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# Mononuclear Pivalates of Metals of the First Transition Series

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**Abstract**—The synthesis and structures of mononuclear Ni(II), Co(II), Mn(II), and Cu(II) pivalates isolated as complex salts $NBu_4[M(Piv)_3]$ ($(NBu_4)^+$ is tetrabutylammonium cation, $Piv^-$ is pivalate anion) and polynuclear complexes $[Ni_6(L)_2(HL)_2(Piv)_6(HPiv)_8]$, $(NBu_4)_2[Co_4(Piv)_8(AcO)_2(H_2O)_4]$, $NBu_4[Co_2(Piv)_5(H_2O)_2]$, and $(NBu_4)_2[Cu_4(Piv)_8(AcO)_2(H_2O)_2]$ ($L^{2-}$, $HL^-$, and $AcO^-$ is lactic acid dianion, lactic acid monoanion, and acetate anion, respectively) are discussed. The formation of the compounds is detected during the development of the synthesis of $NBu_4[M(Piv)_3]$.

*Keywords*: complexes, mononuclear compounds, polynuclear compounds, pyvalates, cobalt, nickel, copper, manganese

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Transition metal complexes with pivalates as ligands providing a good solubility in organic solvents are actively used as components of the catalytic systems for hydrocarbon oxidation [1, 2], matrices for metal transport on the support surface [3, 4], models of metal-containing proteins [5, 6], and magnetically active blocks for assembling molecular magnetics [7–12]. Therefore, easily available transition metal pivalates are valuable initial compounds. For instance, binuclear pivalates of Cu(II) [13–15], Ni(II), and Co(II) [16] and hexanuclear Mn(II,III) pivalate [17, 18] are widely used in synthesis practice. The fact that, as a rule, the initial Cu(II) carboxylates are binuclear compounds with the “Chinese lantern” structure has long ago become a commonly accepted point of view described in textbooks [19–21]. Mononuclear transition metal complexes in which carboxylates perform only the chelate function resulting in the formation of tetranuclear metallocycles are very rare. The structures of more than two thousands of various transition metal compounds containing coordinated pivalates are presented at the Cambridge Crystallographic Data Centre (CCDC) [22]. Only in one of them, oxovanadium(V) tris(pivalate) $[VO(Piv)_3]$ [23], the coordination sphere of the transition metal contains pivalate anions performing exclusively the function of chelating ligands, although oxygen forming the oxo cation $VO^{3+}$ is also observed in the coordination sphere of vanadium(V) along with coordinated pivalates.

We have recently discovered an approach to synthesis that allowed us to obtain a whole series of mononuclear transition metal pivalates $NBu_4[M(Piv)_3]$, where $(NBu_4)^+$ is the tetrabutylammonium cation, $Piv^-$ is the pivalate anion, and M = Ni(II), Co(II), Mn(II), and Cu(II) [24]. Concentrated aqueous solutions of $MCl_2$ and KPiv are introduced into the reaction to obtain mononuclear pivalates. The reaction is rather unusual. The product suspended in the whole bulk is rapidly accumulated upon the mixing of the solutions, and the reaction mixture solidifies almost completely. Then the powder formed begins to spontaneously eliminate water to form a two-phase system consisting of a finely dipersed precipitate and a transparent layer of the mother liquor above the precipitate. Hexane, ethyl acetate, and an aqueous solution of $NBu_4OH$ are added. After this, the reaction mixture is stirred and an organic layer is separated. Crystals of $NBu_4[M(Piv)_3]$ are isolated from the organic layer upon storage for several days. The crystals are yellow in the case of the Ni(II) complex, they are lilac for the Co(II) complex, the Mn(II) crystals are pink, and those of Cu(II) are blue [25].

The crystals of complex salts $NBu_4[M(Piv)_3]$ (M = Mn, Co, and Ni) are isostructural. The fragment of structure $NBu_4[Mn(Piv)_3]$, whose crystals are formed by alternating layers of the $(NBu_4)^+$ cations and $[M(Piv)_3]^-$ anions, is shown as an example in Fig. 1. The values of M–O bonds lengths and chelate angles for $NBu_4[M(Piv)_3]$ are given in the table.

The study of structure $NBu_4[Co(Piv)_3]$ at 240 K for $[Co(Piv)_3]$ revealed the disordering of two of three carboxylate groups over two positions in the 0.7/0.3 ratio. The disordering disappears on cooling the crys-

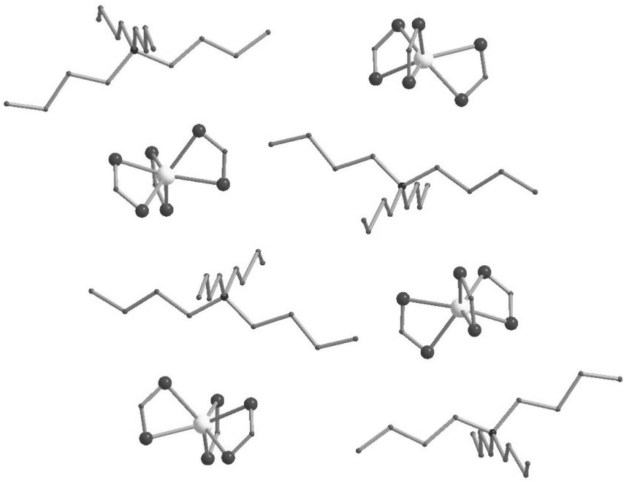

**Fig. 1.** Fragment of structure $NBu_4[Mn(Piv)_3]$. Hereinafter, hydrogen atoms and *tert*-Bu groups are omitted.

tal to 85 K (table). There is no disordering of positions of the COO groups at 240 K for $NBu_4[Mn(Piv)_3]$ and $NBu_4[Ni(Piv)_3]$. In $NBu_4[Mn(Piv)_3]$ the Mn–O distances are longer than those in the Ni and Co complexes and, hence, the chelate angles are smaller (table). The environment of the central atom in mononuclear $NBu_4[M(Piv)_3]$, especially for M = Mn, is closer in geometry to a trigonal prism than to an octahedron (Fig. 2a). If the metal atom is linked with the atoms of the carboxyl groups, a figure resembling a distorted propeller screw is formed.

In the course of X-ray diffraction analysis of $NBu_4[Cu(Piv)_3]$ at room temperature, 12 oxygen atoms were localized in the environment of the metal; i.e., the disordering of all carboxyl groups was observed. The Cu–O distances were approximately the same and equal to 2.17 Å. In the crystal cooled to 85 K, only two of three carboxylates (ratio 0.85/0.15) remained disordered. In this case, the environment of the Cu atom could be considered as close to a distorted square formed by the O atoms of one Piv coordinated through the bidentate-cyclic coordination mode and two monodentately coordinated Piv (Fig. 2b, table). However, taking into account that the Cu–O distances to two most remote O atoms are 2.439(1) and 2.568(3) Å and taking into account specific features characteristic of the Jahn–Teller distortion in the trigonal prism for the central atom with the electronic configuration $d^9$ [26], it is more correct to consider the environment of the Cu(II) atom in $[Cu(Piv)_3]^-$ and in other $[M(Piv)_3]^-$ as close to a distorted trigonal prism.

It could be assumed that the use of a three-charged metal in the synthesis of $NBu_4[M(Piv)_3]$ would result in electroneutral tris(chelate) $[M(Piv)_3]$. In order to check the assumption, we introduced the $In^{3+}$ cation into the reaction. However, when using $InCl_3$ in the reaction, $NBu_4[In(Piv)_4]$ was always isolated in a high yield. The structure of $NBu_4[In(Piv)_4]$ is presented in Fig. 3. In this structure, the In and N atoms lie on the $C_2$ axis, due to which the alternating $(NBu_4)^+$ and $[In(Piv)_4]^-$ ions form columns (Fig. 3a). The coordination polyhedron of the In atom is a distorted eight-vertex figure in which four In–O distances are noticeably shorter than other contacts (Fig. 3b).

All compounds $NBu_4[M(Piv)_3]$, where M = Ni(II), Co(II), Mn(II), and Cu(II), were reproductively formed when the initial M/Piv ratio was varied from 1/2 to 1/3, indicating in favor of their individual character. However, in several cases, crystals of impurity compounds were formed along with the maximum volume of $NBu_4[M(Piv)_3]$ crystals if the synthesis of the compounds was repeated in the solid phase. In the case of M = Ni, an unusual process was initiated affording hexanuclear complex $[Ni_6(L)_2(HL)_2(Piv)_6(HPiv)_8]$, where L and HL are the di- and monoanion of lactic acid, respectively [23]. The formation of the compound with lactate is a nontrivial result, since $[Ni_6(L)_2(HL)_2(Piv)_6(HPiv)_8]$ is the first hexanuclear transition metal complex, whose ligand shell contains lactate anions [21], each of which

Values of M–O bonds and chelate angles for $NBu_4[M(Piv)_3]$ and $NBu_4[In(Piv)_4]$

| Anion | *T*, K | M–O, Å | Angle OMO, deg | CCDC no.* |
|---|---|---|---|---|
| $[Mn(Piv)_3]^-$ | 240 | 2.117(9)–2.307(11) | 56.7(4), 58.9(4), 58.30(8) | 976097 |
| $[Co(Piv)_3]^-$ | 85 | 2.105(2)–2.147(2) | 61.77(9), 61.98(8), 62.09(9) | 976095 |
| $[Ni(Piv)_3]^-$ | 240 | 2.045(4)–2.108(4) | 60.7(1), 61.5(1), 62.6(2) | 940203 |
| $[Cu(Piv)_3]^-$ | 85 | 1.934(2) 1.942(3)<br>2.047(1) 2.028(1)<br>2.439(1) 2.568(3) | 56.8(1), 59.33(5), 64.59(2) | 976098 |
| $[In(Piv)_4]^-$ | 295 | 2.230(3)<br>2.345(3), 2.348(3) | 56.70(9), 56.36(9) | 1446783 |

* Structure number in the CCDC (http://www.ccdc.cam.ac.uk/).

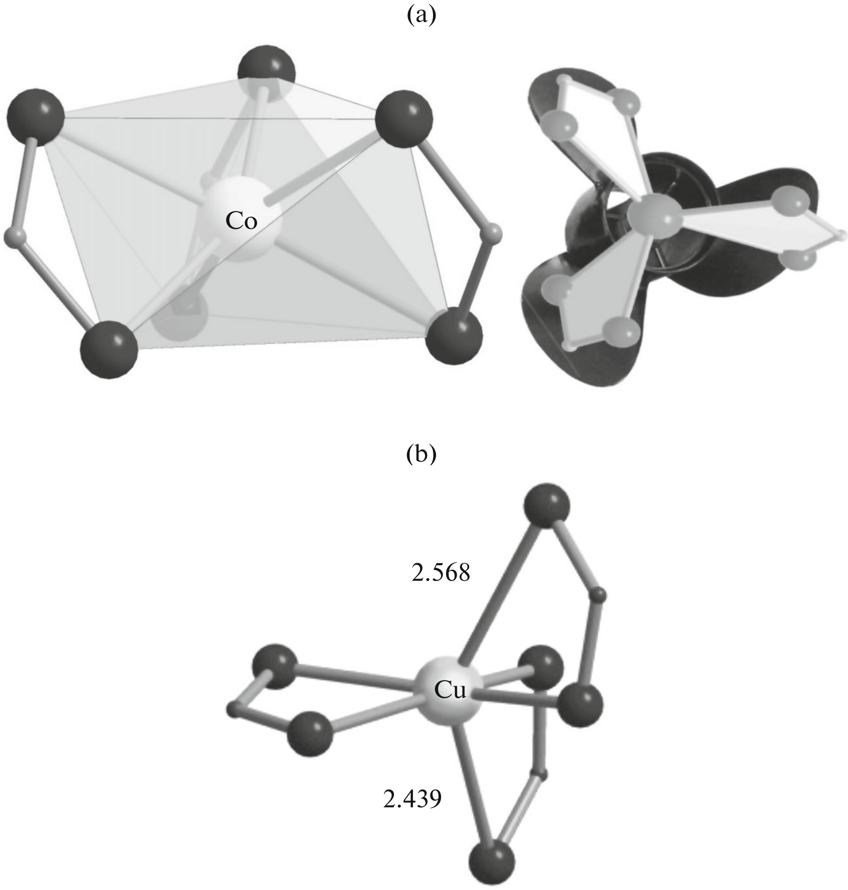

**Fig. 2.** Environment of the (a) Co atom in the $[Co(Piv)_3]^-$ anion and (b) Cu atom in the $[Cu(Piv)_3]^-$ anion.

is coordinated to three $Ni^{2+}$ ions and performs the $\mu_4$-bridging function. Two of the lactate ions participate along with pivalates in the formation of trinuclear fragments and two other lactate ions cross-link these fragments into the 16-membered metallocycle (Fig. 4).

Lactic acid is an important product of carbohydrate degradation in biological systems [5]. However, its formation from fairly simple chemical precursors including $NBu_4[Ni(Piv)_3]$, Ni(II) chloride, and organic solvents represents a result that could hardly be assumed beforehand, although the case of formation of lactic acid or metal lactates from methylglyoxal, 1,3-dihydroxyacetone, or glyceraldehyde in the presence of the chromium [27], rhodium [28], or copper complexes [29, 30] are known. The formation of lactic acid from the indicated precursors is an admissible process, although it is not simple. Rather complicated mechanism of coordinated lactate formation from methylglyoxal was proposed for this process [27, 28]. In the case of $[Ni_6(L)_2(HL)_2(Piv)_6(HPiv)_8]$, even if assuming that lactate is formed from one or several mentioned precursors, the latter should preliminarily be obtained from acetone introduced into the reaction mixture [23]. Note that attempts to obtain $[Ni_6(L)_2(HL)_2(Piv)_6(HPiv)_8]$ in the absence of acetone in the reaction mixture was unsuccessful. Methanol should be introduced into the reaction mixture along with acetone, since the hexanuclear complex cannot either be isolated without methanol. The latter seems important, because polynuclear Ni(II) pivalates often contain bridging methoxy groups [31–34]. They can provoke proton substitution in the methyl group of acetone by hydroxy or methoxy groups followed by the transformation of the products into one of the mentioned intermediates. The reaction mixture also needs the presence of $NBu_4[Ni(Piv)_3]$: $[Ni_6(L)_2(HL)_2(Piv)_6(HPiv)_8]$ was not formed in the presence of only nickel chloride, methanol, and acetone [23].

The formation of single crystals of tetranuclear complexes $(NBu_4)_2[Co_4(Piv)_8(AcO)_2(H_2O)_4]$ and $(NBu_4)_2[Cu_4(Piv)_8(AcO)_2(H_2O)_2]$ ($AcO^-$ is the acetate anion) is detected in several cases for the multiple repetition of the synthesis procedure for $NBu_4[Co(Piv)_3]$ and $NBu_4[Cu(Piv)_3]$. The structures of complex anions $[Co_4(Piv)_8(AcO)_2(H_2O)_4]$ and $[Cu_4(Piv)_8(AcO)_2(H_2O)_2]$ are presented in Fig. 5.

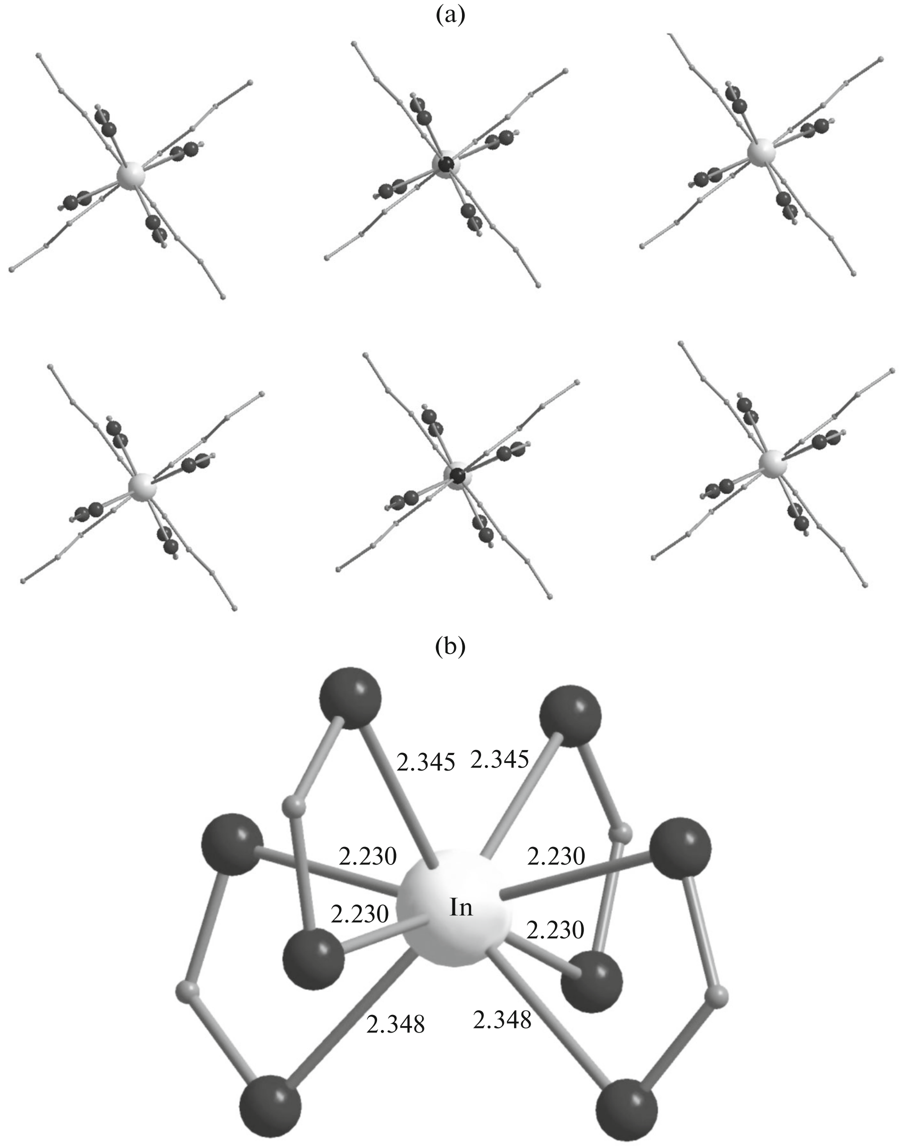

**Fig. 3.** (a) Fragment of structure $NBu_4[In(Piv)_4]$ and (b) the environment of the In atom in the anion.

In $[Co_4(Piv)_8(AcO)_2(H_2O)_4]^{2-}$, the pivalate ions and water molecules act as bridges between the terminal and internal Co atoms, and two O atoms of two acetate ions act as bridges between two internal Co atoms (Fig. 5a). In $[Cu_4(Piv)_8(AcO)_2(H_2O)_2]^{2-}$, the central fragment is a classical binuclear lantern $\{Cu_2(Piv)_4\}$ (Fig. 5b) linked to the terminal Cu atoms by the bidentate-bridging acetate ions. The water molecules in the complex anions form intramolecular hydrogen bonds with the O atoms of the carboxylate ligands. The coordination polyhedron of all Co atoms is a distorted octahedron, and that of the Cu atoms is a square pyramid. The ranges of the distances are as follows: 2.030(5)–2.187(4) Å for Co–O, 1.921(3)–1.986(2) Å for equatorial Cu–O, 2.144(2)–2.154(2) Å for the axial bonds at the Cu atoms forming the lantern, and 2.443(4)–2.463(4) Å for the axial bonds of the terminal Cu atoms.

Tetranuclear $(NBu_4)_2[Co_4(Piv)_8(AcO)_2(H_2O)_4]$ and $(NBu_4)_2[Cu_4(Piv)_8(AcO)_2(H_2O)_2]$ were not known earlier. This implies that mononuclear $(NBu_4)[M(Piv)_3]$ can be used for the synthesis of polynuclear compounds. It can also be assumed that a certain stress of the four-membered metallocycles in the coordination anions $[M(Piv)_3]$ can potentially be a reason for the enhanced reactivity of similar mononuclear forms and, hence, a reason for their absence in the CCDC. As it turned out, a similar assumption is invalid. The experiments showed that $NBu_4[M(Piv)_3]$, where M = Ni, Mn, Co, and Cu, were inert in the

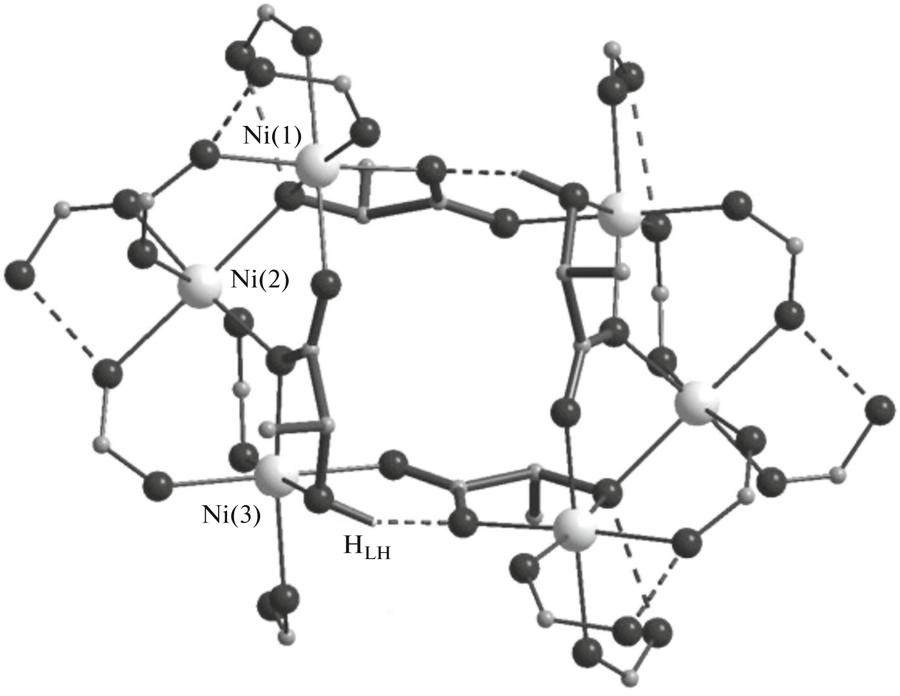

**Fig. 4.** Structure of molecule $[Ni_6(L)_2(HL)_2(Piv)_6(HPiv)_8]$; thin lines show the Ni–O and C–O bonds in the carboxy groups of the pivalate ions, solid lines show bonds in the lactate ions, and dashed lines are hydrogen bonds.

reactions with other compounds of metals of the first transition series [24]. As mentioned above, $NBu_4[Ni(Piv)_3]$ was introduced into the reaction with the Ni(II) salts only by rather complicated manipulations in various organic media. This gave hexanuclear $[Ni_6(L)_2(HL)_2(Piv)_6(HPiv)_8]$ in a yield of ~10–15% [23]. Numerous attempts to obtain $(NBu_4)_2[Co_4(Piv)_8(AcO)_2(H_2O)_4]$ by the reactions of $NBu_4[CoPiv_3]$ with different Co(II) salts (including those with the addition of acetate to the reaction mixture) failed. On the one hand, this indicates in favor of the inertness of mononuclear $NBu_4[Co(Piv)_3]$ and, on the other hand, shows that the formation of impurity crystals of $(NBu_4)_2[Co_4(Piv)_8(AcO)_2(H_2O)_4]$ detected for the development of the synthesis of $NBu_4[Co(Piv)_3]$ is associated with processes occurring in the aqueous phase.

Since the synthesis of $NBu_4[Co(Piv)_3]$ assumes the mixing of aqueous solutions of Co(II) chloride and KPiv followed by the treatment of the mixture with hexane, ethyl acetate, and $NBu_4OH$, it seemed evident that the formation of $(NBu_4)_2[Co_4(Piv)_8(AcO)_2(H_2O)_4]$ in the extract is a consequence of the alkaline hydrolysis of ethyl acetate during the synthesis of the complex, because there is no other source of acetate ions in the reaction mixture [24]. The study of the aqueous phase remained after the extraction of the major part of cobalt during the synthesis of $NBu_4[Co(Piv)_3]$ shows that $NBu_4[Co_2(Piv)_5(H_2O)_2]$ can be isolated from this mixture. If this product is introduced into the reaction with $(NBu_4)AcO \cdot AcOH$, tetranuclear $(NBu_4)_2[Co_4Piv_8(AcO)_2(H_2O)_4]$ can be obtained in a yield higher than 50%. The reaction equation is given below

$$2NBu_4[Co_2(Piv)_5(H_2O)_2] + (NBu_4)AcO \cdot AcOH$$
$$= (NBu_4)_2[Co_4Piv_8(AcO)_2(H_2O)_4] + NBu_4Piv + HPiv.$$

The structure of the $[Co_2(Piv)_5(H_2O)_2]^-$ coordination anion is shown in Fig. 6. In each binuclear anion, the Co atoms have different environments: octahedral and close to trigonal pyramid. In the solid phase, the binuclear anions are joined into a tetranuclear molecule by hydrogen bonds. The formation of the latter involves all hydrogen atoms of the water molecules: the bridging molecule containing the O(1*w*) atom is bound to monodentately coordinated Piv, and water containing the O(2*w*) atom links two coordination anions into one molecule.

Thus, we succeeded to reveal reasons for the formation of tetranuclear compound $(NBu_4)_2$-$[Co_4(Piv)_8(AcO)_2(H_2O)_4]$ accompanying the synthesis of $NBu_4[Co(Piv)_3]$. The formation of crystals of the tetranuclear complex containing simultaneously coordinated anions of pivalic and acetic acids: a consequence of the presence of $NBu_4[Co_2(Piv)_5(H_2O)_2]$ and tetrabutylammonium acetate, which is formed due to the alkaline hydrolysis of ethyl

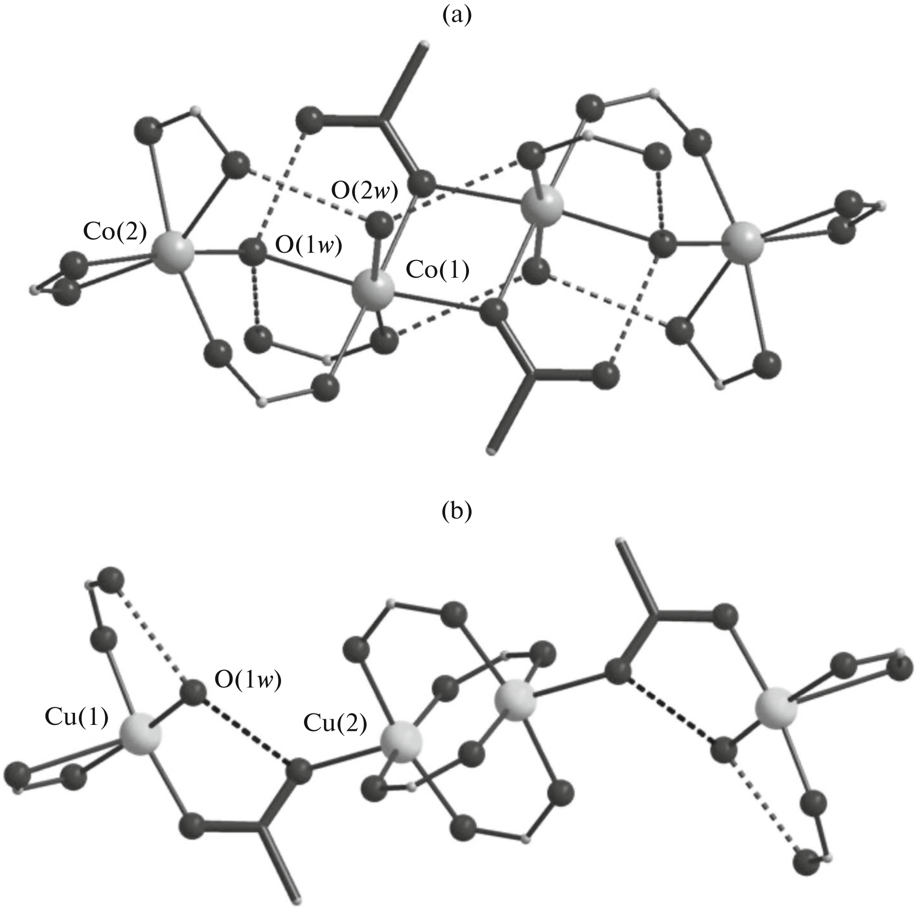

**Fig. 5.** Structures of complex anions (a) $(NBu_4)_2[Co_4(Piv)_8(AcO)_2(H_2O)_4]$ and (b) $(NBu_4)_2[Cu_4(Piv)_8(AcO)_2(H_2O)_2]$. Thin lines show the M–O and C–O bonds in the carboxy groups of the pivalate ions, solid lines are bonds in the acetate ions, and dashed lines are hydrogen bonds of the coordinated water molecules.

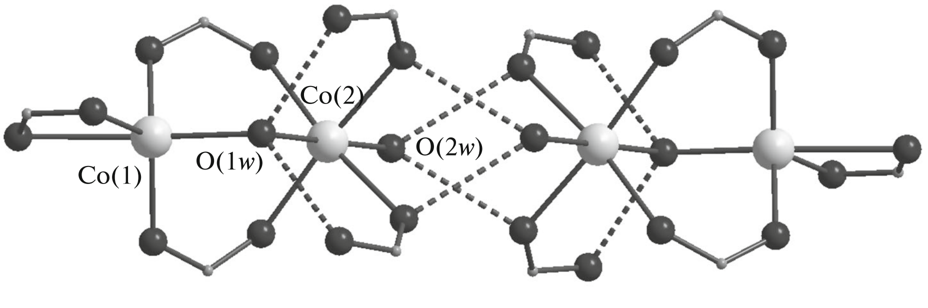

**Fig. 6.** Hydrogen bonding of two coordination anions $[Co_2(Piv)_5(H_2O)_2]$ into a tetranuclear molecule in crystals of $NBu_4[Co_2(Piv)_5(H_2O)_2]$.

acetate, in the aqueous phase. Based on this, we developed the method for the purposeful synthesis of $(NBu_4)_2[Co_4(Piv)_8(AcO)_2(H_2O)_4]$. We failed to find necessary conditions for a similar synthesis of $(NBu_4)_2[Cu_4(Piv)_8(AcO)_2(H_2O)_2]$, although there is no doubt that the reason for the formation of acetate in the coordination sphere of $(NBu_4)_2[Cu_4(Piv)_8(AcO)_2(H_2O)_2]$ is the same as that for $(NBu_4)_2[Co_4(Piv)_8(AcO)_2(H_2O)_4]$, namely, the hydrolysis of ethyl acetate [25].

The studies performed made it possible to discover the general methodical approach to the synthesis of mononuclear $NBu_4[M(Piv)_3]$ (M = Mn, Co, Ni, and Cu, being metals of the first transition series). It is found using $NBu_4[In(Piv)_4]$ as an example that the introduction into the reaction of the metal atom with larger charge and radius than those of the used metals of the first transition series also allows one to obtain the mononuclear complex, whose coordination sphere contains only carboxylate ligands performing exclusively the chelate function resulting in the formation of four-membered metallocycles.

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