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OXIDATION OF TRI(*O*-TOLYL)ANTIMONY BY *TERT*-BUTYL HYDROPEROXIDE. MOLECULAR STRUCTURES OF *BIS*[μ$_2$-OXO- TRI(O-TOLYL)ANTIMONY] AND μ$_2$-OXO-*BIS*[(*TERT*-BUTYLPEROXY)TRI(*O*-TOLYL)ANTIMONY]

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Tri(*o*-tolyl)antimony oxidation by equimolar amount of *tert*-butyl hydroperoxide in diethyl ether led to the formation of *bis*[μ_2-oxo-tri(*o*-tolyl)antimony] (1). At the molar ratio of reactants 1:2 or 1:4 μ$_2$-oxo-*bis*[(*tert*-butylperoxy)tri(*o*-tolyl)antimony] (2) has been formed. According to the X-ray analysis data, antimony atoms are in the trigonal bipyramidal coordination in molecules 1 and 2. The bond lengths Sb–O vary within the ranges 1.937(2)–2.078(2) Å (1) and 1.975(17)–2.216(15) Å (2).

Keywords: tri(ortho-tolyl)antimony, tert-butyl hydroperoxide, oxidation, bis[μ_2-oxo-tri(o-tolyl)antimony], μ_2-oxo-bis[(tert-butylperoxy)tri(o-tolyl)antimony], molecular structures, X-ray analysis.

The interactions between triarylantimony and inorganic and organic oxidizing agents were investigated by many authors, for example [1–4]. The studies of reactions of triarylantimony with hydroperoxides are of great importance, as the products are useful precursors of derivatives with general formula Ar_3SbX_2 (X = acid radical HX) [5–10]. It has been found that hydrogen peroxide oxidizes triarylantimony to produce oxide Ar_3SbO (Ar = Ph, *p*-Tol) or dihydroxide $Ar_3Sb(OH)_2$ (Ar = 2,4,6-$Me_3C_6H_2$), depending on the volume of organic radical bonded with antimony atom, it is believed [10, 11]. The interaction of triarylantimony with *tert*-butyl hydroperoxide was studied on the example of triphenylantimony only. The molecular structure of the reaction product was found to depend on the amount of an oxidizing agent. Triphenylantimony oxide was formed at stoichiometric ratio of reactants and may be dimerized or polymerized [12–14]. Stable antimony peroxides $Ph_3Sb(OOBu\text{-}t)_2$ and $(Ph_3SbOOBu\text{-}t)_2O$ are formed in the presence of the excess of *tert*-butyl hydroperoxide [15].

The reactions of tri(*o*-tolyl)antimony with *tert*-butyl hydroperoxide at various molar ratios of the reactants have been investigated and crystal and molecular structures of the products have been determined in the present paper.

Experimental

Synthesis of *bis*[μ_2-oxo-tri(*o*-tolyl)antimony] (1). Tri(o-tolyl)antimony (200 mg, 0.50 mmol) was dissolved in diethyl ether (20 mL). Then *tert*-butyl hydroperoxide (66 mg of 70 % aqueous solution, 0.50 mmol) was added. The solution was left to stand for 24 hours at temperature 20 °C. When the solvent evaporated, colourless cristalline substance **1** was obtained; the product yield was 199 mg (95 %), MP: 216 °C.

IR spectrum (ν, cm^{-1}): 3048, 2921, 2854, 1584, 1446,1280, 1202, 1160, 1120, 1031,935, 918, 890,764, 750, 740, 655, 636, 491, 471, 435.

Synthesis of μ_2-oxo-*bis*[(*tert*-butylperoxy)tri(*o*-tolyl)antimony] 2. Tri(o-tolyl)antimony (200 mg, 0.50 mmol) was dissolved in diethyl ether (20 mL). Then *tert*-butyl hydroperoxide (132 mg of 70 % aqueous solution, 1.00 mmol) was added. The solution was left to stand for 24 hours at temperature 20 °C. Colorless crystals **2** were obtained; yield 230 mg (92 %), MP: 162 °C.

The reaction with the molar ratio 1:4 was carried out at the same conditons. The product yield of substance **2** was 87 %.

IR spectrum of the substunce **1** was recorded on the Bruker Tensor 27 FT-IR (KBr pellets; 4000–400 cm^{-1}).

The X-ray diffraction analyses of crystalline substances **1** and **2** were made on the Bruker D8 QUEST automatic four-circle diffractometer (Mo K_{α}-emission, $\lambda = 0.71073$ Å, graphite monochromator). The data were collected and analyzed, the unit cell parameters were refined, and the absorption correction was applied using the *SMART* and *SAINT-Plus* programs [16]. All calculations for structure determination and refinement were performed using the *SHELXL/PC* programs [17]. The structures **1** and **2** were determined by the direct method and refined by the least-squares method in the anisotropic approximation for non-hydrogen atoms.

The main crystallographic data and refinement results for structures **1** and **2** are listed in Table 1. The selected bond lengths and bond angles are given in Table 2.

Table 1

Crystallographic data and the experimental and structure refinement parameters for compound 1

Parameter	Value	
	1	**2**
Empirical formula	$C_{42}H_{42}O_2Sb_2$	$C_{50}H_{60}O_5Sb_2$
Formula weight	822.26	984.48
T, K	296(2)	296(2)
Crystal system	Triclinic	Triclinic
Space group	P-1	P1
a, Å	11.0684(3)	10.3355(4)
b, Å	11.1721(3)	11.0049(5)
c, Å	17.0248(5)	11.0848(4)
α, deg	80.7820(10)	69.771(2)
β, deg	86.0600(10)	84.636(2)
γ, deg	61.0370(10)	81.907(2)
V, $Å^3$	1818.06(9)	1169.88(8)
Z	2	1
ρ(calcd.), g/cm^3	1.502	1.397
μ, mm^{-1}	1.520	1.198
F(000)	824.0	502.0
Crystal size, mm	0.17×0.09×0.08	0.55×0.38×0.21
2θ Range of data collection, deg	7.38 – 58.28°	3.98 – 47.5°
Range of refraction indices	$-15 \le h \le 15, -15 \le k \le 15,$ $-23 \le l \le 23$	$-11 \le h \le 11, -12 \le k \le 12,$ $-12 \le l \le 12$
Measured reflections	32558	14775
Independent reflections	9014	6981
R_{int}	0.0480	0.0245
Refinement variables	421	521
GOOF	1.030	1.159
R factors for $F^2 > 2\sigma(F^2)$	$R_1 = 0.0314$, $wR_2 = 0.0551$	$R_1 = 0.0511$, $wR_2 = 0.1274$
R factors for all reflections	$R_1 = 0.0564$, $wR_2 = 0.0611$	$R_1 = 0.0589$, $wR_2 = 0.1361$
Residual electron density (min/max), $e/Å^3$	0.49/–0.33	1.04/–2.17

Table 2

Selected bond lengthes and bond angles in the structures of compounds 1–2

Bond	*d*, Å	Angle	ω, deg	Bond	*d*, Å	Angle	ω, deg
1				**2**			
Sb(1)–Sb(1a)	3.1409(4)	O(1a)Sb(1)C(1)	165.14(10)	Sb(1)–O(1)	1.997(17)	O(1)Sb(1)C(11)	93.7(9)
Sb(1)–O(1)	1.9372(18)	O(1)Sb(1)C(11)	114.54(10)	Sb(1)–C(11)	2.145(14)	O(1)Sb(1)O(2)	167.6(6)
Sb(1)–O(1a)	2.0784(18)	O(1a)Sb(1)C(11)	89.40(9)	Sb(1)–O(2)	2.143(18)	O(1)Sb(1)C(21)	86.7(7)
Sb(1)–C(1)	2180(3)	O(1)Sb(1)C(21)	130.04(10)	Sb(1)–C(21)	2.166(17)	O(1)Sb(1)C(1)	95.3(8)
Sb(1)–C(11)	2.135(3)	C(11)Sb(1) C(1)	103.30(11)	Sb(1)–C(1)	2.18(2)	C(11)Sb(1)C(21)	116.3(10)

Table 2 (end)

Bond	*d*, Å	Angle	ω, deg	Bond	*d*, Å	Angle	ω, deg
1				**2**			
Sb(1)–C(21)	2.150(3)	C(11)Sb(1)C(21)	111.93(11)	Sb(2)–O(1)	1.951(18)	C(11)Sb(1)C(1)	124.4(12)
Sb(2)–Sb(2b)	3.1441(3)	C(21)Sb(1)Sb(1a)	110.58(8)	Sb(2)–C(41)	2.150(17)	O(2)Sb(1)C(11)	87.2(9)
Sb(2)–O(2)	2.0585(17)	C(21)Sb(1)C(1)	96.05(11)	Sb(2)–C(61)	2.114(12)	O(2)Sb(1)C(21)	81.9(7)
Sb(2)–O(2b)	1.9473(17)	O(2b)Sb(2)C(31)	89.87(9)	Sb(2)–C(51)	2.114(12)	O(2)Sb(1)C(1)	94.3(8)
Sb(2)–C(31)	2.186(3)	O(2)Sb(2)C(31)	163.65(9)	Sb(2)–O(4)	2.129(17)	C(1)Sb(1)C(21)	118.9(10)
Sb(2)–C(41)	2.131(3)	O(2b)Sb(2)C(41)	108.70(10)	O(2)–O(3)	1.337(17)	Sb(2)O(1)Sb(1)	169.2(6)
Sb(2)–C(51)	2.151(3)	O(2)Sb(2)C(41)	92.51(10)	O(4)–O(5)	1.356(17)	C(22)C(21)Sb(1)	127.0(19)
O(1)–Sb(1a)	2.0784(18)	O(2b)Sb(2)C(51)	130.20(9)	O(5)–C(35)	1.51(2)	C(46)C(41)Sb(2)	114.2(15)
O(2)–Sb(2b)	1.9473(17)	O(2)Sb(2)C(51)	87.24(9)	O(3)–C(31)	1.451(17)	C(42)C(41)Sb(2)	124.6(15)
Symmetry relation: a) 1–x, –y, 2–z; b) 2–x, 1–y, 1–z							

The full tables of atomic coordinates, bond lengths, and bond angles for the substance **1** was deposited with the Cambridge Crystallographic Data Centre (№ 1052677; deposit@ccdc.cam.ac.uk; http://www.ccdc.cam.ac.uk).

Results and Discussion

It has been found that the oxidation of tri(*o*-tolyl)antimony by *tert*-butylhydroperoxide at the molar ratio 1:1 in diethyl ether goes with the formation of tri(*o*-tolyl)antimony oxide with dimeric structure: *bis*[μ_2-oxo-tri(*o*-tolyl)antimony] (**1**):

$$2\ (o\text{-Tol})_3\text{Sb} + 2\ t\text{-BuOOH} \rightarrow [(o\text{-Tol})_3\text{SbO}]_2 + 2\ t\text{-BuOH}$$

According to X-ray diffraction data the crystal of compound **1** contains two types of crystallographically independent molecules (**A**, **B**). The antimony atoms have intermediate coordination between trigonal-bipyramidal and square-pyramidal coordination (Fig. 1).

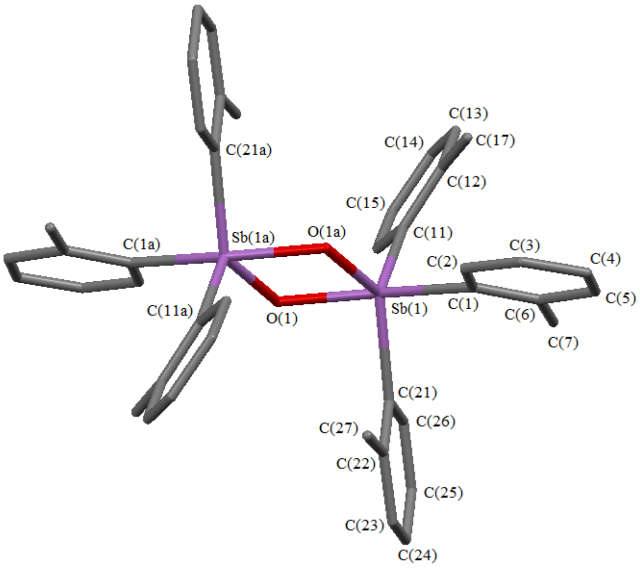

Fig. 1. The structure of compound 1A (hydrogen atoms aren't shown)

Two carbon atoms of the aryl substituents and μ_2-bridging oxygen atom are placed in equatorial plane, the second μ_2-bridging oxygen atom and carbon atom are in axial positions. The sum of equatorial OSbC and CSbC angles is 356.57(10)° for **A** and 350.83(10)° for **B**. The axial OSbC angles are significantly distorted, they are equal to 165.14(10)° and 163.65(9)°. The OSbO and SbOSb angles in the flat cyclic fragment [Sb_2O_2] equal 77.14(8)°, 102.86(8)° (**A**) and 76.62(8)°, 103.13(7)° (**B**). The Sb–C_{eq} bond lengths (2.135(3), 2.150(3) Å **A**; 2.131(3), 2151(3) Å **B**) and Sb–O_{eq} (1.937(2) Å **A**; 1.943(2) Å **B**) are less than Sb–C_{ax} (2.180(3) Å **A**; 2.186(3) Å **B**) and Sb–O_{ax} (2.078(2) Å **A**; 2.058(2) Å **B**). The distances between antimony atoms in the cycle (3.1409(5) (**A**), 3.1441(3) Å (**B**)) are considerably less than the double Van der Waals radius of antimony atom (4.4 Å [18]). The *o*-Tol_3Sb fragments in compound **1** are in staggered conformation with respect to each other. Geometrical parameters of complex **1** are close to geometrical parameters of such compounds as $(Ph_3SbO)_2$ [19] and $[(2\text{-}MeOC_6H_4)_3SbO]_2$ [20].

When the concentration of *tert*-butylhydroperoxide has increased (1:2 or 1:4) the single organoantimony product in the reaction mixture is μ_2-oxo-*bis*[(*tert*-butylperoxo)tri(*o*-tolyl)antimony] (**2**), the product yield is 92 %:

$$2\ (o\text{-Tol})_3Sb + 4\ t\text{-BuOOH} \rightarrow [(o\text{-Tol})_3SbOOBu\text{-}t]_2O + 2\ t\text{-BuOH} + H_2O$$

The coordination polyhedron of antimony atoms in binuclear molecule **2** is an insignificantly distorted trigonal bipyramid (Fig. 2). The bipyramid distortion is characterized by deflection of Sb(1) and Sb(2) atoms from their respective equatorial planes by 0.02 Å and 0.08 Å to the direction of the bridging oxygen atom O(1), which leads to angle deviation between axial and equatorial bonds from the theoretical value 90°. The axial OSb(1,2)O angles are equal to 167.6(6)° and 159.5(5)°.

The equatorial CSb(1,2)C angles are changed in the range of 116.3(8)°–124.4(12)°. The Sb(1)O(1)Sb(2) angle is 169.2(6)°. The SbOSb fragment has linear structure in the centrosymmetric molecule of μ_2-oxo-*bis*[(*tert*-butylperoxo)triphenylantimony] [21].

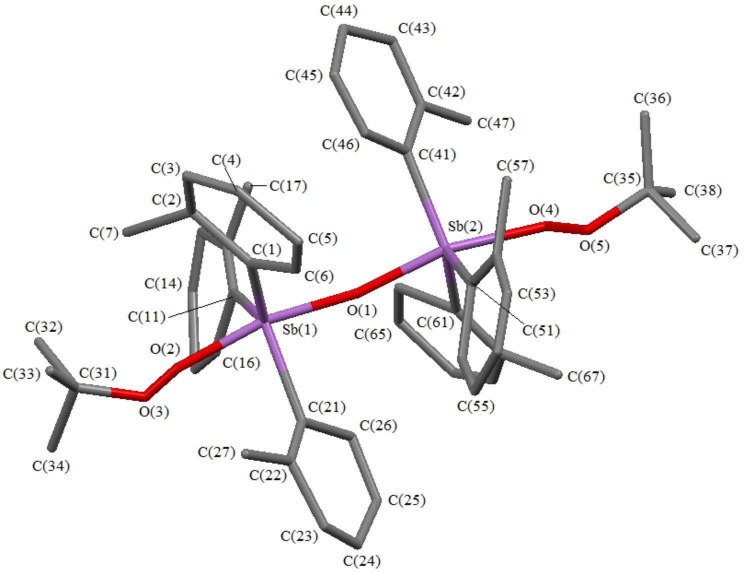

Fig. 2. The structure of compound 2 (hydrogen atoms aren't shown)

The equatorial bonds Sb(1)–C_{eq} and Sb(2)–C_{eq} are changed in the range of 2.14(1)–2.18(2) Å and 2.10(1)–2.15(1) Å.

The Sb(1,2)–O(1) distances are equal to 1.997(7) and 1.951(18) Å, and they are less than terminal distances Sb(1)–O(2) (2.143(18) Å) and Sb(2)–O(4) (2.129(17) Å) like in the molecule of μ_2-oxo-*bis*[(*tert*-butylperoxo)triphenylantimony].

Conclusions

Thus, *tert*-butylhydroperoxide oxidizes tri(*o*-tolyl)antimony at the molar ratio of the reactants 1:1 into tri(*o*-tolyl)antimony oxide, which dimerizes into *bis*[μ_2-oxo-tri(*o*-tolyl)antimony]. With *tert*-butylhydroperoxide in excess (1:2 and 1:4) the reaction proceeds with the formation of the single organoantimony compound: μ_2-oxo-*bis*[(*tert*-butylperoxo)tri(*o*-tolyl) antimony].

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ОКИСЛЕНИЕ ТРИ(*О*-ТОЛИЛ)СУРЬМЫ ТРЕТБУТИЛГИДРОПЕРОКСИДОМ. МОЛЕКУЛЯРНЫЕ СТРУКТУРЫ *БИС*[μ$_2$-ОКСО-ТРИ(*О*-ТОЛИЛ)СУРЬМЫ] И μ$_2$-ОКСО-*БИС*[(*ТРЕТ*БУТИЛПЕРОКСО)ТРИ(*О*-ТОЛИЛ)СУРЬМЫ]

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Окисление три(*о*-толил)сурьмы эквимолярным количеством *трет*бутилгидропероксида в эфире приводит к образованию *бис*[μ$_2$-оксо-три(*о*-толил)сурьмы] (1). При соотношении реагентов 1:2 или 1:4 (мольн.) продуктом реакции является μ_2-оксо-[три(*о*-толил)(*трет*бутилпероксо)-сурьма] (2). По данным РСА, в молекулах 1 и 2 атомы Sb имеют искаженную тригонально-бипирамидальную координацию. Длина связей Sb–O изменяется в интервалах 1.937(2)–2.078(2) Å (1) и 1.975(17)–2.216(15) Å (2).

Ключевые слова: три-о-толилсурьма, третбутилгидропероксид, окисление, бис[μ_2-оксо-три(о-толил)сурьма], μ_2-оксо-бис[(третбутилпероксо)три(о-толил)-сурьма], молекулярные структуры, рентгеноструктурный анализ.

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Oxidation of Tri(*o*-tolyl)antimony by *Tert*-butyl Hydroperoxide. Molecular Structures of *Bis*[μ_2-oxo-tri(o-tolyl)antimony] and μ_2-oxo-*bis*[(*tert*-butylperoxy)tri(*o*-tolyl)antimony] / V.V. Sharutin, O.K. Sharutina, E.V. Artem'eva, M.S. Makerova // Вестник ЮУрГУ. Серия «Химия». – 2015. – Т. 7, № 4. – С. 23–29. DOI: 10.14529/chem150404

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