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Reactivity of 1,4-Diaza-1,3-Butadienes towards Cu(II) Pivalate: A Rare Case of Polymeric Structure Formed by Bridging Diazabutadiene Ligands

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Abstract—The reaction of copper(II) trimethylacetate, $[Cu(Piv)_2]_n$ (Piv = $(Me)_3CCOO^-$), with 1,4-bis(R)-1,4-diaza-1,3-butadienes (R-DAD, R = 2,4,6-trimethylphenyl, Mes, or 2,6-diisopropylphenyl, dpp) in tetrahydrofuran afforded the coordination polymers $[Cu_2(Piv)_4(Mes\text{-}DAD)]_n$ (**I**) and $[Cu_2(Piv)_4(dpp\text{-}DAD)]_n$ (**II**). The structure of complexes in the crystal was determined by X-ray diffraction (CCDC nos. 2205866, 2205867). The products were shown to be 1D coordination polymers in which the binuclear $\{Cu_2(Piv)_4\}$ moieties are linked into chains owing to the bridging function of 1,4-diaza-1,3-butadienes, which is unusual for this type of ligands.

Keywords: copper(II) complexes, carboxylate complexes, coordination polymers, diazadienes, molecular structure

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INTRODUCTION

Coordination polymers are formed due to linking of metal ions or oligonuclear metal-containing blocks by bridging ligands in one, two, or three directions. By varying the structure-forming characteristics of the ligands, it is possible both to control the dimensionality of coordination polymers (1D, 2D, or 3D) [1–10] and to finely tune the magnetic [11–16], luminescent [17–23], sorption [21, 24, 25], and other properties. Coordination polymers containing transition metal ions and ligands with a branched π-system can be used to design new electroactive materials [26–28]; for example, 1D polymers of copper(II) possess electrical conductivity [29–31].

Bi- and polydentate N-donor organic compounds are used most often as bridging ligands for the formation of coordination polymers. Commercially available 4,4'-bipyridine is one of the most popular linkers [32–34]: more than 6000 coordination polymers containing this ligand are included in the Cambridge Crystallographic Data Centre. Despite the ready availability of γ,γ'-bipyridine and some other polypyridines, the search for new types of bridging ligands is a relevant task.

1,4-Diaza-1,3-butadiene derivatives can be considered as a promising class of bridging ligands. On the one hand, they have conjugated π-bonds and, on the other hand, they offer considerable opportunities for varying substituents at nitrogen. A combination of these factors may enable fine tuning of the donor-acceptor, steric, and redox properties of both the ligands and the coordination compounds they form [35–39]. Metal complexes with redox-active diimine ligands are of interest for the development of new catalytic systems [40–42], activation of small molecules [43, 44], and design of compounds possessing magnetic bistability [45–49]. In the vast majority of cases, 1,4-diaza-1,3-butadienes function as chelating ligands; however, there are rare examples of bridging [50–54] or chelating bridging coordination [55–60] of these ligands.

Previously, we have shown that derivatives of *o*-phenylenediamine, which is a typical chelating ligand, act as bridging ligands when react with copper(II) pivalate [61]. The purpose of this work was to study the reactivity of another class of typical chelating ligands, that is, 1,4-diaza-1,3-butadienes (with bulky mesityl and 2,6-diisopropylphenyl substituents at nitrogen atoms), towards copper(II) pivalate. As a result, we synthesized and studied new copper(II) 1D coordination polymers in which diazabutadiene ligands perform unusual bridging function.

EXPERIMENTAL

The target products were synthesized and isolated in air. Tetrahydrofuran (reagent grade, Khimmed) was used as received. The initial copper(II) trimethylacetate $[Cu(Piv)_2]_n$ was prepared similarly to $[Co(Piv)_2]_n$ [62] from copper(II) acetate monohydrate (reagent grade, Ruskhim) in a pivalic acid melt followed by keeping the product at 110°C in a drying oven for 24 h and in a dynamic vacuum at 120°C for 10 h. The starting 1,4-diaza-1,3-butadiene ligands Mes-DAD and dpp-DAD were prepared from commercially available chemicals by a known procedure [63].

IR spectra were measured in the 400–4000 cm^{-1} range on a Perkin Elmer Spectrum 65 spectrophotometer equipped with a Quest ATR Accessory attachment (Specac) by the attenuated total reflectance (ATR) method. Elemental analysis was performed on a EuroEA-3000 automated C,H,N,S-analyzer (EuroVektor).

The X-ray diffraction data for the crystals of compounds **I** and **II** were collected on a Bruker D8 Venture diffractometer equipped with a CCD detector (MoK_α, λ = 0.71073 Å, graphite monochromator). Semi-empirical absorption correction was applied by the SADABS program [64]. The structures were solved by direct methods and refined by the full-matrix least squares in the anisotropic approximation for non-hydrogen atoms. The calculations were carried out by the SHELX-2014 program package [65] using Olex2 1.2 [66]. The crystallographic parameters for **I** and **II** and the structure refinement details are given in Table 1. Crystallographic data for the structures reported in this paper have been deposited with the Cambridge Crystallographic Data Centre (2205866 and 2205867).

Synthesis of $[Cu_2(Piv)_4(Mes\text{-}DAD)]_n$ (I). Mes-DAD (0.146 g, 0.5 mmol) and THF (30 mL) were added to copper(II) pivalate (0.266 g, 1 mmol). The reaction mixture was heated (70 °C) for 20 min under continuous stirring until the reactants completely dissolved. The color of the reaction mixture was invariably green. Cooling down to room temperature and subsequent concentration (48 h in an open glass beaker) afforded parallelepiped-shaped green crystals. The yield was 0.38 g (92%).

For $C_{40}H_{60}Cu_2N_2O_8$

Anal. calcd., %	C, 58.30	H, 7.34	N, 3.40
Found, %	C, 58.27	H, 7.21	N, 3.37

IR (ATR; ν, cm^{-1}): 3637 w, 2957 s, 2920 w, 2866 w, 1606 vs, 1479 s, 1416 s, 1366 s, 1310 w, 1224 s, 1218 m, 1065 w, 1031 m, 929 w, 852 m, 793 m, 735 w, 684 w, 619 s, 530 w, 502 w, 443 s.

Synthesis of $[Cu_2(Piv)_4(dpp\text{-}DAD)]_n$ (II). Tetrahydrofuran (20 mL) was added to weighed amounts of copper(II) pivalate (0.266 g, 1 mmol) and dpp-DAD (0.188 g, 0.5 mmol). The reaction mixture was heated (70°C) for 20 min under continuous stirring until the reactants completely dissolved. The color of the reaction mixture was invariably green. Cooling down to room temperature and subsequent concentration (48 h in an open glass beaker) afforded parallelepiped-shaped dark green crystals. The yield was 0.260 g (57%).

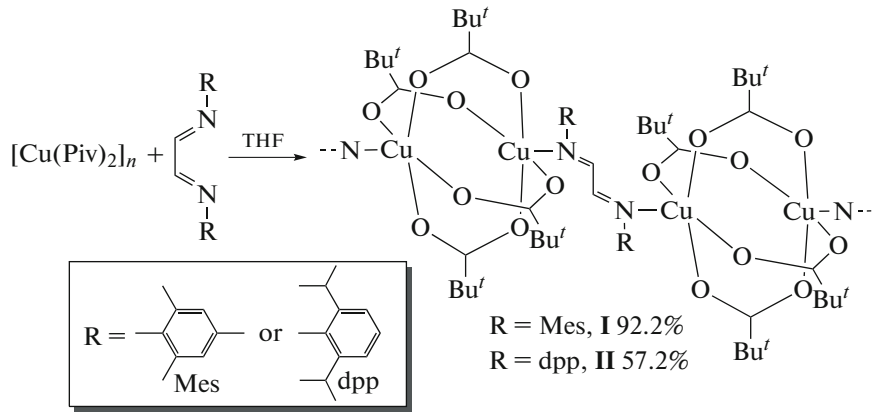
For $C_{46}H_{72}Cu_2N_2O_8$

Anal. calcd., %	C, 60.84	H, 7.99	N, 3.08
Found, %	C, 60.68	H, 7.91	N, 3.04

IR (ATR; ν, cm^{-1}): 3069 w, 2963 m, 2928 w, 2870 w, 1596 vs, 1481 m, 1459 m, 1414 s, 1361 m, 1224 m, 1173 w, 1105 w, 1043 w, 926 w, 895 w, 831 w, 790 m, 762 m, 682 w, 621 s, 520 w, 447 s.

RESULTS AND DISCUSSION

The reaction of copper(II) pivalate with 1,4-diaza-1,3-butadiene ligands Mes-DAD and dpp-DAD in 2 : 1 ratio in THF results in the formation of $[Cu_2(Piv)_4(Mes\text{-}DAD)]_n$ (**I**) and $[Cu_2(Piv)_4(dpp\text{-}DAD)]_n$ (**II**) (Scheme 1).



Scheme 1.

Table 1. Crystallographic data and structure refinement details for complexes **I** and **II**

Compound	**I**	**II**
Chemical formula	$C_{20}H_{30}CuNO_4$	$C_{23}H_{36}CuNO_4$
M_r	411.99	454.07
Crystal system	Monoclinic	Triclinic
Space group	$P2_1/n$	$P\bar{1}$
Temperature, K	100	
a, b, c, Å	9.2413(6) 18.4483(11) 13.1740(8)	9.4487(14) 11.2285(17) 11.8338(17)
α, β, γ, °	109.499(2)	67.392(6) 84.834(5) 85.292(5)
V, Å^3	2117.2(2)	1152.8(3)
Z	4	2
μ, mm^{-1}	1.05	0.97
Crystal size, mm	0.18 × 0.15 × 0.1	0.15 × 0.12 × 0.1
T_{min}, T_{max}	0.315, 0.381	0.331, 0.381
No. of measured reflections	16060	11892
No. of unique reflections	4159	6079
No. of observed [$I > 2\sigma(I)$] reflections	3422	4965
R_{int}	0.037	0.034
$R_1/wR(F^2)$, [$I > 2\sigma$]	0.0445/0.1048	0.0409/0.0855
$R_1/wR(F^2)$, (all data)	0.0575/0.1114	0.0556/0.0913
No. of reflections	4159	6079
No. of parameters	251	272
No. of restraints	36	–
H-atom treatment	H-atom parameters constrained	
$\Delta\rho_{max}$, $\Delta\rho_{min}$, e Å^{-3}	0.84, –0.44	0.77, –0.42

The crystal structure of compounds **I** and **II** is formed by polymer chains consisting of classical copper pivalate "paddlewheels" linked by diimine ligands (Fig. 1). The polymer chains are formed in such a way that metal atoms and diazabutadiene linkers form a planar cage. The carboxylate moieties, being almost perpendicular to each other in both structures (88.13(5)° in **I** and 91.53(4)° in **II**), are arranged in different ways relative to the main plane of the polymer chains; the angles are 24.27(5)° and 64.77(4)° in **I** and 12.69(7)° and 75.62(5)° in **II** (Fig. 2). The distances between the copper atoms within the metal core are 2.6450(8) and 2.6356(7) Å; the distances between the diimine-bridged copper atoms are 6.7722(9) and 6.9286(12) Å in **I** and **II**, respectively.

Since compounds **I** and **II** crystallize in different systems, the packing and relative positions of the chains in the crystal differ (Fig. 3). In the crystals of **II**, the main planes of chains are parallel, while in the crystal of **I**, a more intricate network is formed. Since

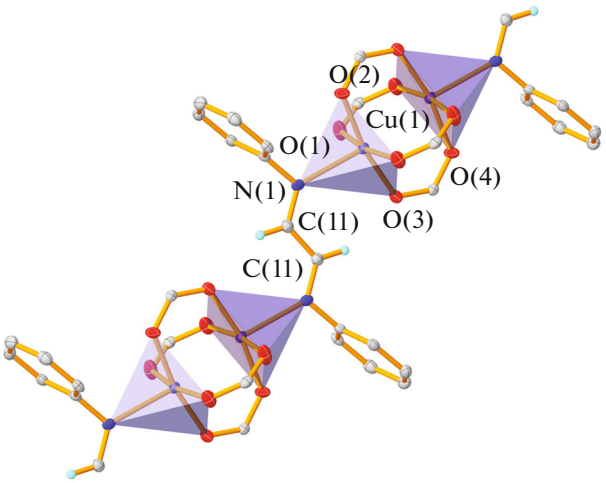


Fig. 1. Molecular structure of compounds **I** and **II** in the crystals exemplified by **II**. The hydrogen atoms and *tert*-butyl and isopropyl groups are omitted for clarity. The ellipsoids are drawn at 50% probability level.

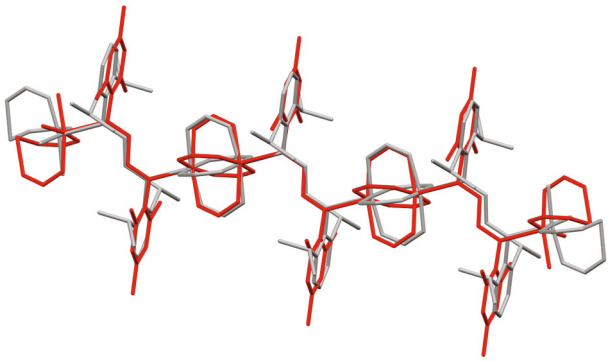


Fig. 2. Structure of 1D polymer chain in compounds **I** (red) and **II** (gray) in the crystals.

all functional groups capable of considerable noncovalent interactions are localized within a polymer chain, all detected CH...O and CH...π type contacts are intramolecular (Tables 2 and 3). The chains in the crystal are connected by weak van der Waals interactions.

It is of interest to analyze the change in the bond length distribution in the diazabutadiene ligands compared with the same ligands in the uncoordinated (free) state. Most information is provided by the distances between the N1–C11 and C11–C11 atoms. In compounds **I** and **II**, the C=N bond lengths are 1.295(4) and 1.290(3) Å, while the C–C bond lengths are 1.440(6) and 1.452(4) Å, respectively. Data on the crystal structures of diazabutadienes in the free state can be found in CCDC [67, 68]. Upon coordination in **I** and **II**, the C=N double bonds of both diimine groups are elongated (in the isolated state, 1.234–1.270 Å for dpp-DAD and 1.265 Å for Mes-DAD), while the C–C single bonds are shortened (1.445–1.471 Å and 1.435 Å for dpp-DAD and Mes-DAD, respectively). Figures 4 and 5 present analysis of data on the corresponding bond lengths in diimine derivatives. The main histogram shows that the C=N bond lengths are generally in the 1.2–1.4 Å range, while the C–C bond lengths are 1.32–1.5 Å; however, the additional histograms a and b, which show data for uncoordinated and chelating diimines, respectively, demonstrate that the coordination is accompanied by a slight elongation of C=N bonds and shortening of the C–C bonds.

Table 2. C-H...π interactions in crystals of **I** and **II**

C–H...π	Symmetry equivalent	H...Cg, Å	H–Perp, Å	Gamma	X–H...C	X...Cg, Å
			$[Cu_2(Piv)_4(Mes–DAD)]_n$ (**I**)			
C3–H3A...Cg1	$1-X, 1-Y, 1-Z$	2.78	2.75	8.30°	176°	3.756(4)
C5–H5A...Cg1	$-X, 1-Y, 1-Z$	2.99	–2.99	3.71°	114°	3.503(3)
			$[Cu_2(Piv)_4(dpp–DAD)]_n$ (**II**)			
C8–H8A...Cg1	$1+X, Y, Z$	2.79	–2.76	8.56°	120°	3.392(2)
C10–H10C...Cg1	X, Y, Z	2.78	2.76	6.32°	159°	3.709(2)

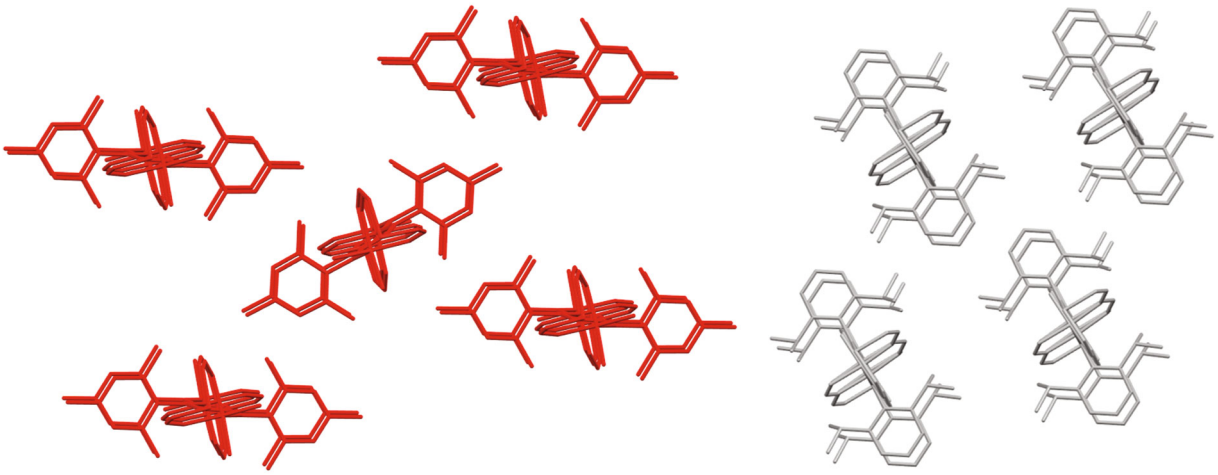


Fig. 3. Packing of molecules in the crystals of **I** (red) and **II** (gray).

The diagrams do not separately show data on the bond lengths in diazabutadienes that occupy a bridging position. Currently, the number of such structures included in CCDC is too low for statistical analysis. However, according to the available data, the C=N bond lengths in these compounds are 1.27–1.32 Å, while the C–C bond lengths are 1.4–1.48 Å, which attests to elongation of the double bond and virtual retention of the single bond. It is yet impossible to draw conclusions about a considerable change in the structure of diimine moieties in such complexes; however, a minor decrease in the degree of electron density delocalization can be assumed.

Thus, we performed one-step synthesis of new 1D coordination polymers in which tetracarboxylate moieties $\{Cu_2(Piv)_4\}$ are connected into a chain by bridging 1,4-diaza-1,3-butadiene ligands. The reaction takes place under mild conditions and gives products in rather high yields. The products are stable on long-term storage under ambient conditions. The presence of bulky substituents at the nitrogen atoms in the N=C–C=N moiety does not prevent implementation of the bridging function unusual for diazabutadiene ligands.

Table 3. H-bonds in crystals of **I** and **II**

H-bond	Symmetry equivalent	D–H, Å	H...A, Å	D...A, Å	D–H...A
		$[Cu_2(Piv)_4(Mes–DAD)]_n$ (**I**)			
C11–H11...O1	$-x, 1-y, 1-z$	0.95	2.39	3.250(4)	150°
C18–H18A...O4	$1-x, 1-y, 1-z$	0.98	2.53	3.460(5)	159°
C20–H20A...O3		0.98	2.57	3.436(4)	148°
		$[Cu_2(Piv)_4(dpp–DAD)]_n$ (**II**)			
C11–H11...O4	$-1+x, y, z$	0.95	2.40	3.280(2)	154°
C18–H18...N1		1.00	2.52	2.882(3)	101°
C18–H18...O2	$1-x, 1-y, 1-z$	1.00	2.55	3.496(3)	158'
C21–H21...O1		1.00	2.56	3.542(3)	167°
C21–H21...N1		1.00	2.52	2.896(3)	102'

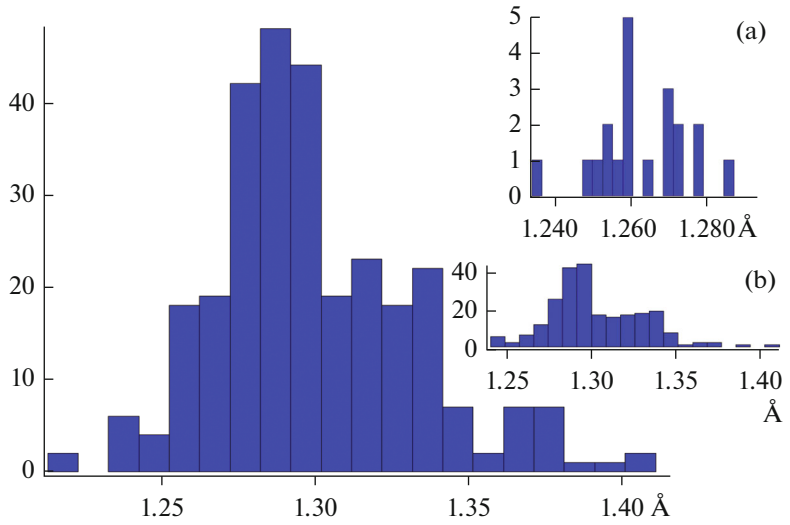


Fig. 4. Histogram of C=N bond length distribution in diimines according to CCDC data: (a) uncoordinated and (b) chelating diimines.

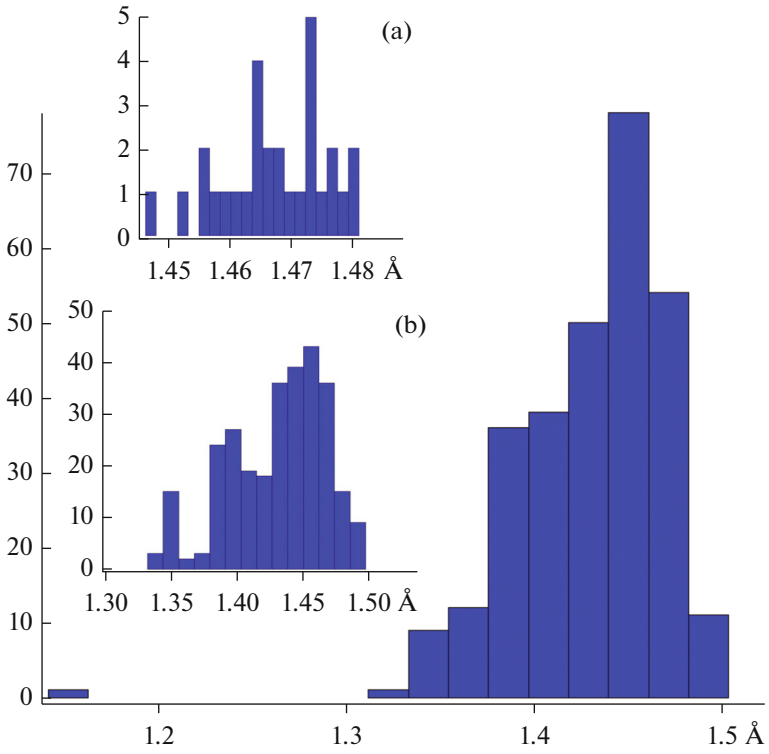


Fig. 5. Histogram of C–C bond length distribution in diimines according to CCDC data (a) uncoordinated and (b) chelating diimines.

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CONFLICT OF INTEREST

The authors declare that they have no conflicts of interest.

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