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Synthesis and Structure of $[Ph_3MeP]_3[Sb_3I_{12}]\cdot Me_2C{=}O$, $[Ph_3MeP]_3[Sb_2I_9]$, $[Ph_3MeP]_2[SbI_5]$, and $[Ph_3MeP]_3[Sb_3I_{12}]$ Antimony Complexes

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Abstract—Complexes $[Ph_3MeP]_3[Sb_3I_{12}]Me_2C{=}O$ (**I**), $[Ph_3MeP]_3[Sb_2I_9]$ (**II**), and $[Ph_3MeP]_2[SbI_5]$ (**III**) were obtained via the reaction of triphenylphosphonium iodide with antimony triiodide in acetone in 1:1, 3:2 and 2:1 molar ratios. Reaction of the complex **III** with antimony triiodide (1:1) affords $[Ph_3MeP]_3[Sb_3I_{12}]$ (**IV**). The structure of the obtained complexes was confirmed by X-ray analysis.

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We have previously shown that the reaction of equimolar amounts of triphenylpentylphosphonium or triphenylpropylphosphonium iodide with antimony triiodide in an aprotic solvent results in the addition complexes containing the binuclear doubly charged anion, and the solvent is involved into the coordination sphere of the antimony atom: $[Ph_3AmP]_2[Sb_2I_8\cdot 2DMSO]$, $[Ph_3PrP]_2[Sb_2I_8\cdot 2C_4H_8O_2]$ [1].

The aim of this work is studying the reactions of antimony triiodide with triphenylphosphonium iodide in different molar ratios in acetone and determining the structure of the obtained complexes with Sb,I-containing anions.

The reaction of Ph_3MePI with SbI_3 in a 1:1 molar ratio produces the red-orange complex containing a trinuclear triply charged anion, which crystallizes as $[Ph_3MeP]_3[Sb_3I_{12}]Me_2C{=}O$ solvate (**I**), mp 138°C.

$$3[Ph_3MeP]I + 3SbI_3 \xrightarrow{Me_2C=O} \underset{\textbf{I}}{[Ph_3MeP]_3[Sb_3I_{12}]\cdot Me_2C{=}O}.$$

Comparison of the structure of complexes obtained in [1] and complex **I** shows that the nature of the alkyl substituent of the phosphonium cation and the solvent nature influence the anions structure. Unlike DMSO and dioxane, acetone is not included into the coordination sphere of the antimony atom, but it presents in the crystal cell as a solvate molecule.

According to the X-ray diffraction (XRD), in the complex **I** crystal there are three types of the crystallographically independent $[Ph_3MeP]^+$ cations, where the phosphorus atoms have a tetrahedral coordination (Fig. 1), the CPC bond angles [107.7(2)°–111.7(3)°] deviate slightly from the theoretical value (109°28'). The P–C bond lengths vary in the range of 1.776(6)–1.805(6) Å (Table 1).

In the anion the antimony atoms are hexa-coordinated. In the octahedron ISbI *trans*-angles vary in the range of 163.45(2)°–178.03(2)°. The antimony atoms lie in one plane. The iodine atoms perform a variety of structural features: there are the terminal iodine atoms (monodentate) I_{term}, bridging (bidentate) I_b and one tridentate iodine atom I_t. The average $Sb–I_{term}$ bond lengths are 2.8231(9) Å [2.8014(9)–2.8450(9) Å], $Sb–I_b$ 3.1833(8) Å [2.9455(8)–3.4118(8) Å], $Sb–I_t$ 3.2867(8) [3.2685(9)–3.3180(9) Å]. The average Sb–I bond length is slightly greater than the sum of the covalent radii of antimony and iodine atoms and is much less than the sum of their van der Waals radii (2.71 and 4.3 Å, respectively [2]). The average bond length value increases with increasing the coordination number of iodine atom.

Note that the anions $[Sb_3I_{12}]^{3-}$ containing μ_3-iodine have not been reported earlier.

The intermolecular contacts involving the oxygen lone pair of the acetone molecules in the crystal are not

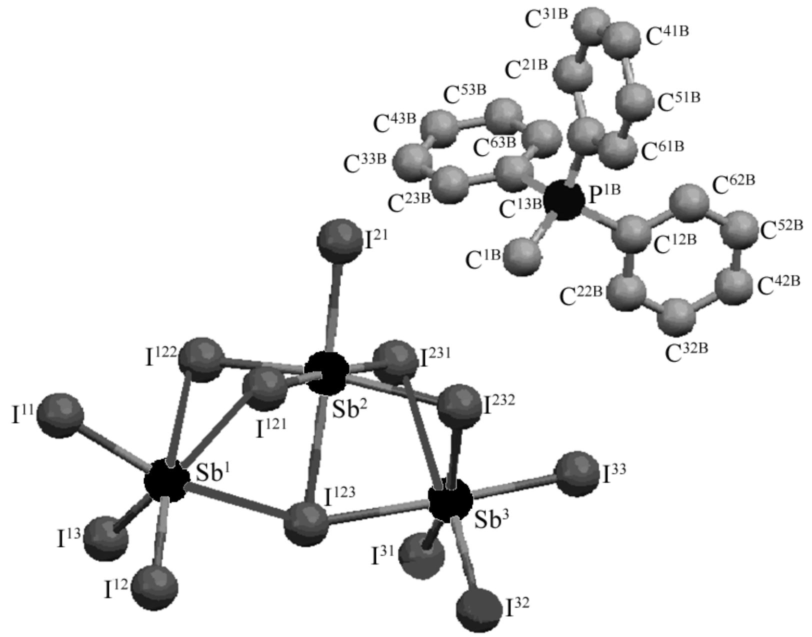

Fig. 1. Structure of one of the cations and anions of **I** (the acetone solvate molecule is not shown).

observed. Apparently, the absence of the significant intermolecular interactions causes the easy acetone removal from the crystals at room temperature. Thus, after 4–5 weeks the melting points of the crystals changes and becomes equal to 136°C.

The anions are united into the chains extending along the crystallographic *a* axis due to the interactions of I···I type (Fig. 2). The I···I distances are 3.922 Å while the van der Waals radii sum in 4.2 Å [2]. Cations are located between the anions chains. The cations and anions are connected by the multiple intermolecular I···H–C hydrogen bonds. The I···H distances are 3.10–3.18 Å for the van der Waals radii sum of 3.3 Å [2].

An increase in the concentration of antimony triiodide in the reaction mixture with triphenylmethylphosphonium iodide does not change the complex composition and anion structure. Thus, regardless of the molar ratio of antimony triiodide and triphenylmethylphosphonium iodide (1:1, 1:1.5, 1:2 and 1:3) the same product **I** was obtained. In contrast, the

Table 1. Bonds lengths and valence angles in the structures **I**, **II** and **IV**

Bond *d*, Å		Angle ω, deg		Bond *d*, Å		Angle ω, deg	
			I				
$\mathrm{Sb^{1}{-}I^{11}}$	2.8132(9)	$\mathrm{I^{11}Sb^{1}I^{12}}$	97.494(16)	$\mathrm{Sb^{2}{-}I^{123}}$	3.2735(10)	$\mathrm{I^{21}Sb^{2}Sb^{3}}$	130.346(13)
$\mathrm{Sb^{1}{-}I^{12}}$	2.8224(7)	$\mathrm{I^{11}Sb^{1}I^{122}}$	89.393(15)	$\mathrm{Sb^{2}{-}I^{231}}$	3.1110(8)	$\mathrm{I^{21}Sb^{2}I^{121}}$	91.139(15)
$\mathrm{Sb^{1}{-}I^{13}}$	2.8042(7)	$\mathrm{I^{11}Sb^{1}I^{123}}$	166.667(16)	$\mathrm{Sb^{2}{-}I^{232}}$	3.0972(8)	$\mathrm{I^{21}Sb^{2}I^{123}}$	178.031(15)
$\mathrm{Sb^{1}{-}I^{121}}$	3.4118(8)	$\mathrm{I^{12}Sb^{1}I^{121}}$	101.39(3)	$\mathrm{Sb^{3}{-}I^{31}}$	2.8314(8)	$\mathrm{I^{21}Sb^{2}I^{231}}$	91.133(14)
$\mathrm{Sb^{1}{-}I^{122}}$	3.3154(8)	$\mathrm{I^{12}Sb^{1}I^{122}}$	172.654(15)	$\mathrm{Sb^{3}{-}I^{31A}}$	2.831(5)	$\mathrm{I^{121}Sb^{2}Sb^{3}}$	123.389(16)
$\mathrm{Sb^{1}{-}I^{123}}$	3.3180(9)	$\mathrm{I^{13}Sb^{1}I^{12}}$	92.68(3)	$\mathrm{Sb^{3}{-}I^{32}}$	2.8014(8)	$\mathrm{I^{121}Sb^{2}I^{123}}$	87.605(13)
$\mathrm{Sb^{2}{-}I^{21}}$	2.8450(9)	$\mathrm{I^{13}Sb^{1}I^{121}}$	163.451(16)	$\mathrm{Sb^{3}{-}I^{32A}}$	2.888(6)	$\mathrm{I^{121}Sb^{2}I^{231}}$	177.312(16)
$\mathrm{Sb^{2}{-}I^{121}}$	2.9505(8)	$\mathrm{I^{13}Sb^{1}I^{122}}$	89.25(3)	$\mathrm{Sb^{3}{-}I^{33}}$	2.8443(8)	$\mathrm{I^{121}Sb^{2}I^{232}}$	94.43(2)
$\mathrm{Sb^{2}{-}I^{122}}$	2.9455(8)	$\mathrm{I^{122}Sb^{1}I^{121}}$	75.73(2)				

Table 1. (Contd.)

Bond *d*, Å		Angle ω, deg		Bond *d*, Å		Angle ω, deg	
			II				
Sb^1–I^{123}	3.1023(4)	I^{123}Sb^{1}I^{121}	85.156(11)	Sb^2–I^{23}	2.8554(4)	I^{22}Sb^{2}I^{123}	171.715(14)
Sb^1–I^{122}	3.2365(4)	I^{13}Sb^{1}I^{122}	94.306(12)	Sb^2–I^{22}	2.8979(5)	C^{1B}P^{1B}C^{11B}	108.0(2)
Sb^1–I^{13}	2.9072(4)	I^{13}Sb^{1}I^{121}	174.166(14)	Sb^2–I^{21}	2.8485(4)	C^{1B}P^{1B}C^{12B}	109.7(2)
Sb^1–I^{12}	2.9369(4)	I^{12}Sb^{1}I^{121}	94.539(12)	P^{1B}–C^{1B}	1.785(5)	C^{1B}P^{1B}C^{13B}	108.8(2)
Sb^1–I^{121}	3.1858(4)	I^{121}Sb^{1}I^{122}	83.840(11)	P^{1B}–C^{11B}	1.786(5)	C^{11B}P^{1B}C^{13B}	111.5(2)
Sb^1–I^{11}	2.8621(4)	I^{11}Sb^{1}I^{122}	172.571(15)	P^{1B}–C^{12B}	1.786(5)	C^{12B}P^{1B}C^{11B}	110.0(2)
Sb^2–I^{123}	3.1895(5)	I^{11}Sb^{1}I^{121}	88.759(12)	P^{1B}–C^{13B}	1.794(5)	C^{12B}P^{1B}C^{13B}	108.7(2)
			IV				
Sb^1–I^{11}	2.7953(9)	I^{11}Sb^{1}I^{12}	94.01(3)	Sb^3–I^{123}	3.2599(9)	I^{232}Sb^{2}I^{231}	83.84(2)
Sb^1–I^{12}	2.8146(9)	I^{11}Sb^{1}I^{121}	161.25(3)	Sb^3–I^{231}	3.3701(11)	I^{31}Sb^{3}I^{123}	93.89(3)
Sb^1–I^{13}	2.8120(9)	I^{11}Sb^{1}I^{123}	91.63(3)	Sb^3–I^{232}	3.2680(10)	I^{31}Sb^{3}I^{232}	169.05(4)
Sb^1–I^{121}	3.4405(10)	I^{12}Sb^{1}I^{121}	102.28(3)	P^{1A}–C^{11A}	1.800(11)	I^{32}Sb^{3}I^{33}	95.45(3)
Sb^1–I^{122}	3.2899(9)	I^{12}Sb^{1}I^{122}	171.59(3)	P^{1A}–C^{12A}	1.805(11)	I^{32}Sb^{3}I^{123}	86.42(3)
Sb^1–I^{123}	3.3017(9)	I^{12}Sb^{1}I^{123}	93.53(3)	P^{1A}–C^{13A}	1.788(11)	I^{32}Sb^{3}I^{231}	167.95(3)
Sb^2–I^{21}	2.8490(9)	I^{13}Sb^{1}I^{123}	166.82(3)	P^{1A}–C^{1A}	1.799(11)	I^{33}Sb^{3}I^{31}	96.01(3)
Sb^2–I^{121}	2.9385(9)	I^{122}Sb^{1}I^{121}	75.15(2)	P^{1B}–C^{11B}	1.801(12)	I^{33}Sb^{3}I^{123}	169.72(3)
Sb^2–I^{122}	2.9497(9)	I^{21}Sb^{2}I^{121}	91.88(3)	P^{1B}–C^{12B}	1.781(10)	I^{232}Sb^{3}I^{231}	76.98(2)
Sb^2–I^{123}	3.2136(9)	I^{21}Sb^{2}I^{123}	177.96(3)	P^{1B}–C^{13B}	1.792(11)	C^{11A}P^{1A}C^{12A}	111.2(5)
Sb^2–I^{231}	3.0963(9)	I^{21}Sb^{2}I^{232}	96.54(3)	P^{1B}–C^{1B}	1.748(12)	C^{13A}P^{1A}C^{11A}	108.5(5)
Sb^2–I^{232}	3.0884(9)	I^{121}Sb^{2}I^{123}	87.48(2)	P^{1C}–C^{11C}	1.786(10)	C^{13A}P^{1A}C^{12A}	110.2(5)
Sb^3–I^{31}	2.8495(12)	I^{121}Sb^{2}I^{231}	176.00(3)	P^{1C}–C^{12C}	1.798(11)	C^{13A}P^{1A}C^{1A}	109.3(5)
Sb^3–I^{32}	2.8105(11)	I^{121}Sb^{2}I^{232}	94.29(3)	P^{1C}–C^{13C}	1.786(10)	C^{1A}P^{1A}C^{11A}	110.0(5)
Sb^3–I^{33}	2.8375(10)	I^{122}Sb^{2}I^{232}	169.58(3)	P^{1C}–C^{1C}	1.775(11)	C^{1A}P^{1A}C^{12A}	107.7(5)

increase in the triphenylmethylphosphonium iodide concentration in the initial solution affects the composition and structure of the complex. The reaction of antimony triiodide with triphenylmethylphosphonium iodide in 3:2 molar ratio results in the formation of $[Ph_3MeP]_3[Sb_2I_9]$ complex (**II**).

$$3[Ph_3MeP]I + 2SbI_3 \rightarrow \underset{\textbf{II}}{[Ph_3MeP]_3[Sb_2I_9]}.$$

The complex **II** is orange crystals melting at 188°C. The structure of the complex **II** was determined by the X-ray diffraction analysis (Fig. 3). Coordination of the phosphorus atoms in the three types of crystallographically independent $[Ph_3PMe]^+$ cations is close to tetrahedral: CPC angles are 107.2(2)°–113.0(2)°, P–C distances vary in the range of 1.783(5)–1.797(5) Å. Thus, the geometric parameters of the cations in the complexes **I** and **II** differ only slightly (Table 1).

In the binuclear anion $[Sb_2I_9]^{3-}$ the antimony atoms have octahedral coordination, the terminal SbI_3 fragments are in virtually eclipsed conformations. The axial ISbI angles are 169.90(1)°–174.17(1)°. The $I_{term}SbI_b$ angles between the terminal and bridging bonds vary in the range of 81.96(1)°–95.88(1)°; the

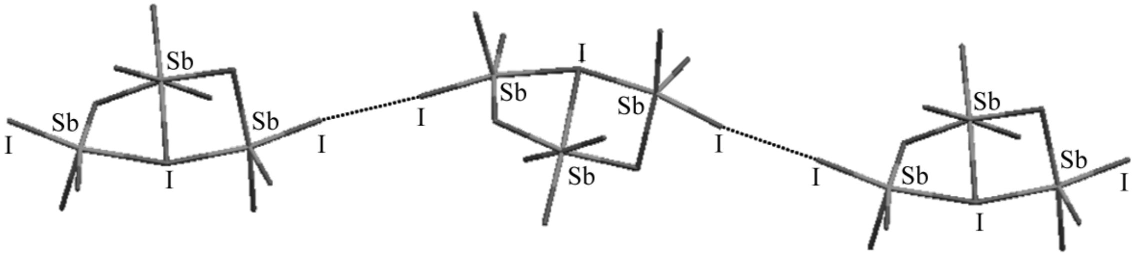

Fig. 2. The $[Sb_3I_{12}]^{3-}$ ions chain in the crystal of **I**.

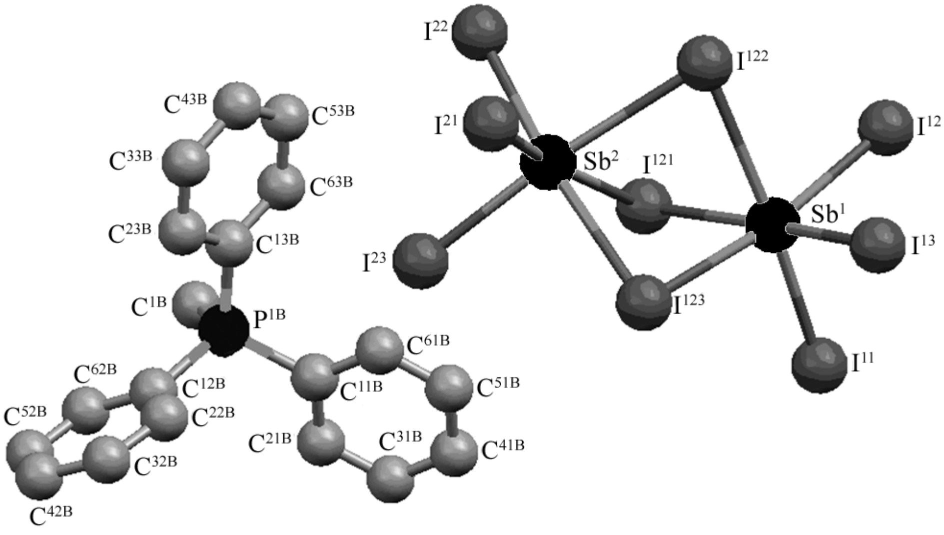

Fig. 3. The structure of **II**.

$I_{term}SbI_{term}$ angles between the terminal bonds are in the range from 90.78(1)° to 95.55(1)°. The SbI_bSb [77.41(1)°–80.67(1)°] and I_bSbI_b angles [84.24(1)°–86.32(1)°] in the $[Sb_2I_2]$ metallocycles are much less than 90°. The pair distances Sb–I_b between the antimony and bridging iodine atoms are not equivalent and equal 3.1023(4)–3.2770(4) Å. The Sb–I_{term} bond lengths [2.8485(4)–2.9369(4) Å] are smaller than Sb–I_{br}. In the crystal of **II** the anions are isolated. Cations and anions are connected by the multiple intermolecular hydrogen I···H–C bonds.

A further increase in the concentration of triphenylmethylphosphonium iodide in the reactions with antimony triiodide (2:1) leads to the formation of $[Ph_3MeP]_2[SbI_5]$ complex (**III**) as bright yellow crystals with mp 180°C.

$$2[Ph_3MeP]I + SbI_3 \rightarrow \underset{\mathbf{III}}{[Ph_3MeP]_2[SbI_5]}.$$

Previously, we obtained similarly a bismuth complex $[Ph_3MeP]_2[BiI_5]$, in which the $[BiI_5]^{2-}$ anion has the ideal trigonal–bipiramidal configuration [3].

When adding an equimolar amount of antimony triiodide to the complex **III** acetone solution, the complex **IV** is formed, which composition differs from that for the complex **I** only in the absence of acetone solvate. The geometric parameters of the cation and anion of **IV** are practically coincided with those parameters of the compex **I**.

$$3[Ph_3MeP]_2[SbI_5] + 3SbI_3 \rightarrow \underset{\mathbf{IV}}{2[Ph_3MeP]_3[Sb_3I_{12}]}.$$

In the crystal of the complex **IV** there are three types of the crystallographically independent cations. Coordination of the phosphorus atoms in the $[Ph_3PMe]^+$ cation (Fig. 4) is close to tetrahedral [CPC 107.(5)°–111.6(5)°, P–C 1.748(12)–1.805(11) Å].

In the $[Sb_3I_{12}]^{3-}$ trinuclear anions the antimony atoms have octahedral coordination; the iodine atoms are characterized by the coordination numbers 1, 2, and 3. The axial ISbI angles are 161.25(3)°–177.96(3)°. The $I_{term}SbI_{term}$ angles between the terminal bonds vary in the range of 91.84(2)°–97.63(3)°. The SbI_3 terminal fragments [Sb–I_{term} 2.7953(9)–3.0963(9) Å] are almost

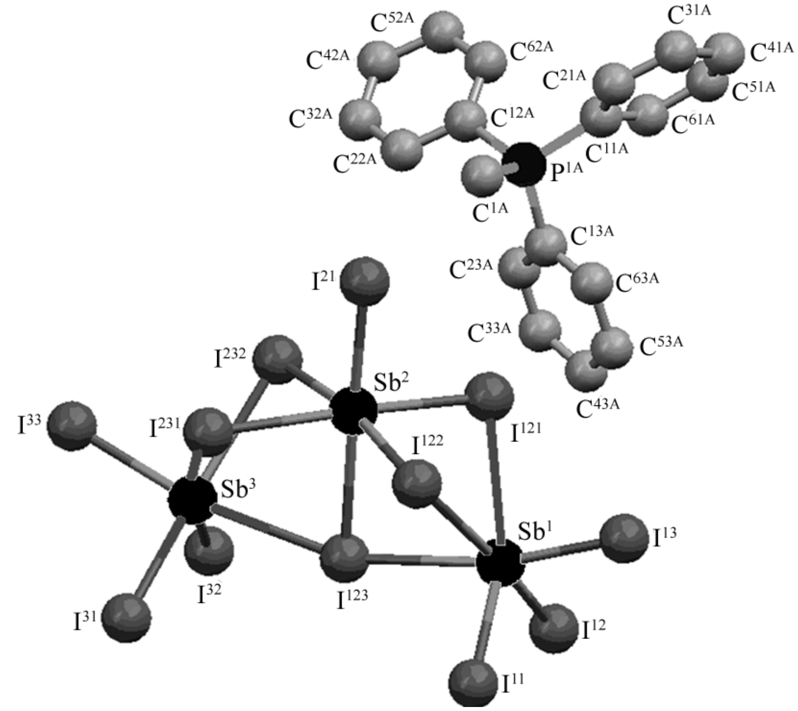

Fig. 4. The structure of one of the cations and anions of **IV**.

in the eclipsed conformations. Pair distances between the antimony and bridging iodine atoms [Sb–I_b 2.9497(9)–3.4405(10) Å] are not equivalent. The distances between the iodine atoms with the coordination number 3 (Sb–I_{term}) and the antimony atoms are 3.2136(9), 3.2599(9) and 3.3017(9) Å. The average values of the Sb–I_{term}, Sb–I_b and Sb–I_t bonds lengths are 2.8764(9), 3.2342(9) and 3.2584(9) Å, respectively.

The geometric parameters of the complexes **I** and **IV** are very similar, suggesting that the presence of the acetone solvate molecules in the crystals of **I** has no significant effect on the bond lengths and angles in the cations and anions. In crystals of **IV** the I···I distance in the anions chains are 3.917 Å. The weak I···H–I hydrogen bonds are observed between the cations and anions.

Thus, the initial reagents ratio defines the composition and structure of the complex anion of the adduct formed in the reactions of antimony iodide with methyltriphenylphosphonium iodide. The complexes **I**–**IV** acetone and dimethyl sulfoxide solutions are found to conduct electricity.

EXPERIMENTAL

The IR spectra were recorded on a FTIR 1201 spectrometer from KBr pellets. X-ray diffraction was performed on a Bruker-Nonius X8Apex diffractometer (MoK_α-radiation, λ 0.71073 Å, graphite monochromator). The data collection as well as the unit cell parameters editing and refinement were made with SADABS [4], SMART and SAINT-Plus programs [5]. All calculations were performed with SHELXL/PC program [6]. The main crystallographic data and refinement of the structures **I**, **II**, **IV** are shown in Table 2, the main bond lengths and angles in Table 1.

Complex I. A mixture of 0.60 g (1.50 mmol) of triphenylmethylphosphonium iodide, 0.75 g (1.50 mmol) of antimony iodide and 30 ml of acetone was kept at room temperature for 24 h. Reddish-orange crystals are formed. Yield 1.21 g (87%), mp 138°C. IR spectrum, ν, cm^{-1}: 616, 685, 717, 741, 785, 996, 1113, 1169, 1189, 1331, 1440, 1484, 1589, 1706, 2901, 2974, 3051. Found, %: C 25.39; H 2.53. $C_{60}H_{60}OI_{12}P_3Sb_3$. Calculated, %: C 25.91; H 2.16.

Complex II. A mixture of 0.60 g (1.50 mmol) of triphenylmethylphosphonium iodide and 0.50 g (1.00 mmol) of antimony(III) iodide and 30 ml of acetone was kept at room temperature for 24 h. Reddish-orange crystals are formed. Yield 1.00 g (91%), mp 188°C. IR spectrum, ν, cm^{-1}: 434, 510, 686, 714, 738, 786, 890, 998, 1118, 1166, 1338, 1438, 1482, 1586, 2903, 2971, 3055. Found, %: C 29.18; H 2.47. $C_{57}H_{54}I_9P_3Sb_2$. Calculated, %: C 29.23; H 2.31.

Table 2. Crystallographic data, experiment and refinement parameters for the structures **I**, **II** and **IV**

Parameter	Value		
	I	**II**	**IV**
M	2778.04	2217.51	2719.96
T, K	100.0(2)	100.0(2)	100.0(2)
Crystal system	Rhombic	Monoclinic	Rhombic
Space group	$P2_12_12_1$	$P2_1/c$	$P2_12_12_1$
a, Å	12.688(4)	24.0594(7)	12.6185(3)
b, Å	20.257(6)	14.6930(4)	20.0383(5)
c, Å	30.300(8)	18.9255(5)	30.1373(8)
β, deg	–	93.0630(10)	–
V, Å^3	7788(4)	6680.7(3)	7620.3(3)
Z	4	4	4
d_{calc}, g cm^{-3}	2.369	2.205	2.371
μ, mm^{-1}	5.887	5.073	6.012
F(000)	5048	4080	4920
Crystal form (size, mm)	Prism (0.39×0.24×0.22)	Plate (0.19×0.06×0.02)	Prism (0.28×0.19×0.17)
Collection data range on θ, deg	1.21–37.65	1.76–31.55	1.22–29.83
Reflections indices intervals	$-21 \le h \le 20$, $-34 \le k \le 19$, $-37 \le l \le 50$	$-32 \le h \le 33$, $-17 \le k \le 20$, $-22 \le l \le 26$	$-16 \le h \le 8$, $-28 \le k \le 27$, $-39 \le l \le 39$
Reflections measured	38330	19136	19085
Independent reflections	30733	13285	16095
Refinement variables	727	643	671
GOOF	1.005	1.027	0.853
R-Factors on $F^2 > 2\sigma(F^2)$	R_1 0.0474, wR_2 0.0765	R_1 0.0357, wR_2 0.0540	R_1 0.0449, wR_2 0.1187
R-Factors on all reflections	R_1 0.0665, wR_2 0.0863	R_1 0.0726, wR_2 0.0616	R_1 0.0590, wR_2 0.1317
Residual electron density (min/max), e A^{-3}	1.468/–1.561	–1.022/1.089	–1.693/1.917

Complex III. A mixture of 0.81 g (2.00 mmol) triphenylmethylphosphonium iodide, 0.50 g (1.00 mmol) of antimony(III) iodide and 30 ml of acetone was kept at room temperature for 24 h. Reddish-orange crystals are formed. Yield 1.20 g (92%), mp 180°C. IR spectrum, ν, cm^{-1}: 440, 508, 691, 722, 738, 790, 889, 1001, 1112, 1163, 1191, 1318, 1334, 1438, 1481, 1589, 2904, 2979, 3047. Found, %: C 34.47; H 2.64. $C_{38}H_{36}I_5P_2Sb$. Calculated, %: C 34.78; H 2.75.

Complex IV. A mixture of 0.39 g (0.30 mmol) of compound **III** and 0.15 g (0.30 mmol) of antimony triiodide of antimony(III) iodide and 30 ml of acetone was kept at room temperature for 24 h. Reddish-orange crystals form. Yield 0.47 g (87%), mp 136°C. IR spectrum, ν, cm^{-1}: 689, 721, 744, 783, 888, 997, 1118, 1169, 1188, 1337, 1442, 1485, 2900, 2974, 3056. Found, %: C 25.07; H 2.02. $C_{57}H_{54}I_{12}P_3Sb_3$. Calculated, %: C 25.14; H 1.98.

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