

SYNTHESIS AND STRUCTURE OF BISMUTH COMPLEXES [p-Tol₄E]⁺₃[Bi₃I₁₂]³⁻·HOCH₂CH₂OC₂H₅ E=P, Sb

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The interaction of equimolar amounts of tetra-*p*-tolylphosphonium and tetra-*p*-tolylstibonium iodides with bismuth triiodide in 2-ethoxyethanol leads to formation of the complexes [p-Tol₄E]⁺₃[Bi₃I₁₂]³⁻·HOCH₂CH₂OC₂H₅ E=P (I), Sb (II). X-ray diffraction analysis of compounds (I) and (II) has shown that in the cations of complexes I and II the coordination of phosphorus and antimony atoms is tetrahedral (angles equal: CPC 107.3(3)°–113.4(4)° (I), CSbC 105.4(3)°–113.7(3)° (II); bond lengths for P–C and Sb–C are 1.761(9)–1.815(7) Å and 2.085(8)–2.099(8) Å, respectively). In trinuclear anions [Bi₃I₁₂]³⁻ the terminal fragments BiI₃ (Bi–I_{term} 2.8714(5)–2.9181(5) Å (I), 2.8867(5)–2.9248(6) Å (II)) are bonded to the central bismuth atom through six μ₂-bridging iodine atoms (Bi–I_{br} 3.0454(6)–3.3891(6) Å (I), 3.0595(4)–3.3694(6) Å (II)).

Keywords: bismuth triiodide, bismuth complexes [p-Tol₄E]⁺₃[Bi₃I₁₂]³⁻·HOCH₂CH₂OC₂H₅, E=P, Sb, synthesis, structure.

Introduction

Ionic bismuth complexes with linear anions [Bi₃I₁₂]³⁻, in which the bismuth atoms are bonded through six μ₂-bridging iodine atoms, are described in the literature as separate examples, where nitrogen-containing acyclic or heterocyclic cations stand as counter-ions [1–3]. Thus far only one similar complex with phosphonium cation is known [4]; like complexes with organoantimony cations are not known.

In the present paper we have synthesized two new phosphorus- and antimony-containing bismuth complexes [p-Tol₄E]⁺₃[Bi₃I₁₂]³⁻·HOCH₂CH₂OC₂H₅ (E=P, Sb) with the linear anion [Bi₃I₁₂]³⁻ and carried out X-ray diffraction analysis of these.

Experimental

tris(Tetra-*p*-tolylphosphonium) hexakis(μ₂-iodo)-hexaiodo-tribismuth 2-ethoxyethanol solvate (I). The solution of 0.100 g (0.19 mmol) tetra-*p*-tolylphosphonium and 0.113 g (0.19 mmol) bismuth triiodide in 15 mL 2-ethoxyethanol was obtained. The solvent was slowly evaporated. The yield was 0.142 g (65%) of orange-red crystals of complex **I** with decomposition temperature 195 °C. Found, %: C 30.83, H 2.74. For C₈₈H₉₄O₂P₃Bi₃I₁₂ calculated, %: C 30.54, H 2.85.

tris(Tetra-*p*-tolylstibonium) hexakis(μ₂-iodo)-hexaiodo-tribismuth 2-ethoxyethanol solvate (II). It was obtained by the similar procedure. Orange-red crystals of complex **II** were isolated (48%) with decomposition temperature 219 °C. Found, %: C 28.56, H 2.54. For C₈₈H₉₄O₂Sb₃Bi₃I₁₂ calculated, %: C 28.43, H 2.62.

The X-ray diffraction analyses of crystals **I** and **II** were performed on the Bruker D8 QUEST automatic four-circle diffractometer (Mo K_α-emission, λ = 0.71073 Å, graphite monochromator). The data were collected and analyzed, the unit cell parameters were refined, and the absorption correction was applied using the SMART and SAINT-Plus programs [5]. All calculations for structure determination and refinement were performed using the SHELXL/PC [6] and OLEX2 programs [7]. The structures were determined by the direct method and refined by the least-squares method in the anisotropic approximation for non-hydrogen atoms. The main crystallographic data and refinement results for the structure are listed in Table 1, the selected bond lengths and bond angles are given in Table 2.

The full tables of atomic coordinates, bond lengths, and bond angles were deposited with the Cambridge Crystallographic Data Centre (CCDC 1053856, 1049481; deposit@ccdc.cam.ac.uk; http://www.ccdc.cam.ac.uk).

Table 1
Crystallographic data and the experimental and structure refinement parameters for compounds I, II

Parameter	Value	
	I	II
Empirical formula	C ₈₈ H ₉₄ O ₂ P ₃ I ₁₂ Bi ₃	C ₈₈ H ₉₄ O ₂ Sb ₃ I ₁₂ Bi ₃
Formula weight	3426.28	3698.62
<i>T</i> , K	296(2)	296(2)
Crystal system	Triclinic	Triclinic
Space group	<i>P</i> $\bar{1}$	<i>P</i> $\bar{1}$
<i>a</i> , Å	12.4517(6)	12.4055(4)
<i>b</i> , Å	18.0680(9)	18.3805(5)
<i>c</i> , Å	24.1711(10)	24.6523(7)
α , deg	87.532(2)	87.401(2)
β , deg	87.039(2)	87.049(2)
γ , deg	75.281(2)	74.124(2)
<i>V</i> , Å ³	5250.0(4)	5396.8(3)
<i>Z</i>	4	2
ρ (calcd.), g/cm ³	2.167	2.276
μ , mm ⁻¹	8.630	9.085
<i>F</i> (000)	3136.0	3352.0
Crystal size, mm	0.26×0.25×0.16	0.33×0.3×0.14
2 θ Range of data collection, deg	4.04 – 46.56	4.1 – 53.54
Range of refraction indices	-13 ≤ <i>h</i> ≤ 13 -20 ≤ <i>k</i> ≤ 20 -26 ≤ <i>l</i> ≤ 26	-15 ≤ <i>h</i> ≤ 15 -23 ≤ <i>k</i> ≤ 23 -31 ≤ <i>l</i> ≤ 31
Measured reflections	94609	102068
Independent reflections	15056	22967
<i>R</i> _{int}	0.0486	0.0587
Refinement variables	990	990
<i>GOOF</i>	1.036	1.007
<i>R</i> factors for <i>F</i> ² > 2 σ (<i>F</i> ²)	<i>R</i> ₁ = 0.0361 <i>wR</i> ₂ = 0.0817	<i>R</i> ₁ = 0.0382 <i>wR</i> ₂ = 0.0705
<i>R</i> factors for all reflections	<i>R</i> ₁ = 0.0579 <i>wR</i> ₂ = 0.0941	<i>R</i> ₁ = 0.0711 <i>wR</i> ₂ = 0.0811
Residual electron density (min/max), e/Å ³	1.42/-0.77	1.29/-0.92

Table 2
Selected bond lengths and bond angles in the structure of compounds 1, 2

Bond <i>d</i> , Å		Angle ω , deg	
I			
P(1) – C(1)	1.788(8)	C(1)P(1)C(11)	109.0(4)
P(1) – C(11)	1.800(7)	C(1)P(1)C(21)	110.0(4)
P(1) – C(21)	1.761(9)	C(1)P(1)C(31)	109.6(4)
P(1) – C(31)	1.799(8)	C(11)P(1)C(21)	110.3(4)
P(2) – C(41)	1.804(7)	C(11)P(1)C(31)	109.9(3)
P(2) – C(51)	1.791(6)	C(21)P(1)C(31)	108.0(4)

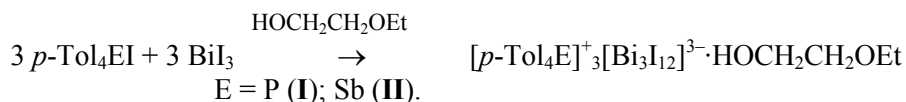
Bond d , Å		Angle ω , deg	
I			
P(2) – C(61)	1.770(7)	C(41)P(2)C(51)	107.5(3)
P(2) – C(71)	1.795(7)	C(41)P(2)C(61)	109.6(3)
P(3) – C(81)	1.800(7)	C(41)P(2)C(71)	111.7(3)
P(3) – C(91)	1.791(7)	C(51)P(2)C(61)	109.3(3)
P(3) – C(101)	1.815(7)	C(51)P(2)C(71)	111.4(3)
P(3) – C(111)	1.800(8)	C(61)P(2)C(71)	107.3(3)
Bi(1) – I (1)	3.0748(5)	C(81)P(3)C(91)	109.7(3)
Bi(1) – I (2)	3.0706(5)	C(81)P(3)C(101)	109.1(3)
Bi(1) – I (3)	3.0444(4)	C(81)P(3)C(111)	107.7(4)
Bi(2) – I (1)	3.3046(5)	C(91)P(3)C(101)	107.8(3)
Bi(2) – I (2)	3.2715(5)	C(91)P(3)C(111)	109.1(3)
Bi(2) – I (3)	3.3556(5)	C(101)P(3)C(111)	113.4(4)
Bi(2) – I (4)	2.9181(5)	I(1)Bi(1)I(2)	91.611(13)
Bi(2) – I (5)	2.8714(5)	I(7)Bi(3)I(9)	88.583(16)
Bi(2) – I (6)	2.9049(5)	I(7)Bi(3)I(8)	85.096(15)
Bi(3) – I (7)	3.0669(5)	I(8)Bi(3)I(9)	88.406(15)
Bi(3) – I (8)	3.0654(5)	I(2)Bi(2)I(3)	79.092(13)
Bi(3) – I (9)	3.0454(6)	I(2)Bi(2)I(4)	88.012(16)
Bi(4) – I (7)	3.3153(6)	I(2)Bi(2)I(5)	93.250(16)
Bi(4) – I (8)	3.3891(6)	I(2)Bi(2)I(6)	169.597(15)
Bi(4) – I (9)	3.2907(7)	I(3)Bi(2)I(4)	94.399(15)
Bi(4) – I (10)	2.8958(6)	I(3)Bi(2)I(5)	165.035(15)
Bi(4) – I (11)	2.9040(7)	I(3)Bi(2)I(6)	92.370(15)
Bi(4) – I (12)	2.9088(7)	I(4)Bi(2)I(5)	98.194(16)
		I(4)Bi(2)I(6)	98.652(17)
		I(5)Bi(2)I(6)	93.689(17)
		I(7)Bi(4)I(8)	76.406(14)
		I(7)Bi(4)I(9)	80.500(15)
		I(7)Bi(4)I(10)	87.219(17)
		I(7)Bi(4)I(11)	93.121(18)
		I(7)Bi(4)I(12)	170.78(2)
		I(8)Bi(4)I(9)	79.245(15)
		I(8)Bi(4)I(10)	163.130(16)
		I(8)Bi(4)I(11)	87.587(17)
		I(8)Bi(4)I(12)	101.94(2)
		I(9)Bi(4)I(10)	94.251(17)
		I(9)Bi(4)I(11)	166.381(18)
		I(9)Bi(4)I(12)	90.28(2)
		I(10)Bi(4)I(11)	97.464(19)
		I(10)Bi(4)I(12)	93.58(2)
		I(11)Bi(4)I(12)	95.88(2)
II			
Sb(1) – C(1)	2.089(6)	C(1)Sb(1)C(11)	107.6(3)
Sb(1) – C(11)	2.099(8)	C(1)Sb(1)C(21)	110.8(3)
Sb(1) – C(21)	2.091(7)	C(1)Sb(1)C(31)	108.8(3)

Table (end)

Bond <i>d</i> , Å		Angle ω, deg	
II			
Sb(1) – C(31)	2.085(8)	C(11)Sb(1)C(21)	112.0(3)
Sb(2) – C(41)	2.086(7)	C(11)Sb(1)C(31)	110.9(3)
Sb(2) – C(51)	2.097(7)	C(21)Sb(1)C(31)	106.9(3)
Sb(2) – C(61)	2.097(7)	C(41)Sb(2)C(51)	109.7(3)
Sb(2) – C(71)	2.086(7)	C(41)Sb(2)C(61)	106.1(3)
Sb(3) – C(81)	2.092(8)	C(41)Sb(2)C(71)	113.6(3)
Sb(3) – C(91)	2.073(9)	C(51)Sb(2)C(61)	110.2(3)
Sb(3) – C(101)	2.090(9)	C(51)Sb(2)C(71)	105.6(3)
Sb(3) – C(111)	2.085(8)	C(61)Sb(2)C(71)	111.7(3)
Bi(1) – I (1)	3.0785(5)	C(81)Sb(3)C(91)	113.7(3)
Bi(1) – I (2)	3.0866(5)	C(81)Sb(3)C(101)	113.1(3)
Bi(1) – I (3)	3.0595(4)	C(81)Sb(3)C(111)	105.4(3)
Bi(2) – I (2)	3.3341(5)	C(91)Sb(3)C(101)	107.9(3)
Bi(2) – I (3)	3.3376(5)	C(91)Sb(3)C(111)	109.6(3)
Bi(2) – I (4)	2.9191(5)	C(101)Sb(3)C(111)	106.8(4)
Bi(2) – I (5)	2.9234(6)	I(1)Bi(1)I(2)	92.813(14)
Bi(2) – I (6)	2.8867(5)	I(1)Bi(1)I(3)	93.202(13)
Bi(3) – I (7)	3.0754(5)	I(2)Bi(2)I(3)	78.388(13)
Bi(3) – I (8)	3.0672(5)	I(2)Bi(2)I(4)	92.235(15)
Bi(3) – I (9)	3.0750(6)	I(2)Bi(2)I(5)	167.390(16)
Bi(4) – I (8)	3.3694(6)	I(2)Bi(2)I(6)	89.205(16)
Bi(4) – I (9)	3.2947(6)	I(3)Bi(2)I(4)	91.055(14)
Bi(4) – I (10)	2.9086(8)	I(3)Bi(2)I(5)	93.163(15)
Bi(4) – I (11)	2.9248(6)	I(3)Bi(2)I(6)	166.958(16)
Bi(4) – I (12)	2.9032(6)	I(4)Bi(2)I(5)	97.312(17)
		I(4)Bi(2)I(6)	93.459(17)
		I(5)Bi(2)I(6)	98.383(17)
		I(7)Bi(3)I(8)	94.620(15)
		I(7)Bi(3)I(9)	91.955(16)
		I(8)Bi(3)I(9)	88.963(16)
		I(8)Bi(4)I(9)	80.444(15)
		I(8)Bi(4)I(10)	101.90(2)
		I(8)Bi(4)I(11)	86.408(17)
		I(8)Bi(4)I(12)	163.561(19)
		I(9)Bi(4)I(10)	89.58(2)
		I(9)Bi(4)I(11)	166.666(19)
		I(9)Bi(4)I(12)	94.318(16)
		I(10)Bi(4)I(11)	95.33(2)
		I(10)Bi(4)I(12)	93.58(2)
		I(11)Bi(4)I(12)	97.730(19)

Results and Discussion

It has been shown that the interaction of equimolar amounts of tetra-p-tolylphosphonium and tetra-p-tolylstybonium iodides with bismuth triiodide in 2-ethoxyetanol leads to formation of the ionic bismuth complexes with the anion [Bi₃I₁₂]³⁻, which contain the solvate molecule of the solvent:



According to X-ray diffraction data, the phosphorus and antimony atoms of the cations have weakly distorted tetrahedral coordination (Fig. 1 and 2). Angles CPC and CSbC equal $107.3(3)^\circ$ – $113.4(4)^\circ$ and $105.4(3)^\circ$ – $113.7(3)^\circ$, respectively. Bond lengths P–C (1.761(9)–1.815(7) Å) and Sb–C (2.085(8)–2.099(8) Å) are near to the sums of covalent radii of phosphorus, carbon (1.88 Å) and antimony, carbon (2.19 Å) [8]. In trinuclear centrosymmetrical anions $[\text{Bi}_3\text{I}_{12}]^{3-}$ the terminal fragments BiI_3 ($\text{Bi}-\text{I}_{\text{term}}$ 2.8714(5)–2.9181(5) Å (**I**), 2.8867(5)–2.9248(6) Å (**II**)) are bonded to the central bismuth atom through six μ_2 -bridging iodine atoms ($\text{Bi}-\text{I}_{\text{br}}$ 3.0454(6)–3.3891(6) Å (**I**), 3.0595(4)–3.3694(6) Å (**II**)). The terminal fragments BiI_3 are in the masked conformation.

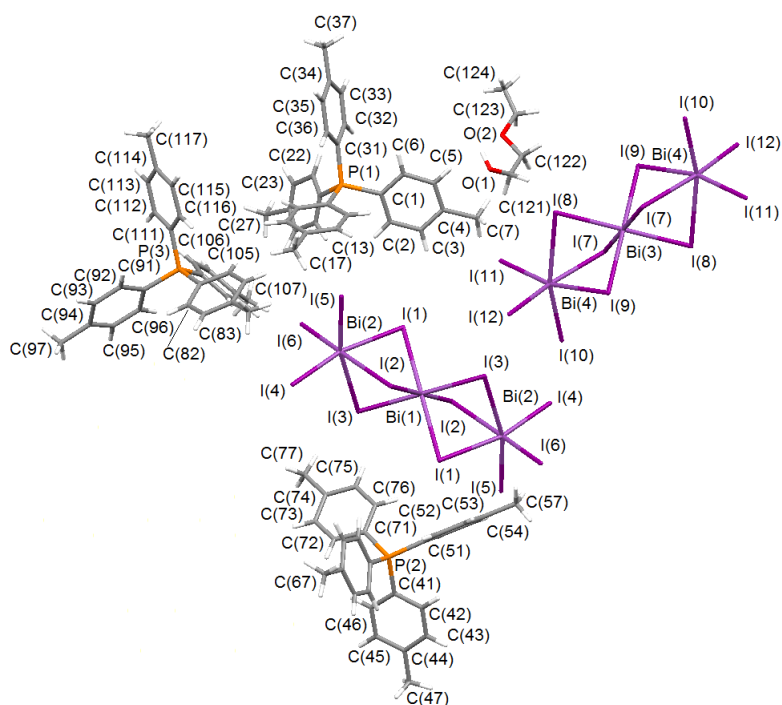


Fig. 1. The structure of complex I

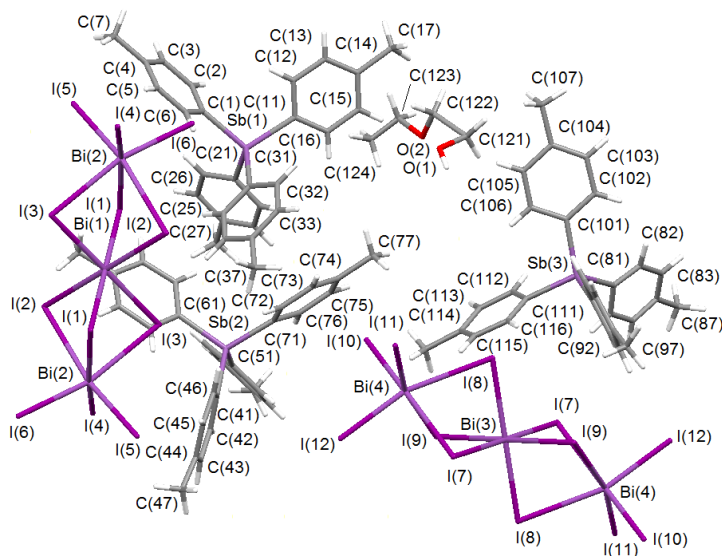


Fig. 2. The structure of complex II

The structural organization in crystals **I** and **II** results from weak interactions of the type H···I and H···O. In **I** the bridging μ₂-atoms of iodine in anions are bonded (2.86–3.16 Å) to the hydrogen atoms of phosphonium cations, in **II** the similar bonds are formed with participation of both bridging (3.10 Å) and terminal (3.15 Å) atoms of iodine (which is somewhat less than the sum of Van der Waals radii of hydrogen and iodine, namely, 3.3 Å [8]). No significant close contacts of the ions with the solvent molecules have been observed, but the molecules of 2-ethoxyethanol are bonded with each other by hydrogen bonds H···O (2.33 Å (**I**) and 2.62 Å (**II**)) (Fig. 3).

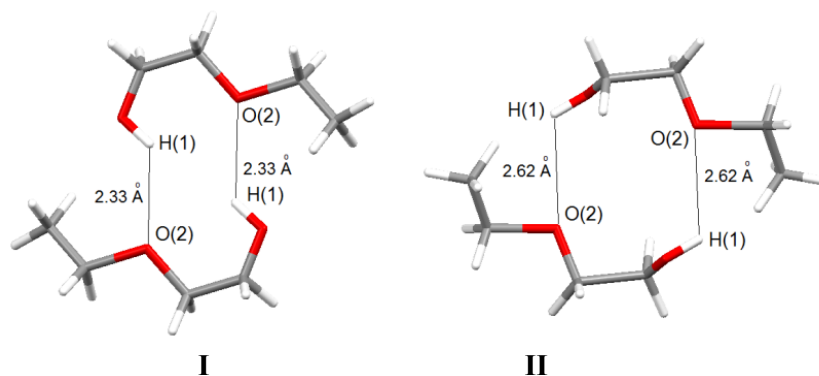


Fig. 3. Association of solvent molecules in crystals **I** and **II**

Conclusions

Complexes [p-Tol₄E]⁺₃[Bi₃I₁₂]³⁻·HOCH₂CH₂OC₂H₅ E=P (**I**), Sb (**II**) have been synthesized by interaction of equimolar amounts of tetra-*p*-tolylphosphonium and tetra-*p*-tolylstibonium iodides with bismuth triiodide in 2-ethoxyethanol. The structure of the products has been established by X-ray diffraction analysis.

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**СИНТЕЗ И СТРОЕНИЕ КОМПЛЕКСОВ ВИСМУТА
[p-Tol₄E]⁺₃[Bi₃I₁₂]³⁻·HOCH₂CH₂OC₂H₅ E=P, Sb**

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Взаимодействием эквимольных количеств иодидов тетра-*n*-толилфосфония и тетра-*n*-толилстибония с трийодидом висмута в 2-этоксигэтанолу получены комплексы [p-Tol₄E]⁺₃[Bi₃I₁₂]³⁻·HOCH₂CH₂OC₂H₅ E=P (I), Sb (II). Проведен рентгеноструктурный анализ I и II. В катионах координация атомов фосфора и сурьмы тетраэдрическая (углы СРС 107.3(3)°–113.4(4)° (I), CSbC 105.4(3)°–113.7(3)° (II); связи P–C 1.761(9)–1.815(7) Å (I), Sb–C 2.085(8)–2.099(8) Å (II). В трехъядерных анионах [Bi₃I₁₂]³⁻ концевые фрагменты BiI₃ (Bi–I_{терм} 2.8714(5)–2.9181(5) Å (I) 2.8867(5)–2.9248(6) Å (II)) связаны с центральным атомом висмута посредством шести μ₂-мостиковых атомов иода (Bi–I_{мост} 3.0454(6)–3.3891(6) Å (I) 3.0595(4)–3.3694(6) Å (II)).

Ключевые слова: трийодид висмута, комплексы висмута [p-Tol₄E]⁺₃[Bi₃I₁₂]³⁻·HOCH₂CH₂OC₂H₅, E=P, Sb, синтез, строение.

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