

## Study of Complexation Patterns in the System $\text{Ni}^{2+}$ , $\text{MoO}_4^{2-}$ , $\text{P}_2\text{O}_7^{4-}$ , $\text{Cit}^{3-}$ for the Development of Poly-Ligand Electrolytes (*Study of complexation patterns*)

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### Abstract

The complexation behavior in systems containing  $\text{Ni}^{2+}$ ,  $\text{MoO}_4^{2-}$ ,  $\text{P}_2\text{O}_7^{4-}$ ,  $\text{Cit}^{3-}$  ions have been thoroughly investigated. The study determined the composition and instability constants of both mono- and biligand complex compounds at a constant ionic strength of the solution ( $I_c=1$ ). By analyzing the concentration ratios of the complexing agent and ligands, the composition of mono- and polyligand complexes was elucidated. The complexation study utilized the potentiometric method, which is based on the functional dependence of the potential of the indicator electrode on the concentration of the complexing agent ions. The results enabled the calculation of the ionic composition of aqueous solutions of nickel complexes with citrate and diphosphate ions, depending on their concentrations. A proposed scheme for the formation of heteronuclear nickel-molybdenum complexes takes into account the sequence of component introduction into the electrolyte to form complexes of a specific composition. These findings were applied to develop electrolyte compositions for coating with alloys based on iron subgroup metals with molybdenum. These alloys exhibit several valuable properties, including corrosion resistance, electrocatalytic activity in hydrogen production, and enhanced operational characteristics.

## 1. Introduction

In recent years, there has been significant progress in the development of electrochemical coatings using alloys and composite materials based on metals from the iron family. This advancement is driven by the high demand for their properties, including anticorrosion, catalytic, magnetic, and other functional attributes [1–4]. As a result, a key objective has been to create new electrolytes, particularly complex ones, that are environmentally safe, cost-effective, and capable of ensuring high produc-

tivity in technological processes, thereby enhancing their competitiveness.

However, comparing the standard electrode potential values of the alloy-forming components suggests that co-depositing nickel and molybdenum into multicomponent alloys or composites is impossible using simple electrolytes [5–7]. Therefore, developing more sophisticated electrolytes is necessary to overcome this challenge and achieve the desired material properties.

The electrochemical behavior of molybdenum exhibits several distinct characteristics [8]. Molybdenum can exist in various oxidation states in its compounds; however, reducing molybdenum from salt solutions with oxidation states of  $+2$  or  $+3$  is not

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feasible because they undergo hydrolysis, forming insoluble compounds (Table 1, Reaction 1). Furthermore, the reduction of molybdates to the metallic state from aqueous solutions is practically impossible due to the competing hydrogen evolution reaction. This reaction results in the formation of oxide compounds with intermediate oxidation states (Table 1, Reaction 2) which have low electrical conductivity and block the electrode surface. The only viable method for the electrochemical deposition of molybdenum from aqueous solutions is its co-reduction with metals from the iron subgroup, forming alloys. In these alloys, the refractory component is fully reduced, enabling the effective incorporation of molybdenum into the material.

**Table 1.** Equilibrium potentials of electrode reactions

#	Reaction	Electrode potential E, B
1	$\text{Mo}^{3+} + 3\text{H}_2\text{O} = \text{MoO}_3 + 6\text{H}^+ + 3e$	$0.317 - 0.1182 \text{ pH} - 0.0197 \text{ lgc}(\text{Mo}^{3+})$
2	$\text{MoO}_4^{2-} + 4\text{H}^+ + 2e = \text{MoO}_2 + 2\text{H}_2\text{O}$	$0.606 - 0.1182 \text{ pH} + 0.0295 \text{ lgc}(\text{MoO}_4^{2-})$

It is well known that the use of complex compounds allows for the convergence of electrode potentials and the co-deposition of metals into alloys or composites [9–11]. The deliberate combination of ligands can have various effects: it can strengthen bonds and form mixed complexes, create intermediates that dissociate with significant inhibition, or even prevent joint coordination altogether.

Modern understanding of the mutual influence of ligands in all types of complex compounds, regardless of their geometry [12–15], suggests that the diphosphate ion [16–19], as a bidentate ligand, forms uncompensated induced dipoles. These dipoles can strengthen the metal bond in the complex with one group while weakening it with another. Introducing a second ligand into an electrolyte solution containing diphosphate complexes, especially one with neutral substituents or coordination groups that slightly reduce the effective charge on the central atom, creates favorable conditions for the formation of complex mixed complexes. The citrate ion is an ideal candidate for this second ligand, as it meets all the aforementioned criteria [20–23]. By using citrate ions, the overall effectiveness of the complexation process is enhanced, leading to the successful formation of stable and efficient mixed complexes.

Understanding the composition of complex compounds based on diphosphate and citrate ions in the citrate-diphosphate electrolyte, as well as the

kinetics and mechanism of alloy and composite deposition, enables precise control over electrode processes. This, in turn, impacts the structure, quality, and functional properties of the resulting coatings. Despite numerous studies, the chemistry of iron subgroup metals continues to be of significant theoretical interest in the field of coordination compounds, underscored by their practical importance. Therefore, mono- and poly-ligand complexes of nickel are particularly noteworthy and hold substantial promise for practical applications.

This study focused on a diphosphate-citrate electrolyte, chosen for its ability to deposit high-quality nickel-molybdenum alloy coatings with predictably higher current yields compared to mono-ligand electrolytes [24–27]. Alloys and composites derived from this system possess a range of valuable physical, chemical, and operational properties, making them indispensable for practical applications and enhancing the demand and competitiveness of these coatings.

The research aimed to understand the regularities of complexation in the  $\text{Ni}^{2+}$ ,  $\text{MoO}_4^{2-}$ ,  $\text{P}_2\text{O}_7^{4-}$ ,  $\text{Cit}^{3-}$ -system, with the goal of utilizing these findings to develop a polyligand electrolyte for the deposition of coatings needed in practical applications. This practical focus guided the study, leading us to limit our analysis to well-documented experimental results described in the literature [28–32].

## 2. Materials and methods

The composition of nickel diphosphate, citrate, molybdate, and citrate-diphosphate complexes was studied using the potentiometric method [28–30]. Measurements were conducted in a sealed standard electrochemical cell YASE-2 TU 25-05 at a temperature of  $293 \pm 1$  K. The cells were thermostated with an automatic temperature regulator RT-049, which included a resistance thermometer. Nickel and molybdenum plates were used as indicator electrodes, while a silver chloride half-cell ( $\text{Ag}|\text{AgCl}|\text{Cl}^-$ ,  $E^0 = 0.222$  V) - EVL-1M1 served as the reference electrode. This reference electrode was connected to the cell through a salt bridge filled with thickened agar-agar containing potassium chloride solution. The potentials of indicator electrodes were measured with a UNI-T UT8802E high impedance voltmeter, all potentials were recalculated and referenced to the normal hydrogen electrode. The pH of the initial solutions and electrolytes was determined and controlled using a pH-150 M instrument with a glass electrode (ESCL-08M).

The instability constants ( $K_n$ ) of the complexes were determined by potentiometry of solutions where the total concentration of nickel ions was  $5\text{--}10^{-3}$  mol/dm<sup>3</sup>, and the concentration of ligands  $c(\text{P}_2\text{O}_7^{4-})$ ,  $c(\text{C}_3\text{H}_5\text{O}(\text{COO})_3^{3-})$  (hereinafter  $\text{Cit}^{3-}$ ) and  $c(\text{MoO}_4^{2-})$  varied in the range of  $10^{-3}$  -  $2\cdot 10^{-1}$  mol/dm<sup>3</sup>. The average value of the potentials, derived from the results of 5–8 parallel measurements in solutions with constant ionic strength ( $I_c=1$ ) maintained by the addition of potassium sulfate, was used to calculate the instability constants.

### 3. Results and discussion

The complexation was studied using the Leden method, a widely adopted approach for investigating composition and stability of complex compounds. This method is based on determination the concentration of free metal ions. Analyzing the equilibrium potential of nickel as a function of ligand concentration in solution (Fig. 1) reveals that as the concentration of  $c(\text{P}_2\text{O}_7^{4-})$  and  $c(\text{Cit}^{3-})$  increases, the electrode potential becomes more noble. This indicates a decrease in the concentration of Ni(II) ions due to their binding in the complex.

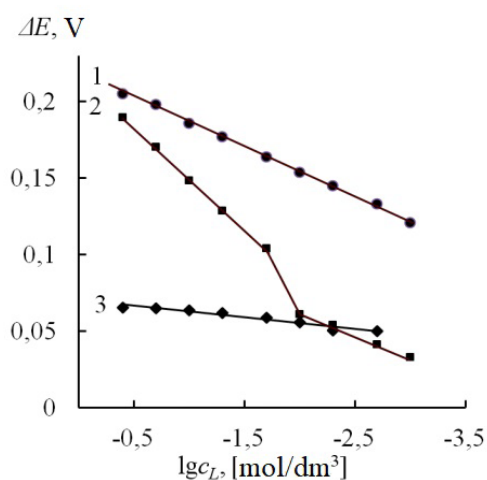
Notably, when the concentration ratio  $c(\text{P}_2\text{O}_7^{4-})/c(\text{Ni}^{2+})$  exceeds 10, a kink is observed in the  $E\text{-lg}c(\text{P}_2\text{O}_7^{4-})$  dependence (Fig. 1). The ratio of the slopes of the linear segments of the  $E\text{-lg}c(\text{P}_2\text{O}_7^{4-})$  dependence indicates that the stepwise instability constants of the complexes present in the solution differ by more than two orders of magnitude. This allows for the determination of the complex compositions at different ligand concentrations. When the ratio of ligand to complexing agent is less than

10, the solution predominantly contains  $\text{Ni}(\text{P}_2\text{O}_7^{2-})$  ions. Increasing the ligand concentration leads to the formation of a more stable complex compound  $\text{Ni}(\text{P}_2\text{O}_7)_2^{6-}$ , considering that in the pH range above 7, the protonated ligand species do not participate in complexation [11].

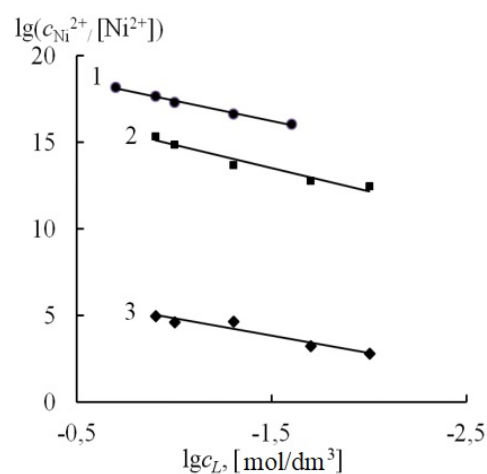
For solutions containing nickel ions and sodium molybdate, the degree of formation or the average number of ligands across entire concentration range is 0.22 (Fig. 1). This observation suggests that the formation of a nickel molybdate complex is highly unlikely. The calculated instability constant  $K_n = 3.6\text{--}10^{-6}$  for the  $\text{Ni}^{2+} - \text{MoO}_4^{2-} - \text{H}_2\text{O}$  system indicates the formation of hydroxocomplex  $\text{Ni}(\text{OH})^+$ , since the pH of these solutions ranges from 8.4 to 9.6.

The linear progression and angular slope coefficient of the equilibrium potential of the nickel electrode as a function of  $\text{Cit}^{3-}$  ion concentration (Fig. 1) indicate the formation of a complex compound with the composition  $\text{NiCit}^-$ . It should be noted that the degree of protonation and the concentration of the various citrate forms change significantly depending on the pH of the solution. As a result, either deprotonated  $\text{Cit}^3$  ions or particles with different degrees of protonation participate in the coordination process. At pH levels of 7 or higher,  $\text{Cit}^{3-}$  ions predominantly exist in the solution.

The instability constants, calculated from the changes in the equilibrium potential of the electrode with varying ligand concentrations (Table 2), are consistent and align closely with literature values [12, 13, 30]. This consistency confirms the validity of the proposed methods for determining the composition and stability of complex compounds.



**Fig. 1.** Dependence of the potential difference of nickel electrodes on the concentration of ligands  $\text{Cit}^{3-}$  (1),  $\text{P}_2\text{O}_7^{4-}$  (2) and  $\text{MoO}_4^{2-}$  ions (3).



**Fig. 2.** Effect of ligand concentration (1, 2 –  $\text{Cit}^{3-}$ ; 3 –  $\text{P}_2\text{O}_7^{4-}$ ) on the  $\lg(c_{\text{Ni}^{2+}}/[\text{Ni}^{2+}])$  ratio for the systems: 1 –  $\text{Ni}^{2+} - \text{P}_2\text{O}_7^{4-} - \text{Cit}^{3-}$ ; 2 –  $\text{Ni}^{2+} - \text{MoO}_4^{2-} - \text{Cit}^{3-}$ ; 3 –  $\text{Ni}^{2+} - \text{MoO}_4^{2-} - \text{P}_2\text{O}_7^{4-}$ .

**Table 2.** Instability constants of nickel complex compounds

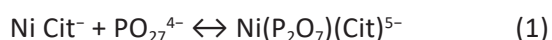
Ligand	Composition of the complex	pH	K <sub>n</sub>	K <sub>n</sub> [30]
Cit <sup>3-</sup>	Ni(Cit) <sup>-</sup>	7.0–7.5	2.8·10 <sup>-13</sup>	5.6·10 <sup>-11</sup>
P <sub>2</sub> O <sub>7</sub> <sup>4-</sup>	Ni(P <sub>2</sub> O <sub>7</sub> ) <sup>2-</sup>	7.5–9.3	1.2·10 <sup>-6</sup>	1.5·10 <sup>-6</sup>
	Ni(P <sub>2</sub> O <sub>7</sub> ) <sub>2</sub> <sup>6-</sup>		1.8·10 <sup>-11</sup>	6.48·10 <sup>-10</sup>

Analyzing the probability of forming complexes with different compositions reveals that mixed compounds are statistically more favorable than monoligand complexes. In most cases, poly-ligand complexes are more stable than monoligand ones, although some studies indicate a weakening of bonds in certain scenarios.

To determine the influence of a second ligand on the composition and stability of nickel complexes, we studied these characteristics by varying the concentrations of the complexing agent and ligands (Fig. 2). Here, c<sub>Ni<sup>2+</sup></sub> represents the total Ni<sup>2+</sup> concentration in the electrolyte, and [Ni<sup>2+</sup>] represents the concentration of ions not bound in complexes. Based on the analysis of the obtained dependencies (see Fig. 2) for complexation in the Ni<sup>2+</sup> - P<sub>2</sub>O<sub>7</sub><sup>4-</sup> - MoO<sub>4</sub><sup>2-</sup> and Ni<sup>2+</sup> - P<sub>2</sub>O<sub>7</sub><sup>4-</sup> - Cit<sup>3-</sup> systems, a Ni<sup>2+</sup>/P<sub>2</sub>O<sub>7</sub><sup>4-</sup> ratio of ≥ 1:10 was used.

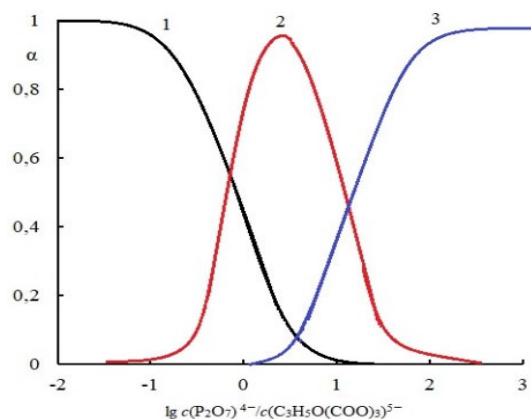
Previous studies have shown that at this specific ratio, there is a noticeable kink in the E-dependencies, indicating the formation of a more stable complex compound Ni(P<sub>2</sub>O<sub>7</sub>)<sub>2</sub><sup>6-</sup> (Table 2).

Complexation in the Ni<sup>2+</sup> - P<sub>2</sub>O<sub>7</sub><sup>4-</sup> - Cit<sup>3-</sup> system exhibits certain peculiarities and is influenced by the order in which the ligands are introduced into the solution. When citrate ions are added to a solution containing diphosphate, the citrate ions do not participate in complex formation, as indicated by the unchanged potential difference upon the addition of the citrate solution. Conversely, when diphosphate ions are added to a solution of nickel citrate complexes, a strong mixed biligand complex forms, characterized by an instability constant K<sub>n</sub> = 2.2·10<sup>-17</sup>. This process can be described by the following equation:



This reaction within the system can be attributed to an increase in the coordination number of the central atom. The formation of a more stable complex is explained by the additional polarization of the ion in the presence of dissimilar ligands.

The dependence character of ΔE = f(lgc<sub>i</sub>) for the Ni<sup>2+</sup> - MoO<sub>4</sub><sup>2-</sup> - P<sub>2</sub>O<sub>7</sub><sup>4-</sup> system is identical to that of

**Fig. 3.** Effect of ligand ratio on the dependence of the degree of formation of citrate (1), citrate-diphosphate (2) and diphosphate (3) nickel (II) complexes.

the Co-P<sub>2</sub>O<sub>7</sub><sup>4-</sup> system, indicating the complete substitution of MoO<sub>4</sub><sup>2-</sup> ions and formation of complex Co(P<sub>2</sub>O<sub>7</sub>)<sub>2</sub><sup>6-</sup>. The study of complexation in the system Ni<sup>2+</sup> - MoO<sub>4</sub><sup>2-</sup> - Cit<sup>3-</sup> (Fig. 2) suggests partial substitution of MoO<sub>4</sub><sup>2-</sup> ions and the formation of complexes [NiMoO<sub>4</sub>(Cit)]<sup>3-</sup>.



The results of these studies were used to calculate the ionic composition of aqueous solutions of nickel complexes with citrate and diphosphate ions, depending on their concentrations (Fig. 3).

Using the established principles of complexation and considering the mutual influence of the nature of ligands, the following scheme for the formation of mono- and biligand complexes of nickel can be proposed (Fig. 4). The determined parameters of these nickel complex compounds, along with the proposed schemes, provide a foundation for developing electrolytes and methods for the electrochemical deposition of alloys and composites based on nickel with refractory metals. The practical application of the proposed scheme provides opportunities to control the electrodeposition process of electroplating alloys or metal-oxide composites within the studied systems. This can be achieved by varying the methods (such as direct or pulsed current) and adjusting polarization parameters to achieve the targeted synthesis of coatings with a specified composition.

The graphical scheme presented in Fig. 4 illustrates the sequence of ionic equilibrium formation, the impact of pH on the protonation and deprotonation of ligands, and the development of mono- and biligand complexes. Additionally, it highlights the

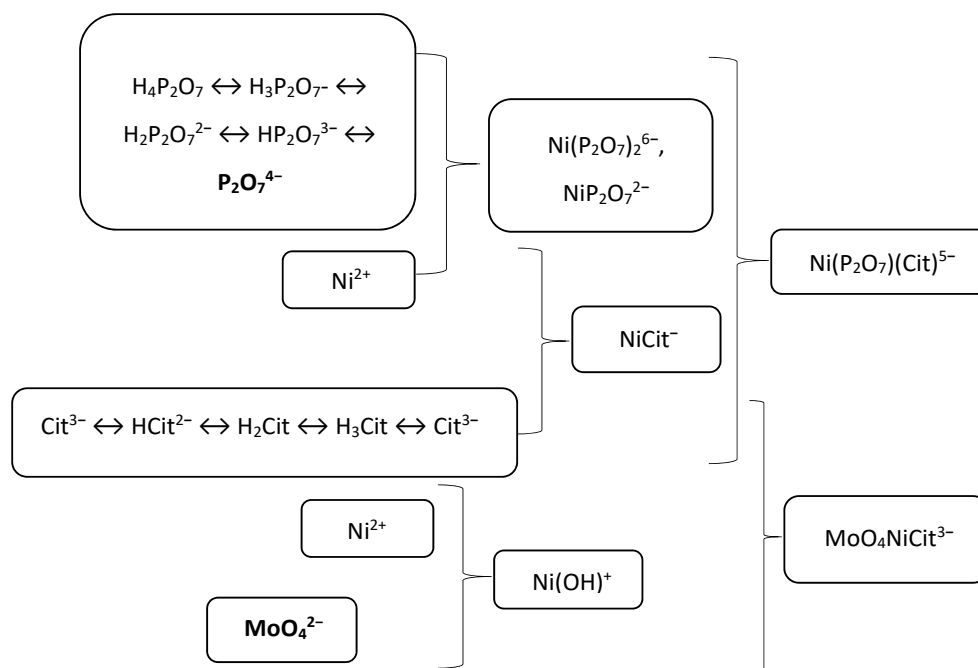


Fig. 4. Scheme of formation of heteronuclear nickel-molybdenum complexes.

significance of the order in which ligands are introduced into the electrolyte solution, as this affects the composition of the resulting complexes. This aspect of the research is crucial for developing polyligand electrolyte compositions for practical electroplating, ensuring a high current yield.

#### 4. Conclusion

By varying the ratio of concentrations of complexing agent ( $\text{Ni}^{2+}$ ) and ligands ( $\text{MoO}_4^{2-}$ ,  $\text{P}_2\text{O}_7^{4-}$ ,  $\text{Cit}^{3-}$ ) in the pH range of 7–9.5, we determined the composition and instability constants of mono- and biligand complex compounds at a constant ionic strength of the solution ( $I_c=1$ ). It was found that at the ligand/complexing agent concentration ratio of  $\geq 10$ , the complexes  $\text{Ni}(\text{P}_2\text{O}_7)_2^{6-}$ , with an instability constant of  $1.8 \cdot 10^{-11}$ , and  $\text{Ni}(\text{Cit})^-$  with an instability constant of  $K_n = 2.8 \cdot 10^{-13}$  are formed. Combining these ligands of different natures within one coordination sphere creates the potential for forming a stronger compound. An algorithm for the formation of heteronuclear nickel-molybdenum complexes has been proposed, which can be used to develop electrolyte compositions for depositing coatings with alloys or metal-oxide composites with predictably high functional properties.

The functional properties of such coatings are multifaceted, including electrochemical and physico-mechanical characteristics, corrosion resistance, and electrocatalytic activity in hydrogen production.

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