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SYNTHESIS AND STRUCTURE OF *BIS*(THIOPHENE-2-ALDOXIMATO)-*TRIS*(5-BROMO-2-METHOXYPHENYL)ANTIMONY

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Synthesis of *bis*(thiophene-2-aldoximato)*tris*(5-bromo-2-methoxyphenyl)antimony (**1**) has been carried out by the oxidative addition reaction of *tris*(5-bromo-2-methoxyphenyl)antimony with thiophene-2-aldoxime in the presence of *tert*-butyl hydroperoxide with the 1:2 molar ratio of the reactants. The compound has been characterized by IR spectroscopy and X-ray diffraction analysis. According to the X-ray diffraction analysis data, in the crystal there are two types of crystallographically independent the molecules, geometrical parameters of which are slightly different. Coordination polyhedron of antimony atoms in a molecule is a distorted trigonal bipyramid. The sum of the CSbC angles equals 360°, the values of the individual angles differ from the theoretical 120° by no more than 8.6(8)°. The axial OSbO angle is 175.8(4)°. The OSbC angles vary within the range 85.8(6)º–97.6(6)º. The average value of the Sb–C bond lengths is 2.13(2) Å. The Sb–O distances equal 2.08(1) Å. The distances between the Sb atom and N atoms of the iminoxy groups are 2.80(2)–2.94(2) Å. The distances between the N and O atoms do not depend on the distances between the Sb and N atoms; they are equal to 1.39(2)–1.43(2) Å. In the molecules there are contacts between the Sb and O atoms of methoxy groups, the corresponding distances are within the range of 3.13(1)–3.23(1) Å. The molecules in a crystal are connected by intermolecular hydrogen bonds between the aromatic H and Br (2.883 Å), S (2.992 Å) and N (2.715 Å) atoms. In the molecules there are intramolecular short contacts between the iminoxy group O atom and S (2.72(1)–2.80(1) Å), as well as the methoxy group O atom (2.93(2)–3.03(2) Å).

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

Introduction

It is known that triarylantimony dioximates are biologically active compounds, having antibacterial, antifungal [1] and antitumor [2, 3] activity. Various triarylantimony dioximates Ar_3SbX_2 (Ar = Ph, *p*-Tol, *o*-Tol, *m*-Tol, 3-F-C_6H_4, 4-F-C_6H_4; X = ONCHR, ONCRR') were obtained by substitution [1, 2, 4–7] and oxidative addition reactions, with the molar ratio of triaryl antimony and oxime 1:2 [8–18]. Synthesis of *tris*(5-bromo-2-methoxyphenyl)antimony oximates has been described in a few papers only [17–19]. Obviously, such compounds have not been studied enough, and a further investigation is required.

The present work concerns the study of the interaction of *tris*(5-bromo-2-methoxyphenyl)antimony with thiophene-2-aldoxime in the presence of *tert*-butyl hydroperoxide at 1:2:1 molar ratio of the reactants, and the structure determination of the reaction product.

Experimental

Synthesis of *bis*(thiophene-2-aldoximato)*tris*(5-bromo-2-methoxyphenyl)antimony (1).

Tris(5-bromo-2-methoxyphenyl)antimony (0.1 g, 0.14 mmol) and thiophene-2-aldoxime (0.037 g, 0.29 mmol) were dissolved in 10 ml of diethyl ether, then 70 % aqueous solution of *tert*-butyl hydroperoxide (0.019 g, 0.14 mmol) was added. The mixture was kept for 24 h at 20 °C. After the solvent evaporation, the solid residue was recrystallized from amyl acetate. 0.151 g (97 %) of colorless crystals of **1** with MP 136 °C was obtained.

IR spectrum, ν, cm^{-1}: 3438, 3096, 3065, 3003, 2961, 2932, 2837, 1572, 1472, 1437, 1420, 1375, 1351, 1283, 1269, 1254, 1209, 1180, 1144, 1092, 1049, 1016, 912, 862, 823, 808, 741, 711, 667, 619, 602, 555, 525, 467, 436.

Found, %: C 39.94, H 2.81. For $C_{62}H_{52}Br_6N_4O_{10}S_4Sb_2$ calculated, %: C 40.00, H 2.75.

IR spectra of compound **1** were recorded on a Shimadzu IRAffinity-1S FTIR-spectrometer; samples were prepared by pelletting 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 [20]. All calculations aimed at solving and refining the structure of compound **1** were performed in *SHELXL/PC* [21] and *OLEX2* software [22]. 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 $C_{62}H_{52}N_4O_{10}Br_6Sb_2S_4$ (*M* 1864.28 g/mol): triclinic, space group Pī, *a* 9.565(10) Å, *b* 17.472(18) Å, *c* 24.42(3) Å, α 97.25(7)°, β 92.12(8)°, γ 98.46(6)°, *V* 3999(8) Å^3, *Z* 2, μ_{Mo} 3.827 mm^{-1}, D_{calc} 1.548 g/cm^3, 29317 reflections measured, 5456 unique reflections (R_{int} 0.0522), the number of refinement variables 800, *GOOF* 1.118, *R* factors for $F^2 > 2\sigma(F^2)$: R_1 0.0580, wR_2 0.1635, *R* factors for all reflections R_1 0.0680, wR_2 0.1700.

The full tables of atomic coordinates, bond lengths, and bond angles were deposited with the Cambridge Crystallographic Data Centre (CCDC 1901674 for compound **1**; deposit@ccdc.cam.ac.uk; http://www.ccdc.cam.ac.uk).

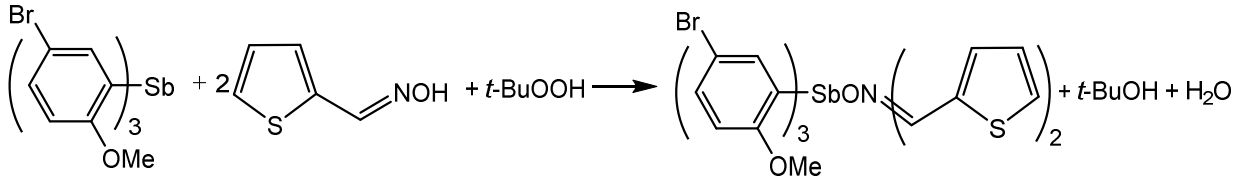
Table 1

Selected bond lengths and bond angles in structure 1

		a			
Bond	*d*, Å	Angle	ω, deg	Angle	ω, deg
Sb(1)–C(1)	2.15(2)	O(4)Sb(1)O(5)	175.8(4)	O(4)Sb(1)C(11)	87.3(6)
Sb(1)–C(11)	2.14(2)	C(1)Sb(1)C(11)	120.6(7)	O(4)Sb(1)C(21)	85.8(6)
Sb(1)–C(21)	2.11(2)	C(1)Sb(1)C(21)	112.5(7)	O(5)Sb(1)C(1)	86.1(6)
Sb(1)–O(4)	2.08(1)	C(11)Sb(1)C(21)	126.9(7)	O(5)Sb(1)C(11)	92.7(6)
Sb(1)–O(5)	2.08(1)	O(4)Sb(1)C(1)	97.6(6)	O(6)Sb(1)C(21)	90.8(6)
N(1)–C(35)	1.30(3)	O(4)N(1)C(35)	110(1)		
N(2)–C(45)	1.28(3)	O(5)N(2)C(45)	113(2)		
		b			
Sb(2)–C(51)	2.16(2)	O(I)Sb(2)O(J)	175.2(5)	O(I)Sb(2)C(61)	87.8(6)
Sb(2)–C(61)	2.12(2)	C(51)Sb(2)C(61)	125.9(8)	O(I)Sb(2)C(71)	85.2(6)
Sb(2)–C(71)	2.13(2)	C(51)Sb(2)C(71)	122.5(7)	O(J)Sb(2)C(51)	89.4(6)
Sb(2)–O(I)	2.10(1)	C(71)Sb(2)C(81)	111.4(8)	O(J)Sb(2)C(61)	87.6(6)
Sb(2)–O(J)	2.04(1)	O(I)Sb(2)C(51)	92.2(6)	O(J)Sb(2)C(71)	97.7(6)
N(3)–C(85)	1.23(3)	O(I)N(3)C(85)	112(2)		
N(4)–C(95)	1.21(4)	O(J)N(4)C(95)	113(2)		

Results and Discussion

It has been found that the oxidative addition reaction of *tris*(5-bromo-2-methoxyphenyl)antimony with thiophene-2-aldoxime in the presence of *tert*-butyl hydroperoxide at 1:2:1 molar ratio goes by a standard pathway with the formation of triarylantimony dioximate:



1

Compound **1** is a crystalline substance, highly soluble in aromatic 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 absorption bands at 2932 cm^{-1} (thiophene C–H, *st*), 708, 712 cm^{-1} (thiophene C–H, δ). Characteristic bands are observed at 1472 cm^{-1} (C=N bond),

1375 cm^{-1} (OH, δ), 912 cm^{-1} (N–O) [23]. Vibrations at 436 cm^{-1} indicate the presence of the Sb–C bond in the molecule of compound **1** [24]. The absorption bands at 1180 cm^{-1} and 1283 cm^{-1} correspond to vibrations of the C_{Ar}–Br and C_{Ar}–OMe bonds, respectively [23].

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 have a distorted trigonal-bipyramidal coordination with oxygen atoms in axial positions (Fig. 1). The SbC_3 fragment lying in the equatorial plane is almost flat. The Sb atom deviates from the [C_3] plane toward the axial oxygen atom by 0.001 Å (***a***) and 0.053 Å (***b***). The sum of the CSbC equatorial angles is 360° for both molecules, the values of the individual angles differ from the theoretical 120° by no more than 8.6(8)°. The axial OSbO angle is 175.8(4)°. The OSbC angles vary within the ranges 85.8(6)º–97.6(6)º. The NOON torsion angles accept large values (140(1)° (***a***), 117(1)° (***b***)) because Sb interacts with N from different sides. The angles between SbON planes equal 40.54° (***a***), 63.42° (***b***).

The average values of the Sb–C bond lengths are 2.13(2) Å (***a***) and 2.14(2) (***b***) Å. The Sb–O distances equal 2.08(1) Å what is approximately equal to the sum of covalent radii of Sb and O atoms (2.07(1) Å). The Sb···N distances between the Sb atom and N atoms of iminoxy groups (2.80(2), 2.94(2) Å (***a***), 2.82(2) Å (***b***)) are considerably less than the sum of Van der Waals radii of the Sb and N atoms (3.8 Å [25]). The N–O distances do not depend on Sb···N distances and are equal to 1.43(2) Å (***a***) and 1.39(2), 1.41(2) Å (***b***). The geometrical parameters of **1** are close to the ones for *bis*(thiophene-2-aldoximato)tri(*o*-tolyl)antimony [12]. The average values for N–O (1.41 Å) and N–C (1.26(4) Å) distances, as well as ONC angles (112(2)°) in **1** are close to the ones for thiophene-2-aldehyde (1.394(3) Å, 1.269(8) Å, 111.6(3)°) as well [12].

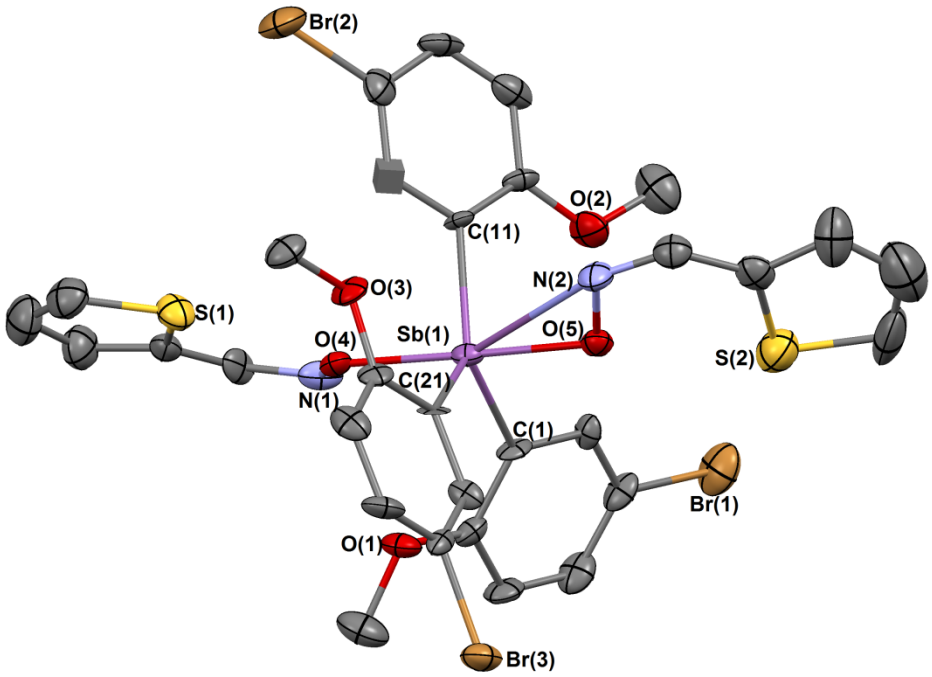


Fig. 1. Structure 1*a* showing thermal ellipsoids at 30% probability. Hydrogen atoms have been omitted for clarity

In molecule **1** there are contacts Sb(1,2)···OMe, the corresponding distances equal 3.18(1)–3.23(1) (***a***), 3.13(1)–3.23(1) Å (***b***).

Molecules ***a*** and ***b*** in crystal **1** are linked by intermolecular contacts H_{Ar}···Br (2.883 Å) and H_{Me}···S (2.992 Å) (Fig. 2). Molecules of ***b*** type are connected via H_{Ar}···N interactions (2.715 Å). In the molecules there are such intramolecular short contacts as S···O (2.80(1) Å (***a***), 2.72(1), 2.74(1) Å (***b***)), O_{MeO}···O (2.97(2), 2.93(2) Å (***a***), 3.03(2), 2.93(2), 2.79(2) Å (***b***)).

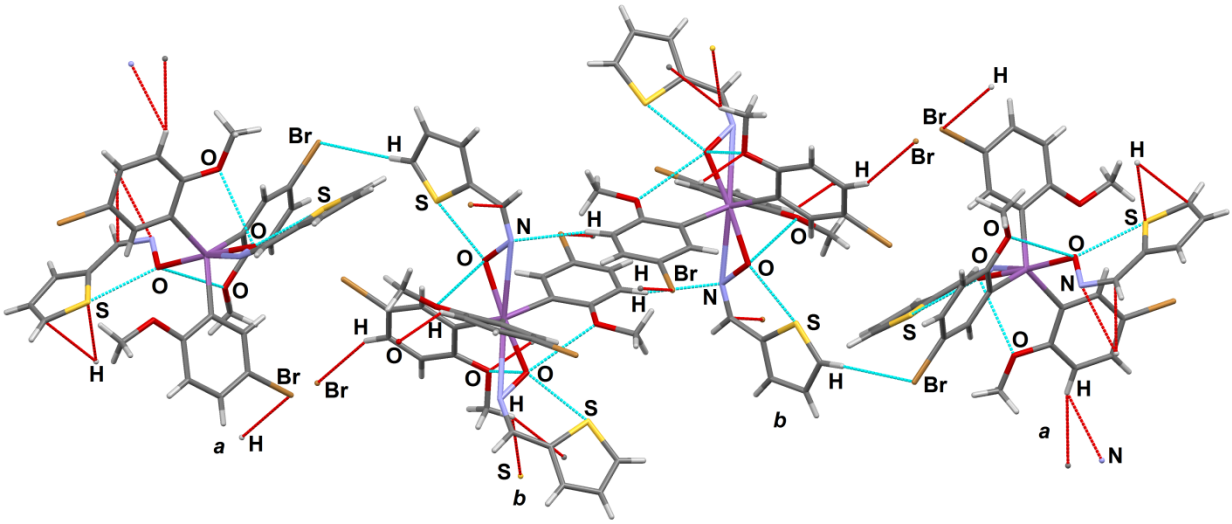


Fig. 2. The intermolecular interactions in 1

Conclusion

The oxidative addition reaction of *tris*(5-bromo-2-methoxyphenyl)antimony with thiophene-2-aldoxime at 1:2 molar ratio leads to formation of *tris*(5-bromo-2-methoxyphenyl)antimony dioximate, the structural organization of which is due to hydrogen bonds and other short contacts.

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СИНТЕЗ И СТРОЕНИЕ *БИС*(ТИОФЕН-2-АЛЬДОКСИМАТО)*ТРИС*(5-БРОМ-2-МЕТОКСИФЕНИЛ)СУРЬМЫ

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По реакции окислительного присоединения *трис*(5-бром-2-метоксифенил)сурьмы с тиофен-2-альдоксимом в присутствии *трет*-бутилгидропероксида при соотношении реагентов 1:2 синтезирована *бис*[(тиофен-2-альдоксимато)*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. Координационный полиэдр атомов сурьмы в молекуле – тригональная бипирамида. Сумма углов CSbC составляет 360°, при этом значения индивидуальных углов отличаются от теоретического 120° не больше, чем на 8,6(8)°. Аксиальные углы OSbO равны 175,8(4)°. Углы OSbC варьируют в пределах 85,8(6)º–97,6(6)º. Средние значения длин связей Sb–C составляют 2,13(2) Å. Расстояния Sb–O равны 2,08(1) Å. Расстояния между атомами Sb и атомами N иминокси-групп составляют 2,80(2)–2,94(2) Å. Расстояния между атомами N и O не зависят от расстояний между атомами Sb и N и равны 1,39(2)–1,43(2) Å. В молекулах имеют место контакты между атомами Sb и атомами O метокси-групп, соответствующие расстояния лежат в пределах 3,13(1)–3,23(1) Å. Молекулы в кристаллах связаны межмолекулярными водородными связями между атомами H и Br (2,883 Å), S (2,992 Å) и N (2,715 Å). В молекулах присутствуют короткие контакты между атомом O иминокси-групп и атомами S (2,72(1)–2,80(1) Å), а также атомами O метокси-групп (2,93(2)–3,03(2) Å).

Ключевые слова: трис(5-бром-2-метоксифенил)сурьма, тиофен-2-альдоксим, окислительное присоединение, строение, рентгеноструктурный анализ, ИК-спектроскопия.

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