ISSN 1070-3632, Russian Journal of General Chemistry, 2021, Vol. 91, No. 7, pp. 1361–1367. © Pleiades Publishing, Ltd., 2021.
Russian Text © The Author(s), 2021, published in Zhurnal Obshchei Khimii, 2021, Vol. 91, No. 7, pp. 1100–1107.

Synthesis and Structure of Antimony Complex Compounds $[(4\text{-}N,N\text{-}Me_2C_6H_4)_3MeSb]I$ and $[(4\text{-}N,N\text{-}Me_2C_6H_4)_3MeSb]_2[Hg_2I_6]\cdot 2DMSO$

I. V. Egorova[a],*, **V. V. Zhidkov**[a], **I. P. Grinishak**[a], **N. A. Rodionova**[a],
I. Yu. Bagryanskaya[b], **and N. V. Pervukhina**[c]

[a] *Blagoveschensk State Pedagogical University, Blagoveschensk, 675000 Russia*
[b] *N.N. Vorozhtsov Novosibirsk Institute of Organic Chemistry, Siberian Branch of the Russian Academy of Sciences, Novosibirsk, 630090 Russia*
[c] *A.V. Nikolaev Institute of Inorganic Chemistry, Siberian Branch of the Russian Academy of Sciences, Novosibirsk, 630090 Russia*
**e-mail: bgpu.chim.egorova@mail.ru*

Received April 26, 2021; revised May 25, 2021; accepted May 27, 2021

Abstract—It was first found that the alkylation of tris(4-*N*,*N*-dimethylaminophenyl)stibine with methyliodide leads to the formation of tris(4-*N*,*N*-dimethylaminophenyl)methylstibonium iodide which decomposes in hot water to tris(4-*N*,*N*-dimethylaminophenyl)stibine. The reaction of $[(4\text{-}N,N\text{-}Me_2C_6H_4)_3MeSb]I$ with HgI_2 in DMSO yields $[(4\text{-}N,N\text{-}(Me)_2C_6H_4)_3MeSb]_2[Hg_2I_6]\cdot 2DMSO$, in which a significant distortion of the tetrahedral cation coordination is caused by the additional interaction of the oxygen atom of the DMSO molecule with the antimony atom.

Keywords: tris(4-*N*,*N*-dimethylaminophenyl)stibine, methyl iodide, mercury(II) iodide, X-ray diffraction analysis, NMR spectroscopy

DOI: 10.1134/S1070363221070148

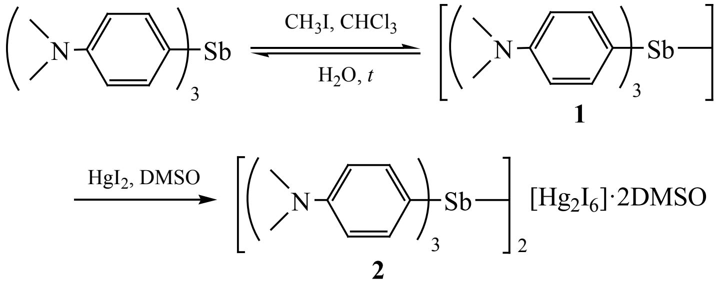
Tetraorganylstibonium halides of the general formula R_4SbHlg, among which the most studied are phenyl derivatives of pentavalent antimony, are used in various fields of analytical chemistry and have a great synthetic potential, in particular, tetraalkylstibonium halides react with aldehydes to form addition products, the hydrolysis of which gives the corresponding alcohols [1–7]. Therefore, it is of interest to study the synthesis and structure of such compounds.

The reaction of pentaarylantimony with hydrohalic acids is the main method for obtaining tetraarylstibonium halides [3, 8]. One of the methods for the synthesis of tetraorganylstibonium halides $[ArAlk_3Sb]Hlg$ containing aryl and alkyl substituents at the antimony atom is the reaction of $ArAlk_2Sb$ with haloalkanes [9]. However, for a long time the attempts to attach alkyl halides to antimony(III) compounds with two or three aromatic substituents were unsuccessful [3, 8]. Therefore, trimethyloxonium borofluorides are applied as alkylating agents to synthesize the stibonium compounds $[Ar_3MeSb]X$ (Ar = Ph, Tol, Mes, $(3,4\text{-}Me)_2C_6H_3$,$(2,4\text{-}Me)_2C_6H_3$, X = BF_4) [10–12].

For the first time, the addition of alkyl halides to triarylstibine was observed in reactions involving $(2,6\text{-}(MeO)_2C_6H_3)_3Sb$. To prove the structure of tetraorganylstibonium halides $[Ar_3RSb]Hlg$ [Ar = $2,6\text{-}(MeO)_2C_6H_3$; R = Me, Et, *n*-Bu, $CH_2CH{=}CH_2$; Hlg = Cl, Br, I], the authors present elemental analysis and NMR spectroscopy data [13]. We studied the alkylation of tris(2,6-dimethoxyphenylstibine) with iodoacetic acid ethyl ether and 1,4-diiodbutane. Structure of the compounds $[Ar_3RSb]^{n+}I^-_n$, Ar = $2,6\text{-}(MeO)_2C_6H_3$; $n = 1$, R = Me, $(CH_2)_4I$, $CH_2C(O)OEt$; $n = 2$, R = $(CH_2)_4SbAr_3$ and $[Ar_3SbCH_2C(O)OEt]_2[Hg_2I_6]$, $[Ar_3MeSb]_2[HgI_4]\cdot DMSO$ was confirmed by the X-ray and NMR spectroscopy methods [14].

Thus, to date the possibility of triarylstibine alkylation with alkyl halides and ethyl iodoacetate has been shown only by the example of $[2,6\text{-}(MeO)_2C_6H_3]_3Sb$ [13, 14]. In

Scheme 1.



this regard, the study of the alkyl halides reactions with other antimony(III) fully substituted aromatic compounds and the determination of the structure of the resulting alkylaryl stibonium compounds is an urgent task.

The aim of this work was to study the alkylation of tris(4-*N*,*N*-dimethylaminophenyl)stibine with methyliodide, to develop the directed synthesis of complexes with the tris(4-*N*,*N*-dimethylaminophenyl)-methylstibonium cation, and to study their structure and properties.

Triphenylstibine does not react with alkyl halides [3]. The presence of strong electron-donor groups $N(Me)_2$ in phenyl substituents as a whole suggests that the basicity (and hence, nucleophilicity) of the tris(4-*N*,*N*-dimethylaminophenyl)stibine molecule increases compared to triphenylstibine, and, as a result, the possibility of alkylation with an antimony atom appears. Another nucleophilic center of the tris(4-*N*,*N*-dimethylaminophenyl) molecule capable of being subjected to an electrophilic attack by methyl is the amino group nitrogen atom. Its basicity decreases due to the p–π conjugation of its unshared electron pair with the benzene ring π-electrons. It is known that the nucleophilicity of atoms decreases along the N–P–As–Sb–Bi series, and methyliodide, as a soft reagent in nucleophilic reactions, prefers soft nucleophiles [8, 15]. It can be expected that the alkylation of the tris(4-*N*,*N*-dimethylaminophenyl)-stibine molecule, containing two competing nucleophiles (nitrogen and antimony atoms), with methyliodide will proceed through the antimony atom to form tris(4-*N*,*N*-dimethylaminophenyl)methylstibonium iodide.

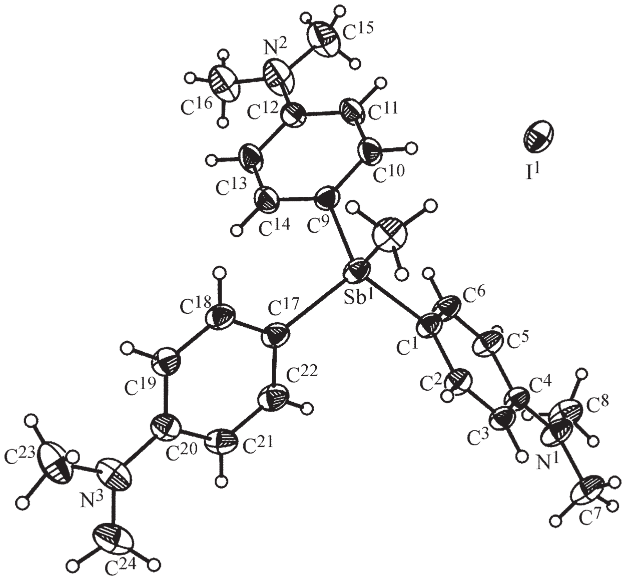


Fig. 1. General view of the cation and anion of the compound **1** (CCDC 2067967).

Alkylation of tris(4-*N*,*N*-dimethylaminophenyl)-stibine with methyliodide was carried out in chloroform at room temperature. It was found that, regardless of the molar ratio of the reagents (from 1 : 1 to 1 : 10) and the experiment duration (24–120 h), alkylation proceeds through the antimony atom to form colorless crystals of the complex $[(4\text{-}N,N\text{-}Me_2C_6H_4)_3MeSb]I$ **1** (Scheme 1).

To determine the space structure of compound **1**, single crystals suitable for X-ray diffraction analysis were obtained by slow crystallization from chloroform. We note that the tetraorganylstibonium derivatives containing the $4\text{-}N,N\text{-}Me_2C_6H_4$ substituent were not structurally characterized. According to the X-ray analysis data, complex **1** consists of I^- and $[(4\text{-}N,N\text{-}Me_2C_6H_4)_3MeSb]^+$ ions. The antimony atom in the cation has a distorted tetrahedral coordination. The values of the CSbC angles are in the range 106.0(1)°–114.6(1)° (Fig. 1). The main crystallographic characteristics, experimental data, and parameters of the structure refinement are given in Table 1.

Tetraphenylstibonium halides are stable during storage, non-volatile, and readily soluble in water,

Table 1. Crystallographic characteristics of compounds **1** and **2**, experimental data, and parameters of structure refinement

Parameter	**1**	**2**
Formula	$C_{25}H_{33}IN_3Sb$	$C_{54}H_{78}Hg_2I_6N_6O_2S_2Sb_2$
M	624.19	2313.42
Temperature, K	296(2)	120(2)
Crystal system	Triclinic	
Space group	$P\overline{1}$	$P\overline{1}$
a, Å	9.1698(3)	10.3319(6)
b, Å	10.9934(4)	12.4940(7)
c, Å	14.3748(5)	14.1746(8)
α, deg	71.618(2)	84.2800(10)
β, deg	79.193(2)	73.0820(10)
γ, deg	84.026(2)	82.8230(10)
V, $Å^3$	1349.19(8)	1732.98(17)
Z	2	1
d_{calc}, g/cm^3	1.536	2.217
$\mu(MoK_\alpha)$, mm^{-1}	2.182	7.959
F(000)	616	1072
Crystal size, mm	0.60 × 0.20 × 0.15	0.21 × 0.18 × 0.16
Data collection range for θ, deg	2.51–30.06	2.071–33.715
Number of measured reflexes	49040	26246
Number of independent reflexes	7901 (R_{int} 0.0299)	12568 (R_{int} 0.0413)
Transmission, min/max	0.354/0.736	0.273/0.375
GOOF by F^2	1.097	0.954
R-Factors for $I > 2\sigma(I)$	R_1 0.0296, wR_2 0.0790	R_1 0.0402, wR_2 0.0570
R-Factors for all reflexes	R_1 0.0385, wR_2 0.0844	R_1 0.0734, wR_2 0.0659
Residual electron density, max/min, $e/Å^3$	1.069/–1.219	1.052/–1.200

therefore they can be purified by recrystallization from an aqueous solution. At the same time, tetraalkylstibonium halides are hygroscopic and decompose in hot aqueous solutions to release alkyl halide and trialkylstibine [3]. Since complex **1** contains aryl and methyl substituents at the antimony atom, it is of interest to study its stability and the possibility of decomposition to triarylstibine.

Heating an aqueous solution of compound **1** at 80°C leads to the formation of an insoluble product, the structure of which was established by NMR spectroscopy, agrees with the elemental analysis and IR spectroscopy data, and corresponds to tris (4-*N*,*N*-dimethylaminophenyl) stibine. According to the ^{1}H NMR data, the spectrum of the obtained compound contains signals of eighteen methyl protons in the region of 2.80 ppm, six aromatic protons in the region of 6.67 ppm, and six aromatic protons in the region of 7.21 ppm. The ^{13}C NMR spectra contain signals in the regions of 113.6, 135.8, 137.1, and 153.9 ppm corresponding to the chemical shifts of carbon nuclei in the benzene ring, and the signal of carbon atoms of methyl fragments in the region of 39.1 ppm. The obtained result points to the decomposition of the stibonium salt with the formation of tris (4-*N*,*N*-dimethylaminophenyl)stibine (Scheme 1).

Organylantimony(III, V) halides, reacting with heavy metal halides (Hg, Cd, and Bi), form complex compounds in which they act as a ligand or a stibonium cation [14, 16, 17]. The reaction of complex **1** with mercury(II) iodide (1 : 1 mol) in DMSO was studied (Scheme 1). The reaction product $[(4\text{-}N,N\text{-}Me_2C_6H_4)_3MeSb]_2[Hg_2I_6]\cdot 2DMSO$ **2** is a crystal highly soluble in acetone, chloroform, and DMSO. The absorption bands in the IR spectra of compounds **1** and **2** were assigned in accordance with the data of [18, 19]. These spectra contain absorption bands at 2798 and 1433 cm^{-1}, i.e. in the region of stretching and bending vibrations of the C-H bonds of the methyl group bound to the nitrogen atom. The band at 1594 cm^{-1} can be attributed to stretching vibrations of aromatic C–C bonds. In the IR

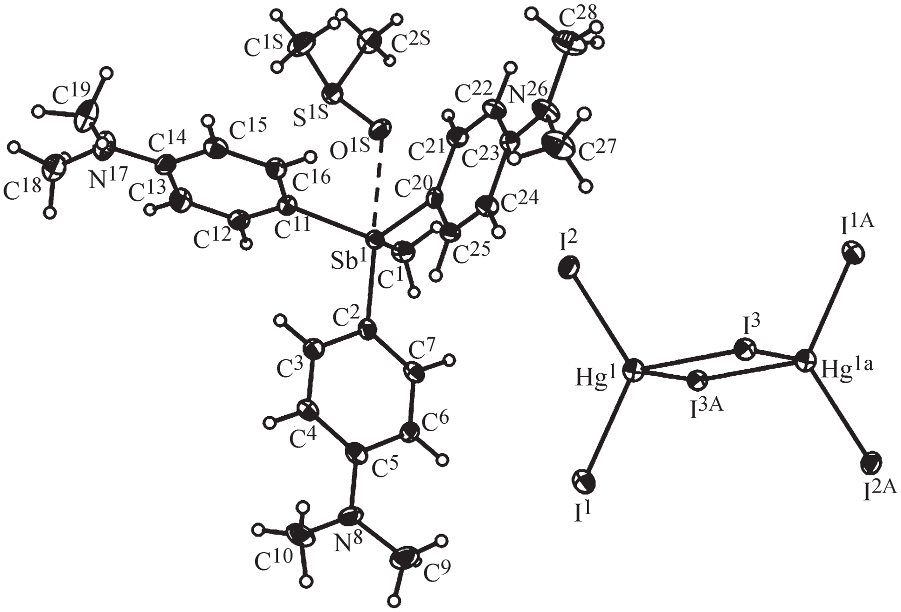


Fig. 2. General view of the compound **2** anion and its cation coordinated with the DMSO molecule (CCDC 2059337).

spectrum of complex **2**, a band of stretching vibrations ν(SO) is observed at 1016 cm^{-1}, the shift of which to the long-wave region of the spectrum in comparison with the free DMSO molecule points to the DMSO coordination through the oxygen atom [20]. The IR spectroscopy data are consistent with the X-ray results.

In the crystal of compound **2**, there is an additional $Sb\cdots O^{1S}$ (2.831 Å) coordination between the antimony atoms of the stibonium cation and the oxygen of the solvate DMSO molecule (the sum of the van der Waals radii 3.58 Å [21]), which determines the stibonium cation configuration as being intermediate between tetrahedral and trigonal-bipyramidal (Figs. 2, 3). The O^{1S} atoms of the DMSO molecule and C^2 atoms of one of the aryl substituents are located in the pseudoaxial plane (the angle $C^2Sb^1O^{1S}$ is 171.97°). The pseudo-equatorial positions are occupied by the C^1, C^{11}, and C^{20} atoms of the methyl and two aryl substituents; the values of the $C_{eq}SbC_{eq}$ angles are 107.21(15)°–119.50(17)°, and of the $C_{ax}SbC_{eq}$ angles: 101.92(16)°–103.09(16)° (Figs. 2, 3).

The Sb–C_{Me} bond lengths are 2.097(3) (**1**) and 2.104(4) Å (**2**), the Sb–C_{Ar} distances lie in the interval 2.078(2)–2.087(3) (**1**), 2.087(4)–2.091(4) Å (**2**) (the sums of the covalent radii of Sb and C_{sp^3} or C_{sp^2} atoms are 2.15 or 2.12 Å) [22]. In the trigonal-bipyramidal tetraphenylstibonium iodide molecule, the Sb–C_{Ph} bonds are longer: Sb–C_{ax} 2.141(3) Å, Sb–C_{eq} 2.103(3)–2.117(3) Å, and the Sb–I distance is 3.341 (1) Å [23]. The reduction of the Sb–C_{Ar} distances in compound **1** is caused by the presence of an amino group in the phenyl substituent, the nitrogen atom of which can participate in the *p*–*π*–*d* conjugation.

Nitrogen atoms of the amino groups in complexes **1** and **2** have the sp^2 hybridization [the CNC angles are 116.7(4)°–121.7(3)° (**1**) and 116.9(4)°–120.9(4)° (**2**)]. Substituents at all the nitrogen atoms form planes; the maximum outgoing of a nitrogen atom from the corresponding root-mean-square planes does not exceed 0.02 Å. Conjugation of a free electron pair of the nitrogen atom with the aromatic ring leads to a decrease in the N–C_{Ar} bond lengths of 1.359(4)–1.374(6) (**1**), 1.373(5)–1.381(5) Å (**2**) compared to the N–C_{Me} distance of 1.420(6)–1.454(4) (**1**), 1.445(6)–1.458(6) Å (**2**) (sum of the covalent radii 1.47 Å).

Centrosymmetric binuclear anions $[Hg_2I_6]^{2-}$ form tetrahedra joined along edges; the IHgI angles are 94.128(10)°–123.833(12)°. The Hg_2I_2 cycle is flat, the torsion angle $I^3Hg^1I^{3a}Hg^{1a}$ is 0.00(1)°. The terminal iodine atoms are more strongly bound to mercury atoms [distances 2.7063(4) and 2.7118(4) Å] than the two-coordinated bridging iodine atoms [Hg–I-μ_2 2.8691(4) and 3.0011(4) Å]. These values are comparable with parameters of the $[Hg_2I_6]^{2-}$ anion of complexes containing $\{[(2,6\text{-MeO})_2C_6H_3]_3SbCH_2C(O)OEt\}^+$, $[Ph_4Sb]^+$, and $[p\text{-Tol}_4Sb]^+$ cations, where these distances vary within the intervals 2.6874(4)–2.7222(3) Å for terminal and

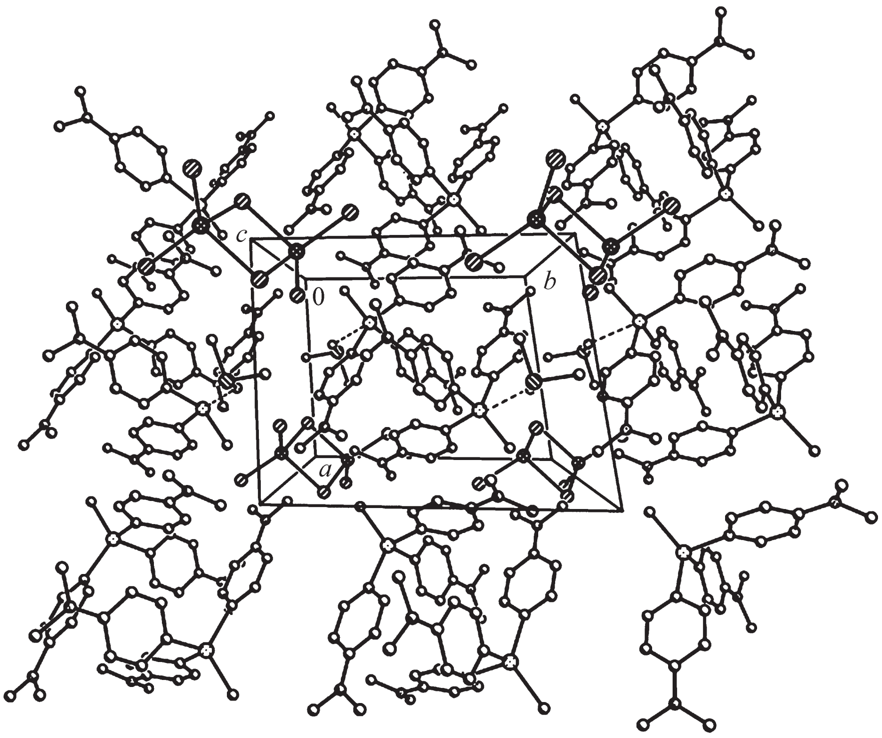


Fig. 3. Fragment of the crystal package of compound **2** (CCDC 2059337). The dotted line indicates the intermolecular contacts between the cation and the DMSO solvate molecule.

2.8250(4)–3.0748(5) Å for bridging iodine atoms [14]. The $Hg^1I^3Hg^{1a}I^{3a}$ and $I^1I^2I^{1a}I^{2a}$ planes are almost perpendicular with the angle between them of 86.31°.

Iodine atoms participate in the formation of the crystal structure **1,** forming weak $I\cdots H–C_{Ar}$ (2.985 Å) and $I\cdots H–C_{Me}–Sb$ (2.991 Å) contacts. Potential coordinating centers—nitrogen atoms of amino groups—participate in weak intermolecular interactions with the DMSO molecule ($N\cdots H–C_{Me}$ 2.628 Å) **2**. The sum of the van der Waals radii of iodine (nitrogen) and hydrogen atoms is 3.08 (2.65) Å [21].

Thus, a method has been proposed for the synthesis of triarylalkylstibonium complexes $[(4\text{-}N,N\text{-}Me_2C_6H_4)_3MeSb]I$ and $[(4\text{-}N,N\text{-}Me_2C_6H_4)_3MeSb]_2[Hg_2I_6]\cdot 2DMSO$, which is based on the alkylation reaction of tris(4-*N*,*N*-dimethylaminophenyl) stibine at the antimony atom. The crystal structure of the complexes was confirmed by the X-ray analysis method. The coordination number of the antimony atom in the $[(4\text{-}N,N\text{-}(Me)_2C_6H_4)_3MeSb]_2[Hg_2I_6]\cdot 2DMSO$ solvate increases to five, which results in the cation configuration as intermediate between tetrahedral and trigonal-bipyramidal.

EXPERIMENTAL

The NMR spectra were recorded on a Bruker Avance AV-300 spectrometer with a proton resonance frequency of 300 MHz. For the 13C nucleus, a cross-polarization technique with suppression of dipole-dipole interactions and magic angle rotation was used. The duration of the 90° pulse for protons was 4 μs, the polarization transfer time was 500-3000 ms, the rotation speed was 5, 7, and 16 kHz, the sample diameter was 4 mm, the time between pulses was 20 s, and the number of accumulations was 512. The hydrogen signals were recorded using a single-pulse sequence, the inter-pulse delay was 3 s. The error in determining the chemical shift did not exceed 1 ppm. The spectra were recorded at 300 K. The IR spectra of the compounds were recorded on a Fourier spectrometer FSM 2202 in tablets with KBr. The elemental analysis was performed on a Carlo Erba CHN analyzer (model 1106).

The X-ray analysis was performed on a Bruker APEX II CCD diffractometer (MoK_α-radiation, graphite monochromator, ω scanning). The structures were interpreted by the direct method and refined by the OLS in the anisotropic full-matrix approximation for F^2. The positions of the hydrogen atoms (except for the hydrogen of the methyl group at the C^1 atom in complex **2**) were calculated geometrically and refined in the rider model (the parameters of the hydrogen atoms were calculated in each refinement cycle according to the coordinates of the corresponding carbon atoms). The hydrogen atoms of the methyl group at the C^1 **2** atom were identified from the difference Fourier synthesis of the electron density. The maximum value of the residual electron density peak in the vicinity of the C^1 atom of complex **2** does not exceed 0.5 *e*. The X-ray analysis parameters of compounds **1** and **2** are deposited at the Cambridge Crystallographic Data Center (CCDC 2067967, 2059337). All calculations were carried out using the SHELX-97 and SHELXL software packages [24-26].

Tris(4-*N*,*N*-dimethylaminophenyl)methylstibonium iodide (1). A solution of 9.00 g (53.41 mmol) of methyliodide in 30 mL of chloroform was added drop by drop with stirring to 4.82 g (10.00 mmol) of tris(4-*N*,*N*-dimethylaminophenyl)stibine [3] in 50 mL of chloroform. The mixture was kept in the dark at room temperature in a closed ampoule for 48 h. The solvent was evaporated. The crystals were washed with diethyl ether (3×15 mL). Yield 6.18 g (99%), mp 220°C (decomp.). IR spectrum, ν, cm^{-1} 3029 (C_{Ar}–H), 2951 [$\nu_{as}(CH_3)$], 2924 [$\nu_{as}(CH_3)$], 2853 [$\nu_s(CH_3)$], 2798 (NC–H), 1594 (C_{Ar}–C_{Ar}), 1460 [$\delta(CH_3)$], 1433 [δ(NC–H)], 1396 [$\delta(CH_3)$], 1202 (CNC), 1165 (CNC), 947 [$\delta(CH_3)$], 939 [$\delta(CH_3)$], 549 (Sb–C). Found, %: C 49.21; H 5.76; N 5.98. $C_{25}H_{33}IN_3Sb$. Calculated, %: C 48.10; N 5.33; N 6.73.

Decomposition of tris(4-*N*,*N*-dimethylaminophenyl)methylstibonium iodide. A solution of 3.00 g of compound **1** in 30 mL of water was heated at 80°C for 0.5 h. The precipitate was filtered, washed sequentially with water (25 mL), diethyl ether (2×15 mL), dried, and recrystallized (chloroform–alcohol). Yield of tris(4-*N*,*N*-dimethylaminophenyl)stibine is 1.39 g (60%), mp 229°C (mp 229°C [3]). IR spectrum, ν, cm^{-1}: 3056 (C_{Ar}–H), 2797 (NC–H), 1588 (C_{Ar}–C_{Ar}), 1199 [ν_s(CNC)], 1167 (CNC), 944 [$\delta(CH_3)$], 519 (Sb–C). ^{1}H NMR spectrum, ppm: 7.21 d (6H, 2.6-H, $J = 9.0$ Hz), 6.67 d (6H, 3.5-H, $J = 9.0$ Hz), 2.80 s (18H, Me). Found, %: C 58.82; H 6.69; N 9.03. $C_{24}H_{30}N_3Sb$. Calculated, %: C 58.93; H 6.82; N 9.16.

Bis[tris(4-*N*,*N*-dimethylaminophenyl)methylantimony] bis(μ_2-iodo)tetraiododimercurate(II), solvate with dimethyl sulfoxide (2). A solution of 0.36 g (0.80 mmol) of mercury(II) iodide in 20 mL of DMSO was added to 0.50 g (0.80 mmol) of compound **1** dissolved in 20 mL of DMSO. The mixture was kept at room temperature for 12 h. The solvent was removed. Yield 0.81 g (87%), mp 163°C. IR spectrum, ν, cm^{-1}: 3032 (C_{Ar}–H), 2952 [$\nu_{as}(CH_3)$], 2924 [$\nu_{as}(CH_3)$], 2853 [$\nu_s(CH_3)$], 2798 (NC–H), 1594 (C_{Ar}–C_{Ar}), 1458 [$\delta(CH_3)$], 1433 [δ(NC–H)], 1399 [$\delta(CH_3)$], 1197 (CNC), 1161 (CNC), 1016 (SO), 954 [$\delta(CH_3)$], 944 [$\delta(CH_3)$], 549 (Sb–C). Found, %: C 26.94; H 2.99; N 4.12. $C_{54}H_{78}Hg_2I_6N_6O_2S_2Sb_2$. Calculated, %: C 28.02; N 3.40, N 3.63.

AUTHOR INFORMATION

I.V. Egorova, ORCID: http://orcid.org/0000-0001-8799-608X

I.P. Grinishak, ORCID: http://orcid.org/0000-0003-2547-1629

I. Yu. Bagryanskaya, ORCID: http://orcid.org/0000-0001-7760-5540

N.V. Pervukhina, ORCID: http://orcid.org/0000-0001-6200-5657

ACKNOWLEDGMENTS

The authors of the work express their gratitude to the Center of the Nesmeyanov Institute of Organoelement Compounds of the Russian Academy of Sciences for the Study of the Structure of Molecules and the Chemical Service Center for Collective Use of the SB RAS for conducting X-ray diffraction analysis.

CONFLICT OF INTEREST

No conflict of interest was declared by the authors.

REFERENCES

1. Potratz, H.A. and Rosen, J.M., *Anal. Chem.*, 1949, vol. 21, no. 10, p. 1276. https://doi.org/10.1021/ac60034a037
2. Willard, H.H., and Perkins, L.R., *Anal. Chem.*, 1953, vol. 25, no. 11, p. 1634. https://doi.org/10.1021/ac60083a016
3. Kocheshkov, K.A., Skoldinov, A.P., and Zemlyanskii, N.N., *Metody elementorganicheskoi khimii. Sur'ma, vismut* (Methods of Organoelement Chemistry. Antimony, Bismuth), Moscow: Nauka, 1976, p. 184.
4. Huang, Y.-Z. and Liao, Y., *J. Org. Chem.*, 1991, vol. 56, p. 1381. https://doi.org/10.1021/jo00004a010
5. Huang, Y.-Z., Zhang, L.-J., Chen, C., and Guo, G.-Z., *J. Organometal. Chem.*, 1991, vol. 412, p. 47. https://doi.org/10.1016/0022-328X(91)86040-W
6. Zhang, L.-J., Huang, Y.-Z., and Huang, Z.-H., *Tetrahedron Lett.*, 1991, vol. 32, no. 45, p. 6579.

https://doi.org/10.1016/0040-4039(91)80226-V

7. Zhang, L.-J., Mo, X.-S., Huang, J.-L, and Huang, Y.-Z., *Tetrahedron Lett*., 1993, vol. 34, no. 10, p. 1621. https://doi.org/10.1016/0040-4039(93)85024-Q
8. Rochow, E.G., Hurd, D.T., and Lewis, R.N., *The Chemistry of Organometallic Compounds*, New York: John Wiley, 1957, p. 344. https://doi.org/10.1002/jps.3030461026
9. Brinnand, M.E., Dyke, W.J.C., Jones, W.H., and Jones, W.J., *J. Chem. Soc*., 1932, p. 1815. https://doi.org/10.1039/JR9320001815
10. Henry, M.C. and Wittig, G., *J. Am. Chem. Soc*., 1960, vol. 82, no. 3, p. 563. https://doi.org/10.1021/ja01488a017
11. Henning, D., Kempter, G., Ahrens, E., Benecke, K.D., and Paul, P., *Z. Chem*., 1967, vol. 7, no. 12, p. 463. https://doi.org/10.1002/zfch.19670071213
12. Henning, D., Kempter, G., and Worlitzer, K.-D., *Z. Chem*., 1969, vol. 9, no. 8, p. 306. https://doi.org/10.1002/zfch.19690090813
13. Wada, M., Miyake, S., Hayashi, S., Ohba, H., Nobuki, S., Hayase, S., and Erabi, T., *J. Organometal. Chem*., 1996, vol. 507, p. 53. https://doi.org/10.1016/0022-328X(95)05716-3
14. Egorova, I.V., Zhidkov, V.V., Grinishak, I.P., Bagryanskaya, I.Yu, Pervukhina, N.V., El'tsov, I.V., and Kurat'eva, N.V., *Russ. J. Inorg. Chem*., 2019, vol. 64, no. 1, p. 28. https://doi.org/10.1134/S0036023619010078
15. Pearson, R.G., *J. Chem. Educ*., 1968, vol. 45, no. 9, p. 581. https://doi.org/10.1021/ed045p581
16. Egorova, I., Zhidkov, V., Zubakina, I., Rodionova, N., and Eltsov, I., *J. Organometal. Chem*., 2020, vol. 907, p. 121077. https://doi.org/10.1016/j.jorganchem.2019.121077
17. Burt, J., Levason, W., and Reid, G., *Coord. Chem. Rev*., 2014, vol. 260, p. 65. https://doi.org/10.1016/j.ccr.2013.09.020
18. Smith, B.C., *Infrared Spectral Interpretation: A Systematic Approach*, Boca Raton: CRC Press, 1998.
19. Nakamoto, K., *Infrared and Raman Spectra of Inorganic and Coordination Compounds*, New York: J. Wiley, 1986.
20. Cotton, F.A., Francis, R., and Horrocks, W.D.Jr., *J. Phys. Chem*., 1960, vol. 64. P. 1534. https://doi.org/10.1021/j100839a046
21. Mantina, M., Chamberlin, A.C., Valero, R., Cramer, C.J., and Truhlar, D.G., *J. Phys. Chem*., 2009, vol. 113, p. 5806. https://doi.org/10.1021/jp8111556
22. Cordero, B., Gomez, V., Platero-Prats, A.E., Reves, M., Echeverria, J., Cremades, E., Barragan, F., and Alvarez, S., *J. Chem. Soc., Dalton Trans*., 2008, vol. 21, p. 2832. https://doi.org/10.1039/B801115J
23. Baker, L.-J., Rickard, C.E.F., and Taylor, M.J., *J. Chem. Soc., Dalton Trans*., 1995, p. 2895. https://doi.org/10.1039/dt9950002895
24. Sheldrick, G.M., *SHELX-97*, Programs for Crystal Structure Analysis, Göttingen: Göttingen University, 1997.
25. Sheldrick, G.M., *Acta Crystallogr*., 2007, vol. 64, p. 112. https://doi.org/10.1107/S0108767307043930
26. Sheldrick, G.M., *Acta Crystallogr. C*, 2015, vol. 71, p. 3. https://doi.org/10.1107/S2053229614024218