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**COORDINATION COMPOUNDS**

# Antimony Complexes $\{[2,6\text{-}(OMe)_2C_6H_3]_3SbCH_2C(O)OEt\}_2^+[Hg_2I_6]^{2-}$ and $\{[2,6\text{-}(OMe)_2C_6H_3]_3SbMe\}_2^+[HgI_4]^{2-} \cdot$ DMSO: Synthesis and Structure

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**Abstract**—It has been established that ethyl iodoacelate and 1,4-diiodobutane alkylate triarylantimony $Ar_3Sb$ with the formation of $[Ar_3SbCH_2C(O)OEt]^+I^-$ and $[Ar_3Sb(CH_2)_4I]^+I^-$, $[Ar_3Sb(CH_2)_4SbAr_3]^{2+}I_2^-$, where Ar = 2,6-$(OMe)_2C_6H_3$. The complexes $[Ar_3SbCH_2C(O)OEt]_2^+[Hg_2I_6]^{2-}$ and $[Ar_3SbMe]_2^+[HgI_4]^{2-} \cdot$ DMSO have been synthesized by the reaction of $[Ar_3SbCH_2C(O)OEt]^+I^-$ and $[Ar_3SbMe]^+I^-$ with mercury diiodide and studied by X-ray diffraction. The antimony and iodine atoms have a distorted tetrahedral coordination. The CSbC and IHgI angles are ranged within 103.28(14)°–116.68(14)°, 103.7(4)°–115.5(4)° and 98.122(9)°–125.590(12)°, 102.66(2)°–115.64(2)°, respectively.

**Keywords:** tris(2,6-dimethoxyphenyl) antimony, ethyl iodoacetate, 1,4-diiodobutane, tris(2,6-dimethoxyphenyl) (ethoxycarbonylmethyl) antimony iodide, tris(2,6-dimethoxyphenyl)methyl antimony iodide, mercury diiodide, X-ray diffraction analysis, nuclear magnetic resonance

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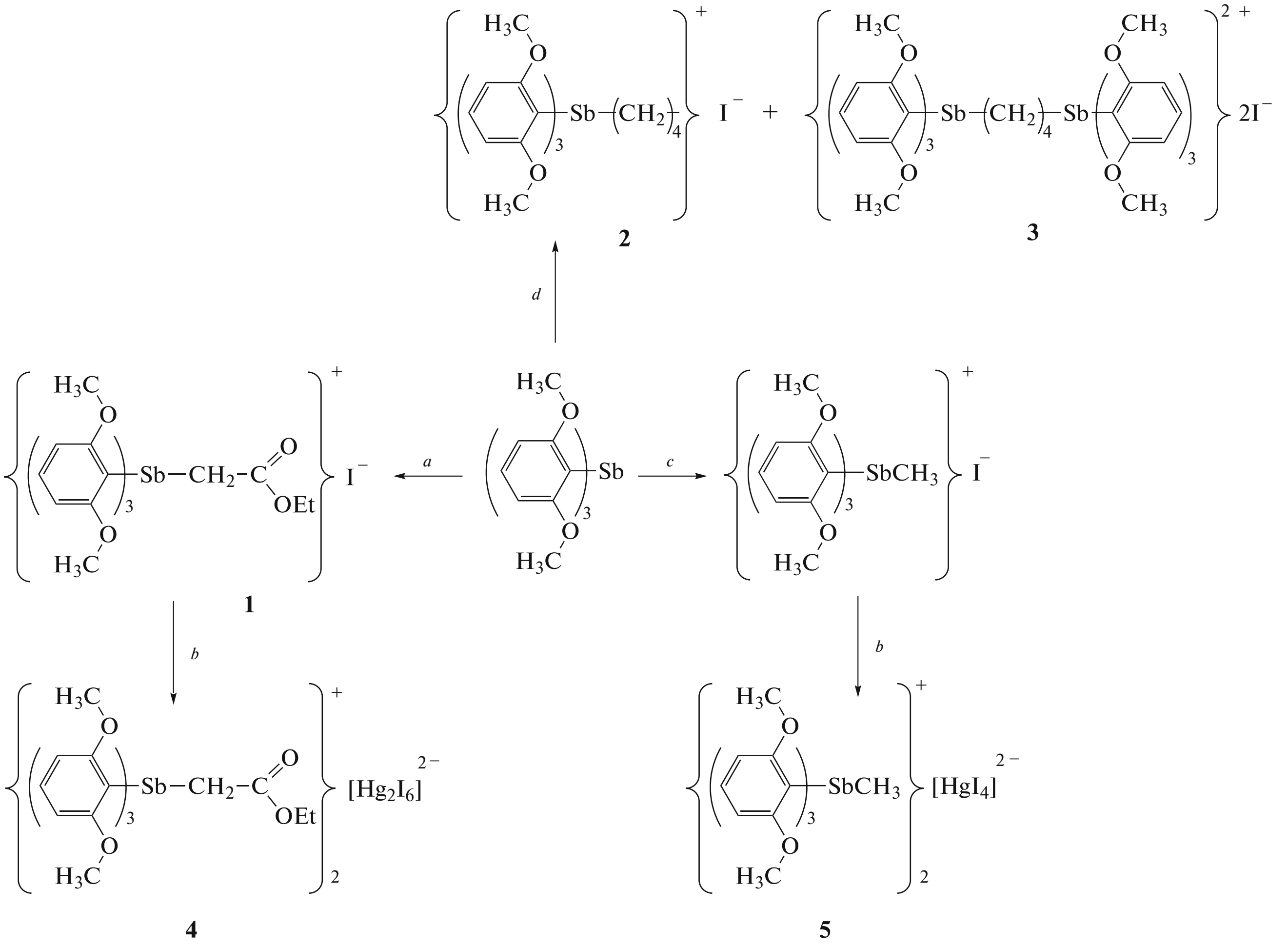
## INTRODUCTION

The addition of an alkyl iodide to alkyl mono- and binuclear antimony(III) derivatives with the formation of Sb–C bonds leads to a wide variety of tetraalkystibonium iodides with the general formula $[R_3R'Sb]^+I^-$ (R = Me, Et, *n*-Pr, *n*-Bu, *iso*-Bu, *n*-Am; R' = Me, Et) and $[R_2SbMe(CH_2)_nMeSbR_2]^{2+}I_2^-$ (R = Et, *tert*-Bu; $n$ = 4, 6), respectively [1–5]. The antimonyorganic compounds with the general formula $[Alk_3SbCH_2E]^+Br^-$, where Alk = *n*-Bu; E = Ph, CH=CH$_2$, CH=CHC(O)OEt, C(O)OMe, CN, CH=CHR; R=Me, *n*-Pr, *iso*-Pr, were synthesized by the reaction of a alkyl bromide and tributylantimony. These complexes are used in the synthesis of secondary alcohols from corresponding aldehydes R'CHO (R' = Ph, 4-ClC$_6$H$_4$, 4-MeC$_6$H$_4$, PhCH=CH, pyridine-2-yl, 4-BrC$_6$H$_4$) [6, 7]. The alkylation of triethyl- and trimethylantimony with methyl was also performed [8, 9].

The reaction between alkyl halides and aryl antimony(III) compounds is less profoundly studied. The alkylation of dimethyl- and diethylantimony with methyliodide leads to the formation of trialkylphenylstibonium iodide [10]. It has not been managed to perform the addition of alkyl halides to antimony(III) compounds with two or three aromatic substituents for a rather long time [3]. For this reason, stibonium salts $[Ar_3RSb]^+[BF_4]^-$ (Ar = Ph, *o*-Tol, *m*-Tol, *p*-Tol, (3,4-Me)$_2$C$_6$H$_3$, (2,4-Me)$_2$C$_6$H$_3$, (2,4,6-Me)$_3$C$_6$H$_2$; R = Me, Ph) are synthesized with the use of more active alkylating agents, such as trimethyloxonium or diphenyliodinium fluoroborates [11–14]. The further effect of sodium iodide leads to corresponding iodide complexes [7, 10]. Trimethyloxonium tetrafluoroborate and methyltrifluoromethanesulfonate alkylate the binuclear antimonyorganic compound 1,2-$(Ph_2Sb)_2C_6H_4$ to result in the complexes $[1,2\text{-}(Ph_2MeSb)_2C_6H_4][BF_4]_2$ and $[1,2\text{-}(Ph_2MeSb)_2C_6H_4]$[OTf], which are efficient catalysts for the hydrosilylation of benzaldehyde under



(a) $ICH_2C(O)OEt$, (b) $HgI_2$, (c) $CH_3I$, (d) $ICH_2(CH_2)_2CH_2I$.

**Fig. 1.** Synthetic scheme for stibonium complexes.

mild conditions [15]. The method for the synthesis of $[Ph_3MeSb]^+Cl^-$ by the reaction between bis(chloromethyl)zinc and triphenylantimony with the further hydrolysis of the formed zincorganic compound is also known [16]. The synthesis of triphenylalkylstibomium zwitterions with the use of bromomethylindium(III) dibromide or phenylmethanesulfonic acid anhydride as alkylating agents in the presence of hydrogen peroxide was also described [17, 18]. Tetraorganylantimony halides $[Ar_3RSb]^+X^-$ [Ar = $2,6\text{-}(OMe)_2C_6H_3$; R = Me, Et, *n*-Bu, $CH_2CH{=}CH_2$; X = Cl, Br, I] were synthesized by the addition of alkyl halides to triarylantimony [19].

The objective of this work was to study the targeted synthesis of stibonium complexes containing potential coordinating centers, which represent the oxygen atoms of carbonyl and/or methoxy groups in the organic substituents at the antimony atom.

## EXPERIMENTAL

The possibility to perform the alkylation of tris(2,6-dimethoxyphenyl)antimony with ethyl ester of iodoacetic acid and 1,4-diiodidebutane was studied. Tris(2,6-dimethoxyphenyl)(ethoxycarbonylmethyl)antimony iodide $\{[2,6\text{-}(OMe)_2C_6H_3]_3SbCH_2C(O)OEt\}^+I^-$ (**1**), tris(2,6-dimethoxyphenyl)(4-iodobytul)antimony iodide $\{[2,6\text{-}(OMe)_2C_6H_3]_3Sb(CH_2)_4I\}^+I^-$ (**2**), and 1,4-di[tris(2,6-dimethoxyphenyl)antimonyl]butane iodide $\{[2,6\text{-}(OMe)_2C_6H_3]_3Sb(CH_2)_4Sb[C_6H_3(OMe)_2\text{-}2,6]_3\}^{2+}I_2^-$ (**3**) were synthesized and characterized by NMR spectroscopy.

The mercury(II) complexes $\{[2,6\text{-}(OMe)_2C_6H_3]_3SbCH_2C(O)OEt\}_2^+[Hg_2I_6]^{2-}$ (**4**) and $\{[2,6\text{-}(OMe)_2C_6H_3]_3SbMe\}_2^+[HgI_4]^{2-}\cdot$ DMSO (**5**) were synthesized by the reaction between mercury diiodide and compounds **1** {[2,6-

$(OMe)_2C_6H_3]_3SbMe\}^+I^-$ and studied by X-ray diffraction.

The reactions were performed at room temperature (Fig. 1).

**Synthesis of complex 1.** To tris(2,6-dimethoxyphenyl)antimony (1.03 g, 1.93 mmol) in chloroform (10 mL), ethyl ester of monoiodoacetic acid (0.41 g, 1.93 mmol) was added under stirring. The reaction mixture was allowed to stand for 12 h. The solvent was evaporated, and the solid residue was washed with diethyl ester (5 × 10 mL). Complex **1** (1.40 g, 97%) with $T_m$ = 184°C (decomp.) was thus synthesized. IR spectrum (ν, $cm^{-1}$): 3059, 3000, 2973, 2941, 2835, 1728, 1716, 1587, 1576, 1475, 1429, 1400, 1388, 1363, 1305, 1256, 1184, 1173, 1126, 1102, 1022, 891, 866, 804, 792, 775, 740, 713, 615, 594, 499.

For $C_{28}H_{34}IO_8Sb$, anal. calcd. (%): C, 45.00; H, 4.59; O, 17.13.

Found (%): C, 44.56; H, 4.10; O, 16.52.

$^1H$ NMR spectrum (δ, ppm): 0.88 t (3H, $CH_2$–$C\underline{\mathbf{H}}_3$, *J* 7.2 Hz), 3.63 s (18H, $CH_3$–O), 3.65 s (2H, $CH_2$–Sb), 3.85 q (2H, $C\underline{\mathbf{H}}_2$–$CH_3$, *J* 7.2 Hz), 6.65 d (6H, 3,5-Ph, *J* 8.4 Hz), 7.49 t (3H, 4-Ph, *J* 8.4 Hz).

$^{13}C\{^1H\}$ NMR spectrum (δ, ppm): 13.65 ($\underline{C}H_3$–$CH_2$), 29.74 ($CH_2$–Sb), 56.52 ($CH_3$–O), 61.69 ($CH_3$–$\underline{C}H_2$), 104.77 (1-Ph), 104.87 (3,5-Ph), 135.78 (4-Ph), 162.15 (2,5-Ph), 167.32 (C=O).

**Synthesis of complexes 2 and 3.** To 1,4-diiodobutane (0.60 g, 1.94 mmol) in chloroform (70 mL), a solution of tris(2,6-dimethoxyphenyl)antimony (1.00 g, 1.88 mmol) in chloroform (100 mL) was added drop by drop under stirring for 30 min. The solution was stirred for 15 min. The solvent was evaporated, and the residue was washed with diethyl ester (5 × 20 mL). A colorless finely crystalline product (1.42 g) representing a mixture of two compounds **2** (~90%) and **3** (~10%) according to NMR data was separated.

**Complex 2.** $^1H$ NMR spectrum (δ, ppm): 1.74 m (2H, 2-Bu), 1.88 m (2H, 3-Bu), 2.77 m (2H, 1-Bu), 3.12 t (2H, 4-Bu, *J* 6.6 Hz), 3.62 s (18H, $CH_3$–O), 6.64 d (6H, 3,5-Ph, *J* 8.3 Hz), 7.47 t (3H, 4-Ph, *J* 8.4 Hz).

$^{13}C\{^1H\}$ NMR spectrum (δ, ppm): 6.11 (1-Bu), 25.65 (2-Bu), 25.73 (1-Bu), 35.00 (3-Bu), 56.52 ($CH_3$–O), 104.29 (1-Ph), 104.73 (3,5-Ph), 135.43 (4-Ph), 162.40 (2,5-Ph).

**Complex 3.** $^1H$ NMR spectrum (δ, ppm): 1.73 m (4H, 2,3-Bu), 2.77 m (4H, 1,4-Bu), 3.57 s (18H, $CH_3$–O), 6.63 d (6H, 3,5-Ph), 7.47 t (3H, 4-Ph).

$^{13}C\{^1H\}$ NMR spectrum (δ, ppm): 26.00 (1,4-Bu), 27.32 (2,3-Bu), 56.71 ($CH_3$–O), 104.13 (1-Ph), 104.87 (3,5-Ph), 135.52 (4-Ph), 162.40 (2,5-Ph).

**Synthesis of complexes 4 and 5.** To complex **1** (0.50 g, 0.66 mmol) in DMSO (20 mL), a solution of mercury diiodide (0.30 g, 0.66 mmol) in DMSO (10 mL) was added under stirring. The solvent was evaporated. Light-green crystals of complex 4 (0.78 g, 97%) with $T_m$ = 185°C were thus synthesized.

IR spectrum (ν, $cm^{-1}$): 3086, 3069, 3001, 2972, 2937, 2834, 1724, 1587, 1576, 1471, 1427, 1385, 1362, 1306, 1286, 1257, 1180, 1102, 1032, 1017, 889, 869, 804, 800, 781, 771, 740, 715, 708, 617, 596, 584, 499.

For $C_{56}H_{68}Hg_2I_6O_{16}Sb_2$, anal. calcd. (%): C, 27.99; H, 2.85; O, 10.65.

Found (%): C, 27.60; H, 2.73; O, 10.60.

Complex **5** was obtained by the same method from tris(2,6-dimethoxyphenyl)methylstibonium iodide (0.50 g, 0.74 mmol) synthesized as described in [19] and mercury diiodide (0.17 g, 0.37 mmol). Light-green crystals of complex 5 (0.65 g, 97%) with $T_m$ = 168°C was thus separated.

IR spectrum (ν, $cm^{-1}$): 3151, 3066, 3024, 3006, 2991, 2970, 2937, 2848, 2835, 1587, 1576, 1471, 1427, 1303, 1257, 1172, 1151, 1102, 1035, 1020, 950, 891, 829, 781, 773, 740, 717, 696, 594.

For $C_{50}H_{60}HgI_4O_{12}Sb_2 \cdot 0.92C_2H_6SO$ ($C_{51.84}H_{65.52}HgI_4O_{12.92}S_{0.92}Sb_2$), anal. calcd. (%): C, 33.18; H, 3.53; O, 11.02.

Found (%): C, 33.07; H, 3.61; O, 10.70.

**IR spectra** of these complexes were recorded on a FSM 2202 Fourier-transform IR spectrometer in the region of 500–7000 $cm^{-1}$ ad KBr pellets. Elemental analysis was performed on a Carlo Erba 1106 CHN-analyzer.

**NMR spectra** were recorded on a Bruker Avance III 500 spectrometer with a working frequency of 500.03 MHz for $^1H$ and 125.73 MHz for $^{13}C$. The spectra were taken in the solution of a complex (25 mg) in $CDCl_3$ (0.6 mL). The solvent signals used as standards were δ = 7.26 ppm for residual protons in the $^1H$ NMR spectrum and δ = 77.23 ppm for the $^{13}C$ NMR spectra. The assignment of signals was accomplished on the basis of homonuclear $^1H,^1H$-COSY and heteronuclear $^{13}C,^1H$-HMBC and $^{13}C,^1H$-HSQC two-dimensional correlations.

**X-ray diffraction analysis** of complexes **4** and **5** was performed on a Bruker KAPPA APEX II diffractometer with an area CCD-detector (Mo$K_\alpha$ radiation, graphite monochromator, ω–φ-scanning). The structures were solved by direct methods and refined by the full-matrix least-squares technique in the anisotropic approximation using the SHELXL-97 software [20]. The positions of hydrogen atoms were calculated geometrically and refined "as riding" (the parameters of hydrogen atoms were calculated in every refinement cycle from the coordinates of corresponding carbon atoms). In complex **5**, the $[HgI_4]^{2-}$ anion and a DMSO molecule are partially disordered (the second position of a solvent molecule has not been determined). The selected crystallographic characteristics, results of X-ray diffraction experiment, and refinement details

**Table 1.** Crystallographic characteristics, data of X-ray diffraction experiment, and refinement details for the structures of complexes **4** and **5**

| Parameter | **4** | **5** |
|---|---|---|
| Formula | $C_{56}H_{68}Hg_2I_6O_{16}Sb_2$ | $C_{51.84}H_{65.52}HgI_4O_{12.92}S_{0.92}Sb_2$ |
| Formula weight | 2403.18 | 1876.57 |
| Temperature, K | 200(2) | 173(2) |
| Symmetry system | Monoclinic | |
| Space group | $C2/c$ | $P2_1/n$ |
| $a$, Å | 28.2103(12) | 17.8459(9) |
| $b$, Å | 11.2798(5) | 30.1024(16) |
| $c$, Å | 23.1211(8) | 24.3373(13) |
| β, deg | 104.929(1) | 106.236(2) |
| $V$, Å$^3$ | 7108.9(5) | 12552.7(11) |
| $Z$ | 4 | 8 |
| $\rho_{calcd}$, g/cm$^3$ | 2.245 | 1.986 |
| $\mu(\mathrm{Mo}K_\alpha)$, mm$^{-1}$ | 7.721 | 5.347 |
| $F$(000) | 4448 | 7109 |
| Crystal size, mm | 0.60 × 0.45 × 0.20 | 0.65 × 0.45 × 0.35 |
| θ angle range, deg | 2.07–30.07 | 1.26–26.37 |
| Number of measured reflections | 81829 | 295857 |
| Number of independent reflections | 10421 ($R_{int}$ = 0.0386) | 25677 ($R_{int}$ = 0.0626) |
| Transmittance min/max | 0.0903/0.3073 | 0.1287/0.2562 |
| GOOF on $F^2$ | 1.012 | 1.089 |
| $R$-factor for $I > 2\sigma(I)$ | $R_1$ 0.0305, $wR_2$ 0.0799 | $R_1$ 0.0524, $wR_2$ 0.0967 |
| $R$-factor for all reflections | $R_1$ = 0.0376, $wR_2$ = 0.0839 | $R_1$ = 0.1002, $wR_2$ = 0.1092 |
| Residual electron density, max/min, e/Å$^3$ | 2.491/–2.252 | 1.773/–2.342 |

for the structures of complexes **4** and **5** are given in Table 1. The X-ray diffraction data for complexes **4** and **5** were deposited with the Cambridge Structure Database (CCDC nos. 1549982 and 1549983).

## RESULTS AND DISCUSSION

The positions and intensities of signals in the one-dimensional $^1$H and $^{13}$C NMR spectra of complexes **1**–**3** was in agreement with the presumable addition of ethyliodoacetate and 1,4-diiodobutane to tris(2,6-dimethoxyphenyl)antimony, but did not unambiguously confirmed it. To perform the targeted assignment of signals and confirm the structures of these stibonium compounds, two-dimensional $^{13}$C,$^1$H-corelation spectra were recorded. The analysis of the obtained data argues for the formation of the new Sb–$C_{Alk}$ bond in complexes **1** and **2**. Cross-peaks corresponding to the interaction of the methylene group bonded to the antimony atom with the *unco*-carbon atom of the phenyl moiety can be clearly seen in the two-dimensional spectra of these complexes (Fig. 2). It has not been managed to detect this signal for complex **3** due to a small content of this product in the analyzed mixture (~10%). However, the absence of signals typical for a moiety with the C–I bond from this complex in the high-field $^{13}$C NMR spectrum region allows us to presume the substitution of both halide atoms in 1,4-diiodobutane. Another confirmation of symmetry in the structure of complex **3** is the existence of only two signals from the aliphatic moiety in the $^1$H and $^{13}$C NMR spectra.

The composition of complexes **4** and **5** formed by the reactions of $HgI_2$ with complex **1** and $\{[2,6\text{-}(OMe)_2C_6H_3]_3SbMe\}^+I^-$ is governed by the molar ratio of reagents (1 : 1, 1 : 2). According to X-ray diffraction data, the mercury atoms in the centrosymmetric binuclear $[Hg_2I_6]^{2-}$ anion have a distorted tetrahedral coordination (IHgI angles are 98.122(9)°–125.590(12)°, Fig. 3).

The terminal iodine atoms are more strongly bonded to the mercury atoms (Hg–$I_{term}$, 2.6874(4), 2.6945(3) Å) in comparison with the bicoordinated bridging iodine atoms (Hg–$I_{bridge}$, 2.9246(4), 2.9260(4) Å). Their values are comparable with the parameters of the complexes $[Ph_4Sb]_2^+[Hg_2I_6]^{2-}$ and $[p\text{-}Tol_4Sb]_2^+[Hg_2I_6]^{2-}$ (Hg–$I_{term}$, 2.6910(4) and 2.6996(4) Å; 2.7028(3) and 2.7222(3) Å; Hg–$I_{bridge}$,

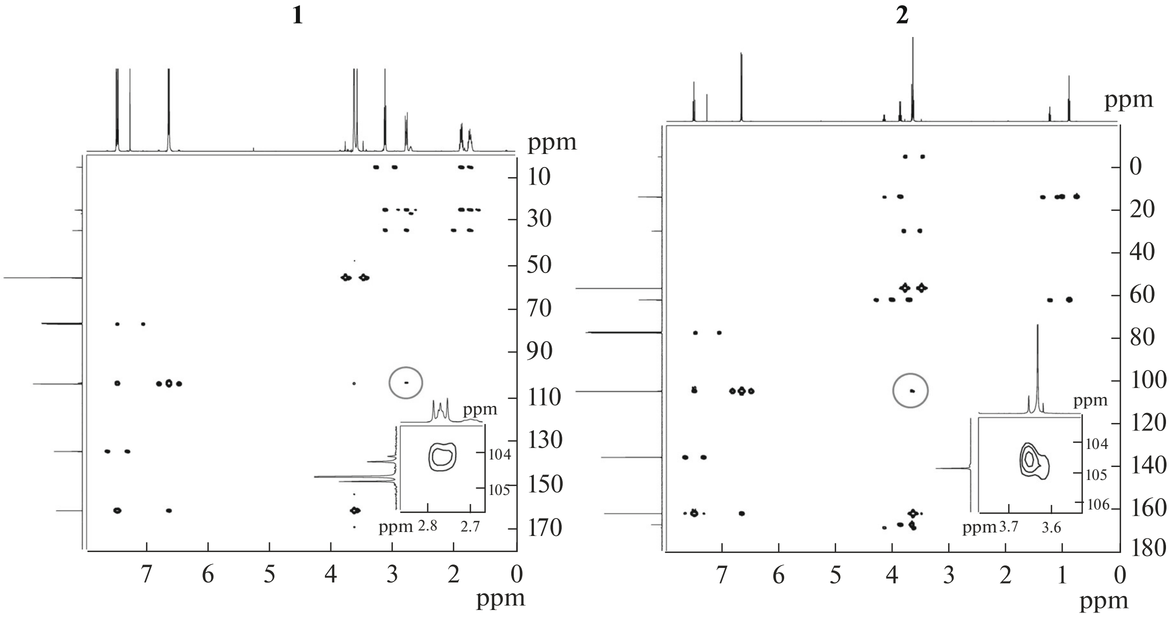


**Fig. 2.** $^{13}C,^{1}H$-HMBC spectra of complexes **1** and **2**.

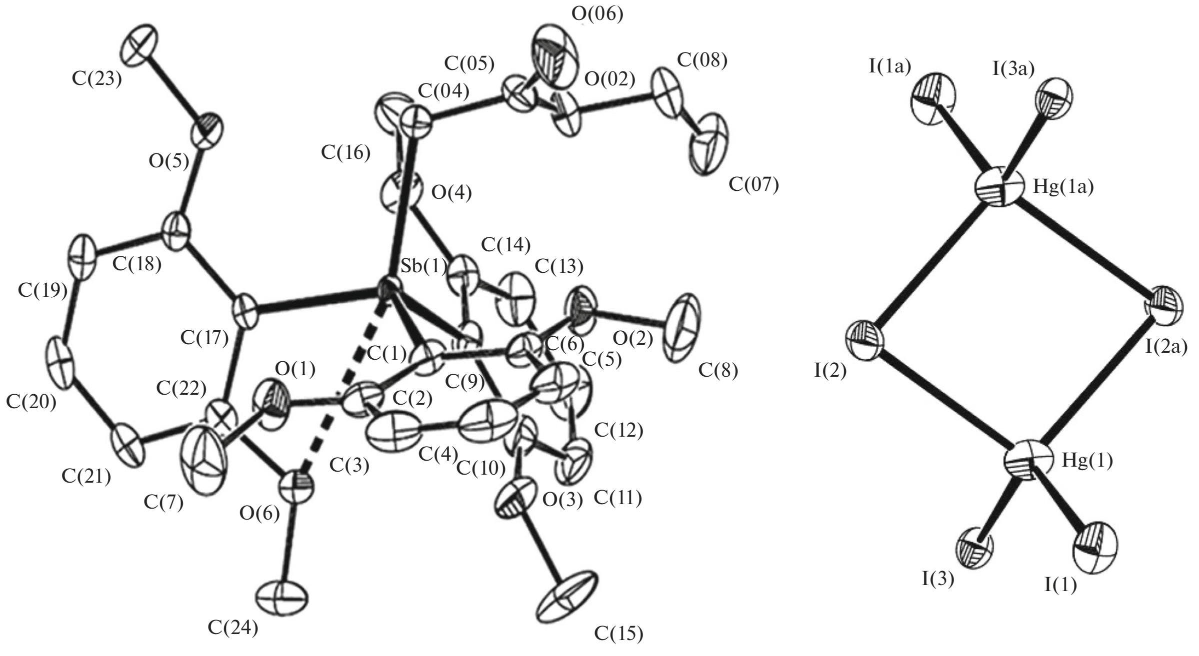


**Fig. 3.** General view of the $\{[2,6\text{-}(OMe)_2C_6H_3]_3SbCH_2C(O)OEt\}^+$ cation and the $[Hg_2I_6]^{2-}$ anion of complex **4**. Hydrogen atoms are omitted.

2.8250(4) and 3.0748(5)Å; 2.8539(3) and 2.9163(3) Å) [21]. The Hg(1)I(2)Hg(1a)I(2a) and I(1)I(3)I(1a)I(3a) are almost mutually perpendicular, and the angle between them is 87.47°. The terminal iodine atoms link $[Hg_2I_6]^{2-}$ anions into chains running along axis *c* by means of I···I contacts (3.944 Å) (Fig. 4).

The unit cell of complex **5** contains four structurally nonequivalent $\{[2,6\text{-}(OMe)_2C_6H_3]_3SbMe\}^+$ cations and two $[HgI_4]^{2-}$ anion (Fig. 5). The IHgI angles in the tetrahedral anions are 102.66(2)°–115.64(2)°. The Hg–I distances are nonequivalent (2.670(5)–2.956(7) Å), as also takes place in the complex$[n\text{-}Tol_4Sb]_2^+[HgI_4]^{2-}$ (2.7719(13)–2.7908(12) Å) [21].

In the cations of complexes **4** and **5**, the antimony and oxygen atoms of methoxy groups are characterized by additional coordination (Sb···O distances are

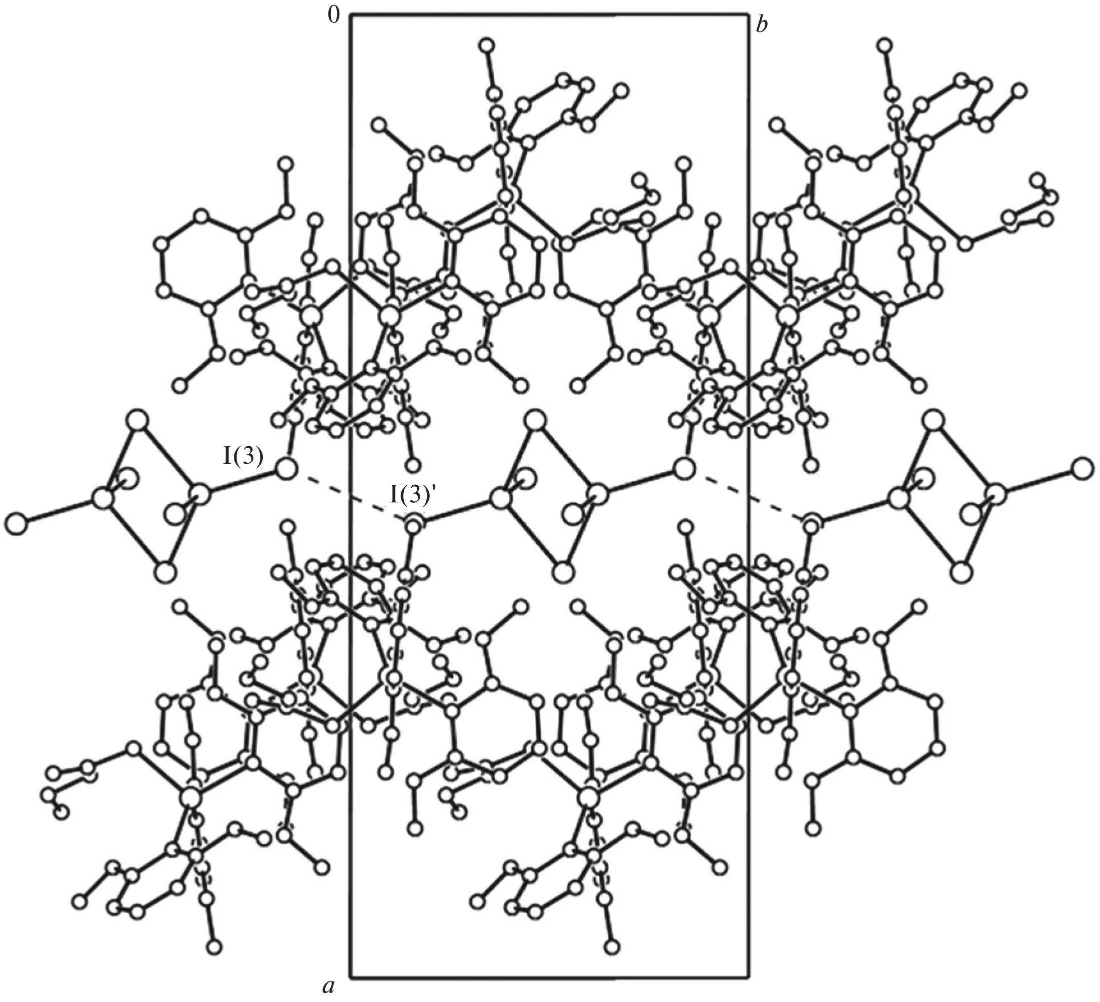


**Fig. 4.** Fragment of the structural packing of complex **4**. Hydrogen atoms are omitted.

2.853–2.895 Å, and the van der Waals radii of Sb and O in sum are 3.7 Å [22]), which leads to the appearance of the contribution from trigonal-bipyramidal component to the tetrahedral structure (Figs. 3 and 5). The CSbC bond angles (103.28(14)°–116.68(14)° in complex **4** and 103.7(4)°–115.5(4)° in complex **5**) deviate from the value ideal for a tetrahedron. The bond lengths are the following: Sb–$C_{Alk}$, 2.140(4); Sb–$C_{Ar}$, 2.085(3)–2.093(3) Å in complex **4**; Sb–$C_{Me}$, 2.085(10)–2.113(9); Sb–$C_{Ar}$, 2.051(9)–2.114(8) Å in complex **5** (the Sb and C covalent radii in sum are 2.18 Å [22]).

Let us mention that the Sb–$C_{Ar}$ bond lengths and CSbC bond angles in cations **4** and **5** and oxide tris(2,6-dimethoxyphenyl)antimony have close values (2.073(4)–2.098(5) Å and 107.47(18)°–116.24(18)° [23]).

The carbonyl oxygen atoms participate in the formation of hydrogen bonds $C_{Ar}(21)$–H(21)···O(06) (H···O 2.493 Å), linking the cations of complex **4** into a chain running along axis *b* (Fig. 6). The incorporation of a solvent molecule into the coordination sphere of the mercury atom takes place in the $[HgI_3(DMSO)]^-$ anion [24]. However, DMSO molecules in complex **5** participate only in the formation of weak hydrogen bonds O···H–$C_{Ar}$ (O···H, 2.48–2.67 Å) with the stibonium cation.

The absorption bands in the IR spectra of complexes **1**–**5** were identified in compliance with the data [23, 25]. The vibration frequencies of bonds in the spectra of complexes **1**, **4**, and **5** appear at 1102 $cm^{-1}$ [$\nu_s$(O–$C_{Alk}$)]; 1022, 1256 (**1**), 1017, 1257 (**4**), 1020, 1257 (**5**) $cm^{-1}$ [$\nu_{as}$($C_{Me}$–O–$C_{Ar}$)]; 2835, 2941 (**1**), 2834, 2937 (**4**), 2835, 2937 (**5**) $cm^{-1}$ [$\nu_{s,as}$(C–H)]; 1716, 1728 (**1**), 1724 (**4**) $cm^{-1}$ [$\nu_{as}$(OCO)].

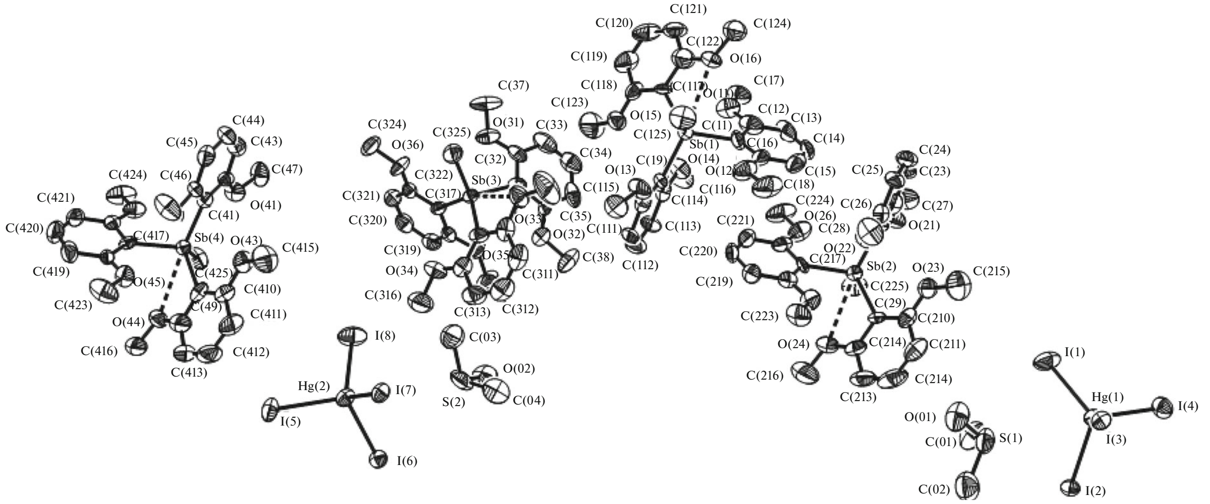


**Fig. 5.** Indepenent part in the structure of complex **5**. Hydrogen atoms, second anion–solvent disordering component, and some atom notations are omitted.

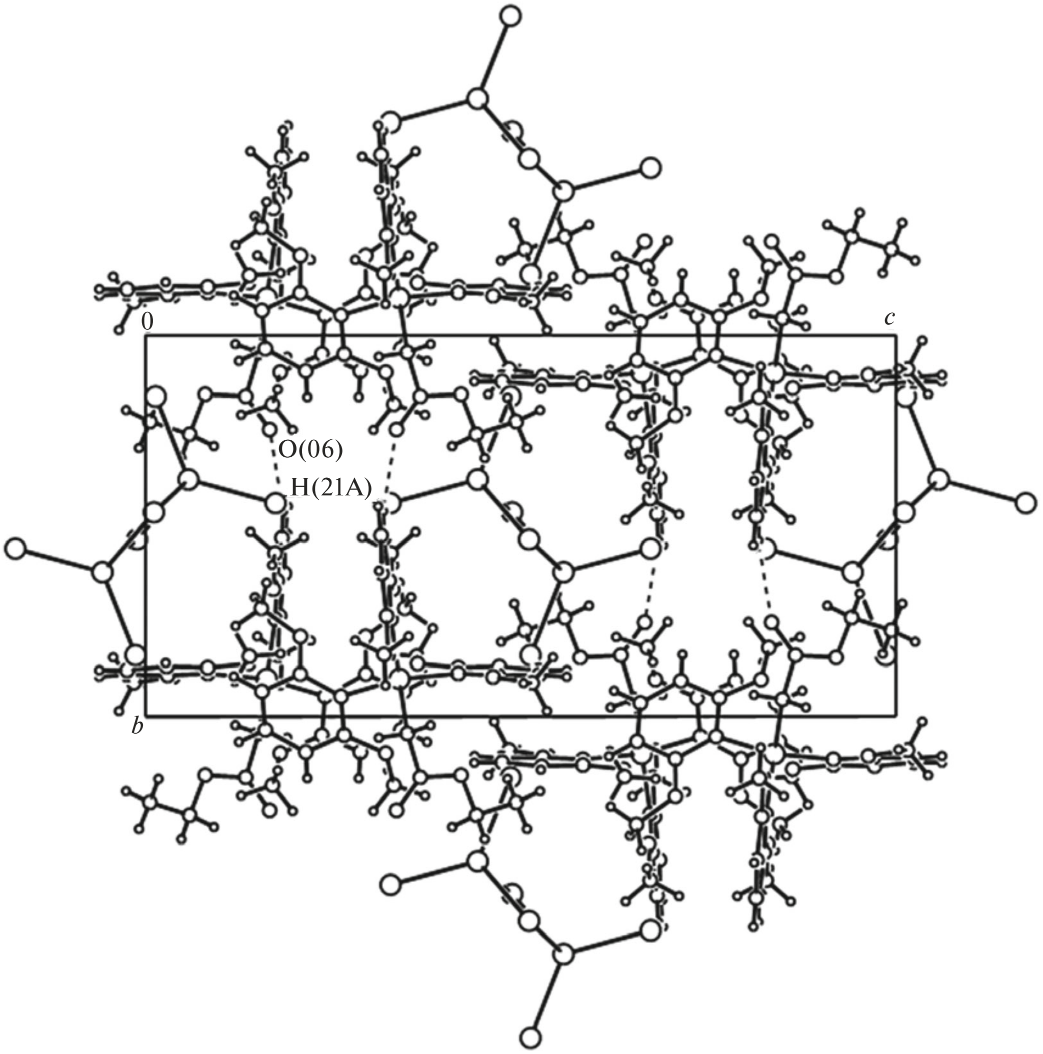


**Fig. 6.** Fragment of the structural packing of complex **4** with hydrogen bonds (dashed lines) forming the chains of cations along axis *b*.

## CONCLUSIONS

Tris(2,6-dimethoxyphenyl)antimony alkylation providing the possibility to broaden the series of stibonium salts has been studied. The composition of the stibonium cations $\{[2,6\text{-}(OMe)_2C_6H_3]_3SbCH_2C(O)OEt\}^+$, $\{[2,6\text{-}(OMe)_2C_6H_3]_3Sb(CH_2)_4I\}^+$, and $\{[2,6\text{-}(OMe)_2C_6H_3]_3Sb(CH_2)_4Sb[C_6H_3(OMe)_2\text{-}2,6]_3\}^{2+}$ is governed by the nature of an alkylating agents, ethyl iodoacetate and 1,4-diiodobutane. The synthetic approach has been proposed, and the earlier unknown triarylalkylstibonium mercurates $\left\{[2,6\text{-}(OMe)_2C_6H_3]_3SbCH_2C(O)OEt\right\}_2^+[Hg_2I_6]^{2-}$ and $\left\{[2,6\text{-}(OMe)_2C_6H_3]_3SbMe\right\}_2^+[HgI_4]^{2-}$ containing potential coordination centers in the organic substituents at the antimony atom have been structurally characterized.

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