Sb(2)–C равны 2,12(2) Å, расстояния Sb–O(μ_2) – 1,89(1), 1,98(1) Å. Карбоксилатные лиганды в молекуле монодентатны. Расстояния между атомами сурьмы и соответствующими терминальными атомами кислорода равны 2,22(1), 2,39(1) Å, расстояния Sb···O=C равны 3,38(2), 3,71(1) Å. Отношения d(Sb···O=C)/d(Sb–O), которыми можно оценить асимметрию координации лигандов на атом металла, составляют 1,67 и 1,41. В кристалле **1** наблюдаются контакты Sb···OMe (2,88(1)–3,16(1), 3,01(1)–3,20(1) Å), водородные связи с участием атомов галогенов H···F (2,16; 2,50; 2,58 Å), H···Br (2,92; 2,96; 3,04 Å) и карбонильных атомов кислорода H···O (2,43; 2,59 Å), а также взаимодействия галоген···галоген F···F (2,70; 2,71 Å).

Ключевые слова: трис(5-бром-2-метоксифенил)сурьма, пентафторпропионовая кислота, окислительное присоединение, строение, рентгеноструктурный анализ.

Литература

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