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Mendeleev Commun., 2024, **34**, 195–197

Mendeleev
Communications

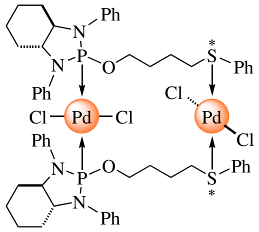
Neutral dinuclear palladium(II) complex containing chiral *P*,*S*-bridging diamidophosphite-thioether ligands

**Vladislav S. Zimarev,[a,b] Ilya V. Chuchelkin,[b] Konstantin N. Gavrilov,[b] Ilya A. Zamilatskov,[c]
Victor A. Tafeenko,[a] Boris V. Lokshin[d] and Nataliya S. Goulioukina*[a,b,c]**

[a] *Department of Chemistry, M. V. Lomonosov Moscow State University, 119991 Moscow, Russian Federation. Fax: +7 495 932 8846; e-mail: goulioukina@org.chem.msu.ru*
[b] *Department of Chemistry, S. A. Esenin Ryazan State University, 390000 Ryazan, Russian Federation. Fax: +7 4912 281 435; e-mail: k.gavrilov@365.rsu.edu.ru*
[c] *A. N. Frumkin Institute of Physical Chemistry and Electrochemistry, Russian Academy of Sciences, 119071 Moscow, Russian Federation*
[d] *A. N. Nesmeyanov Institute of Organoelement Compounds, Russian Academy of Sciences, 119334 Moscow, Russian Federation. Fax: +7 499 135 5085; e-mail: bloksh@ineos.ac.ru*

DOI: 10.1016/j.mencom.2024.02.011

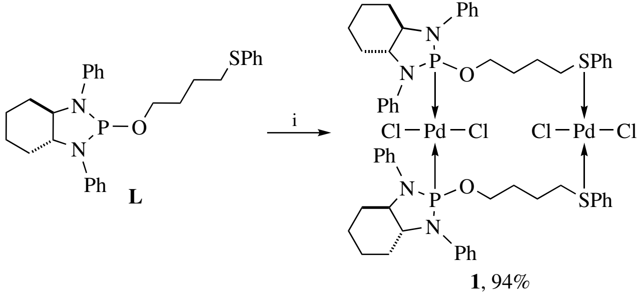
An unusual neutral dinuclear palladium(II) complex with two head-to-head orientated μ-*P*,*S*-bridging ligands was obtained by the reaction of $Pd(MeCN)_2Cl_2$ with (1*R*,5*R*)-3-(4-phenylthiobutoxy)-2,4-diphenyl-2,4-diaza-3-phosphabicyclo[3.4.0]nonane. The dimeric structure of the complex in the solid state and in solution was thoroughly confirmed using appropriate analytical techniques: single crystal X-ray diffraction analysis, HRMS, FTIR and NMR spectroscopy.



Keywords: dinuclear complexes, palladium(II) complexes, *P*,*S*-bidentate ligands, 1,3,2-diazaphospholidines, X-ray crystal structures.

The potential of chiral *P*,*S*-bidentate ligands in transition metal-mediated asymmetric transformations has been widely developed and explored.[1] Undoubtedly, the use of these stereoselectors will be expanded in the near future, which requires an in-depth study of their coordination behavior. Numerous examples show that the formation of five to eight-membered *P*,*S*-chelate complexes, both cationic and neutral, is common in the coordination chemistry of palladium.[2–4] We recently described a family of diamidophosphite-thioethers derived from 1,4-thioether alcohols with a different type of coordination behavior whereby the ligands formed μ-*P*,*S*-bridged dinuclear or polynuclear (allyl)-palladium(II) cationic complexes.[5,6] Here we report the first example of a neutral dinuclear palladium(II) complex with μ-*P*,*S*-bridging ligands.

Complex **1** was prepared in 94% yield by treatment of (1*R*,5*R*)-3-(4-phenylthiobutoxy)-2,4-diphenyl-2,4-diaza-3-phosphabicyclo[3.4.0]nonane[5] **L** with equimolar amount of $Pd(MeCN)_2Cl_2$ in CH_2Cl_2 (Scheme 1; see also Online Supplementary Materials).



Scheme 1 *Reagents and conditions*: i, $Pd(MeCN)_2Cl_2$ (1 equiv.), CH_2Cl_2, 20 °C, 1 h.

The solid-state structure of product **1** was unambiguously confirmed by X-ray diffraction.[†] The unit cell consists of two neutral dinuclear complexes **1** and four CH_2Cl_2 solvate molecules. In the molecule, two palladium atoms are coordinated by two mutually *trans* chlorido ligands and are bridged by two diamidophosphite-thioether ligands **L** which show a head-to-head orientation (Figure 1). The intramolecular metal–metal distance is very large (*ca.* 5.61 Å) and does not imply Pd–Pd interaction (the sum of the equilibrium van der Waals radii for two palladium atoms is 4.7 Å).[9] The palladium atoms show a slightly distorted square-planar coordination [sum of bond

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[†] *Crystal data for* **1**. $C_{56}H_{66}Cl_4N_4O_2P_2Pd_2S_2$ ($M = 1307.88$), monoclinic, space group $P2_1$ at 295(2) K, $a = 13.4740(5)$, $b = 18.0706(7)$ and $c = 14.6689(8)$ Å, $\alpha = 90°$, $\beta = 115.850(4)°$, $\gamma = 90°$, $Z = 4$, $d_{calc} = 1.527$ g cm^{-3}, $V = 3214.2(3)$ Å^3, μ(MoKα) = 1.050 mm^{-1}, $F(000) = 1504$. A total of 81543 reflections were collected (13224 independent reflections, $R_{int} = 0.2244$), GOOF 0.849, final R indices [$I > 2\sigma(I)$]: $R_1 = 0.0791$ and $wR_2 = 0.1622$, R indices (all data): $R_1 = 0.2409$, $wR_2 = 0.2211$, 703 refined parameters. Crystallographic data were collected on a STOE diffractometer with a Pilatus100K detector with λ(MoKα) = 0.7071 Å at 295 K. Structure solution and refinement were carried using the program SHELX,[7] DIAMOND software[8] was used to prepare the material for publication. Full matrix least-squares refinement was carried out by minimizing $(Fo^2 - Fc^2)^2$. All nonhydrogen atoms were refined anisotropically. Hydrogen atoms attached to carbon atoms were placed in the geometrically idealized positions and refined as riding on their parent atoms, with C–H = 0.93–0.98 Å with $U_{iso}(H) = 1.2\,U_{eq}(C)$.

CCDC 2207922 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from the Cambridge Crystallographic Data Centre *via* http://www.ccdc.cam.ac.uk.

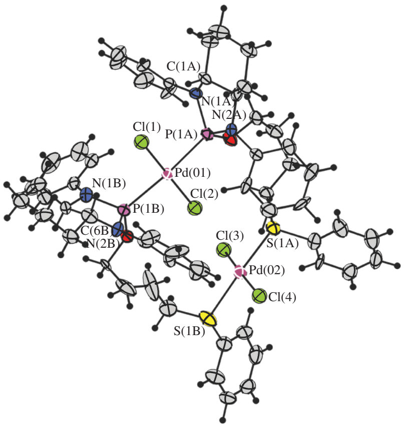

Figure 1 Molecular structure of complex **1**. Solvate molecules of CH_2Cl_2 are omitted for clarity.

angles: Pd(1) 360.3°, Pd(2) 360.1°]. The Pd(2)–S(1A) and Pd(2)–S(1B) bond lengths of 2.325(6) and 2.323(6) Å as well as the Pd(2)–Cl(3) and Pd(2)–Cl(4) bond lengths of 2.290(8) and 2.307(8) Å, respectively, are quite normal and are nearly identical to those found in *trans*-[$PdCl_2(SMePh)_2$] complexes.[10]

The stereogenic sulfur atoms adopt opposite absolute configurations, namely, (*R*)-S(1A) and (*S*)-S(1B). As a result, complex **1** containing two equivalent chiral 1,3,2-diazaphospholidine moieties lacks symmetry. Due to the opposite configurations, the S-substituents are turned towards the Cl(3) atom. This effect is apparently responsible for the significant expansion of Cl(3)–Pd(2)–S(1A) and Cl(3)–Pd(2)–S(1B) angles above 90° [95.9(2) and 93.3(3)°, respectively] and the significant compression of Cl(4)–Pd(2)–S(1A) and Cl(4)–Pd(2)–S(1B) angles [84.0(2) and 86.9(3)°, respectively]. One of the hydrogen atoms from the S(1B)-CH_2 group and one of the *ortho*-H atoms from the S(1B)-Ph substituent approach the Cl(3) atom. The corresponding interatomic distances Cl(3)···H (2.93 and 2.76 Å) are consistent with hydrogen bonding.

The Pd–P distances, *viz*. Pd(1)–P(1A) 2.301(6) Å and Pd(1)–P(1B) 2.294(6) Å, are nearly equal within the limits of error and on average 0.076 Å longer than in the neutral palladium complex *cis*-[$PdCl_2$(P–P)], where (P–P) is a bis(diamidophosphite) ligand containing diazaphospholidine terminal fragments.[11] This elongation can be attributed to the stronger *trans* influence of the diamidophosphite ligand relatively to the chlorido ligand.

The coordination geometry at the endocyclic nitrogen atoms exhibits a slight deviation from planarity [sum of bond angles: N(1A) 353.4°, N(1B) 354.1°, N(2A) 351.4°, N(2B) 351.2°]. The P–N bond distances for both diazaphospholidine groups range between 1.649(19) and 1.728(18) Å and suggest a partial double-bond character when compared to the commonly accepted bond lengths (P–N bond 1.77 Å and P=N bond 1.57 Å).[12] As reported for *N*-alkyl or *N*-benzyl substituted diazaphospholidines coordinated to a transition metal or bonded to borane, the length of the bond from nitrogen atom to substituent (N–Csp^3) varies over the range 1.455(13)–1.551(10) Å.[5,11,13,14] Complex **1** bearing *N*-phenyl substituents shows the N–Csp^2 distances of 1.44(3)–1.45(3) Å [for N(1B) and N(2A)] and 1.39(3)–1.40(3) Å [for N(1A) and N(2B) atoms]. It is doubtful whether the significant shortening of the bonds in the last case can be solely attributed to the carbon hybridization state in the phenyl N-substituent. In general, the differences in the N–Csp^2 bond lengths reflect the degree of their double-bond character, the conjugation between the unshared electron pair on the nitrogen atom and the aromatic ring depending not only on the flattening for the nitrogen atom configuration, but also on the angle of rotation of the aromatic ring on the N–Csp^2 bond.[15] The dihedral angles between the plane of the atoms surrounding the nitrogen atom (P–N–C) and the corresponding benzene ring plane are about 43° for P(1A)–N(1A)–C(1A) and 41° for P(1B)–N(2B)–C(6B), which does not contradict the possibility of conjugation. For comparison, in aniline the dihedral angle between the amino (NH_2) plane and the benzene plane is 38° and the length of the Ph–NH_2 bond is 1.392 Å.[16] Rotation of phenyl rings relatively to the planes of the environment of nitrogen atoms N(1B) and N(2A) is greater. The torsion angles are 50 and 55°, respectively, which probably eliminates conjugation. There is also a tendency on which the changes in the N–Csp^2 and P–N bond distances are correlated. The shorter N(1A)–Csp^2 and N(2B)–Csp^2 bonds correspond to the lengthening of the adjacent P(1A)–N(1A) and P(1B)–N(2B) bonds, which are about 0.04 Å longer than P(1A)–N(2A) and P(1B)–N(1B).

From a limited number of structures containing a diazaphospholidine skeleton derived from *trans*-cyclohexane-1,2-diamines,[5,11,13,14,17] it should be noted that in this class of ligands the conformational location of N-substituent is not strongly locked. As for complex **1**, the *N*-phenyl substituents in both five-membered rings are located in *pseudo*-equatorial positions. Thus, in each moiety these substituents are situated *anti* to each other, and are also positioned *anti* to the adjacent methylene carbons of the cyclohexane ring.

The most striking structural feature of complex **1** is the mutual orientation of coordination planes of Pd(1) and Pd(2). The tilt (dihedral) angle between them is *ca*. 79° so that Cl(2) atom is oriented inside the cavity of the complex. The Pd(1)–Cl(2) bond is sufficiently short [2.263(5) Å] and angles Cl(2)–Pd(1)–P(1A) and Cl(2)–Pd(1)–P(1B) are expanded above 90° [92.5(2) and 91.3(2)°, respectively]. In contrast, Pd(1)–Cl(1) bond is longer [2.310(5) Å] and angles Cl(1)–Pd(1)–P(1A) and Cl(1)–Pd(1)–P(1B) are compressed to 88.0(2) and 88.5(2)°, respectively.

Far-infrared ATR spectra of the solid sample of complex **1** and the parent ligand **L** have been recorded over the frequency range 100–600 cm^{-1} (see Online Supplementary Materials, Figure S7). In the spectrum of the complex, the absorption bands absent in the ligand spectrum appear below 400 cm^{-1}. Two most intense bands at 363 and 339 cm^{-1} may be attributed to the asymmetric Pd–Cl stretching vibrational modes of two different *trans*-ClPdC1 moieties.[18–20] Most of the bands of medium intensity can be assigned taking into account previously established frequency ranges of characteristic vibrations for square-planar complexes of the type *trans*-$PdCl_2(SR_2)_2$.[18] In particular, the band at 280 cm^{-1} may be presumably attributed to the asymmetric Pd–S stretching vibration, the band at 220 cm^{-1} – to the Pd–S–C bending vibration, the bands at 183 and 133 cm^{-1} – to the in-plane and out-of-plane Cl–Pd–Cl bending vibrations, the bands at 118 and 106 cm^{-1} – to the in-plane and out-of-plane S–Pd–S bending vibrations. Metal–phosphorus stretching frequency is expected to occur as a weak band in the range 370–420 cm^{-1}.[19] The assignment of this band is difficult due to overlap with the fundamental vibrations of ligand **L**.

To determine whether the solid-state structure is the same as in solution, complex **1** was studied by NMR spectroscopy. A single sharp singlet appears in the $^{31}P\{^1H\}$ NMR spectrum and one set of signals is observed in the ^1H and $^{13}C\{^1H\}$ NMR spectra at room temperature in $CDCl_3$. A significant upfield shift in $^{31}P\{^1H\}$ NMR from 130.6 ppm (free ligand **L**) to 88.4 ppm in complex **1** (Figure S6) attests the coordination of phosphorus to palladium, and the magnitude of the shift corresponds to the values reported in the literature for similar 1,3,2-diazaphospholidine ligands derived from *trans*-cyclohexane-1,2-diamines.[11,14] In the $^{13}C\{^1H\}$ NMR spectrum, clearly distinguishable signals for the quaternary carbons in the NPh groups as well as carbon OCH_2 appear as virtual triplets (in contrast to doublets in the uncoordinated ligand **L**). Such virtual coupling is relevant for metal complexes with two phosphorus ligands in a mutual *trans*-position, where $^2J_{\mathrm{PP}}$ is large.[21] For complex **1**, it confirms the presence of *trans*-PPdP motif. The singlet corresponding to the methylene carbon adjacent to sulfur moves downfield (from 33.29 ppm for free **L** to 39.02 ppm for **1**), indicating electron depletion at sulfur atom and hence coordination to palladium.[3,5] As mentioned above, in the solid state molecule **1** has no symmetry. A noteworthy feature of complex **1** in solution is that it gives rise to only one set of signals in the NMR spectra, suggesting the C_2-symmetry of the molecule. This result can be explained by the well-known inversion of the coordinated sulfur atom occurring on the NMR time scale,[3] especially as in the crystal structure both epimers are observed and there is quite a lot of open space around sulfur atoms.

The mass spectrum is also consistent with the proposed dinuclear structure of complex **1**. The ESI HRMS spectrum in MeOH shows a quite strong signal for the cation $[\mathrm{Pd_2Cl_3L_2}]^+$ (see Online Supplementary Materials). Note that this type of fragmentation (*i.e.*, molecular ion minus Cl) is common to neutral dichloropalladium(II) complexes.[2,11,14,22]

In summary, an unusual neutral dinuclear palladium(II) complex **1** containing two chiral diamidophosphite-thioether ligands **L** was obtained. As determined by X-ray diffraction, the complex is composed of two *trans*-ClPdCl moieties μ-*P*,*S*-bridged by two ligands orientated head-to-head. In contrast, in the dinuclear allylpalladium(II) complex $[\mathrm{Pd}(\eta^3\text{-}\mathrm{C_3H_5})\mathbf{L}]_2(\mathrm{BF_4})_2$, where only *cis*-positions in the coordination sphere of the metal center are available, these ligands are coordinated in a head-to-tail manner.[5] The features of the solid-state structure of complex **1** are the almost orthogonal mutual orientation of the coordination planes of the two palladium centers and the lack of symmetry stemming from the opposite absolute configurations of coordinated donor sulfur atoms. NMR studies confirm that the molecular structure of the complex is maintained in solution, although due to the unhindered inversion of S-atoms the molecule acquires C_2-symmetry.

We thank the Russian Science Foundation (grant no. 19-13-00197) for financial support of this work. The X-ray diffraction study was supported by the M. V. Lomonosov Moscow State University Program for Development. The registration of FTIR spectra was supported by the Ministry of Science and Higher Education of the Russian Federation (contract no. 075-03-2023-642) and was performed employing the equipment of the Center for Molecular Composition Studies of INEOS.

Online Supplementary Materials

Supplementary data associated with this article can be found in the online version at doi: 10.1016/j.mencom.2024.02.011.

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Received: 13th November 2023; Com. 23/7304