

# Novel Organoantimony Compounds [2,6-(OMe)<sub>2</sub>C<sub>6</sub>H<sub>3</sub>]<sub>3</sub>SbO and [2,6-(OMe)<sub>2</sub>C<sub>6</sub>H<sub>3</sub>]<sub>3</sub>Sb(NCO)<sub>2</sub>·0.5(CH<sub>3</sub>)<sub>2</sub>CO. Synthesis and Structure

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**Abstract**—Novel organic compounds of antimony(V), tris(2,6-dimethoxyphenyl)antimony diisocyanate and oxide, have been prepared and studied by X-ray diffraction analysis. Antimony atoms in the diisocyanate molecule and in two symmetrically independent molecules of the oxide exhibit trigonal-bipyramidal and tetrahedral coordination, respectively.

**Keywords:** tris(2,6-dimethoxyphenyl)antimony, derivative, X-ray diffraction analysis

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Studies of synthesis and structure of antimony compounds, Ar<sub>3</sub>SbO and Ar<sub>3</sub>SbX<sub>2</sub>, are inspired by their synthetic capacity. A series of reports have mentioned the application of Ph<sub>3</sub>SbO (or Ph<sub>3</sub>SbO–P<sub>4</sub>S<sub>10</sub> mixture) as catalyst of synthesis of amides, substituted urea, thiocarboxylic acids, carbocyclic esters, imidazolidin-2-ones, carbodiimides, polyesters, and cyclic carbonates [1–7]. Triphenylantimony oxide has been used in synthesis of dipeptides [8, 9]. Ph<sub>3</sub>SbO, PhSbO<sub>2</sub>, 4-CH<sub>3</sub>C<sub>6</sub>H<sub>4</sub>SbO<sub>2</sub>, and 4-ClC<sub>6</sub>H<sub>4</sub>SbO<sub>2</sub> have exhibited catalytic activity in polymerization of alkene oxides and substituted oxiranes [10–12]. Triphenylantimony diacylate has been used as catalyst of synthesis of thiocarboxylic acids and amides [3, 13].

Structures of many Ph<sub>3</sub>SbX<sub>2</sub> compounds (X being mono- or bidentate ligand) have been characterized so far, whereas the antimony compounds containing potentially coordinating groups in the aromatic ring substituents have been scarcely studied.

Triarylantimony(V) oxides (their structure has been elucidated by means of X-ray diffraction analysis) exist in dimeric, polymeric, and monomeric forms. Triphenylantimony oxide formed upon heating of Ph<sub>4</sub>SbOCH<sub>3</sub> solution in xylene under nitrogen atmosphere or via Ph<sub>3</sub>SbC(SO<sub>2</sub>C<sub>6</sub>H<sub>4</sub>CH<sub>3</sub>)<sub>2</sub> hydrolysis consists of dimeric (Ph<sub>3</sub>SbO)<sub>2</sub> molecules containing

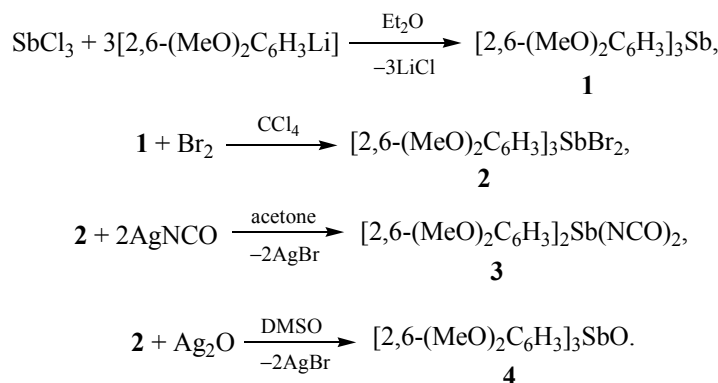
planar Sb<sub>2</sub>O<sub>2</sub> ring, coordination polyhedron of antimony being close to trigonal bipyramid [14, 15].

Similar structure has been revealed for [(2-MeC<sub>6</sub>H<sub>4</sub>)<sub>3</sub>SbO]<sub>2</sub> (prepared via oxidation of triarylantimony with hydrogen peroxide), [(2-MeOC<sub>6</sub>H<sub>4</sub>)<sub>3</sub>SbO]<sub>2</sub> (isolated from the products of tris(2-methoxyphenyl)antimony reaction with potassium *tert*-butylate), and [(2-PhOC<sub>6</sub>H<sub>4</sub>)O(C<sub>6</sub>H<sub>4</sub>)<sub>2</sub>Sb]<sub>2</sub>O<sub>2</sub> [a product of (oxydi-2,1-phenylene)(2-phenoxyphenyl)antimony oxidation with air oxygen in dichloromethane] [16–18].

DFT studies have revealed that dimers of triphenylantimony oxide are more stable than the corresponding monomers [17].

Using X-ray powder diffraction (EXAFS), polymeric triphenylantimony oxide with trigonal-bipyramidal coordination of the central atom has been studied [19]. Oxygen atoms in this compound occupy axial positions, and phenyl substituents are found in equatorial positions. The oxide has been prepared via treatment of Ph<sub>3</sub>Sb with hydrogen peroxide in acetone. (Ph<sub>2</sub>Sb)<sub>4</sub>O<sub>6</sub> has been prepared in a similar manner; it exhibited the resonance structures with two hexacoordinated antimony atoms bearing negative charge and two antimony atoms that are either tetravalent (both positively charged) or pentavalent (with positive charges at two oxygen atoms) [14]. This oxo cluster is

## Scheme 1.



isostructural with  $[(2\text{-MeC}_6\text{H}_4)_2\text{Sb}]_4\text{O}_6$  formed via oxidation of  $2\text{-MeC}_6\text{H}_4\text{Sb}(\text{SiMe}_3)_2$  in THF during several weeks [18].

The only oxide of the considered class with confirmed monomeric structure is the  $[2,4,6\text{-(CH}_3)_3\text{C}_6\text{H}_2]_3\text{SbO}\cdot\text{HO}_3\text{SPh}$  adduct with antimony atom in the distorted tetrahedral coordination [20]. This compound has been prepared in the reaction of trimesitylantimony dihydroxide with sulfonic acid in acetone.

Extending the studies of methods of synthesis of antimony(III,V) compounds bearing potential coordination sites (in particular, oxygen atoms of methoxy groups) in the aromatic substituents, we prepared tris(2,6-dimethoxyphenyl)antimony **1** as well as its dibromide **2**, diisocyanate **3**, and oxide **4** for the first time.

The interaction of 2,6-dimethoxyphenyllithium and antimony(III) chloride resulted in the formation of colorless crystals of compounds **1**, readily soluble in carbon tetrachloride. Subsequent oxidation of compound **1** with bromine afforded tris(2,6-dimethoxyphenyl)antimony dibromide **2** in 94% yield. Compound **2** was further used in the preparation of corresponding diisocyanate **3** and oxide **4**. Reactions of bromide **2** with silver isocyanate and oxide were carried out in acetone and DMSO at room temperature in dark during 1 day. The prepared antimony(V) compounds were soluble in acetone, toluene, DMF, DMSO, and THF. Compound **3** crystallized from acetone in the form of solvate (Scheme 1).

Spatial structure of compounds **3** and **4** was elucidated by means of X-ray diffraction analysis. Structure of triarylantimony(V) cyanates, triphenylantimony diisocyanate and  $\mu$ -oxobis[(isocyanato)triphenylantimony], has been earlier described in [21, 22].

Crystal structure of compound **3** was based on the molecules with antimony atoms in distorted trigonal-bipyramidal coordination (Fig. 1). The apical positions were occupied by isocyanate groups, and the aryl substituents were located in the equatorial plane. In the  $\text{Ar}_3\text{Sb}$  fragment, the  $\text{C}^1\text{Sb}^1\text{C}^{17}$ ,  $\text{C}^1\text{Sb}^1\text{C}^9$ , and  $\text{C}^9\text{Sb}^1\text{C}^{17}$  angles  $[117.09(8)^\circ, 120.94(8)^\circ, \text{and } 121.87(8)^\circ]$  were close to  $120^\circ$ , typical of the angles between equatorial groups in similar compounds.

The aromatic rings took the least sterically hindered configuration of propeller with respect to the base plane of the  $[\text{SbC}_3]$  bipyramid, the corresponding dihedral angles being  $27.27^\circ, 32.29^\circ, \text{and } 36.88^\circ$ . The angles between the axial and equatorial bonds  $[87.09(7)^\circ\text{--}92.61(7)^\circ]$  were close to the theoretical value ( $90^\circ$ ). The antimony atom was shifted towards the  $\text{N}^{\text{I}}$  atom by  $0.038 \text{ \AA}$ . The carbon atoms  $\text{C}^1\text{--C}^6$ ,  $\text{C}^9\text{--C}^{14}$  of two aryl substituents and the antimony atom were located practically in the same plane, in contrast to carbon atoms  $\text{C}^{17}\text{--C}^{22}$  of the third benzene ring, as confirmed by the values of the  $\text{Sb}^1\text{C}^1\text{C}^6\text{C}^5$ ,  $\text{Sb}^1\text{C}^9\text{C}^{14}\text{C}^{13}$ , and  $\text{Sb}^1\text{C}^{17}\text{C}^{18}\text{C}^{19}$  dihedral angles  $[178.79(15)^\circ, 178.92(17)^\circ, \text{and } 168.46(16)^\circ, \text{ respectively}]$ . Let us mark the close values of the axial angles and sums of equatorial angles for compound **3**  $[178.79(7)^\circ \text{ and } 359.9^\circ]$ , triphenylantimony diisocyanate  $\{178.6(6)^\circ \text{ and } 360.0^\circ [21]\}$ , and  $\mu$ -oxobis[(isocyanato)triphenylantimony]  $\{177.30(19)^\circ, 179.12(19)^\circ; 359.3^\circ, 359.1^\circ [22]\}$ .

$p$ - $\pi$ - $d$ -Conjugation involving the lone-electron pairs of methoxy groups oxygen atoms,  $\pi$ -electrons of aryl rings, and free  $d$ -orbitals of antimony atom resulted in the decrease in the Sb-C interatomic distances  $[2.0937(19), 2.0973(19), \text{and } 2.1014(19) \text{ \AA}]$  in compound **3** by  $0.02 \text{ \AA}$  on the average as compared

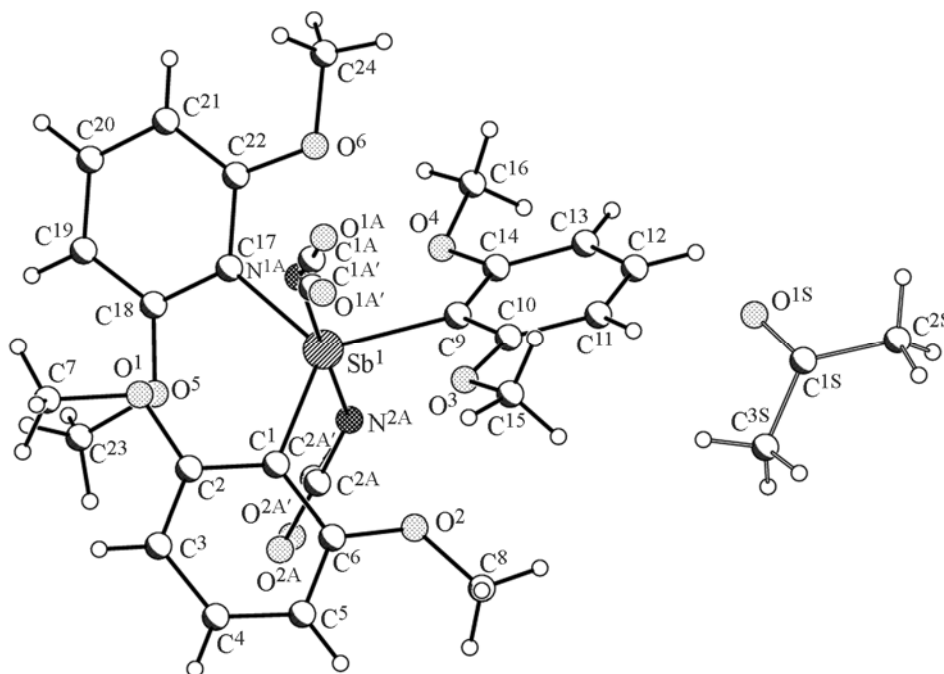


Fig. 1. General view of independent part of unit cell of tris(2,6-dimethoxyphenyl)antimony diisocyanate **3**.

to the corresponding bonds in triphenylantimony diisocyanate {2.109(6)–2.124(6) Å [21]}.

The Sb–N bonds length was increased [2.1412(17), 2.1478(17) Å] as compared to the corresponding bonds in triphenylantimony diisocyanate [2.122(4), 2.125(4) Å]. The SbNC angles in molecules of compound **3** equaled 153.3(6)°, 136.0(6)°; 132.1(16)°, 133.2(11)°. The isocyanate groups were disordered. The ratio of populations of the atomic positions was 3 : 2. The N–C [1.156(4), 1.165(4) Å; 1.161(5), 1.181(4) Å] and C–O [1.188(4), 1.200(4) Å; 1.215(5), 1.196(5) Å] distances in molecules of compound **3** were more even as compared to those in the described triarylantimony(V) cyanates {[1.111(7), 1.146(7) Å; 1.186(7), 1.189(8) Å [21]; 0.973(8), 1.110(8) Å; 1.232(8), 1.285(9) Å [22]} and close to the N–C (1.183 Å) and C–O (1.184 Å) distances in isocyanic acid [23]. The NCO angles [177.0(8)°, 175.1(12)°; 173.9(19)°, 164.0(3)°] were distorted and deviated from the standard bond angles for the *sp*-carbon atom.

Compound **3** exhibited weak intermolecular interactions of oxygen atoms of methoxy groups with antimony atom, with the Sb⋯O distance 3.080–3.108 Å (sum of Van der Waals radii 3.7 Å [24]).

Crystal of the oxide **4** was built of symmetrically nonequivalent molecules A and B (Fig. 2). Antimony

atoms in compound **4** revealed coordination of distorted tetrahedron with three carbon atoms of the aryl groups and an oxygen atom in vertexes.

The CSbC and OSbC angles were different [108.52(18)°, 115.29(18)°, 116.24(18)°, and 97.03(16)°, 109.4(2)°, 109.59(16)° in molecule A; 107.47(18)°, 111.68(17)°, 115.9(2)° and 101.6(2)°, 109.99(18)°, 110.13(16)° in molecule B] and deviated from the ideal value, similarly to molecule of [2,4,6-(CH<sub>3</sub>)<sub>3</sub>C<sub>6</sub>H<sub>2</sub>]<sub>3</sub>SbO with the bond angles in the 99.8(2)°–119.1(3)° range [20].

The Sb–C bonds in compound **4** were shorter than those in the [2,4,6-(CH<sub>3</sub>)<sub>3</sub>C<sub>6</sub>H<sub>2</sub>]<sub>3</sub>SbO·HO<sub>3</sub>SPh adduct {2.104(7)–2.128(7) Å [20]}, (Ph<sub>3</sub>SbO)<sub>2</sub> {2.121(6)–2.164(6) Å [14]}, (Ph<sub>2</sub>Sb)<sub>4</sub>O<sub>6</sub> {2.108(18)–2.154(18) Å [14]}, (Ph<sub>3</sub>SbO)<sub>2</sub> 2.129(2)–2.161(2) Å [15]}, [(2-MeC<sub>6</sub>H<sub>4</sub>)<sub>3</sub>SbO]<sub>2</sub> {2.122(3)–2.183(3) [16]}, [(2-MeOC<sub>6</sub>H<sub>4</sub>)<sub>3</sub>SbO]<sub>2</sub> {2.123(4)–2.180(3) [17]}, and [(2-MeC<sub>6</sub>H<sub>4</sub>)<sub>2</sub>Sb]<sub>4</sub>O<sub>6</sub> {2.099–2.172 Å [18]}, due to the presence of two methoxy groups in the phenyl substituents, providing oxygen atoms to be involved in *p*- $\pi$ -*d*-conjugation.

The Sb–O bonds length in crystal of compound **4** [1.914(3) Å for molecule A and 1.918(3) Å for molecule B] and in the [2,4,6-(CH<sub>3</sub>)<sub>3</sub>C<sub>6</sub>H<sub>2</sub>]<sub>3</sub>SbO·HO<sub>3</sub>SPh molecule [1.894(5) Å] exceeded that of the double Sb=O bond (1.86 Å), the Sb–O bond length

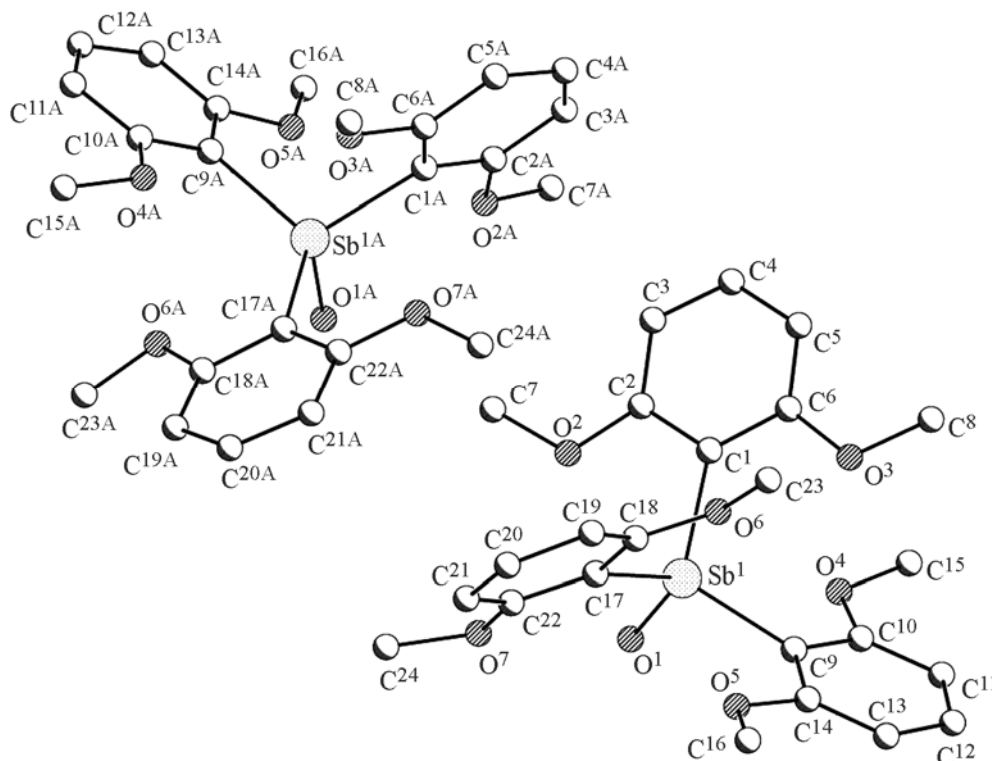


Fig. 2. General view of independent part of unit cell of tris(2,6-dimethoxyphenyl)antimony oxide **4**. Hydrogen atoms are omitted.

being 2.07 Å [25]. Those distances were shorter than the Sb–O bond length in dimeric and polymeric oxides and oxo complexes of antimony {1.881(12)–2.283(10) Å [14–20], average value 2.020 Å} as well as the Sb–O distances for the  $\text{Ph}_3\text{Sb}(\text{OH})_2$  {1.963(6), 2.079(7) Å [26]},  $\text{Ph}_3\text{Sb}(\text{OMe})_2$  {2.027(8), 2.039(8) Å [27]}, and  $\text{Ph}_3\text{Sb}(\text{OObu-}t)_2$  {2.057, 2.065 Å [28]} structures (sum of covalent radii of antimony and oxygen atoms 2.14 Å [24]).

Due to the contacts of nitrogen and carbon atoms with methyl groups [ $\text{N}^{1A}\cdots\text{C}^8$  (3.200 Å),  $\text{C}^{2A}\cdots\text{C}^7$  (3.331 Å)] the cyanate substituents formed the chains in the crystal of compound **3**, those chains being united in the layers through oxygen atoms [ $\text{O}^{2A}\cdots\text{H}-\text{C}^{15}$  (2.655 Å) and  $\text{O}^{1A}\cdots\text{H}-\text{C}^{23}$  (2.477 Å)] (sum of Van der Waals radii of those atoms being 3.3, 3.4, 2.7 Å [25]) (Fig. 3). Formation of the chains involved the short intermolecular contacts C–H $\cdots$ C of methyl groups and (or) benzene rings [ $\text{C}_{\text{Me}}-\text{H}\cdots\text{C}_{\text{Ar}}$  2.810–2.889 (**3**), 2.657–2.836 (**4**),  $\text{C}_{\text{Ar}}-\text{H}\cdots\text{C}_{\text{Ar}}$  2.852, 2.889 Å (**3**)], evidencing the C–H $\cdots$  $\pi$  as well as the  $\text{C}_{\text{Me}}-\text{O}\cdots\text{H}-\text{C}_{\text{Me}}$  2.637 Å contacts (**4**). In the crystal of compound **4**, molecular layers were formed owing to the interactions between the  $\text{C}_{\text{Me}}-\text{H}\cdots\text{H}-\text{C}_{\text{Me}}$ ,  $\text{C}_{\text{Me}}\cdots\text{H}-\text{C}_{\text{Me}}$ ,  $\text{C}_{\text{Me}}\cdots\text{C}_{\text{Ar}}$ ,  $\text{C}_{\text{Me}}-\text{H}\cdots\text{C}_{\text{Ar}}$  chains, their parameters being 2.307, 2.739, 2.352, 2.875 Å, respectively (Fig. 4).

Absorption bands in IR spectra of compounds **1–4** were assigned using the reference data [29, 30]. IR spectrum of tris(2,6-dimethoxyphenyl)antimony **1** contained the absorption bands with maximums at 763 [ $\delta_s(\text{C}-\text{H}_{\text{Ar}})$ ], 1423 [ $\delta_s(\text{CH}_3)$ ], 1462 and 1579 [ $\nu_{\text{as,s}}(\text{C}-\text{C}_{\text{Ar}})$ , 2993, 3056, and 3070  $\text{cm}^{-1}$  (C–H)].

Vibration frequency of the bands of methoxy group in the spectrum of compound **1** (absent in the spectrum of triphenylantimony [31]) were found at 1101 [ $\nu_s(\text{O}-\text{C}_{\text{Me}})$ ], 1041, 1234 [ $\nu_{\text{as}}(\text{C}_{\text{Me}}-\text{O}-\text{C}_{\text{Ar}})$ ], 2828, and 2934  $\text{cm}^{-1}$  [ $\nu_{\text{s,as}}(\text{C}-\text{H})$ ].

Vibration frequencies of methoxy groups in the spectra of the dibromide **2**, diisocyanate **3**, and oxide **4** were increased as compared to compound **1**: 1107  $\text{cm}^{-1}$  (**2**, **3**, **4**) [ $\nu_s(\text{O}-\text{C}_{\text{Me}})$ ]; 1034, 1257 (**2**), 1030, 1255 (**3**), 1020, 1261  $\text{cm}^{-1}$  (**4**) [ $\nu_{\text{as}}(\text{C}_{\text{Me}}-\text{O}-\text{C}_{\text{Ar}})$ ]; 2833, 2936 (**2**), 2835, 2938 (**3**), 2844, 2946  $\text{cm}^{-1}$  (**4**) [ $\nu_{\text{s,as}}(\text{C}-\text{H})$ ].

The change in the frequency of stretching of the NCO was in line with the increase in the length of the corresponding bonds in molecule **3** in comparison with triphenylantimony diisocyanate and  $\mu$ -oxobis[(isocyanato)triphenylantimony]: the vibrations frequency was decreased from 2210, 2209 [ $\nu_{\text{as}}(\text{N}-\text{C}-\text{O})$ ] and 633, 642  $\text{cm}^{-1}$  [ $\delta(\text{N}-\text{C}-\text{O})$ ] to 2189 and 617  $\text{cm}^{-1}$ . Vibration of the Sb=O bond was assigned to the band at 664  $\text{cm}^{-1}$

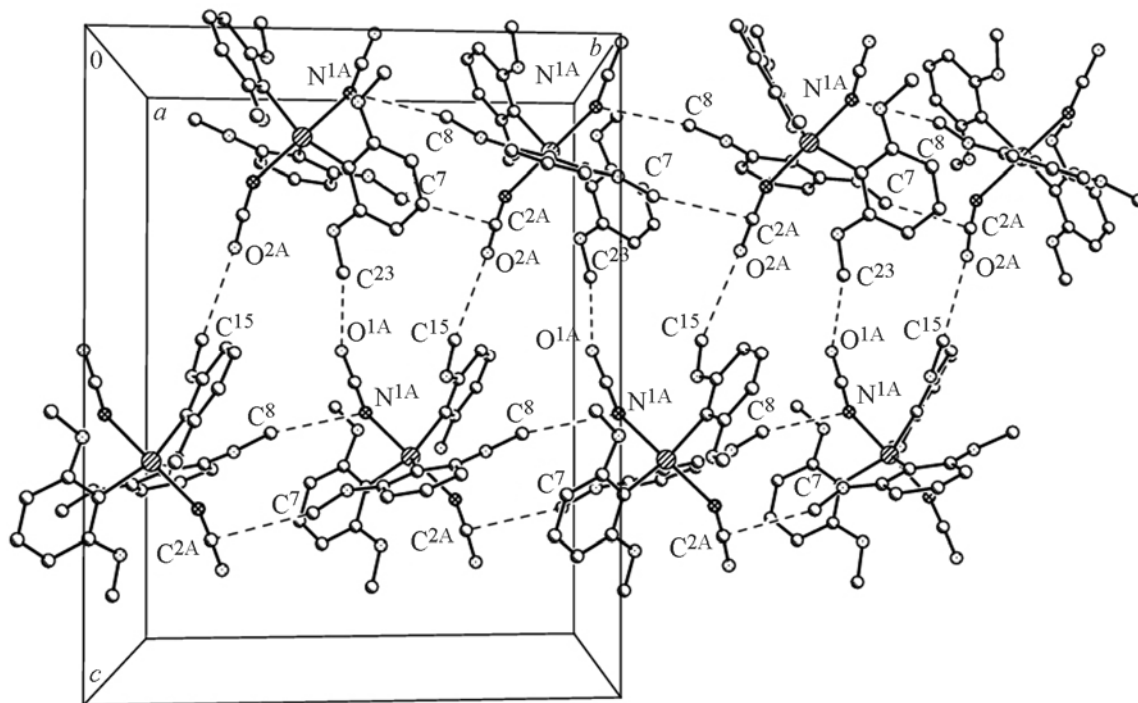


Fig. 3. Fragment of chains and layer (along *bc* plane) of molecules in crystal of compound 3. Hydrogen atoms are omitted.

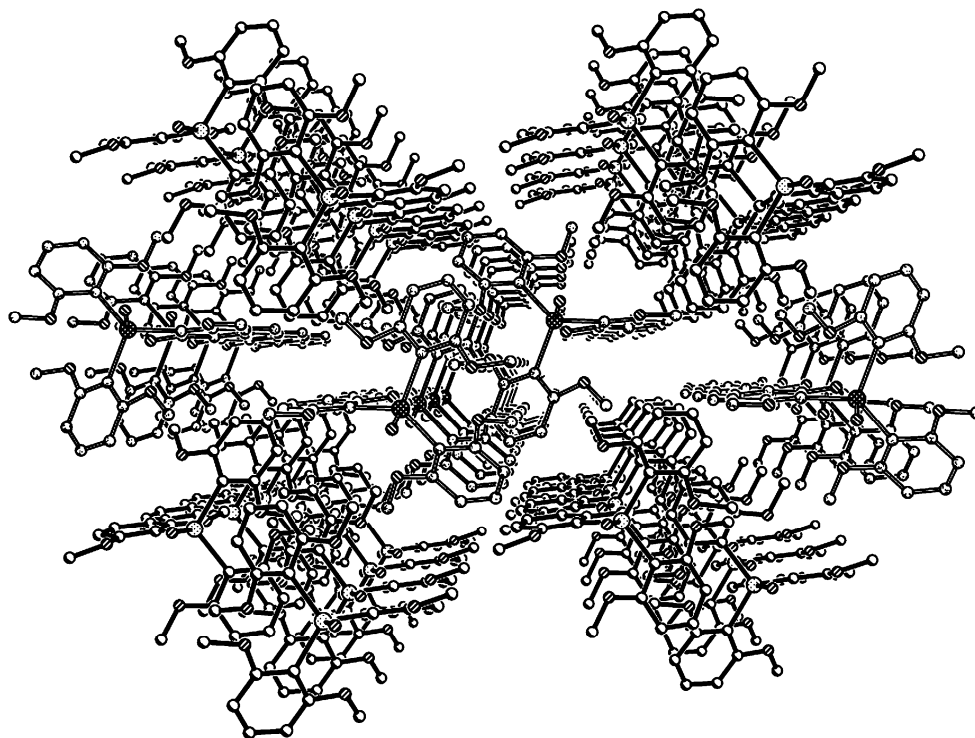


Fig. 4. Fragment of crystal packing of compound 4. Types of independent molecules are distinguished by the lines type. Hydrogen atoms are omitted.

Crystallographic parameters of compounds **3** and **4**, experimental data, and parameters of the structure refinement

Parameter	<b>3</b>	<b>4</b>
Formula	C <sub>27.50</sub> H <sub>30</sub> N <sub>2</sub> O <sub>8.50</sub> Sb <sub>1</sub>	C <sub>24</sub> H <sub>27</sub> O <sub>7</sub> Sb <sub>1</sub>
Crystal system	Orthorhombic	Monoclinic
<i>M</i>	646.28	549.20
Space group	<i>Pbca</i>	<i>P2</i> <sub>1</sub>
<i>a</i> , Å	16.8887(8)	14.9005(5)
<i>b</i> , Å	16.2758(8)	11.5017(4)
<i>c</i> , Å	20.5100(10)	15.9428(6)
$\alpha$ , deg	90	90
$\beta$ , deg	90	92.1270(10)
$\gamma$ , deg	90	90
<i>V</i> , Å <sup>3</sup>	5637.7(5)	2730.41(17)
<i>Z</i>	8	4
<i>T</i> , K	120(2)	120(2)
<i>d</i> <sub>calc</sub> , g/cm <sup>3</sup>	1.523	1.336
<i>F</i> (000)	2624	1112
$\theta$ range	1.986° ≤ $\theta$ ≤ 28.998°	1.278° ≤ $\theta$ ≤ 32.436°
Indexes range	-23 ≤ <i>h</i> ≤ 23, -22 ≤ <i>k</i> ≤ 22, -27 ≤ <i>l</i> ≤ 27	-21 ≤ <i>h</i> ≤ 22, -17 ≤ <i>k</i> ≤ 17, -23 ≤ <i>l</i> ≤ 23
Measured reflections/independent	67075/7487	40414/19011
<i>R</i> <sub>int</sub>	0.0500	0.0381
Number of refined parameters	392	590
$\Delta\rho_{\max}/\rho_{\min}$ , eÅ <sup>-3</sup>	0.694/-0.411	0.849/-0.529
GooF	1.016	0.922
<i>R</i> -Factors [ <i>I</i> > 2 $\sigma$ ( <i>I</i> )]		
<i>R</i>	0.0272	0.0394
<i>R</i> <sub>w</sub>	0.0639	0.0688

[32]; in the case of compound **4**, that vibration was observed at 677 cm<sup>-1</sup>.

In summary, we proposed a method to prepare earlier unknown tris(2,6-dimethoxyphenyl)antimony and its derivatives.

#### EXPERIMENTAL

IR spectra of KBr pellets were recorded using an FSM 1201 Fourier spectrometer at 400–4000 cm<sup>-1</sup>. Elemental analysis was performed using a Carlo Erba (model 1106) CHN-analyzer. Bromine content was determined by means of gravimetry in the AgBr form.

X-ray diffraction studies of monocrystals of compounds **3** and **4** were performed using a Bruker APEX II CCD area detector diffractometer at 120(2) K (MoK $\alpha$ -radiation, graphite monochromator). The independent reflections obtained by averaging the equivalent reflections were used for the structure elucidation and refinement. Absorption was accounted for using SADABS software [33]. The structures were solved via the direct method; all non-hydrogen atoms were localized in the differential syntheses of electronic density and refined over  $F_{hkl}^2$  in anisotropic approximation. Positions of hydrogen atoms were calculated from geometry considerations and refined

using a *riding* model under isotropic approximation. The calculations were performed using software [34].

Major crystallographic parameters of compounds **3** and **4**, experimental data, and parameters of the structure refinement were deposited in The Cambridge Crystallographic Data Centre [CCDC 1468102 (**3**) and 1468103 (**4**)].

**Tris(2,6-dimethoxyphenyl)antimony (1).** 6.50 g of antimony(III) chloride (0.03 mol) in 100 mL of anhydrous diethyl ether was added to an ethereal solution of 2,6-dimethoxyphenyllithium prepared [35] from 13.8 g (0.10 mol) of 1,3-dimethoxybenzene and 0.10 mol of phenyllithium. The mixture boiled up, and white precipitate was formed. After addition of antimony(III) chloride, the reaction mixture was stirred during 2 h, excess of 2,6-dimethoxyphenyllithium was decomposed with 5 mL of water, and the solution was filtered. Ether was evaporated off on a water bath, and the residue was poured onto a Petri dish. After the solvent evaporation, 10.9 g (72%) of compound **1** was isolated, mp 162°C. IR spectrum,  $\nu$ ,  $\text{cm}^{-1}$ : 3070, 3056, 2993, 2934, 2828, 1579, 1569, 1462, 1423, 1300, 1234, 1168, 1101, 1041, 1022, 763, 737, 707, 589. Found, %: C 54.23; H 4.93.  $\text{C}_{24}\text{H}_{27}\text{O}_6\text{Sb}$ . Calculated, %: C 54.06; H 5.10.

**Tris(2,6-dimethoxyphenyl)antimony dibromide (2).** 0.48 mL of bromine (9.38 mmol) in 50 mL of  $\text{CCl}_4$  was added to a solution of 5.00 g (9.38 mmol) of compound **1** in 150 mL of  $\text{CCl}_4$ . The precipitate was filtered off, washed with diethyl ether (3×5 mL), and dried. Yield 6.1 g (94%), mp 174°C. IR spectrum,  $\nu$ ,  $\text{cm}^{-1}$ : 2997, 2963, 2936, 2833, 1586, 1571, 1475, 1426, 1257, 1172, 1107, 1034, 774, 743, 708. Found, %: C 41.77; H 3.70; Br 23.24.  $\text{C}_{24}\text{H}_{27}\text{Br}_2\text{O}_6\text{Sb}$ . Calculated, %: C 41.59; H 3.92; Br 23.06.

**Tris(2,6-dimethoxyphenyl)antimony diisocyanate (3).** A suspension of 0.43 g (2.88 mmol) of silver isocyanate in 150 mL of acetone [36] was added to a solution of 1.00 g (1.44 mmol) of compound **2** in the same solvent [36]. Precipitated silver bromide was filtered off and washed with acetone (3×5 mL). After the solvent evaporation, 0.88 g (95%) of compound **3** was isolated, mp 142°C. IR spectrum,  $\nu$ ,  $\text{cm}^{-1}$ : 3007, 2967, 2938, 2835, 2359, 2341, 2189, 1587, 1472, 1425, 1302, 1292, 1255, 1173, 1107, 1030, 899, 773, 744, 711, 617, 594.

**Tris(2,6-dimethoxyphenyl)antimony oxide (4).** 0.50 g (2.16 mmol) of silver oxide [37] was added to a solution of 1.00 g (1.44 mmol) of compound **2** in

150 mL of DMSO. The mixture was stirred during 30 min in the dark. The precipitate was filtered off and washed with DMSO (3×5 mL). After the solvent evaporation, 0.71 g (89%) of compound **4** was isolated, mp 152°C (decomp.). IR spectrum,  $\nu$ ,  $\text{cm}^{-1}$ : 3084, 3016, 2946, 2844, 1594, 1578, 1472, 1432, 1342, 1261, 1107, 1020, 889, 830, 776, 740, 711, 677, 665, 589. Found, %: C 52.29; H 5.139.  $\text{C}_{24}\text{H}_{27}\text{O}_7\text{Sb}$ . Calculated, %: C 52.48; H 4.959.

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