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Ligand Metathesis in Nickel(II) Complexation with *closo*-Decaborate Anion

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Abstract—Nickel(II) complexation with the *closo*-decaborate anion in water and acetonitrile is studied. Complexes $[Ni(solv)_6][B_{10}H_{10}]$ (solv = H_2O (**I**) or CH_3CN (**II**)) are isolated. The complexes are characterized by elemental analysis and IR spectroscopy. Complex $[Ni(CH_3CN)_5(H_2O)]_{0.75}[Ni(CH_3CN)_4(H_2O)_2]_{0.25}[B_{10}H_{10}]\cdot 0.5H_2O$ (**III**) is isolated from an acetonitrile–water system. The structure of complex **III** is solved by X-ray diffraction (XRD) (CIF file CCDC no. 2224702). A mechanism of ligand metathesis in the complexation of nickel(II) is proposed.

Keywords: boron cluster anions, XRD, aqua complexes, acetonitrile

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INTRODUCTION

Boron cluster anions $[B_nH_n]^{2-}$ (n = 6–12) [1–4] attract attention of researchers owing to their structures and properties. The three-dimensional aromaticity of these systems [5–8] explains their high chemical and thermal stability, which predetermines areas of their practical application [9–16]. Boron cluster anions can be involved in the substitution of *exo*-polyhedral hydrogen atoms by various functional groups to form many substituted derivatives [17–22]. At the same time, they can serve as ligands in the complexation of metals–soft acids (according to Pearson's classification) [23–25].

The boron cluster anions interact with metals M(II) assigned to acids of Pearson's intermediate group to form mainly cation-anionic compounds [26–28]. As a rule, the formed compounds consist of the cationic metal complex with ligands $[ML_x]^{2+}$ and boron cluster anions as counterions.

A new trend in materials science related to the synthesis of metal borides using complexes with boron cluster anions started an active development in the recent time. The energy capacity of boron cluster anions makes it possible to decrease the synthesis temperature of borides facilitating the synthesis and decreasing power expenses. In particular, the thermal treatment of a number of the nickel and cobalt complexes resulted in the synthesis of boron–oxide, boron–nitride, and boride phases of various compositions [29–31]. A possibility of preparing solid solutions $Ni_3C_{1-x}B_x$ by the thermal treatment of the nickel complex $[Ni(DMF)_6][B_{10}H_{10}]$ at 600°C was found [30].

Several nickel(II) complexes with organic ligands and boron cluster anions as counterions were described. The following complexes were synthesized: nickel aqua complex $[Ni(H_2O)_6][B_{12}H_{12}]\cdot 6H_2O$ and mixed-cationic aqua complex $[Ni(H_2O)_6](H_3O)_2[B_{12}H_{12}]_2\cdot 6H_2O$ [32, 33], tris(chelate) complexes with organic ligands $[NiL_3][B_nH_n]$ (n = 10, 12) (L is benzoylhydrazine, *meta*-nitrobenzoylhydrazine [34]; 2,2'-bipyridyl (Bipy), 1,10-phenanthroline (Phen) [35–38]; ethylenediamine [39]; and benzimidazole derivatives [40, 41]), complexes with solvent molecules as ligands $[Ni(solv)_6][B_{10}H_{10}]$ (L = DMF, DMSO [30, 42, 43]), and heteroligand nickel(II) complexes $[NiL_2(H_2O)_2][B_nH_n]$ (n = 10, 12; L is Bipy or 1,2-diaminobenzene (DAB)) [36]. The tris(chelate) nickel(II) complexes were described for the OH-substituted derivative of the *closo*-decaborate anion $[NiL_3][B_{10}H_9OH]$ (L = Bipy, Phen, and 2,2'-bipyridylamine (BPA) or DAB) [44] and dimeric cluster $[NiL_3][B_{20}H_{18}]$ (L = Bipy, Phen) [45]. Complexes $[Ni(solv)_6][B_{10}Cl_{10}]$ (L = CH_3CN, DMF, DMSO) [46], $[NiL_3][B_{10}Cl_{10}]$ (L = Bipy, Phen) [46] and $[Ni(CH_3CN)_6][B_{12}Hal_{12}]$ (Hal = Cl, Br) [47] were isolated for the perhalogenated boron cluster anions. The structures of the majority of the described compounds were solved by XRD.

According to the data of physicochemical analyses, including XRD, the complex nickel(II) cations $[NiL_3]^{2+}$ or $[NiL_2(H_2O)_2]^{2+}$ are octahedral and the $[An]^{2-}$ cluster anions are located in the external coordination sphere in all indicated compounds. No nickel(II) complexes with the metal(II) environment different from the octahedral one were described.

Note that the nickel(II) complex isomers $[Ni(En)[2\text{-}B_{10}H_9O(CH_2)_2O(CH_2)_2NH(CH_2)_2NH_2)]]\cdot H_2O$ and $[Ni(H_2O)(En)[2\text{-}B_{10}H_9O(CH_2)_2O(CH_2)_2NH(CH_2)_2NH_2)]]$ with the coordinated substituted derivative of the boron cluster anion and different arrangements of the water molecule (coordinated and solvate) were isolated for the nickel(II) complex with ethylenediamine and substituted derivative of the *closo*-decaborate anion with the pendant NH_2 group separated from the boron cluster by the alkoxy spacer [48]. The complexes differ by the denticity of the monosubstituted derivative: the $[2\text{-}B_{10}H_9O(CH_2)_2O(CH_2)_2NH(CH_2)_2NH_2)]^{2-}$ anion acts as the tetradentate and tridentate ligand, respectively.

The complexation of nickel(II) with the *closo*-decaborate anion in water, acetonitrile, and an acetonitrile–water system was studied in this work. The mechanism of ligand metathesis in the reaction solution was proposed.

EXPERIMENTAL

Nickel oxide (99%, Aldrich), nickel(II) carbonate (98%, Khimmed), and acetonitrile (HPLC, Aldrich) were used as received. Triethylammonium decahydro-*closo*-decaborate $(Et_3NH)_2[B_{10}H_{10}]$ was synthesized from decaborane-14 via the step of 6,9-bis(triethylamine)decaborane formation according to a described procedure [49]. Potassium *closo*-decaborate was synthesized by refluxing $(Et_3NH)_2[B_{10}H_{10}]$ in an aqueous solution of KOH until the complete removal of triethylamine. Acid $(H_3O)_2[B_{10}H_{10}]$ (20%) was synthesized by the cation exchange on the KU-2 strong-acid cation-exchange resin.

Synthesis of $[Ni(H_2O)_6][B_{10}H_{10}]$ (I). A solution of nickel(II) oxide or carbonate (3 mmol) in water (10 mL) was added to a solution of acid $(H_3O)_2[B_{10}H_{10}]$ (3 mmol) in water (10 mL). The formation of a polycrystalline precipitate colored as green apple was observed after several days. The precipitate was filtered off and dried in air. The yield of compound **I** was 76%.

For $H_{22}O_6B_{10}Ni$

Anal. calcd., %	H, 7.78	B, 37.94	Ni, 20.60
Found, %	H, 7.71	B, 37.66	Ni, 20.72

IR (NaCl; ν, cm^{-1}): 3500–3200 ν(OH); 2493, 2447 ν(BH); 1648 δ(HOH); 1040 δ(BBH).

Synthesis of $[Ni(CH_3CN)_6][B_{10}H_{10}]$ (II). Solid nickel(II) chloride (3 mmol) was added with stirring to a solution of salt $(Et_3NH)_2[B_{10}H_{10}]$ (3 mmol) in acetonitrile (10 mL). The gradual dissolution of metal chloride with the formation of a sea-green solution and the gradual formation of a blue-colored polycrystalline precipitate were observed. The precipitate was filtered off and dried in air. The yield of compound **II** was 89%.

For $C_{12}H_{28}N_6B_{10}Ni$

Anal. calcd., %	C, 34.06	H, 6.67	N, 19.86	B, 25.55	Ni, 13.87
Found, %	C, 33.88	H, 6.54	N, 19.79	B, 25.46	Ni, 13.83

IR (NaCl; ν, cm^{-1}): 2532, 2440 ν(BH); 2326, 2291 ν(CN); 1015 δ(BBH).

Synthesis of $[Ni(CH_3CN)_5(H_2O)]_{0.75}[Ni(CH_3CN)_4(H_2O)_2]_{0.25}[B_{10}H_{10}]\cdot 0.5H_2O$ (III·0.5H$_2$O). Solid nickel(II) chloride (3 mmol) was added with stirring to a solution of salt $(Et_3NH)_2[B_{10}H_{10}]$ (3 mmol) in acetonitrile (10 mL). The gradual dissolution of metal chloride with the formation of a sea-green solution and the gradual formation of a blue polycrystalline precipitate were observed. Water (2 mL) was added to the formed mixture, which resulted in the instant dissolution of the precipitate and formation of a transparent bright blue solution. Blue crystals suitable for XRD precipitated from the reaction solution in a day. The crystals were filtered off and dried in air. The yield of compound **III**·0.5H$_2$O was 69%.

For $C_{38}H_{107}N_{19}O_5B_{40}Ni_4$

Anal. calcd., %	C, 28.93	H, 6.84	N, 16.87	B, 27.41	Ni, 14.88
Found, %	C, 28.89	H, 6.75	N, 16.79	B, 26.92	Ni, 14.69

IR (NaCl; ν, cm^{-1}): 3499, 3435 ν(OH); 2480 ν(BH); 2327, 2289 ν(CN); 1612 δ(HOH); 1017 δ(BBH).

Elemental analysis was carried out on a CHNS-3 FA 1108 Elemental Analyser automated gas analyzer (Carlo Erba). The boron and nickel contents were determined by the ICP MS method on an iCAP 6300 Duo atomic emission spectrometer with inductively coupled plasma. The samples for analyses were dried to a constant weight.

The IR spectra of the starting compounds and thermolysis products were recorded on an Infralyum FT-02 FT-IR spectrophotometer (Lumex, Russia) as suspensions in Nujol (Aldrich) on NaCl plates in an absorption range of 4000–400 cm^{-1} with a resolution of 1 cm^{-1}.

X-ray diffraction analysis on a Bruker D8 Advance diffractometer (CuK_α radiation) was carried out in low-background cells with the support of oriented silicon single crystal in a range of 2θ 5°–80° with an increment of 0.01125°.

XRD. A set of diffraction reflections for a crystal of compound **III** was obtained at the Center for Collective Use of the Kurnakov Institute of General and Inorganic Chemistry (Russian Academy of Sciences) on a Bruker SMART APEX2 automated diffractometer (MoK_α radiation, $\lambda = 0.71073$ Å, graphite monochromator, ω–ϕ scan mode). The data were indexed

Table 1. Crystallographic data, experimental parameters, and structure refinement characteristics for compound **III**$\cdot 0.5H_2O$

Parameter	Value
Empirical formula	$C_{9.5}H_{27.75}B_{10}N_{4.75}O_{1.75}Ni$
FW	403.42
T, K	150.15
Crystal system	Monoclinic
Space group	$P2_1/c$
a, Å	8.5172(7)
b, Å	14.9767(13)
c, Å	16.9977(15)
β, deg	92.149(2)
V, Å^3	2166.7(3)
Z	4
ρ_{calc}, g/cm^3	1.237
μ, mm^{-1}	0.907
F(000)	840.0
Range of data collection over θ, deg	4.234–60.06
Collected reflections	18502
Number of independent reflections	4465 (($R_{int} = 0.0548$), $R_{sigma} = 0.0487$)
GOOF	1.021
R_1, wR_2 for *N*o	$R_1 = 0.0499$, $wR_2 = 0.1250$
R_1, wR_2 for *N*	$R_1 = 0.0789$, $wR_2 = 0.1401$
Residual electron density (max/min), e/Å^3	1.31/–0.29

and integrated using the SAINT program [50]. An absorption correction based on measurements of equivalent reflections was applied (SADABS) [51]. The structure was solved by a direct method followed by the calculation of the difference Fourier syntheses. All non-hydrogen atoms were refined in the anisotropic approximation. All hydrogen atoms of the CH and BH groups were refined by the riding model with the thermal parameters $U_{iso} = 1.2U_{eq}(U_{iso})$ of the corresponding non-hydrogen atom ($1.5U_{iso}$ for CH_3 groups). All calculations were performed using the SHELXTL program [52]. The structure was solved and refined using the OLEX2 software [53]. The main crystallographic data, experimental parameters, and structure refinement characteristics are given in Table 1.

The crystallographic data were deposited with the Cambridge Crystallographic Data Centre (CIF file CCDC no. 2224702; deposit@ccdc.cam.ac.uk; http://www.ccdc.cam.ac.uk).

RESULTS AND DISCUSSION

The nickel aqua complex $[Ni(H_2O)_6][B_{10}H_{10}]$ was previously synthesized by the reaction of acid $[(H_3O)_2][B_{10}H_{10}]$ and nickel(II) hydroxide [30]. The procedure of the synthesis of the nickel(II) aqua complex from nickel oxide or carbonate was used in this work.

$$NiO/Ni(CO_3)_2 + [(H_3O)_2][B_{10}H_{10}] \\ \rightarrow [Ni(H_2O)_6][B_{10}H_{10}].$$

Complex $[Ni(H_2O)_6][B_{10}H_{10}]$ (**I**) was characterized by IR spectroscopy and elemental analysis. The IR spectrum of compound **I** contains ν(BH) stretching vibration bands of BH bonds about 2500 cm^{-1} and a broad band in a range of 3500–3200 cm^{-1} corresponding to the ν(OH) stretching vibrations of the water molecules. This band broadening is explained by numerous hydrogen bonds that are formed in the structure of the compound.

A similar nickel complex containing acetonitrile molecules as ligands is formed by complexation in acetonitrile.

$$NiCl_2 + [(Et_3NH)_2][B_{10}H_{10}] \rightarrow [Ni(CH_3CN)_6][B_{10}H_{10}].$$

Compound $[Ni(CH_3CN)_6][B_{10}H_{10}]$ (**II**) precipitates as a blue polycrystalline substance. The solvent is rapidly blown away from the crystal surface so that the formed crystals are not suitable for XRD studies. The IR spectrum of complex **II** exhibits an absorption band in the range about 2300 cm^{-1} characteristic of ν(CN) vibrations of the solvent molecules and a broad intense band with two maxima at 2532 and 2440 cm^{-1} corresponding to the ν(BH) vibrations.

After a minor amount of water (approximately 1 : 5, vol/vol) was added to the reaction solution containing complex **II** in acetonitrile, the crystals are instantly dissolved and a bright blue solution is formed from which blue crystals of complex $[Ni(CH_3CN)_5(H_2O)]_{0.75}[Ni(CH_3CN)_4(H_2O)_2]_{0.25}[B_{10}H_{10}]\cdot0.5H_2O$ (**III**$\cdot0.5H_2O$) precipitated.

$$[Ni(CH_3CN)_6][B_{10}H_{10}] + H_2O \rightarrow [Ni(CH_3CN)_5(H_2O)]_{0.75}[Ni(CH_3CN)_4(H_2O)_2]_{0.25}[B_{10}H_{10}].$$

The IR spectrum of compound **III** exhibits a broad intense band at 2480 cm^{-1} corresponding to the ν(BH) vibrations and bands in ranges of 3500–3200 and ~2290–2320 cm^{-1} caused by the ν(OH) and ν(CN) vibrations of the water and acetonitrile molecules, respectively.

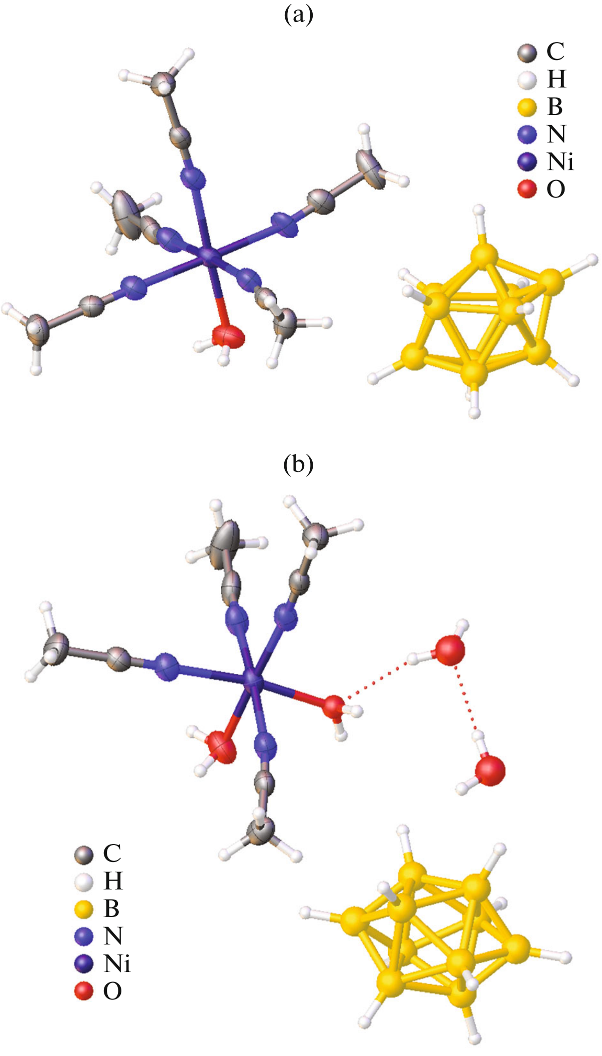


Fig. 1. Structures of cations (a) $[Ni(CH_3CN)_5(H_2O)]^{2+}$ and (b) $[Ni(CH_3CN)_4(H_2O)_2]^{2+}$ cocrystallized in compound **III**$\cdot0.5H_2O$.

According to the XRD data (Fig. 1), complex **III**$\cdot0.5H_2O$ consists of two complexes $[Ni(CH_3CN)_5(H_2O)][B_{10}H_{10}]$ and $[Ni(CH_3CN)_4(H_2O)_2][B_{10}H_{10}]\cdot2H_2O$ in a ratio of 0.75 : 0.25. Similarly to the disordered cationic moiety of the complex, the anion is disordered over two positions with the corresponding populations. The environment of the metal atom in the complex cations is distorted octahedral, the Ni–N bond lengths in the complexes range from 2.046(3) to 2.099(6) Å, and the Ni–O bonds are 2.086(2) Å for the Ni–O(1) bond and 1.988(17) Å for the Ni–O(2) bond, which corresponds to the average bond lengths for the nickel acetonitrile complexes [54–57] and nickel aqua complexes [58–60].

In the crystal, the complex cations form dimeric pairs bound by hydrogen bonds via the water molecules of the aqua complex, and the cations in complex $[Ni(CH_3CN)_4(H_2O)_2]^{2+}$ are additionally linked by hydrogen bonds of the solvate water molecules (Fig. 2). The dimeric pairs form stacks parallel to the *a* axis, which, in turn, form layers parallel to the *ab* plane. The $[B_{10}H_{10}]^{2-}$ anions are arranged between the layers of the cations and bound them between each other by weak BH...CH interactions.

Compound **III** can be considered as an intermediate of ligand metathesis during nickel(II) complexation in the presence of the *closo*-decaborate anion. The data obtained suggest the mechanism of a stepwise metathesis of solvent molecules as ligands in a $solv_1/solv_2$ system during nickel(II) complexation in the presence of the *closo*-decaborate anion. In the ideal case, the process proceeds via Scheme 1.

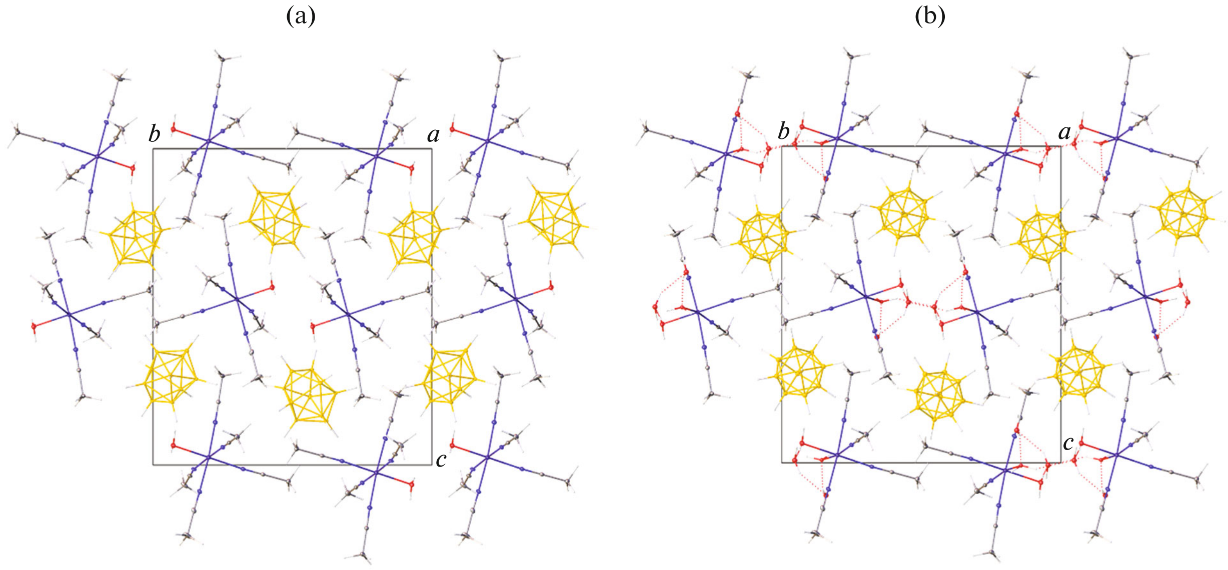
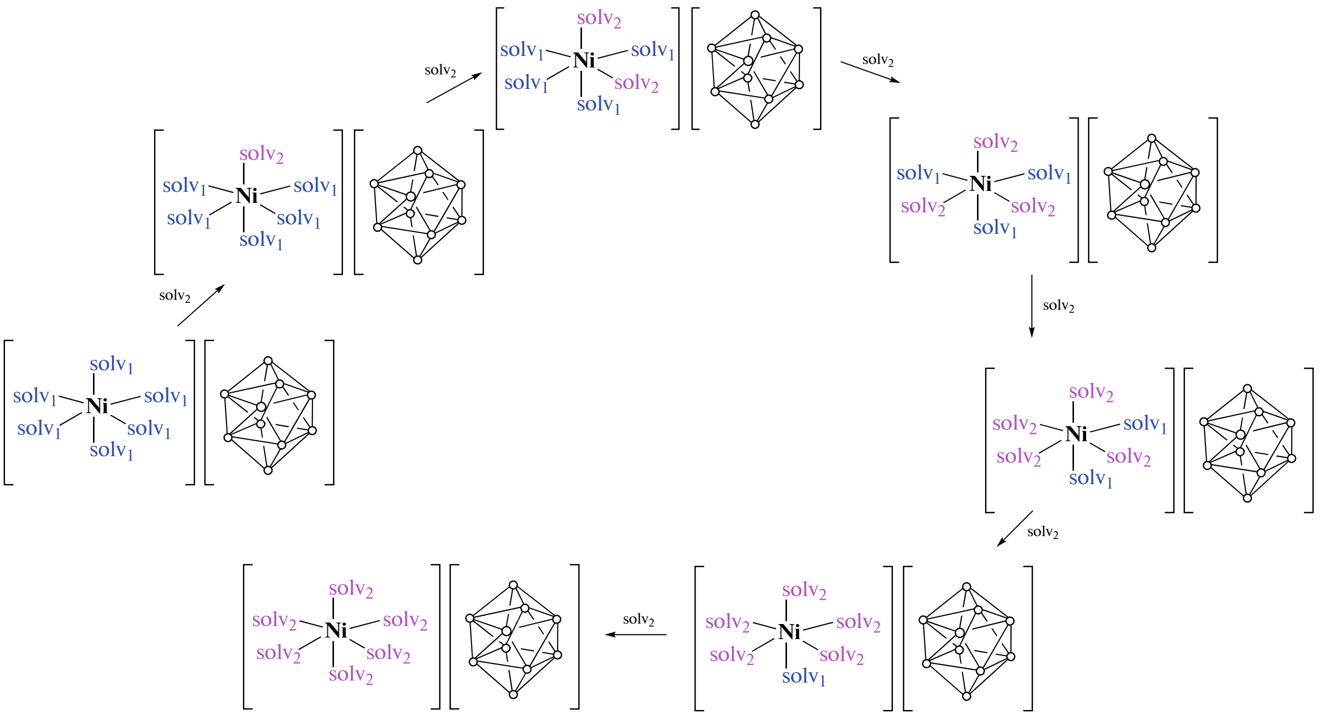


Fig. 2. Fragments of the crystal packings of complexes (a) $[Ni(CH_3CN)_5(H_2O)][B_{10}H_{10}]$ and (b) $[Ni(CH_3CN)_4(H_2O)_2][B_{10}H_{10}] \cdot 2H_2O$ cocrystallized in compound **III**$\cdot 0.5H_2O$.



Scheme 1.

In fact, when the reaction is carried out in an acetonitrile–water mixture, several possible cations with the close ratio of solvents are observed, most likely, in the reaction solution depending on the solvent ratio. This is indicated by the cocrystallization of two nickel cations with different contents of water and acetonitrile. A compound with the lowest solubility precipitates from the reaction solution. When the complexation is carried out in one solvent, the compound containing six molecules of the corresponding solvent is formed: $[Ni(solv)_6][B_{10}H_{10}]$ (solv = H_2O or CH_3CN). This mechanism can be extended over any pair of solvents $solv_1/solv_2$. If complex $[Ni(solv_1)_6][B_{10}H_{10}]$ is soluble in the $solv_2$ solvent, the ligand metathesis $solv_1 \rightarrow solv_2$ can be assumed.

The mechanism of ligand metathesis has previously been studied for the starting complex $[Cu_2(CH_3CN)_4[B_{10}H_{10}]]$ in the complexation of copper(I) with triphenylphosphine in acetonitrile with the formation of the final compound $[Cu_2(Ph_3P)_4$-$[B_{10}H_{10}]]$ and intermediates $[Cu_2(CH_3CN)_{4-x}$-$(Ph_3P)_x[B_{10}H_{10}]]$ (x = 3, 2, 1) [61]. The latter were characterized by XRD and other methods. The selective formation of intermediates of certain compositions with different contents of the organic ligand and acetonitrile molecules was observed when the content of ligand Ph_3P was varied over the copper(I) content in the reaction solution.

Taking into account the above described possibility of using complexes of the type $[Ni(solv)_6][B_{10}H_{10}]$ (solv = DMF, H_2O) in the synthesis of borides and carbides, it seems evident that the complexes synthesized in this work can also be used further as precursors for the preparation of borides, since the acetonitrile molecules are removed from the complexes during thermolysis at a lower temperature in the unchanged form and, therefore, do not contaminate the obtained product.

Thus, the metathesis of the ligands during the complexation of nickel(II) in the presence of the *closo*-decaborate anion in an acetonitrile–water system was studied in this work. Complexes $[Ni(solv)_6][B_{10}H_{10}]$ (solv = H_2O or CH_3CN) were isolated and characterized by IR spectroscopy and elemental analysis. The structure of the $[Ni(CH_3CN)_5(H_2O)]_{0.75}$-$[Ni(CH_3CN)_4(H_2O)_2]_{0.25}[B_{10}H_{10}]$ intermediate was determined by XRD. The mechanism of ligand metathesis during nickel(II) complexation was proposed.

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CONFLICT OF INTEREST

The authors declare that they have no conflicts of interest.

ADDITIONAL INFORMATION

This article is prepared for the memorial issue in tribute to the Corresponding Member of the Russian Academy of Sciences K.Yu. Zhizhin on his 50th birthday.

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