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Palladium Complexes $[Ph_3PEt][PdBr_3(DMSO)]$, $[Ph_3PCH_2OMe][PdBr_3(DMSO)]$, $[Ph_3PC_5H_9$-*cyclo*$][PdBr_3(DMSO)]$, and $[Ph_3PCH_2CH{=}CHCH_2PPh_3][PdBr_3(DMSO)]_2$: Synthesis and Structure

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Abstract—Ionic complexes of palladium(II) with mononuclear anions $[Ph_3PEt][PdBr_3(DMSO)]$ (**I**), $[Ph_3PCH_2OMe][PdBr_3(DMSO)]$ (**II**), $[Ph_3PC_5H_9$-*cyclo*$][PdBr_3(DMSO)]$ (**III**), and $[Ph_3PCH_2CH{=}CHCH_2PPh_3][PdBr_3(DMSO)]_2$ (**IV**) are synthesized from organyl triphenylphosphonium bromide and palladium(II) bromide in the presence of hydrobromic acid. The structures of the complexes are determined by X-ray diffraction (CIF files CCDC nos. 2115506 (**I**), 2115228 (**II**), 888748 (**III**), and 2115007 (**IV**)). The complexes contain organyltriphenylphosphonium cations and $[PdBr_3(DMSO)]$ anions.

Keywords: bromopalladate(II) anions, organyltriphenylphosphonium cations, dimethyl sulfoxide, X-ray diffraction studies

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INTRODUCTION

The coordination chemistry of palladium complexes is interesting due to their efficient application in catalysis [1–10] and wide use in reactions of organic and organoelemental synthesis [11–15]. Structural features of coordination palladium compounds are intensively studied at present. Numerous structurally characterized ionic halogen-containing palladium complexes are mainly presented by mononuclear [PdHal4]2– anions and, to a less extent, binuclear [Pd2Hal6]2– anions [16]. The influence of solvents on the design of the Pd-containing anions and mutual transformations of the anions into each other in various solvents were considered [17]. The synthesis of the palladium(II) complexes with the S-coordinated dimethyl sulfoxide molecule in the [PdHal3(DMSO)]– mononuclear anions was reported [18–28], but the bromine-containing derivatives among them are not numerous.

The study of the synthesis and structural features of the earlier unknown ionic palladium complexes $[Ph_3PR]^+[PdBr_3(DMSO)]^-$ is continued in this work.

EXPERIMENTAL

The following reagents were used: 48% hydrobromic acid (Sigma-Aldrich); palladium(II) bromide (ABCR); ethyl triphenylphosphonium, methoxymethyl triphenylphosphonium, and cyclopentyl triphenylphosphonium bromides; and butene-2-bis(triphenylphosphonium) dibromide (Alfa Aesar). The solvent was dimethyl sulfoxide (reagent grade, used as received).

Synthesis of $[Ph_3PEt][PdBr_3(DMSO)]$ (I). Palladium(II) bromide (0.15 g, 0.56 mmol) was dissolved in 48% hydrobromic acid (2 mL), and a solution of ethyl triphenylphosphonium bromide (0.42 g, 1.12 mmol) in hot water (20 mL) was added with stirring to the resulting solution. A brown precipitate was formed, filtered off, dried, and dissolved in dimethyl sulfoxide (5 mL). Brown crystals of complex **I** were obtained after the evaporation of the solvent in air for 3 weeks. The yield was 0.31 g (78%). T_{decomp} = 114°C.

IR (ν, cm^{-1}): 3053, 3022, 2988, 2941, 2909, 1585, 1479, 1437, 1186, 1117, 1022, 995, 976, 914, 733, 721, 690, 529, 503, 488, 424.

For $C_{22}H_{26}OSPBr_3Pd$		
Anal. calcd., %	C, 36.93	H, 3.66
Found, %	C, 36.68	H, 3.71

Synthesis of $[Ph_3PCH_2OMe][PdBr_3(DMSO)]$ (II) was carried out using a method similar to that for com-

plex **I** from methoxymethyl triphenylphosphonium bromide (0.43 g, 1.12 mmol) and palladium(II) bromide (0.15 g, 0.56 mmol). Complex **II** represents brown crystals with T_{decomp} = 118°C. The yield was 0.30 g (72%). IR (ν, cm^{-1}): 3061, 3021, 2992, 2909, 2828, 1585, 1483, 1439, 1314, 1188, 1113, 1022, 997, 972, 947, 934, 918, 893, 793, 745, 721, 691, 532, 501, 426.

For $C_{22}H_{26}O_2SPBr_3Pd$

Anal. calcd., %	C, 36.12	H, 3.58
Found, %	C, 36.03	H, 3.66

Synthesis of $[Ph_3PC_5H_9$-*cyclo*$][PdBr_3(DMSO)]$ (III) was carried out using a method similar to that for complex **I** from cyclopentyl triphenylphosphonium bromide (0.46 g, 1.12 mmol) and palladium(II) bromide (0.15 g, 0.56 mmol). Complex **III** represents brown crystals with T_{decomp} = 166°C. The yield was 0.37 g (86%). IR (ν, cm^{-1}): 3052, 3040, 3027, 3010, 2993, 2962, 2924, 2914, 2872, 1586, 1487, 1440, 1343, 1317, 1294, 1111, 1024, 996, 908, 759, 750, 725, 693, 530, 515, 457, 433.

For $C_{25}H_{30}OSPBr_3Pd$

Anal. calcd., %	C, 39.73	H, 3.97
Found, %	C, 40.12	H, 4.07

Synthesis of $[Ph_3PCH_2CH{=}CHCH_2PPh_3]$-$[PdBr_3(DMSO)]_2$ (IV) was carried out using a method similar to that for complex **I** from butene-2-bis(triphenylphosphonium) dibromide (0.41 g, 0.56 mmol) and palladium(II) bromide (0.15 g, 0.56 mmol). Complex **IV** represents brown crystals with T_{decomp} = 202°C. The yield was 0.26 g (65%). IR (ν, cm^{-1}): 3051, 3013, 2974, 2935, 2907, 2851, 1634, 1607, 1585, 1481, 1435, 1311, 1184, 1161, 1113, 995, 974, 812, 746, 721, 689, 540, 507, 461, 447, 428.

For $C_{44}H_{48}O_2S_2P_2Br_6Pd_2$

Anal. calcd., %	C, 37.031	H, 3.39
Found, %	C, 36.95	H, 3.71

IR spectra were recorded on a Shimadzu IRAffinity-1S FT-IR spectrometer in KBr pellets. Elemental analysis was carried out on a Euro EA3028-HT analyzer.

X-ray diffraction (XRD) studies were carried out on a D8 QUEST automated four-circle diffractometer (Bruker) with a graphite monochromator at 293 K. Data were collected and primarily processed, unit cell parameters were refined, an absorption correction was applied, and the structures were determined and refined using the known programs [29–31]. The structures were solved by a direct method and refined by least squares in the anisotropic approximation for non-hydrogen atoms.

The full tables of atomic coordinates, bond lengths, and bond angles were deposited with the Cambridge Crystallographic Data Centre (CIF files CCDC nos. 2115506 (**I**), 2115228 (**II**), 888748 (**III**), 2115007 (**IV**); deposit@ccdc.cam.ac.uk or http://www.ccdc.cam.ac.uk/data_request/cif).

Compound **I**: triclinic brown crystals, space group $P\bar{1}$, a = 9.571(6), b = 11.169(6), c = 12.978(10) Å, α = 77.51(3)°, β = 77.26(4)°, γ = 70.21(2)°, V = 1257.9(14) $Å^3$, Z = 2, ρ_{calc} = 1.889 g/cm^3; μ = 5.661 mm^{-1}, $F(000)$ = 696.0. The total number of measured reflections was 29674, the number of independent reflections was 5588 (R_{int} = 0.0590), and 265 refinement parameters: R_1 = 0.0509, wR_2 = 0.1243.

Compound **II**: monoclinic brown crystals, space group $P2_1$, a = 12.596(15), b = 9.141(10), c = 22.96(2) Å, β = 91.81(5)°, V = 2643(5) $Å^3$, Z = 2, ρ_{calc} = 1.839 g/cm^3; μ = 5.394 mm^{-1}, $F(000)$ = 1424.0. The total number of measured reflections was 36366, the number of independent reflections was 8088 (R_{int} = 0.0480), and 547 refinement parameters: R_1 = 0.0290, wR_2 = 0.0625.

Compound **III**: monoclinic brown crystals, space group Cm, a = 12.5097(2), b = 14.1923(3), c = 17.6335(7) Å, β = 116.3960(10)°, V = 1418.05(5) $Å^3$, Z = 2, ρ_{calc} = 1.810 g/cm^3; μ = 5.142 mm^{-1}, $F(000)$ = 740.0. The total number of measured reflections was 30522, the number of independent reflections was 5948 (R_{int} = 0.0423), and 291 refinement parameters: R_1 = 0.0173, wR_2 = 0.0191.

Compound **IV**: triclinic brown crystals, space group $P\bar{1}$, a = 9.755(75), b = 11.241(6), c = 12.276(7) Å, α = 92.48(2)°, β = 111.02(3)°, γ = 96.03(3)°, V = 1244.8(13) $Å^3$, Z = 2, ρ_{calc} = 1.904 g/cm^3; μ = 5.720 mm^{-1}, $F(000)$ = 692.0. The total number of measured reflections was 30998, the number of independent reflections was 5560 (R_{int} = 0.0345), and 264 refinement parameters: R_1 = 0.0387, wR_2 = 0.0926.

RESULTS AND DICUSSION

For the synthesis of the ionic palladium(II) complexes, an aqueous solution of alkyl triphenylphosphonium bromide was poured to a solution of palladium(II) bromide in hydrobromic acid. A brown precipitate was formed, filtered off, dried, and dissolved in dimethyl sulfoxide.

Alkyl triphenylphosphonium dimetylsulfoxidotribromopalladate $[Ph_3PR]^+[PdBr_3(DMSO)]^-$ was obtained as brown crystals after the solvent was evaporated.

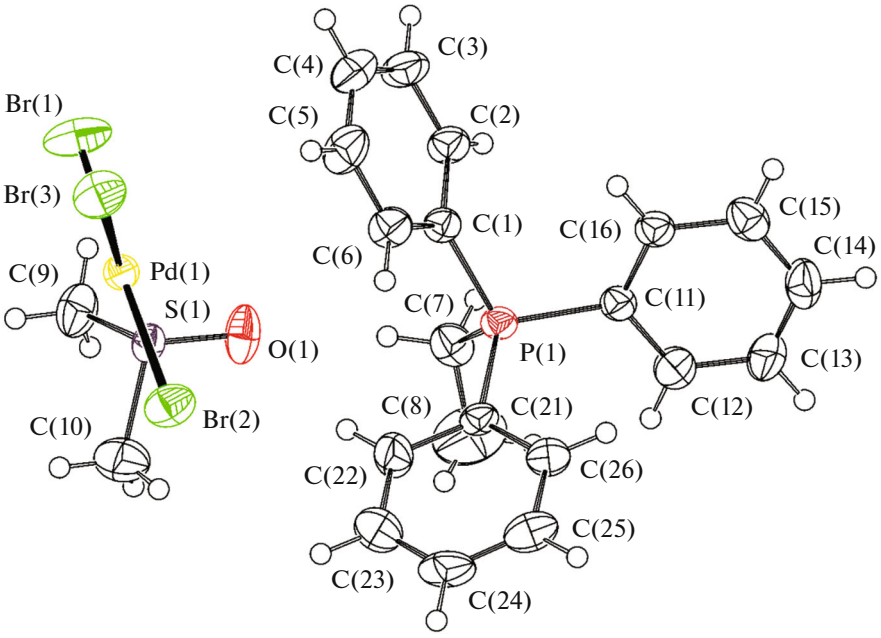

Fig. 1. General view of complex **I**. Bond lengths Pd(1)−Br(1) 2.4258(13), Pd(1)−Br(2) 2.4320(13), Pd(1)−Br(3) 2.4429(17), Pd(1)−S(1) 2.2669(18), P(1)−C(1) 1.792(4), and P(1)−C(7) 1.811(5) Å and bond angles Br(1)Pd(1)Br(2) 178.13(3)°, S(1)Pd(1)Br(3) 177.30(3)°, C(1)P(1)C(7) 106.2(2)°, and C(7)P(1)C(11) 113.5(2)°.

$$[Ph_3PR]^+[Br]^- + PdBr_2 \xrightarrow[2.\,DMSO]{1.\,H_2O/HBr} [Ph_3PR]^+[PdBr_3(DMSO)]^-$$
$$R = Et\ (\mathbf{I}), CH_2OMe\ (\mathbf{II}), C_5H_9\text{-}cyclo\ (\mathbf{III})$$

The use of the phosphonium salt with a more complicated cation in the reaction resulted in the formation of the complex also with the mononuclear anion.

$$[Ph_3PCH_2CH{=}CHCH_2PPh_3]Br_2 + 2PdBr_2 \xrightarrow[2.\,DMSO]{1.\,H_2O/HBr} [Ph_3PCH_2CH{=}CHCH_2PPh_3]^{2+}[PdBr_3(DMSO)]_2^-$$
(IV)

Compounds **I**–**IV** are brown crystalline substances resistant to air moisture and oxygen, highly soluble in acetonitrile, chloroform, and tetrahydrofuran, and insoluble in aliphatic hydrocarbons.

The IR spectra of compounds **I**–**IV** exhibit intense absorption bands in ranges of 1440–1435 and 997–995 cm^{-1} corresponding to stretching vibrations of the P–C_{Ph} bond [32]. The observed shift of the absorption bands of the S=O bond (1117–1111 cm^{-1}) to the short-wavelength range compared to unbound dimethyl sulfoxide (1055 cm^{-1}) is characteristic of similar complexes with *S*-coordinated dimethyl sulfoxide [33].

According to the XRD data, the crystals of complexes **I**–**IV** consist of organyltriphenylphosphonium cations and mononuclear anions (Figs. 1–4), and two types of crystallographically independent cations and anions are observed in the crystal of complex **II**.

The phosphorus atoms in the cations have a slightly distorted tetrahedral coordination with the CPC bond angles 106.2(2)°–113.5(2)° (**I**), 105.4(2)°–111.5(2)° (**II**), 108.0(2)°–111.4(2)° (**III**), and 107.51(16)°–111.77(17)° (**IV**), which differ slightly from the theoretical value, and close P–C bond lengths (1.792(4)–1.811(5), 1.788(5)–1.832(5), 1.783(5)–1.804(4), and 1.787(4)–1.806(3) Å, respectively). The P–C_{Alk} bonds are longer than P–C_{Ph}. In the planar square $[PdBr_3(DMSO)]^-$ anions, the Pd–Br bond lengths vary in the ranges 2.4258(13)–2.4429(17), 2.437(3)–2.444(2) and 2.431(2)–2.447(2), 2.428(3)–2.436(3), 2.4099(12)–2.4343(14) Å, respectively, and the S–Pd distances (2.2669(18), 2.265(3) and 2.270(3), 2.2691(19), 2.2571(16) Å) are

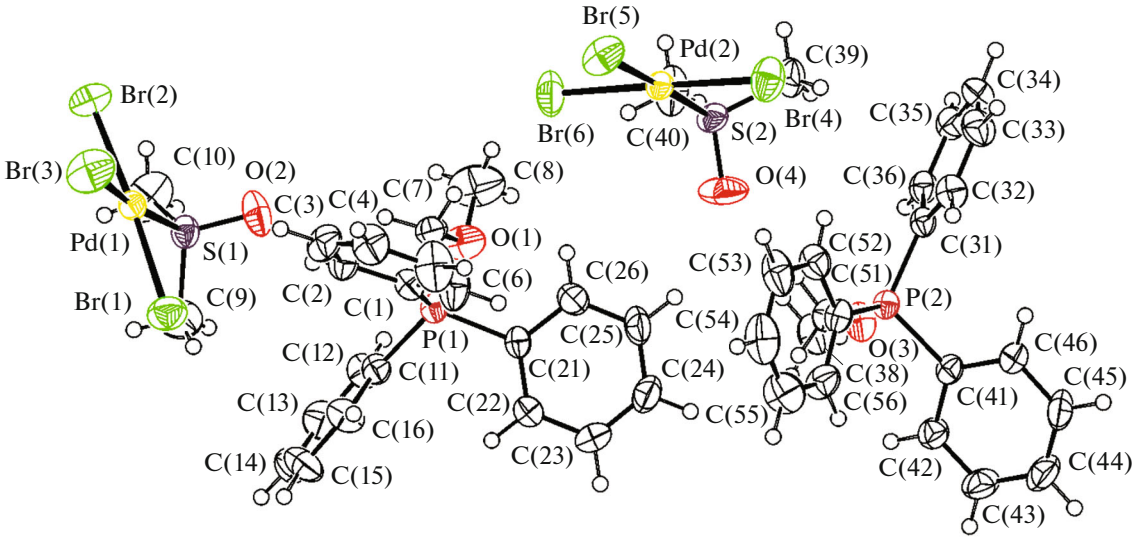

Fig. 2. General view of complex **II**. Bond lengths Pd(1)–Br(1) 2.441(2), Pd(1)–Br(2) 2.444(2), Pd(1)–Br(3) 2.437(3), Pd(1)–S(1) 2.265(3), Pd(2)–Br(4) 2.431(2), Pd(2)–Br(5) 2.447(3), Pd(2)–Br(6) 2.427(2), Pd(2)–S(2) 2.270(3), P(1)–C(11) 1.788(5), P(1)–C(7) 1.832(5), P(2)–C(31) 1.795(5), and P(2)–C(37) 1.813(5) Å and bond angles Br(1)Pd(1)Br(2) 176.43(3)°, S(1)Pd(1)Br(3) 172.12(4)°, C(1)P(1)C(7) 105.7(2)°, C(7)P(1)C(11) 110.6(3)°, Br(4)Pd(2)Br(6) 178.01(3)°, S(2)Pd(2)Br(5) 177.51(4)°, C(37)P(2)C(51) 105.4(2)°, and C(31)P(2)C(51) 111.5(2)°.

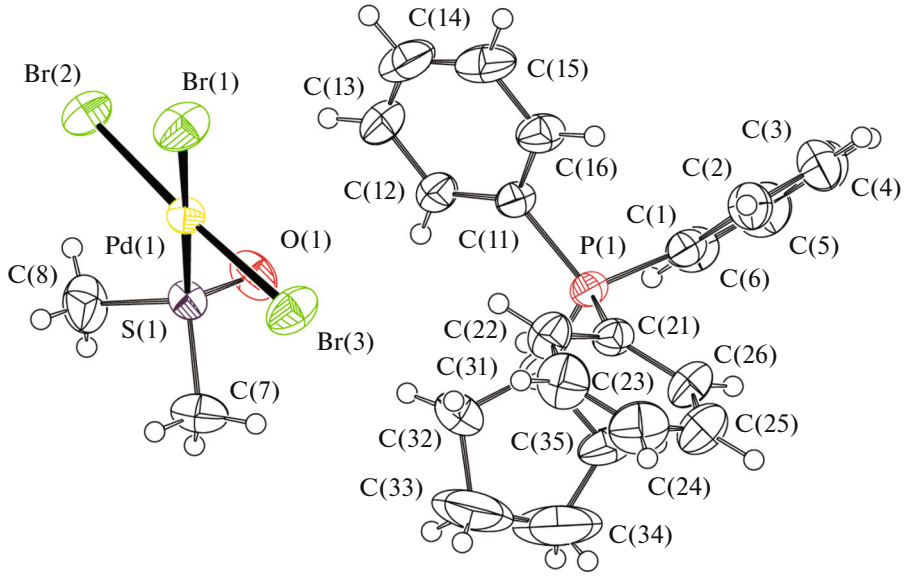

Fig. 3. General view of complex **III**. Bond lengths Pd(1)–Br(1) 2.4298(19), Pd(1)–Br(2) 2.436(3), Pd(1)–Br(3) 2.428(3), Pd(1)–S(1) 2.2691(19), P(1)–C(1) 1.783(5), and P(1)–C(31) 1.804(4) Å and bond angles Br(2)Pd(1)Br(3) 176.30(2)°, S(1)Pd(1)Br(3) 178.85(3)°, C(11)P(1)C(21) 108.0(2)°, and C(11)P(1)C(31) 111.4(2)°.

less than the sum of covalent radii of palladium and sulfur atoms (2.44 Å [34]). The *cis*-BrPdBr angles (89.19(4)° and 90.24(4)°, 89.23(3)°–91.33°, 89.32(6)° and 89.65(6)°, 89.80(4)° and 89.80(4)°) do not nearly differ from the theoretical value (90°). The *trans*-BrPdBr and *trans*-SPdBr angles are comparable to each other and equal to 178.13(3)° and 177.30(3)° for complex **I**; 176.43(3)°, 172.12(4)° and 178.01(3)°, 177.51(4)° for complex **II**; 176.30(2)° and 178.85(3)° for complex **III**; and 175.42(3)° and 177.42(3)° for complex **IV**. In the anions of complexes **I**–**IV**, the deviation of the palladium atom from the Br_3S plane is

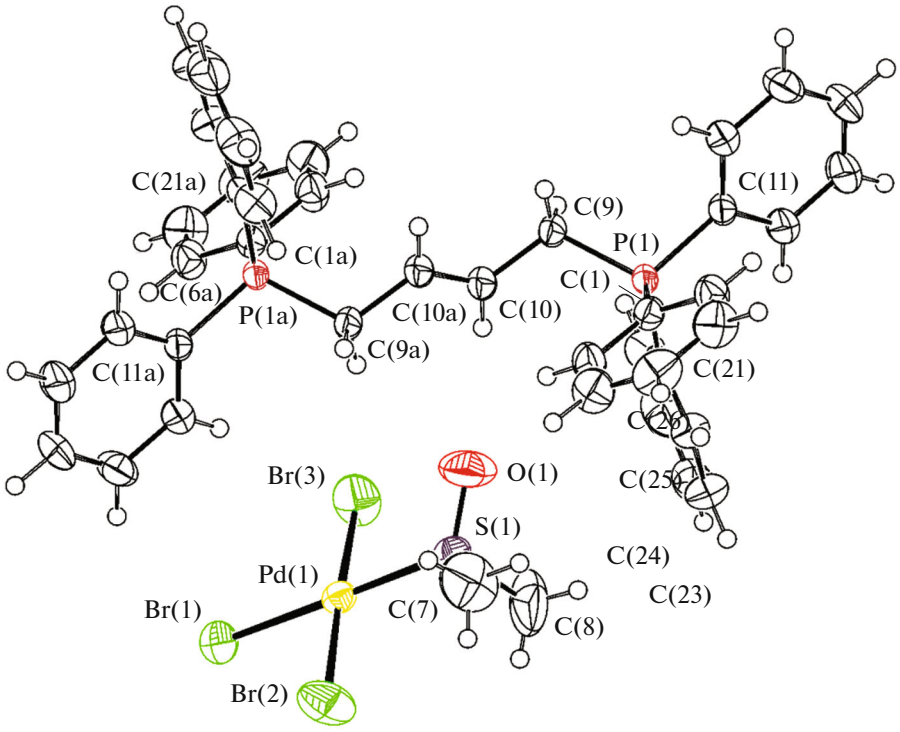

Fig. 4. General view of complex **IV**. Bond lengths Pd(1)–Br(1) 2.4343(14), Pd(1)–Br(2) 2.4240(13), Pd(1)–Br(3) 2.4099(12), Pd(1)–S(1) 2.2571(16), P(1)–C(21) 1.787(4), and P(1)–C(9) 1.806(3) Å and bond angles Br(2)Pd(1)Br(3) 175.42(3)°, S(1)Pd(1)Br(1) 177.42(3)°, C(9)P(1)C(11) 107.51(16)°, and C(1)P(1)C(9) 111.77(17)°.

insignificant (0.026–0.057 Å), and the minimum and maximum values are observed for the structures of complexes **III** and **IV**, respectively.

The structural organization in the crystals of complexes **I**–**IV** is formed by interionic contacts S=O···H–C (2.35–2.71 Å) and Pd–Br···H–C (2.72–3.04 Å), which is close to the sum of van der Waals radii of oxygen and hydrogen atoms (2.62 Å), as well as of bromine and hydrogen atoms (2.93 Å) [35].

To conclude, the reactions of organyl triphenylphosphonium bromides and palladium(II) bromide in the presence of hydrobromic acid afforded the ionic palladium complexes with the mononuclear anions. When the complexes are dissolved in dimethyl sulfoxide, an easy ligand exchange occurs in them to form complexes $[Ph_3PEt][PdBr_3(DMSO)]$ (**I**), $[Ph_3PCH_2OMe][PdBr_3(DMSO)]$ (**II**), $[Ph_3PC_5H_9\text{-}cyclo][PdBr_3\text{-}(DMSO)]$ (**III**), and $[Ph_3PCH_2CH{=}CHCH_2PPh_3][PdBr_3\text{-}(DMSO)]_2$ (**IV**). The anions exhibit the *trans*-effect of the dimethyl sulfoxide substituent leading to the elongation of the *trans*-Pd–Br bonds by 0.014–0.018 Å compared to the average values of *cis*-bonds.

CONFLICT OF INTEREST

The authors declare that they have no conflicts of interest.

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