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Heterometallic Palladium-Copper and Palladium-Nickel Complexes with Pivalate Bridges

A. A. Pasynskii*, S. S. Shapovalov, I. V. Skabitskii, and O. G. Tikhonova

Kurnakov Institute of General and Inorganic Chemistry, Russian Academy of Sciences, Leninskii pr. 31, Moscow, 119991 Russia

**e-mail: aapas@rambler.ru*

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Abstract—Reactions of $(\alpha\text{-Pic})_2Pd(OOCCMe_3)_2$ (**I**) with dinuclear copper pivalate dihydrate and polymeric nickel bispivalate afforded the complexes $(\alpha\text{-Pic})_2Pd(\mu\text{-}OOCCMe_3)_2Cu_2(\mu\text{-}OOCCMe_3)_4$ (**II**) and $Pd(\mu\text{-}OOCCMe_3)_4Ni(\alpha\text{-Pic})$ (**III**), respectively, which were structurally characterized. The lantern dimers in complex **II** show no Cu···Cu bonds (Cu···Cu, 2.671(3) Å) and are united to form chains through the axial bridging pivalate groups inherited from palladium monomer **I**. In contrast, complex **III** features heterometallic palladium-nickel lanterns in which the Ni atom has an axial α-picoline ligand, while the Pd atom has no axial ligand; instead, a short Pd–Ni bond is formed (2.4976(3) Å). For triplet-state complex **III** and its zinc analog $Pd(\mu\text{-}OOCCMe_3)_4Zn(\alpha\text{-Pic})$ (**IV**), quantum chemical calculations and topological analysis of the electron density were performed.

Keywords: carboxylates, copper, palladium, nickel, metal–metal bond, X-ray diffraction, DFT calculations, electron density

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INTRODUCTION

In the mid-1950s, an enormous body of transition metal complexes with carboxylate bridges [1] was supplemented with a large family of dimeric complexes $LM(\mu\text{-}OOCR)_4ML$ shaped like a “Chinese lantern” or, more precisely, a paddle-wheel: from antiferromagnetic Cu(II) carboxylates $LCu(\mu\text{-}OOCR)_4CuL$ [2] to diamagnetic Cr(II) complexes with acetate dehydrate ligands, $(H_2O)Cr(\mu\text{-}OAc)_4Cr(H_2O)$. The latter contain a short (2.46 Å) (hypothetically, quadruple) bond between the chromium atoms [3], although the quadruple Mo–Mo bond in dinuclear molybdenum(II) tetraacetate is even shorter (2.13 Å) [4]. It should be noted that such lantern dimers are unknown for palladium carboxylates forming the triangles Pd3(OOCR)6 and showing no Pd–Pd bonds [5]. The synthesis of heterometallic acetate complexes of palladium(II) with divalent metals of the formulas $PdCo(OAc)_4 \cdot 2AcOH \cdot 2H_2O$, $PdNi(OAc)_4 \cdot AcOH \cdot$ H2O, and $PdCd(OAc)_4 \cdot H_2O \cdot AcOH$ were first reported in 1968, albeit without rigorous proof of the chemical individuality and structures of these complexes [6]. However, a research team of Russian chemists synthesized and structurally characterized several years ago a new large family of heterometallic palladium carboxylate having binuclear lantern core. In these complexes, the Pd atom has no axial ligand and the Pd–M distances (M = Co(II) [7], Zn, Cu(II), and Ni(II) [8]) are very short (~2.5 Å) and probably binding. Note that only with the high-spin Mn^{2+} ion is the Pd–Mn distance extended to 2.6566(9) Å [8, 9]. The geometry of the complexes does not vary greatly when acetates are replaced by pivalates [10], nor when pivalic acid in the axial position is replaced by acetonitrile, urea, or dibenzyl ether.

Unexpected transformations of heterometallic palladium carboxylate lantern dimers have been observed in the presence of pyridine and aniline (L'): some of the palladium atoms yield the mononuclear complex *trans*-$(L')_2Pd(OOCR)_2$ whose carboxylate groups serve as axial ligands to the M atoms (M = Zn, Ni, Co, or Mn) of two heterometallic acetate or pivalate lanterns [11]. The same complexes can be obtained in an independent synthesis involving specially prepared *trans*-$Py_2Pd(OAc)_2$ as a coupling agent between two heterometallic lanterns [11].

We found it interesting to continue studying a mononuclear palladium complex as a ligand to carboxylate lantern dimers of two types: stable copper pivalates and unstable nickel pivalates requiring sterically hindered α-substituted pyridines like α-picoline [12] or quinaldine [13] as axial ligands for stabilization. For this reason, we studied reactions of $(\alpha\text{-Pic})_2Pd(OOCCMe_3)_2$ (**I**) with dinuclear copper pivalate (as dihydrate) and polymeric nickel bispivalate.

EXPERIMENTAL

All manipulations dealing with the synthesis and isolation of complexes were carried out under argon in dehydrated solvents. The starting pivalates $Ni_9(OH)_6(HO_2CCMe_3)(O_2CCMe_3)_{12}$, $Cu_2(O_2CCMe_3)_4(HO_2CCMe_3)_2$, and $(\alpha\text{-Pic})_2Pd(O_2CCMe_3)_2$ were prepared as described in [14–16]. Chemical analysis was performed on a CHNS analyzer (Carlo Erba). IR spectra were recorded on a BrukerAlpha spectrometer.

Synthesis of $(\alpha\text{-Pic})_2Pd(\mu\text{-OOCCMe}_3)_2Cu_2(\mu\text{-OOCCMe}_3)_4$ (II). Complex **I** (0.05 g, 0.1 mmol) and $Cu_2(O_2CCMe_3)_4(HO_2CCMe_3)_2$ (0.1 g, 0.136 mmol) were stirred in CH_2Cl_2 (5 mL) for 12 h. The solvent was evaporated to dryness, and the residue was recrystallized from $MeCN–Et_2O$ and then crystallized at the CH_2Cl_2–hexane interface. The yield of complex **II** was 0.044 g (42%), green crystals.

For $C_{42}H_{68}N_2O_{12}Cu_2Pd$ (M = 1027)

anal. calcd., %:	C, 49.14;	H, 6.68.
Found, %:	C, 48.79;	H, 5.25.

IR (FTIR; ν, cm^{-1}): 2957 w, 2925 w, 2869 w, 1571 s, 1457 m, 1415 vs, 1375 s, 1360 s, 1224 s, 1158 w, 1030 w, 896 w, 788 m, 763 w, 677 w, 618 m, 453 m, 439 m.

Synthesis of $Pd(\mu\text{-OOCCMe}_3)_4Ni(\alpha\text{-Pic})$ (III). The complex $Ni_9(OH)_6(HO_2CCMe_3)(O_2CCMe_3)_{12}$ (0.21 g, 0.093 mmol) was added to complex **I** (0.13 g, 0.26 mmol) in CH_2Cl_2 (10 mL). The resulting solution was stirred for 24 h and concentrated to one third of its initial volume. Hexane was added until a green precipitate formed. The mother liquor was filtered and kept at –18°C. The yield of complex **III** was 0.11 g (63%), yellow prismatic crystals.

For $C_{26}H_{43}NO_8NiPd$ (M = 663)

anal. calcd., %:	C, 47.11;	H, 6.54.
Found, %:	C, 46.87;	H, 5.13.

IR (FTIR; ν, cm^{-1}): 2957 m, 2917 m, 2849 w, 1601 vs, 1561 m, 1480 s, 1458 m, 1421 m, 1405 m, 1388 m, 1355 m, 1321 s, 1208 s, 1160 m, 1120 w, 1064 w, 1028 w, 888 m, 808 m, 785 s, 760 m, 724 m, 635 m, 562 w, 459 m, 436 m.

X-ray diffraction was studied on Bruker Smart Apex II CCD AXS diffractometer. An absorption correction was applied by multiple measurements of equivalent reflections with the SADABS program [17]. Structures **II** and **III** were solved by direct methods and refined anisotropically (for non-hydrogen atoms) by the least-squares method on F^2 with the SHELX-2014 program package [18]. The disordered atoms of the pivalate and 2-methylpyridine ligands were refined isotropically under a constraint of equal distances (the SAME and SADI instructions). The H atoms were located geometrically. The crystallographic parameters and the data collection and refinement statistics for structures **II** and **III** are summarized in Table 1. Selected bond lengths and bond angles are given in the captions to Figs. 1 and 2. The atomic coordinates and other parameters of structures **II** and **III** have been deposited with the Cambridge Structural Database (nos. 1440550 and 1440549; http://www.ccdc.cam.ac.uk/data_request/cif).

Quantum chemical DFT calculations were performed with the ORCA 3.03 program package [19]. Scalar relativistic effects were allowed for using the ZORA approximation [20, 21]. The geometry of the complexes was optimized using the functional PBE [22, 23] with the doubly split all-electron basis set def2-SVP [24, 25] and the empirical correction D3BJ to dispersion interactions [26, 27]. Selected Pd–M and M–L distances are given in Table 2. The electron density was calculated using the hybrid functional PBE0 [28] with the triply split all-electron basis set def2-TZVP [24, 25]. Topological electron density analysis QTAIM [29] was performed with the AIMAll program [30]. Delocalization indices [31] were calculated using the Mueller approximation for a two-electron density matrix [32]. Selected density properties at bond critical points (ρ_{BCP} is the electron density, $\Delta\rho_{BCP}$ is the Laplacian of the electron density, G_{BCP} is the kinetic energy density, V_{BCP} is the potential energy density, and H_{BCP} is the total energy density) and the delocalization indices δ between the atoms forming the corresponding bond are listed in Table 2.

RESULTS AND DISCUSSION

In our review [33], we noted that aniline and its analogs coordinated axially can break down even stable copper lantern dimers because of unfavorable repulsion between the carboxylate O atoms and the phenyl C atoms of aniline that are *ortho* to nitrogen. At the same time, α-picoline coordinated, together with pivalate ligands, to palladium is innocuous to copper lantern dimers, which was observed in the formation of a heterometallic complex (Scheme 1). According to the X-ray diffraction data, this complex contains no copper–copper bonds (Cu(1)···Cu(1*A*), 2.671(3) Å; this distance is substantially longer than 2.56 Å in metallic copper) (Fig. 1, Table 1):

$$\underset{\textbf{(I)}}{(\alpha\text{-Pic})_2Pd(OOCCMe_3)_2} \xrightarrow{Cu_2(O_2CCMe_3)_4(HO_2CCMe_3)_2} \underset{\textbf{(II)}}{(\alpha\text{-Pic})_2Pd(\mu\text{-OOCCMe}_3)_2Cu_2(\mu\text{-OOCCMe}_3)_4}$$

Scheme 1.

Table 1. Crystallographic parameters and the data collection and refinement statistics for structures **II** and **III**

Parameter	**II**	**III**
M	513.23	662.72
Radiation (λ, Å)	MoK_α (0.71073)	
Temperature, K	150(2)	173(2)
Space group	$P2_1/n$	$P\bar{1}$
a, Å	12.588(1)	11.0345(8)
b, Å	11.846(1)	11.7506(9)
c, Å	16.512(2)	13.418(1)
α, deg	90	103.990(1)
β, deg	90.520(2)	95.926(1)
γ, deg	90	107.703(1)
V, Å^3	2462.3(5)	1578.6(2)
Z	4	2
ρ_{calcd}, g cm^{-3}	1.384	1.394
μ, mm^{-1}	1.273	1.208
F(000)	1068	688
θ scan range, deg	1.233–25.123	2.225–27.469
Scan mode	ω	
Number of unique reflections (N_1)	4343 (R_{int} = 0.0354)	7221 (R_{int} = 0.0173)
Number of reflections with $I > 2\sigma(I)$ (N_2)	3563	6554
Number of parameters refined	253	319
GOOF (F^2)	1.011	1.047
R_1 for N_2	0.0361	0.0313
wR_2 for N_1	0.1029	0.0840
$\Delta\rho_{max}/\Delta\rho_{min}$, e Å^{-3}	0.830/–0.569	0.658/–0.602

In contrast, a reaction of complex **I** with polymeric nickel pivalate is accompanied by breakdown of the palladium monomer and preferential formation of heterometallic lantern **III** (Fig. 2, Table1) probably containing the palladium–nickel bond (Pd(1)–Ni(2), 2.4976(3) Å), which is appreciably shorter than the sum of the covalent radii of the Pd and Ni atoms (1.39 + 1.24 = 2.63 Å [34]). Complex **III** shows a strong axial Ni(2)–N(1) bond (2.044(3) Å), while the palladium atom has no axial ligand (Scheme 2):

$$\underset{\textbf{(I)}}{(\alpha\text{-Pic})_2\text{Pd}(\text{OOCCMe}_3)_2} \xrightarrow{\text{Ni}_9(\text{OH})_6(\text{HO}_2\text{CCMe}_3)(\text{O}_2\text{CCMe}_3)_{12}} \underset{\textbf{(II)}}{\text{Pd}(\mu\text{-OOCCMe}_3)_4\text{Ni}(\alpha\text{-Pic})}$$

Scheme 2.

Using the DFT approach, Markov et al. [35] calculated the geometrical and electronic structures of the lantern complexes $\text{Pd}^{\text{II}}(\mu\text{-OOCMe})_4\text{M}^{\text{II}}\text{L}$ (M = Zn, Ni, Cu, Co, or Fe) to find out why the Pd···M distances are so short. They concluded that this shortening is only due to the contracting effect of the acetate bridges and that direct electron interactions between Pd(II) and M(II) are absent. Note, however, that the relatively shorter Pd(II)···M(II) distances were previously found in complexes with M = Ca, Sr, and Ba (3.291(4), 3.346(1), and 3.570(1) Å, respectively) [36]:

$$\text{Pd}_3(\mu\text{-OOCMe})_6 + 3\text{M}(\text{OOCMe})_2 + 12\text{MeCOOH} \rightarrow 3\text{Pd}(\mu\text{-OOCMe})_4\text{M}(\text{HOOCMe})_4.$$

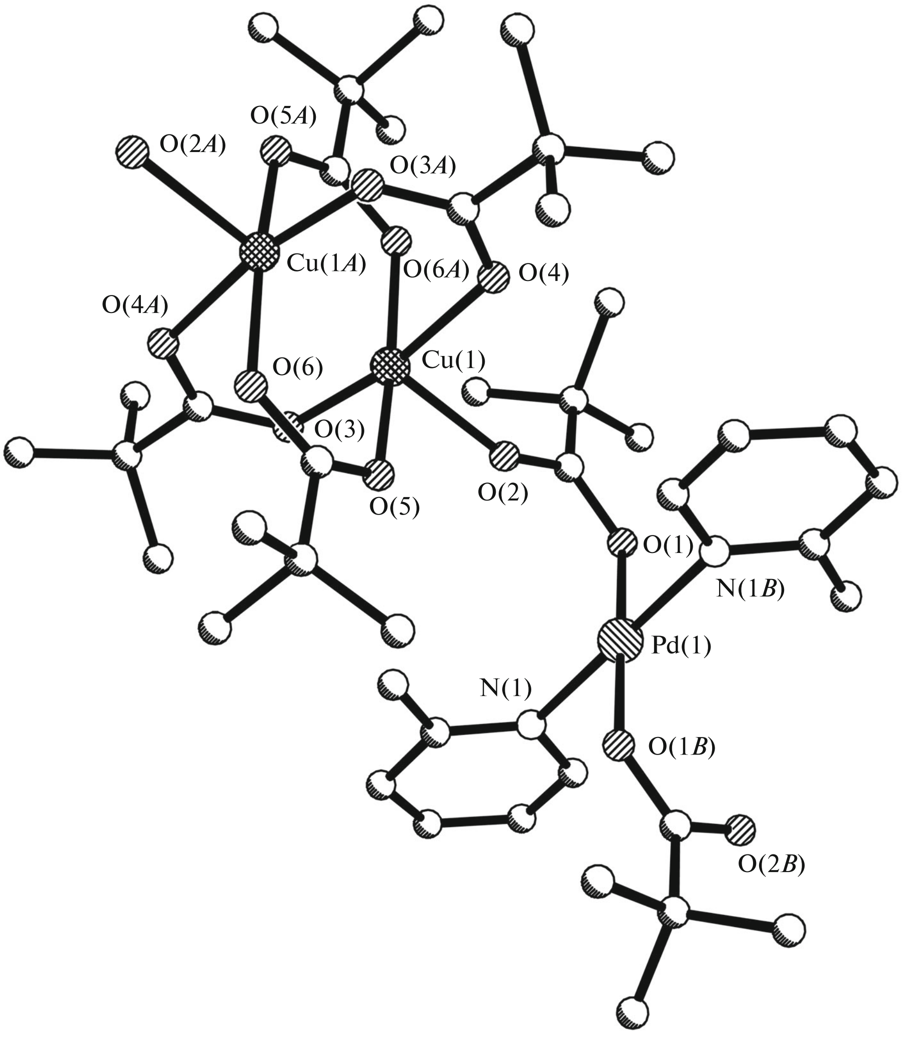

Fig. 1. Molecular structure **II**. For the disordered moieties, only one position is shown. Selected bond lengths and bond angles: Pd(1)–O(1), 2.006(2) Å; Pd(1)–N(1'), 2.024(7) Å; Pd(1)–N(1), 2.028(8) Å; Cu(1)–O(3), 1.788(4) Å; Cu(1)–O(5), 1.989(6) Å; Cu(1)–O(6*A*), 1.990(7) Å; Cu(1)–O(4), 2.129(4) Å; Cu(1)–Cu(1*A*), 2.671(3) Å; Cu(1)–O(2), 2.182(7) Å; O(1)Pd(1)N(1), 91.3(2)°; O(2)C(1)O(1), 122.7(5)°.

Here each palladium atom has a square environment made up of the O atoms of four acetate bridges of the lantern. The M atom is additionally coordinated by four AcOH molecules through the O atoms and greatly deviates from the plane of the O atoms of the acetate bridges. Nevertheless, the Pd(II)···M(II) distances are appreciably shorter than the sum of their covalent radii (3.42, 3.61, and 3.81 Å, respectively [34]), while the M–O bond lengths (2.46, 2.57, and 2.76 Å) are comparable with the sum of the covalent radii (2.42, 2.61, and 2.81 Å, respectively [34]). That is the reason why the shortened distance between the metal atoms can hardly be explained by the contracting effect alone of the bridges without considering the donation of the lone electron pair of the palladium atom to the M atoms.

Apparently, similar reasons are valid for the palladium–nickel binding in complex **III**. The participation of the lone electron pair on the d_{z}^{2}-orbital of palladium in the formation of the Pd–M bonds in heterometallic lanterns is argued for by the aforementioned absence of purely palladium carboxylate lanterns since each Pd atom in them is, as shown by calculations in [37], in the singlet state and only removal of one electron results in a single-electron Pd···Pd bond (2.634 Å, bond order 0.5). This agrees with the detection of the dinuclear cation $Pd_2(OOCR)_4^+$ in the mass spectrum of the thermolysis product of *trans*-$Py_2Pd(OAc)_2$ [37]. At the same time, Pt(II) carboxylates yield the tetramer $Pt_4(OOCR)_8$ containing a square framework in which each Pt atom forms two strong short Pt–Pt σ-bonds (2.492–2.498 Å) [38], thus lengthening the

Pt–O bonds that are *trans* to the Pt–Pt bonds (av. 2.162 Å) compared to the Pt–O bonds involving the carboxylate ligands outside the plane of the Pt_4 ring (av. 2.002 Å) [38].

To gain better insight into the binding pattern in complex **III**, we performed DFT calculations of triplet-state complex **III** and its zinc analog Pd(μ-OOCCMe$_3$)$_4$Zn(α-Pic). The calculated Pd–Ni and Pd–Zn distances (2.491 and 2.575 Å, respectively) agree well with the X-ray diffraction data for complex **III** (2.497 Å) and Pd(μ-OOCMe)$_4$Zn(H_2O) (2.576 Å) [8]. The QTAIM method [29] often used earlier to analyze metal–metal binding in coordination compounds [39] was employed for topological analysis of the calculated electron density. The calculated properties of selected bond critical points and the delocalization indices are given in Table 2.

The electron densities of Ni- and Zn-containing complexes show a bond critical point between the metal atoms. Both the density properties at the critical points and the delocalization indices suggest a Pd–M interaction, though somewhat weaker but qualitatively comparable with the M–O interactions. However, replacement of Ni by Zn weakens all bonds at the Zn atom. This is not surprising because the electrons come to the M–L antibonding orbitals when the electron configuration changes from d^8 to d^{10}.

To sum up, the Pd–M bond in heterometallic lanterns and specifically in Pd-Ni complex **III** is probably of coordination nature (Pd → M), competing with the bond between the metal atom and the axial ligand.

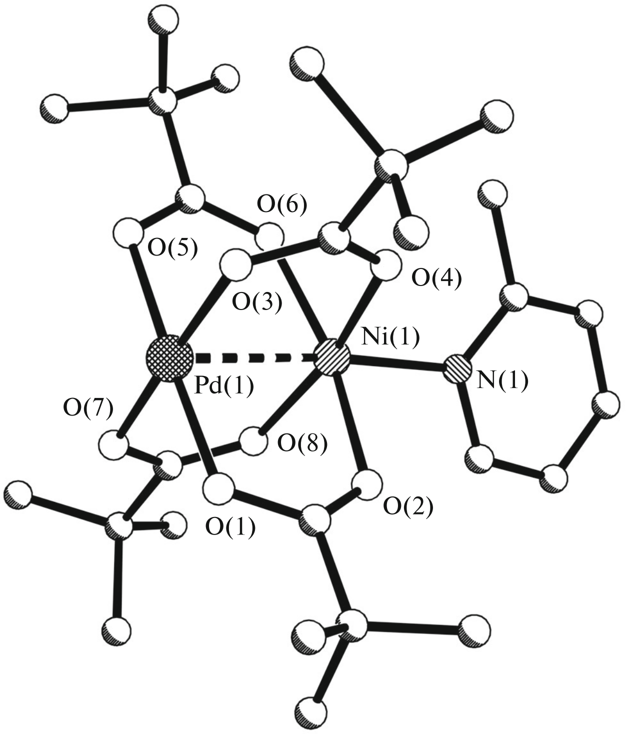

Fig. 2. Molecular structure **III**. For the disordered moieties, only one position is shown. Selected bond lengths and bond angles: Pd(1)–Ni(1), 2.4976(3) Å; Pd(1)–O(7), 1.985(2); Pd(1)–O(1), 1.9930(2; Pd(1)–O(3), 1.997(2) Å; Pd(1)–O(5), 2.002(2); Ni(1)–O(2), 2.030(2); Ni(1)–O(4), 2.038(2); Ni(1)–O(6), 2.045(2); Ni(1)–O(8), 2.0472(2); Ni(1)–N(1*A*), 2.051(12); Ni(1)–N(1), 2.044(3) Å; O(7)Pd(1)O(3), 178.14(7)°; O(1)Pd(1)O(5), 178.01(7)°; O(2)Ni(1)O(6), 166.85(7)°; O(4)Ni(1)O(8), 167.08(7)°.

Table 2. Selected calculated geometrical parameters (Å), the properties of selected bond critical points (au), and the delocalization indices (δ) in complex **III** and Pd(μ-OOCCMe$_3$)$_4$Zn(α-Pic)

Bond	d	ρ_{BCP}	$\Delta^2\rho_{BCP}$	G_{BCP}	V_{BCP}	H_{BCP}	δ
Pd(μ-OOCCMe$_3$)$_4$Ni(α-Pic)							
Pd–Ni	2.491	0.047	0.149	0.045	–0.053	–0.008	0.33
Ni–N	2.017	0.084	0.380	0.116	–0.137	–0.021	0.47
Ni–O	2.054 2.056	0.066	0.337	0.096	–0.107	–0.012	0.35 0.36
Pd–O	2.022	0.103	0.452	0.138	–0.164	–0.025	0.63
Pd(μ-OOCCMe$_3$)$_4$Zn(α-Pic)							
Pd–Zn	2.575	0.040	0.112	0.033	–0.038	–0.005	0.23
Zn–N	2.050	0.079	0.343	0.100	–0.114	–0.014	0.40
Zn–O	2.100 2.120	0.058 0.060	0.283 0.300	0.075 0.079	0.079–0.085	–0.004 –0.005	0.28–0.29
Pd–O	2.024	0.102	0.454	0.138	–0.163	–0.025	0.63

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