



SYNTHESIS OF THE COMPLEX COMPOUND OF SALICYLAMIDE WITH NI(II) NITRATE SALT

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Annotatsiya: Ushbu maqolada $[\text{Ni}(\text{Sal})_2(\text{NO}_3)_2(\text{H}_2\text{O})_2]$ (Sal-salitsilamid) tarkibli kompleks birikmaning sintezi va uning kristall tuzilishi muhokama qilingan. Sintez qilingan kompleks birikma tarkibi element analizi va tuzilishi rentgen strukturaviy analiz usuli yordamida o'rganildi. Nikel tanlangan ligand salitsilamid bilan karbonil guruhidagi kislorod atomi orqali koordinatsiyaga uchrashi, shuningdek koordinatsiyalanishda gidroksil guruhi qatnashmayotganligi rentgen tuzilish tahlili (RTT) usuli yordamida ko'rsatib berildi. Sintezlangan koordinatsion birikma monokristali o'stirilib, unga tegishli parametrlar ya'ni bog' uzunligi, burchaklar kattaligi va kompleks birikma molekulasidagi atomlar orasida kuzatilayotgan vodorod bog'lar aniqlandi hamda struktura tahlil qilindi. Sintezlangan kompleks birikmada nikelning koordinatsion soni 6 ga teng bo'lib, uning gibridlanishi sp^3d^2 ekanligi aniqlandi.

Kalit so'zlar: Nikel(II)nitrat, salitsilamid, ligand, kompleks birikma, sintez, analiz, rentgen tuzilish tahlili (RTT), element analizi, koordinatsion bog', eruvchanlik, suyuqlanish harorati, reaksiya unumi.

Аннотация: В данной статье рассматривается синтез и кристаллическая структура комплексного соединения $[\text{Ni}(\text{Sal})_2(\text{NO}_3)_2(\text{H}_2\text{O})_2]$ (Sal-салициламид). Синтезированное комплексное соединение исследовано методами элементного анализа и рентгеноструктурного анализа. Методом рентгеновской дифракции (РФА) было показано, что никель координируется с выбранным лигандом салициламидом через атом кислорода карбонильной группы, а гидроксильная группа не участвует в координации. Был выращен монокристалл синтезированного координационного соединения, определены его важные параметры, а именно длина связи, величина угла и водородные связи, наблюдаемые между атомами в молекуле комплексного соединения, а также проанализирована структура. Координационное число никеля в синтезированном комплексном соединении равно 6, а его гибридизация имеет вид sp^3d^2 .

Ключевые слова: Нитрат никеля(II), салициламид, лиганд, комплексное соединение, синтез, анализ, рентгеноструктурный анализ (РФА), элементный



анализ, координационная связь, растворимость, температура ликвидуса, выход реакции.

Abstract: *In this study, the synthesis and crystal structure of the complex compound $[\text{Ni}(\text{Sal})_2(\text{NO}_3)_2(\text{H}_2\text{O})_2]$ (Sal – salicylamide) are discussed. The composition of the synthesized complex was analyzed using elemental analysis, and its structure was investigated by X-ray structural analysis. X-ray structural analysis (XSA) revealed that nickel coordinates with the selected ligand, salicylamide, through the oxygen atom of the carbonyl group, while the hydroxyl group does not participate in coordination. In the synthesized complex compound, the coordination number of nickel was determined to be 6, and its hybridization was identified as sp^3d^2 .*

Key words: *Nickel(II) nitrate, salicylamide, ligand, complex compound, synthesis, analysis, X-ray structural analysis (XRD), elemental analysis, coordination bond, solubility, melting point, reaction yield.*

Nowadays, it can be acknowledged that the world's population is growing at a rapid pace. As the population increases, people naturally strive to create comfortable living conditions for themselves. Like all living organisms, humans are also influenced by abiotic and biotic factors in their living environment. As a result, due to the serious ecological changes occurring today, the number of bacteria, viruses, and fungi affecting human health is significantly increasing. Currently, more than 3 million harmful bacteria and over 2,000 types of viruses have been studied [1]. These bacteria and viruses negatively impact human health, the well-being of wild and domestic animals, and the growth and development of plants, disrupting their normal functional state. By studying such adverse effects, research was conducted on the synthesis, composition, and structure of salicylamide (2-hydroxybenzamide), a derivative of salicylic acid, which has a positive influence on plant growth and productivity [2]. The coordination compound of salicylamide with nickel, one of the 3d metals, was synthesized in a solution medium, and its monocrystals were grown. The X-ray structural analysis of the synthesized complex was performed, and the corresponding parameters were determined.

Numerous scientific database records and literature analyses reveal that leading scientific centers and chemists worldwide have synthesized coordination compounds of salicylic acid and its derivatives, studying their composition, structure, and biological activity [3]. The English scientist E. Furia, in his article titled "Study of the Complexation Equilibrium Between Iron (III) Ion and 2-Hydroxybenzamide in Aqueous Solution", provided insights into the interaction of salicylamide and iron (III) salts in aqueous solution, leading to the formation of a coordination compound and discussing the reaction equilibrium [4]. In the article "Serendipitous Synthesis of an Octahedral Ni(II) Complex: Single-Crystal Structure, Hirshfeld Surface, and Void Analysis" by T.Göktürk, C.G.Topkaya, T.Hökelek, and R.Güpları, a newly synthesized Schiff base ligand reacted with PPh_3 , resulting in the serendipitous formation of a $\text{C}_{50}\text{H}_{42}\text{N}_4\text{NiO}_6\text{P}_2 \cdot 2(\text{ClO}_4)$ complex. Its molecular and crystal structures were determined by single-crystal X-ray diffraction analysis. The complex crystallized in a triclinic system with the following parameters: $a =$



10.7936(4) Å, $b = 12.2926(4)$ Å, $c = 19.6272(6)$ Å, $\alpha = 92.574(3)^\circ$, $\beta = 95.527(3)^\circ$, $\gamma = 102.898(4)^\circ$, space group P , $Z = 2$, and unit cell volume $V = 2520.73(15)$ Å³ [5].

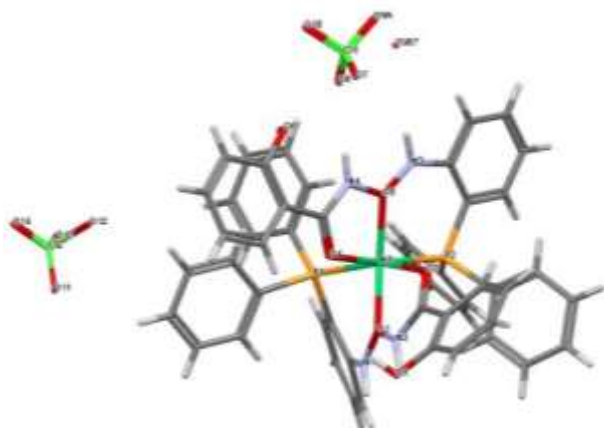
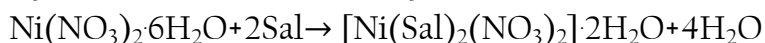


Figure 1. Molecular Structure of the Complex Compound $C_{50}H_{42}N_4NiO_6P_2 \cdot 2(ClO_4)$

In the crystal structure, $N-H \cdots O$ hydrogen bonds and uncoordinated perchlorate anions link to the parent molecule. Additionally, weak $C-H \cdots \pi$ interactions are observed. The Hirshfeld surface analysis of the crystal structure indicates that the most significant contributions to crystal packing interactions are $H \cdots H$ (43.9%), $H \cdots O/O \cdots H$ (30.9%), and $H \cdots C/C \cdots H$ (18.5%) interactions. Hydrogen bonding and Van der Waals interactions are the dominant forces in crystal packing. The analysis of crystal voids confirms the absence of large empty spaces in the crystal packing [6].

During the synthesis of nickel-salicylamide complex compounds, the following nickel salts were used: $Ni(NO_3)_2 \cdot 2H_2O$, $Ni(NO_3)_2 \cdot 4H_2O$, $Ni(NO_3)_2 \cdot 6H_2O$, and $Ni(NO_3)_2$. Initially, 2-hydroxybenzamide (L_1) was dissolved in ethanol, and a 0.02 mol/L, 100 mL solution was prepared. Subsequently, $Ni(NO_3)_2 \cdot 6H_2O$ was dissolved in water to prepare a 0.01 mol/L, 100 mL solution. Once the solutions were prepared, a round-bottom flask ($V = 200$ mL) was taken, and 20 mL of the nickel salt solution was first added. Then, 40 mL of the ligand solution was gradually added in four portions, each at 15-minute intervals. The flask was secured on a stand and placed on a magnetic stirrer, where the mixture was stirred for 60–80 minutes.

After stirring, the resulting mixture was left undisturbed at room temperature to allow sedimentation. To obtain monocrystals of the complex compound, the prepared mixture was divided equally into four beakers and kept in a thermostat at $30^\circ C$ for 5–7 days [7]. The obtained complex compound monocrystals were recrystallized in dimethylformamide. The reaction yield was 78.6%.



Scheme 1. Reaction for the Synthesis of the Coordination Compound $[Ni(Sal)_2(NO_3)_2] \cdot 2H_2O$

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