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Bromoantimonates(III) vs. Bromobismuthates(III): Differences in the Tendency for the Formation of Polynuclear Complexes

A. N. Usoltsev[a], I. V. Korol'kov[a], and S. A. Adonin[a, *]
[a] *Nikolaev Institute of Inorganic Chemistry, Siberian Branch, Russian Academy of Sciences, Novosibirsk, Russia*
**e-mail: adonin@niic.nsc.ru*

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Abstract—Pyridine-based Sb(III) bromide complexes with doubly charged cations, $(PyC_3)_3[Sb_2Br_9]_2$ (**I**), $(PyC_4)[Sb_2Br_8]$ (**II**), $(PyC_5)_2[\alpha\text{-}Sb_4Br_{16}]$ (**III**), $(PyC_6)_2[Sb_2Br_{10}]$ (**IV**), $(4\text{-}MePyC_2)_2[Sb_2Br_{10}]$ (**V**), $(4\text{-}MePyC_3)_2[\alpha\text{-}Sb_4Br_{16}]$ (**VI**), and $(4\text{-}MePyC_5)_2[\alpha\text{-}Sb_4Br_{16}]$ (**VII**), were synthesized and characterized by X-ray diffraction (CCDC nos. 2204718–2204724). The structures of these compounds were compared with the structures of related bromobismuthates(III).

Keywords: antimony, bismuth, halide complexes, polynuclear complexes, X-ray diffraction analysis

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INTRODUCTION

The homoligand halide complexes, or halometallates (HMs) form an extensive class of coordination compounds. These compounds have been known for many decades and remain objects of keen interest to inorganic chemists [1, 2]. Their potential applications include photocatalysis (especially for Cu [3, 4] and Ag [5, 6] derivatives), piezoelectric materials [7–9], and other fields. Particular attention is attracted by Pb(II) iodide complexes, which can be used [10–18] as light-absorbing components of solar cells and photodetectors.

The greatest crystal chemical diversity is inherent in HMs formed by Group 14 and 15 elements: there are anions of various composition and geometry (up to 8- and 18-nuclear structures for Bi(III) [19, 20] and Pb(II) [21], respectively) and 1D, 2D, and 3D (for Pb(II) and Sn(II)) metal-organic frameworks. More than 40 structural types of HMs are known for Bi(III), and it may be expected that this is not the final number [22].

The question of why HM of type X is formed in reaction Y is still unanswered. It is believed that fragments of low nuclearity exist in solutions, and they are self-assembled to give more complex structures only when crystalline phases are formed [23]. It was proved that the outcome of HM formation (i.e., formation of HM of a definite type) is affected by several factors, in particular, the used solvent and the reactant ratio. However, the most important factor is the nature of the cation the salt of which is used for the synthesis [24]. Unfortunately, numerous attempts to find correlations between the nature of cation–anion noncovalent interactions and the preferred formation of one or another type of HM [23, 25] have not yet met with success. It cannot be ruled out that there is no general answer to this question. Nevertheless, it is clear that new studies in this area require a larger amount of experimental data on the structures of various HMs.

Previously, it was noted [26] that haloantimonates (HAs) and halobismuthates (HBs) often have structural similarity (this seems reasonable, considering positions of these elements in the Periodic Table). Our goal was to evaluate the similarity of HA and HB structures formed under analogous conditions in the presence of the same organic cations. For our experiments, we chose 1,1'-(alkane-1,ω-diyl)-bis(pyridinium) dibromides. A considerable advantage of these salts is the possibility of obtaining series of target compounds in nearly quantitative yields by reactions of specified substituted pyridines with 1,ω-dibromoalkanes (both types of reagents are relatively cheap and available) [27–30]. In what follows, these compounds are designated as PyC_n^{2+} (Py is substituted pyridine, n is the number of methylene groups).

In this study, we obtained seven new bromoantimonates(III): $(PyC_3)_3[Sb_2Br_9]_2$ (**I**), $(PyC_4)[Sb_2Br_8]$ (**II**), $(PyC_5)_2[\alpha\text{-}Sb_4Br_{16}]$ (**III**), $(PyC_6)_2[Sb_2Br_{10}]$ (**IV**), $(4\text{-}MePyC_2)_2[Sb_2Br_{10}]$ (**V**), $(4\text{-}MePyC_4)_2[\alpha\text{-}Sb_4Br_{16}]$ (**VI**), and $(4\text{-}MePyC_5)_2[\alpha\text{-}Sb_4Br_{16}]$ (**VII**). The crystal structures of the products are discussed, and comparison with relevant Bi(III) complexes prepared and discussed previously is presented.

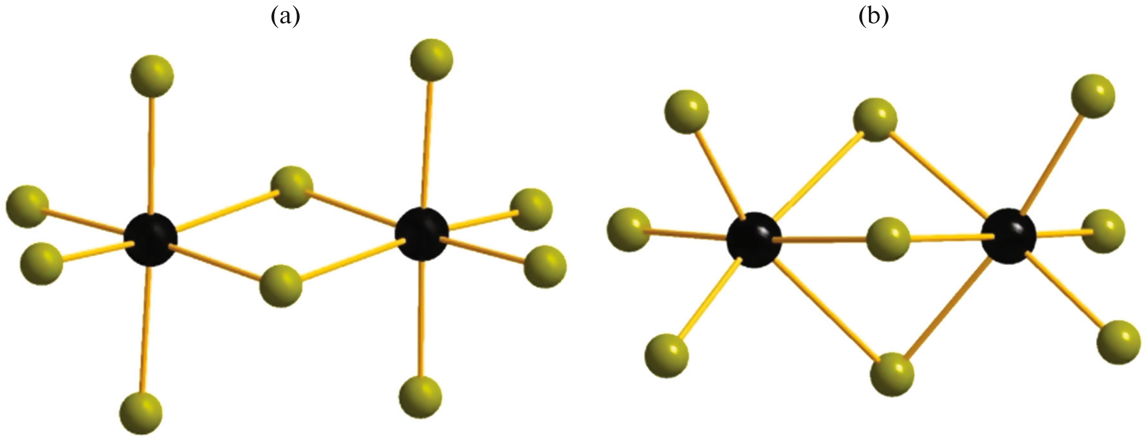

Fig. 1. (a) $[Sb_2Br_{10}]^{4-}$ and (b) $[Sb_2Br_9]^{3-}$ anions. Hereinafter, Sb atoms are shown in black and Br is colored olive green.

EXPERIMENTAL

The synthesis was performed in air. The starting compounds were received from commercial sources. 1,1'-(Alkane-1,ω-diyl)-bis(pyridinium) dibromides were prepared using the general procedure described in the literature [31]: the specified pyridine and 1, ω-dibromoalkane (2.1 : 1) were dissolved in boiling acetonitrile and refluxed for 12 h. This gave products as white powders in nearly quantitative yields.

Synthesis of I–VII. In initial experiments, Sb_2O_3 (100 mg, 0.34 mmol) was dissolved in HBr (4 mL), and a solution of an equimolar amount of PyC_nBr_2 in HBr (4 mL) was added. In all cases, pale yellow crystals suitable for X-ray diffraction formed after some period of time (from a few tens of minutes to 12 h). After structure determination, the reactant ratio in the subsequent syntheses was appropriately changed, which resulted in a higher yield (73–87%).

X-ray diffraction. The experimental details are given in Supplementary Information. The single-crystal X-ray diffraction data were collected at 130 K on an Agilent Xcalibur diffractometer with an AtlasS2 array detector (MoK_α radiation, λ = 0.71073 Å, ω-scan mode) at T = 140 K. The integration, application of absorption corrections, and determination of unit cell parameters were carried out using the CrysAlisPro program package. The crystal structures were solved using the SHELXT program package and refined by the full-matrix least-squares method in the anisotropic (except for hydrogen atoms) approximation using the SHELX 2014/7 [32] and ShelXle [33] software.

The full tables of interatomic distances and bond angles, atom coordinates, and atomic displacement parameters were deposited with the Cambridge Crystallographic Data Center (CCDC no. 2204718–2204724; https://www.ccdc.cam.ac.uk/structures/).

Powder X-ray diffraction study was carried out on a Shimadzu XRD-7000 diffractometer. The X-ray diffraction patterns were constructed and the data were processed using the X'Pert Plus program package.

Table 1. Sb–Br_{term} and Sb–μ_2-Br bond lengths in **I**, **IV**, and **V**

Complex	Sb–Br_{term}, Å	Sb–μ_2-Br, Å
I	2.567–2.670	2.988–3.216
IV	2.668–2.792	2.953–3.064
V	2.602–2.809	3.138–3.208

RESULTS AND DISCUSSION

Structures **I–VII** correspond to four types. Structures **I**, **IV**, and **V** contain binuclear $[Sb_2Br_{10}]^{4-}$ and $[Sb_2Br_9]^{3-}$ anions, respectively, which consist of two $\{SbBr_6\}$ octahedra sharing a face or an edge (Fig. 1). These motifs are fairly typical of HMs of Group 15 elements [26], but not Sb(III): according to CCDC, only 4 and 15 structures with the $[Sb_2Br_{10}]^{4-}$ [34–36] and $[Sb_2Br_9]^{3-}$ [35, 37–45] anions, respectively, are known.

The Sb–Br_{term} and Sb–μ_2-Br bond lengths in **I**, **IV**, and **V** are summarized in Table 1. Their comparison with the published data for the corresponding bromobismuthates(III) shows that the M–Br_{term} distances are similar, while M–μ_2-Br distances are markedly longer for Sb(III) compounds. According to CCDC data, the longest Bi–μ_2-Br distances for $[Bi_2Br_{10}]^{4-}$ and $[Bi_2Br_9]^{3-}$ are 3.172 [46] and 3.229 Å [25]. The most probable cause is the effect of lone pair, which should be much more pronounced for Sb(III).

The alpha-isomers (according to classification that we proposed previously [2]) of the $[M_4X_{16}]^{4-}$ anions are not unusual for Bi(III) [2]. For Sb(III), they have been described in several iodoantimonates [47, 48] and two bromoantimonates [49, 50]. Among **I–VII**,

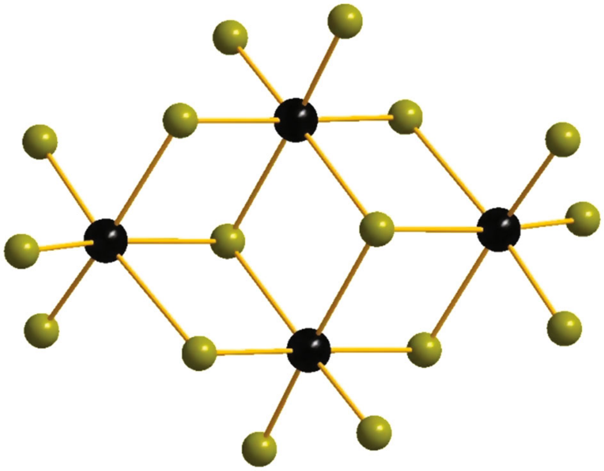

Fig. 2. [α-Sb_4Br_{16}]$^{4-}$ anion.

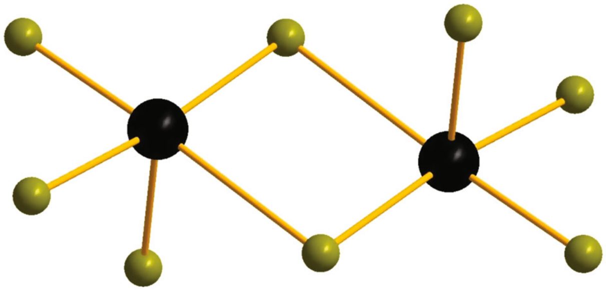

Fig. 3. [α-Sb_2Br_8]$^{2-}$ anion.

there are three compounds with these types of anions (**III**, **VI**, and **VII**) (Fig. 2).

Complex **II** contains binuclear [Sb_2Br_8]$^{2-}$ anions, in which the Sb atoms are five-coordinate (Fig. 3). Structures of this type are very rare for Bi(III) [51, 52], which most often forms an octahedral coordination environment in halide complexes. The Sb–Br_{term} and Sb–μ_2-Br bond lengths are 2.524–2.659 and 2.940–3.156 Å, respectively.

Table 2 summarizes the currently available data on the structures of bromoantimonates(III) and bromobismuthates(III) with 1,1'-(alkane-1,ω-diyl)-bis(pyridinium) dications including both the results described in this study and those reported previously (some of which were obtained in our previous studies). As follows from original publications, virtually all of these compounds were prepared by the same (or, at least, a similar) route (reaction of acidic Sb(III) solutions with an excess of the bromide ligand). This fact allows a direct comparison of the data.

A few interesting points deserve mentioning. First, in the case of Sb(III), there are six cases in which the structure contains discrete tetranuclear anions ([α-Sb_4Br_{16}]$^{4-}$ almost in all cases), whereas there are no such bromobismuthates(III) (compounds with similar cations contain binuclear anions almost in all cases). Second, in none of the cases, complexes with mononuclear [$SbBr_6$]$^{3-}$ are formed. In the cases where Bi(III) forms [$BiBr_6$]$^{3-}$, Sb(III) forms bi- or even trinuclear anions (a case of unusual polymorphism with the (4-MePy)C_4^{2+} cation was described in our previous study [57] and appears especially interesting). These observations can be summarized as follows: under

Table 2. Bromoantimonates(III) and bromobismuthates(III) with 1,1'-(alkane-1,ω-diyl)-bis(pyridinium) dications (integrated data)

Cation	Sb(III)	Ref.	Bi(III)	Ref.
PyC_2	[Sb_2Br_{10}]$^{4-}$/[$SbBr_5$]$^{2-}$	[53]	[Bi_2Br_{10}]$^{4-}$	[54]
PyC_3	[Sb_2Br_9]$^{3-}$ (**I**)	This work	[Bi_2Br_9]$^{3-}$	[55]
PyC_4	[Sb_2Br_8]$^{2-}$ (**II**)	″	[$BiBr_6$]$^{3-}$	[56]
PyC_5	[α-Sb_4Br_{16}]$^{4-}$ (**III**)	″	[Bi_2Br_9]$^{3-}$	[56]
PyC_6	[Sb_2Br_{10}]$^{4-}$ (**IV**)	″	[$BiBr_6$]$^{3-}$	[28]
(4-MePy)C_2	[Sb_2Br_{10}]$^{4-}$ (**V**)	″	[Bi_2Br_{10}]$^{4-}$	[27]
(4-MePy)C_3	[α-Sb_4Br_{16}]$^{4-}$ (**VI**)	″	n/a	n/a
(4-MePy)C_4	[Sb_2Br_8]$^{2-}$/[α-Sb_4Br_{16}]$^{4-}$	[57]	[$BiBr_6$]$^{3-}$	[27]
(4-MePy)C_5	[α-Sb_4Br_{16}]$^{4-}$ (**VII**)	This work	[Bi_2Br_{10}]$^{4-}$	[27]
(3-MePy)C_2	[Sb_2Br_9]$^{3-}$	[55]	[Bi_2Br_{11}]$^{5-}$/[$BiBr_6$]$^{3-}$	[58]
(3-MePy)C_3	[β-Sb_4Br_{16}]$^{4-}$	[55]	[Bi_2Br_9]$^{3-}$	[55]
(3-MePy)C_4	[α-Sb_4Br_{16}]$^{4-}$	[55]	n/a	n/a

similar conditions, bismuth tends to form discrete HM anions with lower nuclearity than antimony.

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CONFLICT OF INTEREST

The authors declare that they have no conflicts of interest.

SUPPLEMENTARY INFORMATION

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ADDITIONAL INFORMATION

This article is prepared for the memorial issue in tribute to the Corresponding Member of the Russian Academy of Sciences K.Yu. Zhizhin on his 50th birthday.

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