Palladium Complexes [Ph₃PCH₂CN]₂[PdBr₄], [Ph₄P]₂[PdBr₄], [Ph₃PC₅H₉-*cyclo*][PdBr₃(Et₂SO)], and [Ph₄P]₂[Pd₂Br₆]. Synthesis and Structure

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Abstract—Palladium phosphonium complexes with mononuclear anions [Ph₃PCH₂CN][PdBr₄], [Ph₄P][PdBr₄], and [Ph₃PC₅H₉-*cyclo*][PdBr₃(Et₂SO)] were synthesized by the reaction of organyltriphenylphosphonium bromides with palladium(II) bromide in the presence of hydrobromic acid followed by recrystallization from acetonitrile, dimethyl sulfoxide, or diethyl sulfoxide. Holding the [Ph₄P][PdBr₄] complex in acetonitrile with the addition of diamyl sulfoxide led to the formation of the [Ph₄P]₂[Pd₂Br₆] complex. According to single crystal X-ray diffraction analysis, mononuclear [PdBr₄]^{2–}, [PdBr₃(Et₂SO)][–] and binuclear [Pd₂Br₆]^{2–} anions in the complexes have a planar structure.

Keywords: palladium complexes, tetraorganylphosphonium cations, diethyl sulfoxide, X-ray diffraction studies

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Palladium complexes are of interest because of their effective application in catalysis [1–5] and also in organic and organoelement synthesis [6–10]. Structural features of palladium coordination compounds are being intensively studied. As a rule, palladium-containing anions in numerous structurally characterized ionic complexes have the composition [PdHlg₄]^{2–} and, more rarely, [Pd₂Hlg₆]^{2–} [11]. The effect of solvents on the design of anions in these complexes was considered [12]. The synthesis of complexes with the S-coordinated dimethyl sulfoxide molecule in mononuclear [PdCl₃(DMSO)][–] anions was reported [13–23]. We synthesized new palladium complexes and studied the solvent effect on the design of bromopalladate(II) anions.

To obtain ionic phosphonium complexes of palladium, an aqueous solution of tetraorganylphosphonium bromide was added to a solution of palladium(II) bromide in hydrobromic acid. The formation of a brown precipitate was observed, which, after filtration and drying, was recrystallized from acetonitrile. After removal of the solvent, tetrabromopalladates of cyanomethyltriphenylphosphonium **1** and tetraphenylphosphonium **2** were obtained as brown crystals (1). Under similar conditions, in reaction (2) with the participation of cyclopentyltriphenylphosphonium bromide, the formation of a brown precipitate was also observed, during recrystallization of which from diethyl sulfoxide, bromine in the coordination sphere of the palladium atom was replaced by diethyl sulfoxide to form complex **3**. Keeping a mixture of equimolar amounts of compound **2** and diamyl sulfoxide in acetonitrile did not lead to the replacement of the bromine atom in the coordination sphere of the metal by the *n*-donor ligand, whereas dimerization (3) of the anion occurred with the formation of the [Ph₄P]₂[Pd₂Br₆] complex (**4**).

Compounds 1–4 are brown crystalline substances resistant to moisture and atmospheric oxygen, readily soluble in acetonitrile, chloroform, and tetrahydrofuran, and insoluble in aliphatic hydrocarbons. In the IR spectra of complexes 1–4, intense absorption bands are observed in the regions 1439–1475 and 997–995 cm⁻¹, which characterize stretching vibrations of the P–C_{Ph} bond. The cyano group in complex 1 manifests itself as a low-intensity narrow absorption band at 2255 cm⁻¹ [24].

According to XRD data, the crystals of complexes 1-3 consist of tetrahedral organyltriphenylphosphonium

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Parameter	1	2	3	4
М	1030.67	1104.78	781.69	685.50
Syngony	Monoclinic	Triclinic	Monoclinic	Triclinic
Space group	$P2_1/n$	<i>P</i> 1	C2/c	<i>P</i> 1
<i>a</i> , Å	9.579(8)	10.458(14)	17.446(14)	10.571(5)
<i>b</i> , Å	12.294(10)	14.204(13)	9.951(10)	11.055(7)
<i>c</i> , Å	17.13(2)	15.060(13)	35.78(3)	11.109(6)
α, deg	90.00	91.78(3)	90.00	79.67(3)
β, deg	91.91(4)	92.54(6)	99.09(2)	79.308(19)
γ, deg	90.00	100.92(4)	90.00	71.74(2)
<i>V</i> , Å ³	2017(3)	2193(4)	6134(9)	1201.2(11)
Ζ	2	2	8	2
$d_{\text{calc}}, \text{g/cm}^3$	1.697	1.673	1.693	1.895
μ , mm ⁻¹	4.532	4.174	4.651	5.837
<i>F</i> (000)	1008.0	1088.0	3072.0	660.0
Crystal size, mm	0.46×0.29×0.1	0.64×0.51×0.19	0.36×0.23×0.08	0.63×0.51×0.18
Data collection area for 2θ , deg	5.8-54.4	5.84-46.68	5.64-65.42	5.78-79.1
Reflection index intervals	$-11 \le h \le 12$	$-11 \le h \le 11$	$-26 \le h \le 26$	$-18 \le h \le 18$
	$-15 \le k \le 15$	$-15 \le k \le 15$	$-15 \le k \le 15,$	$-19 \le k \le 19$
	$-21 \le l \le 21$	$-16 \le l \le 16$	$-53 \le l \le 54$	$-19 \le l \le 19$
Reflections measured	24190	32130	71447	83175
Independent reflections	4466 (<i>R</i> _{int} 0.0437)	$6249 (R_{int} 0.0448)$	10914 (<i>R</i> _{int} 0.0980)	14292 (<i>R</i> _{int} 0.0679)
Reflections with $I > 2\sigma(I)$	3602	5229	5705	7117
Refined parameters	223	499	309	263
GOOF	1.031	1.030	1.063	1.021
<i>R</i> -Factors for $F^2 > 2\sigma(F^2)$	$R_1 0.0291,$	$R_1 0.0409,$	$R_1 0.0846$,	$R_1 0.0586$,
	$wR_2 0.0618$	$wR_2 0.0930$	$wR_2 0.1992$	$wR_2 0.1093$
<i>R</i> -Factors for all reflections	$R_1 0.0429,$	$R_1 0.0526,$	$R_1 0.1611,$	$R_1 0.1406,$
	$wR_2 0.0665$	$wR_2 0.1012$	$wR_2 0.2296$	$wR_2 0.1360$
Residual electron density (max/min), $e/Å^3$	0.38/-0.62	0.83/-0.91	1.21/-1.56	1.73/-1.70

Table 1. Crystallographic data, experimental parameters, and refinement of structures of complexes 1-4

cations and mononuclear square anions $[PdBr_4]^{2-}$ or $[PdBr_3(Et_2SO)]^-$ (Table 1, Figs. 1–3). The crystal of

complex **2** is formed by two types of crystallographically independent cations and anions.

$$2[Ph_{3}PR]Br + PdBr_{2} \xrightarrow{(2) MeCN} [Ph_{3}PR]^{+}_{2}[PdBr_{4}]^{2-}$$
(1)

$$R = CH_{2}CN (1), Ph (2).$$

$$2[Ph_{3}PC_{5}H_{9}-cyclo]Br + PdBr_{2} \xrightarrow{H_{2}O, HBr} [Ph_{3}PC_{5}H_{9}-cyclo]^{+}_{2}[PdBr_{4}]^{2-}$$
(2)

$$\xrightarrow{Et_{2}SO} [Ph_{3}PC_{5}H_{9}-cyclo]^{+}[PdBr_{3}(Et_{2}SO)]^{-} + [Ph_{3}PC_{5}H_{9}-cyclo]Br$$
(2)

$$3$$

$$2 \xrightarrow{Am_{2}SO, MeCN} [Ph_{4}P]^{+}_{2}[Pd_{2}Br_{6}]_{2}$$
(3)



Fig. 1. General view of the molecule of complex 1 in the crystal.

Phosphorus atoms in the cations have slightly distorted tetrahedral coordination with close P–C bond lengths [1.780(3)–1.816(3), 1.791(5)–1.809(5), and 1.776(13)–1.812(8) Å, respectively] and with valence CPC angles of 106.69(15)–111.29(14) (1), 106.6(2)–113.5(2) (2), and 108.0(4)–111.1(4)° (3), which practically do not differ from the theoretical value. The P–C_{Alk} bonds are longer than the P–C_{Ph} bonds (Table 2).

In the centrosymmetric square $[PdBr_4]^{2-}$ anions, the Pd–Br bond lengths [2.4363(16), 2.4438(14) Å (1) and 2.4403(18)-2.466(3) Å (2)] are comparable with similar distances in the square asymmetric anion **3** [2.4337(19)-2.4517(19) Å]; the S–Pd distance [2.269(3) Å] is somewhat smaller than the sum of the covalent radii of palladium and sulfur atoms (2.34 Å [25]). In complex **3**, the *cis*-angles BrPdBr [89.49(7) and 89.64(8)°] differ slightly from the theoretical value (90°); the *trans*-angles BrPdBr and SPdBr are 176.47(5) and 175.65(8)°, respectively. In the anions of complexes **1** and **2** the palladium atom is in the plane formed by four Br atoms, whereas in anion **3** the palladium atom deviates from the Br_3S plane by 0.008 Å.

The crystal of complex **4** (Fig. 4) consists of tetrahedral tetraphenylphosphonium cations [CPC angles $108.50(15)^{\circ}-110.61(15)^{\circ}$, P–C bonds 1.803(3)-1.806(3) Å] and centrosymmetric binuclear anions $[Pd_2Br_6]^{2-}$, in which the distances between the palladium atom and the terminal bromine atoms [2.4064(13) and 2.4141(11) Å] are much shorter than the bond lengths involving bridging bromine atoms [2.4541(14) and 2.4740(12) Å].

Packing of ions in the crystals of complexes 1–4 is caused by weak interionic contacts involving hydrogen atoms and Br, N, and O heteroatoms. Three types of contacts are observed in complex 1: $C\equiv N\cdots H-C$ 2.58 Å, Pd–Br \cdots H–C_{methylene} 2.61, 3.01 Å, and Pd–Br \cdots H–C_{Ph} 3.00 Å. In crystals of complexes 2 and 4, tetraphenylphosphonium cations are bound to anions by Pd–Br \cdots H–C hydrogen bonds [2.93–3.02 (2) and 2.97 Å (4)]. The contacts in complex 3 are more diverse. Cations



Fig. 2. General view of the molecule of complex 2 in the crystal.



Fig. 3. General view of the molecule of complex 3 in the crystal.

U	U		
Bond	d, Å	Angle	ω, deg
	1		
Pd ¹ –Br ^{1A}	2.4438(14)	Br ^{1A} Pd ¹ Br ¹	180.000(12)
Pd ¹ –Br ¹	2.4438(14)	Br ^{2A} Pd ¹ Br ^{1A}	90.73(7)
Pd^1-Br^{2A}	2.4363(16)	$Br^2Pd^1Br^1$	90.73(7)
Pd^1-Br^2	2.4364(16)	$Br^{2A}Pd^{1}Br^{1}$	89.27(7)
$P^1 - C^{11}$	1.783(3)	$Br^{2A}Pd^{1}Br^{2}$	180.0
$P^{1}-C^{1}$	1.780(3)	$C^{11}P^1C^{21}$	111.25(15)
$P^{1}-C^{21}$	1.790(3)	$C^{11}P^1C^7$	106.69(15)
P^1-C^7	1.816(3)	$C^{1}P^{1}C^{11}$	111.29(14)
$N^{1}-C^{8}$	1.136(5)	$C^{21}P^1C^7$	107.14(15)
Symmetry transformations: a) 1-	-x, 1-y, 1-z.		
	2		
Pd^1-Br^1	2.454(3)	$Br^{1A}Pd^{1}Br^{1}$	180.00(3)
Pd ¹ –Br ^{1A}	2.454(3)	Br ² Pd ¹ Br ^{1A}	90.24(6)
Pd^1-Br^2	2.4402(18)	$Br^2Pd^1Br^1$	89.76(6)
Pd ² –Br ³	2.4428(18)	Br ^{3b} Pd ² Br ³	179.999(1)
Pd ² –Br ^{3B}	2.4428(18)	Br ³ Pd ² Br ⁴	89.59(6)
Pd^2-Br^4	2.466(3)	$\mathrm{Br}^{\mathrm{3B}}\mathrm{Pd}^{\mathrm{2}}\mathrm{Br}^{\mathrm{4}}$	90.41(6)
$P^{1}-C^{21}$	1.809(5)	$\mathrm{Br}^{4}\mathrm{Pd}^{2}\mathrm{Br}^{4\mathrm{B}}$	180.00(3)
$P^{1}-C^{31}$	1.791(5)	$C^{1}P^{1}C^{31}$	106.6(2)
$P^2 - C^{71}$	1.806(5)	$C^{11}P^1C^1$	113.5(2)
$P^2 - C^{61}$	1.795(5)	$C^{71}P^2C^{41}$	111.2(2)
Symmetry transformations: a) 1-	-x, 2-y, 1-z; b) -x, 1-y, 2-z.		'
	3		1
Pd^1-Br^1	2.4337(19)	$Br^1Pd^1Br^2$	89.49(7)
Pd^1-Br^2	2.4357(19)	Br ¹ Pd ¹ Br ³	176.47(5)
Pd^1-Br^3	2.4517(19)	$Br^2Pd^1Br^3$	89.64(8)
Pd^1-S^1	2.269(3)	$S^{1}Pd^{1}Br^{1}$	90.50(9)
S^1-O^1	1.457(7)	$S^{1}Pd^{1}Br^{2}$	175.65(8)
$S^{1}-C^{9}$	1.800(12)	$S^{1}Pd^{1}Br^{3}$	90.62(9)
P^1-C^1	1.795(8)	$O^1S^1Pd^1$	118.7(3)
$P^{1}-C^{21}$	1.792(9)	$O^1S^1C^9$	108.5(5)
$P^{1}-C^{11}$	1.778(8)	$O^1S^1C^7$	105.9(6)
$P^1 - C^{31}$	1.812(8)	$C^9S^1Pd^1$	110.8(4)
- 1 - 1	4	- 1 1 - 2	1
$Pd^{1}-Br^{1}$	2.4141(11)	$O^1Sb^1O^2$	174.16(5)
Pd ¹ –Br ^{2a}	2.4541(14)	O ¹ Sb ¹ C ²¹	94.75(7)
Pd^1-Br^2	2.4740(12)	$C^{1}Sb^{1}O^{2}$	88.74(8)
Pd ¹ –Br ³	2.4064(13)	$C^{21}Sb^1C^1$	119.95(9)
$Br^2 - Pd^{1a}$	2.4542(14)	$C^{21}Sb^1C^{11}$	118.71(9)
$P^{1}-C^{1}$	1.805(3)	$C^{11}Sb^1C^1$	120.02(9)
$P^1 - C^{31}$	1.806(3)	$O^{2}S^{1}C^{31}$	103.70(11)
$P^1 - C^{21}$	1.804(3)	$O^3S^1O^2$	110.92(12)
$P^1 - C^{11}$	1.803(3)	$O^{3}S^{1}C^{31}$	105.16(12)
Symmetry transformations: a) – <i>x</i>	z, 2–y, 1–z.		

 Table 2. Main bond lengths and bond angles in structures 1–4



Fig. 4. General view of the molecule of complex 4 in the crystal.



Fig. 5. System of interionic contacts in complex 3 (projection along the *b* crystallographic axis).

and anions are bound by hydrogen bonds (Pd–Br···H– $C_{cyclopent}$ 2.90, Pd–Br···H– C_{Ph} 2.94, and S=O···H– C_{Ph} 2.61, 2.65 Å). The shortest contact (S=O···H– $C_{sulfoxide}$ 2.43 Å) is less by 0.19 Å than the sum of the van der Waals radii of oxygen and hydrogen atoms (2.62 Å [26]) and is observed between diethyl sulfoxide anion ligands. The packing of ions and the system of interionic contacts in the crystal of complex **3** are shown in Fig. 5.

Thus, palladium ionic complexes with mononuclear anions $[Ph_3PCH_2CN][PdBr_4]$, $[Ph_4P][PdBr_4]$, and $[Ph_3PC_5H_9$ -*cyclo*][PdBr_3(Et_2SO)] were obtained from organyltriphenylphosphonium bromides and

palladium dibromide in the presence of hydrobromic acid. Holding a mixture of equimolar amounts of $[Ph_4P][PdBr_4]$ and diamyl sulfoxide in acetonitrile leads to the formation of the $[Ph_4P]_2[Pd_2Br_6]$ complex. According to X-ray diffraction data, phosphonium complexes of palladium $[Ph_3PCH_2CN][PdBr_4]$, $[Ph_4P][PdBr_4]$, and $[Ph_3PC_5H_9-cyclo][PdBr_3(Et_2SO)]$ contain square mononuclear $[PdBr_4]^{2-}$ and $[PdBr_3(Et_2SO)]^{-}$ anions, and the $[Ph_4P]_2[Pd_2Br_6]$ complex – planar binuclear $[Pd_2Br_6]^2$ anions.

EXPERIMENTAL

The IR spectra of samples in KBr pellets were recorded on a Shimadzu IRAffinity-1S IR Fourier spectrometer in the range 4000–400 cm⁻¹; elemental analysis was performed on a Euro EA3028-NT analyzer. X-ray diffraction analysis of crystals of complexes 1-4 was performed on a Bruker D8 QUEST automatic four-circle diffractometer (graphite monochromator) at 293 K. Data collection and primary processing, refinement of the unit cell parameters, absorption accounting, determination and refinement of structures were carried out according to programs [27–29]. The structures were determined by the direct method and refined by the least squares method in the anisotropic approximation for nonhydrogen atoms. Complete tables of atomic coordinates, bond lengths, and bond angles have been deposited at the Cambridge Crystallographic Data Center [CCDC 2115219 (1), 1898988 (2), 1908755 (3), 1898990 (4)].

Complex [Ph₃PCH₂CN]₂[PdBr₄] (1). Palladium dibromide (0.15 g, 0.56 mmol) was dissolved in 2 mL of 48% hydrobromic acid, and a solution of 0.43 g (1.12 mmol) of triphenylcyanomethylphosphonium bromide in 20 mL of hot water was added with stirring. The brown precipitate was filtered off, dried, and dissolved in 20 mL of acetonitrile. After evaporation of the solvent, 0.51 g (88%) of brown crystals were obtained, t. decomp. 215°C. IR spectrum, v, cm⁻¹: 3057, 3030, 2922, 2830, 2731, 2255, 1584, 1483, 1437, 1375, 1337, 1231, 1196, 1167, 1113, 995, 822, 743, 725, 5085, 509, 492, and 442. Found, %: C 46.54; H 3.38. C₄₀H₃₄Br₄N₂P₂Pd. Calculated, %: C 46.61; H 3.33.

Complex $[Ph_4P]_2[PdBr_4]$ (2) was obtained similarly. Yield 93%, brown crystals, t. decomp. 248°C. IR spectrum, v, cm⁻¹: 3075, 3051, 3021, 3005, 2990, 1584, 1481, 1437, 1337, 1315, 1186, 1161, 1107, 1026, 995, 752, 723, 689, 615, 5627, and 432. Found, %: C 52.09; H 3.61. C₄₈H₄₀Br₄P₂Pd. Calculated, %: C 52.18; H 3.65. **Complex** [Ph₃PC₅H₉-*cyclo*][PdBr₃(Et₂SO)] (3) was obtained by a similar procedure followed by recrystallization of the precipitate from diethyl sulfoxide. Yield 72%, brown crystals, t. decomp. 149°C. IR spectrum, v, cm⁻¹: 3075, 3051, 3034, 2968, 2932, 2920, 2909, 2868, 1585, 1483, 1439, 1406, 1325, 1277, 1252, 1188, 1136, 1111, 995, 789, 762, 745, 725, 692, 525, and 449. Found, %: C 41.48; H 4.22. $C_{27}H_{32}Br_{3}OPPdS$. Calculated, %: C 41.49; H 4.13.

Complex $[Ph_4P]_2[Pd_2Br_6]$ (4) was obtained by a similar procedure followed by recrystallization of the precipitate from a diamyl sulfoxide–acetonitrile mixture. Yield 63%, brown crystals, t. decomp. 252°C. IR spectrum, v, cm⁻¹: 3075, 3051, 3005, 2988, 1583, 1481, 1437, 1339, 1315, 1186, 1161, 1107, 1026, 997, 752, 723, 689, 527, 461, and 434. Found, %: C 41.94; H 3.02. $C_{48}H_{40}Br_6PPd_2$. Calculated, %: C 42.05; H 2.94.

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CONFLICT OF INTEREST

No conflict of interest was declared by the authors.

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