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SYNTHESIS AND STRUCTURE OF TETRAPHENYLANTIMONYβ-ISATOXIMATE

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(β-Isatoximato)tetraphenylantimony (**1**) was obtained by the reaction of pentaphenylantimony with β-isatoxime. X-ray diffraction analysis showed that the antimony atom in complex **1** had distorted trigonal-bipyramidal coordination..

Keywords: pentaphenylantimony, β-isatoxime, dephenylation, (β-isatoximato)tetraphenylantimony, molecular structures, X-ray diffraction analysis.

Introduction

It is known from the literature sources that pentaarylantimony reacts with inorganic and organic acids HX (X = OR, OAr, OSO_2R, OC(O)R, Hal, NO_3) to form antimony derivatives Ar_4SbX in high yield [1–6]. In some articles the dephenylation of pentaphenylantimony was studied in the reaction with oximes [7–10], leading to formation of tetraphenylantimony oximes. It has been found that when ketoximes are used, the separation of two phenyl groups and the formation of triphenylantimony dioximates are sometimes observed [7].

Another method for the preparation of tetraaryl antimony oxymates is based on a substitution reaction with the use of tetraarylantimony halides. Thus, Ar_4SbX derivatives were synthesized (Ar = Ph, 4-ClC_6H_4) by the reaction of tetraarylantimony bromide with N-hydroxy-demethylcantharimide with the addition of Et_3N; the reaction products were characterized by IR and NMR spectroscopy methods [11]. Similarly, organoantimony arylhydroxamates of the general formula $(4\text{-}CH_3C_6H_4)_4SbX$ were synthesized, the structure of the compounds thus obtained was established by the X-ray diffraction method [12]. By the means of MTT and SRB, it was found that those compounds had antitumor effect [11, 12].

Tetraarylantimony oximates were obtained in high yield as a result of a ligand redistribution reaction of pentaphenyl- and penta(*p*-tolyl)antimony with triarylantimony dioximates [7, 13].

According to X-ray diffraction analysis data, antimony atoms in tetraaryl antimony oximates, as a rule, have distorted trigonal-bipyramidal coordination with the iminoxy group oxygen atom in the axial position. Short distances between the iminoxy group nitrogen atom and the antimony atom are observed [7–10, 13].

The present paper describes further study of the interaction of pentaphenylantimony with oximes, in particular, β-isatoxime, and the establishment of the molecular structure of the obtained product.

Experimental

Synthesis of β-isatoximato tetraphenylantimony (1)

a) 300 mg of pentaphenylantimony (0.59 mmol) and 96 mg (0.59 mmol) of β-isatoxime were dissolved in benzene, sealed in an ampoule and heated for several hours in a water bath until the precipitate dissolved. The mixture was held for several days at 20 °C. After removing the solvent, the solid residue was recrystallized from chloroform with addition of heptane. Yellow crystals with the mass of 0.348 g (99 %) and melting point of 193 °C were obtained.

IR spectrum, ν, cm^{-1}: 3148, 3065, 1705, 1612, 1591, 1551, 1508, 1474, 1458, 1435, 1375, 1340, 1296, 1291, 1182, 1151, 1096, 1057, 1020, 997, 972, 785, 754, 729, 692, 650, 582, 492, 457, 419.

Similarly, an experiment was carried out with 1:2 molar ratio of the reactants. The solid residue was recrystallized from ethanol.

IR spectra were recorded on a Shimadzu IRAffinity-1S FTIR spectrometer (pellets with KBr; 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, $\lambda = 0.71073$ Å, graphite monochromator).

Data collection and editing, unit-cell parameters refinement, and correction for absorption were carried out in *SMART* and *SAINT-Plus* software. All calculations aimed at solving and refining the structure of compound **1** were performed in *SHELXL/PC* software [14, 15]. Structure **1** was determined by direct methods and refined with LS method in the anisotropic approximation for non-hydrogen atoms. The selected crystallographic data and the structure refinement results are listed in Table 1. Selected bond lengths and bond angles are summarized in Table 2.

The full tables of atomic coordinates, bond lengths, and bond angles were deposited with the Cambridge Crystallographic Data Centre (CCDC 1814675 for compound **1** deposit@ccdc.cam.ac.uk; http://www.ccdc.cam.ac.uk).

Table 1

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

Parameter	Value
	1
Empirical formula	$C_{64}H_{50}N_4O_4Sb_2$
Formula weight	1182.65
T, K	293.15
Crystal system	triclinic
Space group	P–1
a, Å	10.106(12)
b, Å	10.340(14)
c, Å	26.82(3)
α, deg	83.36(6)
β, deg	79.45(4)
γ, deg	84.63(7)
V, Å^3	2729(6)
Z	2
$\rho_{(calcd.)}$, g/cm^3	1.4389
μ, mm^{-1}	1.042
F(000)	1190.0
Crystal size, mm	0.42 × 0.13 × 0.1
2θ Range of data collection, deg	5.66 – 49.54
Range of refraction indices	$-11 \le h \le 11$, $-12 \le k \le 12$, $-31 \le l \le 31$
Reflections collected	45577
Independent reflections	9232
R_{int}	0.0583
Refinement variables	667
GOOF	1.045
R factors for $F^2 > 2\sigma(F^2)$	$R_1 = 0.0973$, $wR_2 = 0.2376$
R factors for all reflections	$R_1 = 0.1105$, $wR_2 = 0.2444$
Residual electron density (min/max), *e*/Å^3	2.46/–2.94

Table 2

Selected bond lengths and bond angles in the structure of compound 1

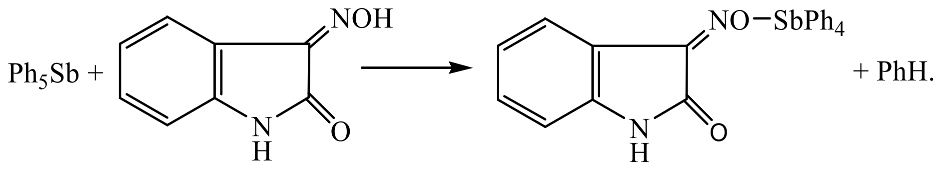
Bond	*d*, Å	Angle	ω, deg
Sb(1)–C(1)	2.128(13)	C(11)Sb(1)O(1)	177.9(4)
Sb(1)–C(21)	2.115(12)	C(1)Sb(1)C(21)	112.9(5)
Sb(1)–C(31)	2.116(13)	C(1)Sb(1)C(31)	117.2(5)
Sb(1)–O(1)	2.231(9)	C(21)Sb(1)C(31)	125.9(5)
Sb(1)–C(11)	2.167(12)	N(1)O(1)Sb(1)	108.5(7)
O(1)–N(1)	1.331(14)	O(1)N(1)C(41)	112.7(11)
N(1)–C(41)	1.330(16)	C(1)Sb(1)O(1)	81.1(4)

Table 2 (end)

Bond	*d*, Å	Angle	ω, deg
O(2)–C(42)	1.206(16)	C(21)Sb(1)O(1)	85.5(4)
N(2)–C(42)	1.388(17)	C(31)Sb(1)O(1)	83.2(4)
Sb(2)–C(51)	2.122(14)	C(81)Sb(2)O(3)	168.1(6)
Sb(2)–C(61)	2.122(14)	C(51)Sb(2)C(61)	121.4(6)
Sb(2)–C(71)	2.103(18)	C(51)Sb(2)C(71)	115.3(7)
Sb(2)–O(3)	2.195(11)	C(61)Sb(2)C(71)	120.8(6)
Sb(2)–C(81)	2.179(14)	N(3)O(3)Sb(2)	127.5(11)
O(3)–N(3)	1.300(18)	O(3)N(3)C(91)	111.0(15)
N(3)–C(91)	1.33(2)	C(51)Sb(2)O(3)	87.4(5)
O(4)–C(92)	1.21(2)	C(61)Sb(2)O(3)	74.9(5)
N(4)–C(92)	1.36(2)	C(71)Sb(2)O(3)	92.3(6)

Results and Discussion

We have studied the reactions of pentaphenylantimony with β-isatoxime containing a large organic radical with several functional groups. It has been found that, regardless of the molar ratio of the reactants (1:1 or 1:2), the reaction product is (β-isatoximato)tetraphenylantimony:



1

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

The resulting compound has been studied by IR spectroscopy and X-ray diffraction analysis.

In the IR spectrum of compound **1** there are absorption bands at 3148 and 3065 cm^{-1}, characterizing the presence of the N–H bond, and a band at 1705 cm^{-1}, which appears as a result of valence vibrations of the C=O bond. Characteristic bands are observed at 1551 cm^{-1} (C=N bond), 1020 cm^{-1} (N–O bond). In comparison with the initial oxime spectrum, the absorption bands in the spectrum of compound **1** are shifted to longer wavelengths. Vibrations at 419 cm^{-1} indicate the presence of the Sb–C bond in the molecule of compound **1** [16].

According to the X-ray diffraction analysis data, in the crystal of compound **1** there are two crystallographically independent molecules (***a*** and ***b***). Antimony atoms have distorted trigonal-bipyramidal coordination with the iminoxy group oxygen atom and the carbon atom of one of phenyl groups in the axial positions (Fig. 1).

The SbC_3 fragment lying in the equatorial plane is not flat. The Sb atom deviates from the $[C_3]$ plane by 0.246 Å (***a***) and 0.198 Å (***b***) toward the axial carbon atom. The sums of the valence angles of $C_{eq}SbC_{eq}$ in the equatorial planes are 356.0(5)° (***a***) и 357.5(6)° (***b***), the values of the individual angles differ from the theoretical 120° by not more than 7.1°. The axial angle $OSbC_{ax}$ is 177.9(4)° (***a***) and 168.1(6)° (***b***). The values of the angles $C_{ax}SbC_{eq}$ and $C_{eq}SbO$ lie within the limits of 92.4(5)°–100.0(5)° (***a***), 93.5(6)°–97.0(6)° (***b***) and 81.1(4)°–85.5(4)° (***a***), 74.9(5)°–92.3(6)° (***b***), respectively, which differs from the theoretical value of 90° and is caused by the deviation of the Sb atom from the equatorial plane.

The variation intervals of the $Sb–C_{eq}$ bond lengths are 2.115(12)–2.128(13) Å (***a***), 2.103(18)–2.122(14) Å (***b***). The axial bonds Sb–C (2.167(12) Å (***a***), 2.179(14) Å (***b***)) are considerably longer than the equatorial bonds. The distances Sb–O (2.231(9) Å (***a***), 2.195(11) Å (***b***)) significantly exceed the sum of the covalent radii of Sb and O atoms (2.07 Å [17]), as well as the distance between the Sb atom and the axial C atom, similarly, as in other compounds, where the O–N=C fragment is linked to electron-accepting functional groups [18–20]. Interestingly, the Sb–O distances (2.121(4)–2.179(1) Å) in previously studied tetraphenylantimony oximates, containing electron-donating substituents, are smaller than those in compound **1** and do not exceed the $Sb–C_{ax}$ distances (2.167(8)–2.207(1) Å) [8–10].

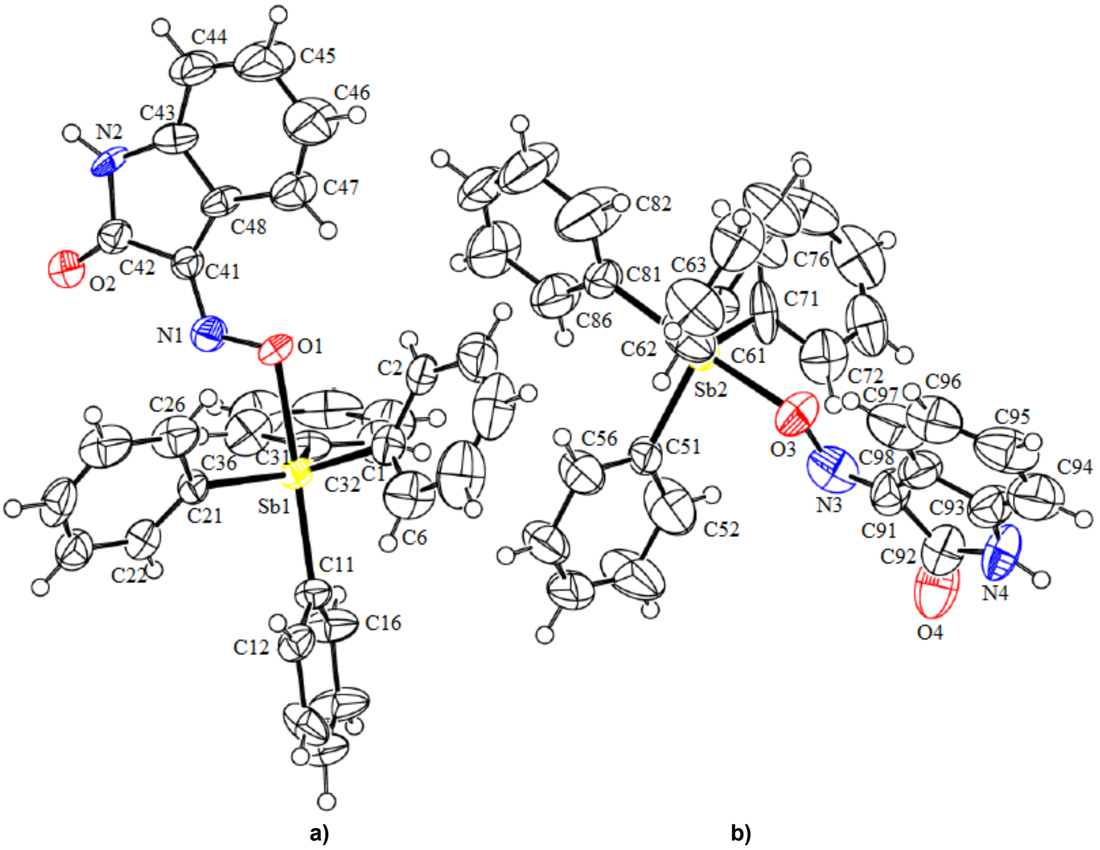


Fig. 1. The structure of compound 1 (a, b)

In the molecules ***a*** and ***b***, the intramolecular contacts Sb···N are observed. The distances between the Sb atom and the iminoxy-group N atom Sb···N [2.94(1) Å (***a***), 3.16(2) Å (***b***)] are shorter than the sum of van der Waals radii of these atoms (3.8 Å [17]). The reduction of the distances Sb···N is accompanied by the decrease of the SbON angle values (108.5(7)° (***a***), 127.5(11)° (***b***)), the SbON angle in molecule ***b*** is abnormally large, in comparison with the corresponding value in analogous compounds (102.7(4)°–109.81(7)°) [8–10]. The values of the ONC angles in the molecules are close: 113(1)° (***a***), 111(1)° (***b***). No dependence in the change of the O–N and N–C bond lengths (1.331(14) Å, 1.330(16) Å (***a***) and 1.300(18) Å, 1.33(2) Å (***b***)) is observed.

In the condensed heterocycle in molecule ***a***, the iminoxy group is located in the heterocycle plane, while in molecule ***b*** the O atom deviates from the plane by 0.308 Å and the N atom deviates by 0.192 Å. The angles between the heterocycle plane and the plane of the benzene ring are 3.50° (***a***) and 6.02° (***b***).

Conclusions

Thus, regardless of the molar ratio of the reactants, as a result of the reaction of pentaphenylantimony with β-isatoxime, (β-isatoximato)tetraphenylantimony is formed. In spite of the fact that β-isatoxime contains several different functional groups (the amino- and the keto-group), it reacts with pentaphenylantimony in a conventional manner. The antimony atom in the resulting compound has distorted trigonal-bipyramidal coordination with the iminoxy-group oxygen atom and the carbon atom of one of the phenyl groups.

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СИНТЕЗ И СТРОЕНИЕ β-ИЗАТОКСИМАТА ТЕТРАФЕНИЛСУРЬМЫ

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Взаимодействием пентафенилсурьмы с β-изатоксимом синтезирована (β-изатоксимато)тетрафенилсурьма (1). Методом рентгеноструктурного анализа установлено, что атом сурьмы в комплексе (1) имеет искаженную тригонально-бипирамидальную координацию.

Ключевые слова: пентафенилсурьма, β-изатоксим, дефенилирование, (β-изатоксимато)тетрафенилсурьма, молекулярные структуры, рентгеноструктурный анализ.

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