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Synthesis and Study of the Complex Compound of Isonicotinamide with Zinc Nitrate

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Abstract. A complex compound of zinc nitrate with isonicotinamide was synthesized. The advantages of the mechanochemical (solid-phase) method, the optimal conditions of synthesis are presented IR- and 1H NMR-spectroscopic analysis provide information about ligands and the complex compound formed on their basis, the nature of the bond, central atom surrounding, polyhedra, valence and deformation vibrations, shifts in proton signals. Crystallographic data of the complex compound were obtained using a Malvern Panalytical Empyrean diffractometer. Analysis of the obtained results was carried out using FULLPROF and VESTA programs. When the lengths and angles of the valence bonds were analyzed using the MOGUL program adapted to the MERCURY complex, it was found that there were no bonds and angles with non-standard values between them. The state of the central atomic spatial structure, hybridization, binding of the isonicotinamide molecule and the nitric acid residue to the zinc atom were studied. Based on the data obtained, it was concluded.

Key words: Complex compound, Infrared and ^{1}H NMR spectroscopy, Mechanochemical method, Zinc nitrate, Physicochemical methods of analysis.

Introduction

Huge amounts of mixed-ligand complex compounds of transition metals are synthesized in the world. In order to accelerate and increase the yield of the main crops, much attention is paid to stimulants, in particular to groups of metal complexes (Boldyrev, 2006). Metal complexes containing various N,O-donor centers in the ligand environment occupy a special place in modern coordination chemistry (Lomovsky, 2001). Due to the specific effect of their environment on the stereochemistry of polyhedra, they are good models for studying the problem of competitive coordination in the chemistry of complex compounds (Sharipova, 2022). In this regard, it seems important and relevant to search for ways of directed synthesis of polydentate ligands and, based on

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them, metal complexes of a certain composition and structure in order to solve theoretical and practical problems of creating new generation materials with predetermined properties (Sharipova et al., 2019). At the same time, the synthesis of effective, new types of complex compounds to increase productivity and their widespread use in agriculture remains an urgent problem (Ibragimova et al., 2016).

The author established the structure of new coordination compounds $[M(HCO_2)_2(NC_5H_4CONH_2)_2(H_2O)_2]$, (M=Co, Ni) using X-ray diffraction analysis and the results were entered into the Cambridge Crystallographic Database, (deposits were entered into the Cambridge Crystallographic Database, (deposits №. 2081143 and №. 2092828) (Jumaniyozova, 2021)

The compounds are isostructural and consist of neutral complexes (CN = 6), in which the metal atom is bonded to two nicotinamide molecules through nitrogen heteroatoms, two water molecules, and two monodentate formate ions. The coordination polyhedron is a distorted octahedron with the trans arrangement of the nitrogen heteroatoms of the nicotinamide molecules. Between nicotinamide molecules and formate ions, as well as between nicotinamide molecules and water molecules, intracomplex hydrogen bonds are realized in the complex (Jumaniyozova et al, 2021).

The structure of the coordination compound $[Ca(H_2O)_2(C_5H_4NC(O)NH_2)_2(NO_3)_2]$ was established by X-ray diffraction analysis. It was determined that water and nicotinamide molecules are coordinated monodentately through water oxygen atoms and oxygen atoms of the carbonyl group of nicotinamide, nitrate - anions are coordinated bidentately through oxygen atoms. This structure was entered into the Cambridge Crystallographic Database, deposit № 1850646 (Jumanazarova, 2018).

Method

All reagents were readily available from commercial sources and were used as received without further purifcation. The amount of metal in the synthesized complex was determined on the Novaa 300 apparatus of Analytic Jena (Germany) (Charlot, 2007). Analysis of C H and N were performed on an EuroVector EA3000 Series of CHNS-O Elemental Analysers (Bazhenova, 2008).

Synthesis and Crystallization

Synthesis of the coordination compounds of zinc nitrate with isonicotinamide was carried out by mechanochemical method (solid phase) (Sharipova, 2022). To determine the optimal conditions for the reaction, the mechanochemical reaction was carried out in a ball mill for 0.5 hours using blanks (working part) 1 and 2 (a ball with a diameter of 20 mm). The mass of the working part is 67 grams. The rotation number is 150 rpm. the duration of one is 30 seconds. Three such mixings constitute one cycle, the time between mixing cycles is 2-3 seconds (Sharipova, 2023). Zinc nitrate and isonicotinamide molecules were mixed in an equimolar ratio of 1:2 ($Zn(NO_3)_2{\cdot}6H_2O{:}2NC_5H_4CONH_2$). Yield of the complex is 68%. $[ZnC_{12}H_{14}O_9N_6]$ white crystalline substance, T_m=111. Elemental analysis for complex $ZnC_{12}H_{14}O_9N_6$ (451,68): calcd. C 31.93; H 3,10; N 18,63%; found: C 32,08; H 3,13; N 18,59% (Sharipova, 2018).

Methods and Refinement

The determination of the structure of the compounds was carried out using a Malvern Panalytical Empyrean diffractometer. XRD data were recorded using CuKα radiation (λ = 1.54 Å). In this experiment, the accelerating voltage of the radiation generator was set to 45 kV, and the current emission was set to 40 mA. X-ray diffraction radiographs were recorded at 2Th = 200–1200 in a Bregga–Brentano beam geometry with a continuous scanning speed of 0.33 degrees/min (Yakimov & Dubinin 2008). Analysis of the obtained results was carried out using FULLPROF and VESTA programs (Khudoyberganov et al., 2022). When the lengths and angles of the valence bonds were analyzed using the MOGUL program adapted to the MERCURY complex, it was found that there were no bonds and angles with non-standard values between them (Khasanov et al., 2023).

The absorption regions of the IR spectra were recorded on an IR Tracer-100 spectrometer (500–4000 cm^{-1}) from SHIMADZU (Nakamoto, 1991). The NMR method is very important in modern chemistry. This is due to the fact that the resonant frequencies of the nuclei depend on the interaction of the magnetic field (Ibragimov et al., 2022) and the distribution of electron densities in the molecule (Sharipova et al., 2023).

The ^{1}H NMR spectra of the complex compound were recorded on a JNM-ECZ400R spectrometer (Jeol, Japan) at an operating frequency of 400 MHz in methanol deuterium (CD_3OD) solution. Tetramethyl silane (TMS - $Si(CH_3)_4$) (ppm) was used as an internal standard to obtain ^{1}H NMR spectra (Volovenko et al., 2011). Chemical shifts of protons occupy a limit close to δ 10 ppm and their uncetanty found in experiment equal to± 0,001 ppm.

Results and Discussion

Description of IR-Analysis

In the IR spectrum of the uncoordinated isonicotinamide molecule, the ring frequency is observed at 1585 cm^{-1}, the frequency of this field increased to 1593 cm-1 in the complex state. The ring vibration frequency of isonicotinamide is in the region of $\nu_{(CN)}$=1019 cm^{-1} and $\delta_{(CCN)}$=733 cm^{-1}, and the vibration frequencies are shifted to the region of 1026 cm-1 and 743 cm^{-1}. The valence frequency of $\nu_{(CO)}$ bond of isonicotinamide remained unchanged, i.e. 1681 cm^{-1}. This indicates coordination of the isonicotinamide pyridine ring through a nitrogen heteroatom. In the complex compound, the nitrate anion was represented by ν s(NO_3), a low intensity band at 1027 cm^{-1}, ν as(NO_3) at 1303 cm-1, and δ (NO_3) at 815 cm^{-1}.

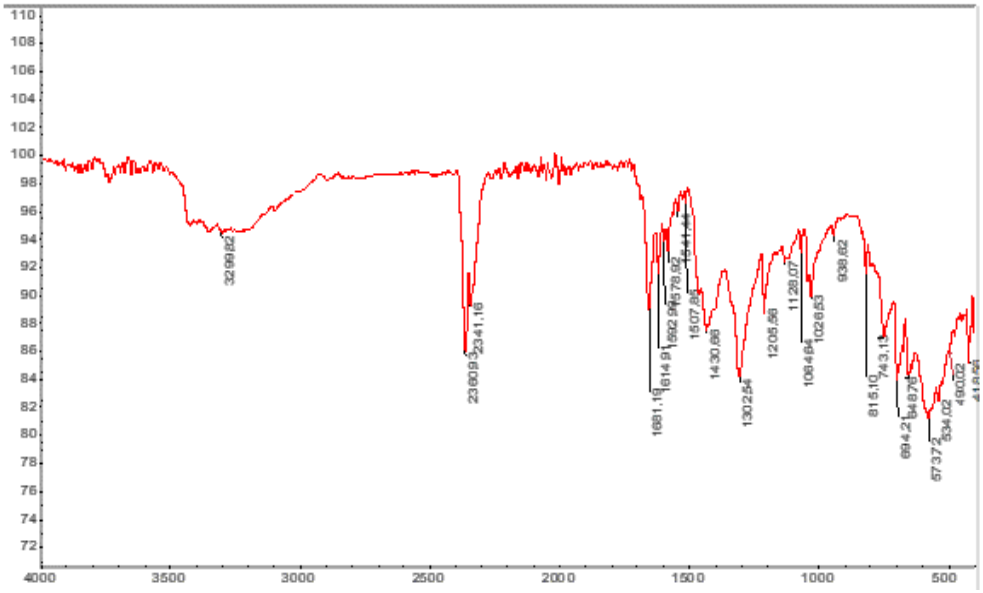

Figure 1. IR-spectrum of the complex compound

Description of NMR Analysis

In the ^{1}H NMR spectrum of isonicotinic acid amide, the signal of four hydrogen protons in the pyridine ring δ=9.028 ppm at (Hα) and multiplet signals (Hα') δ=8.686 ppm., (Hγ) δ=8.282 ppm., (Hβ) δ=547, 7.545 ppm. observed in weak areas (Fig. 3.10). The following chemical shift is observed in the ^{1}H NMR spectrum of $[Zn(NO_3)_2{\cdot}2NC_5H_4CONH_2]{\cdot}H_2O$ complex compound: δ=9.040, 9.035 ppm. at (Hα) and multiplet signals (Hα') δ =8.698 ppm, (Hγ) δ= 8.348, 8.328 ppm., (Hβ) δ=7.596, 7.594 ppm.

Comparing the chemical shift values of the protons in the free and coordinated pyridine ring shows that the signals are shifted towards the weak field. This shift indicates that the process of complex generation has gone. The strong shielding of the hydrogen located in the b state in the ligand pyridine ring and the strong descreening when isonicotinamide goes to the complex state are related to the metaorientation property of the nitrogen in the pyridine ring. Descreening in the pyridine ring is caused by the interaction of the unshared electron pair on the nitrogen atom with the 4s and 4p3 orbitals of zinc. As a result of this interaction, a molecular orbital is formed in which the electron density is transferred to the metal ion. This process causes descreening of hydrogen protons in the pyridine ring. Therefore, based on the data of 1N NMR spectroscopy, it can be concluded that isonicotinamide is coordinated through the nitrogen atom in the pyridine ring.

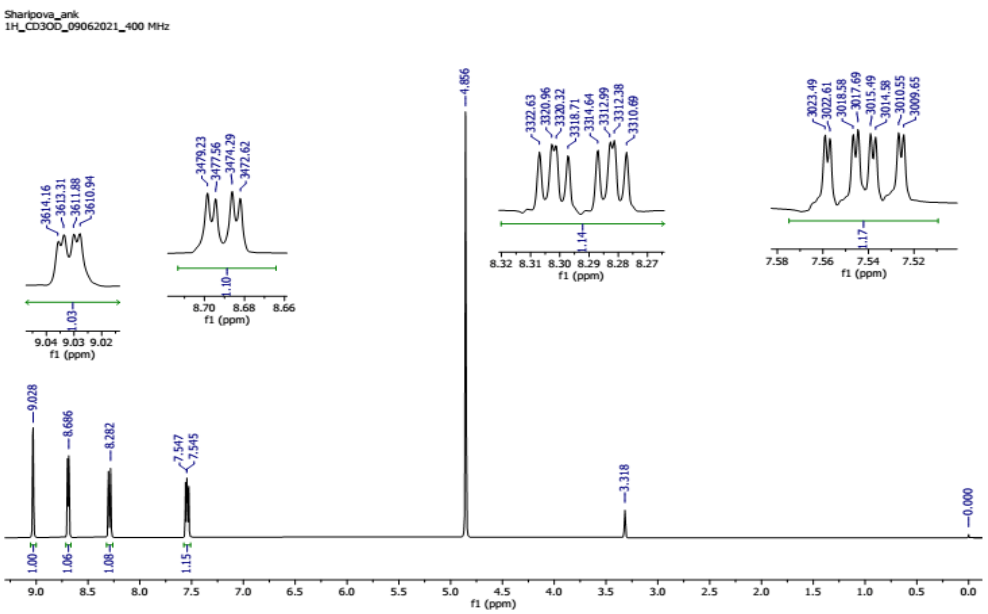

Figure 2. ^{1}H NMR spectrum of isonicotinamide molecule in CD_3OD

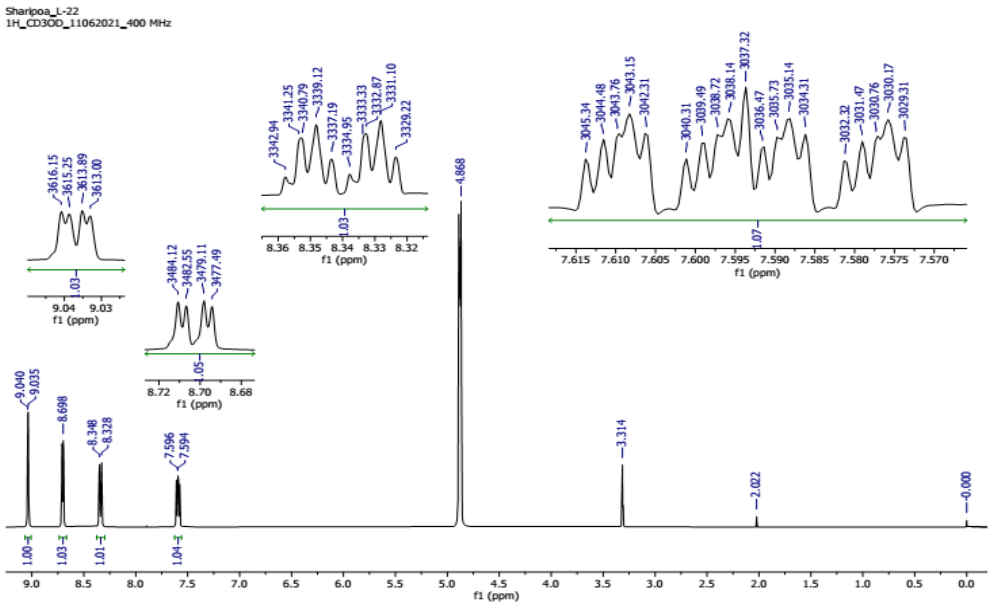

Figure 3. ^{1}H NMR spectrum of complex compound

Description of Molecular Structure

In the structure of the complex compound, the central zinc atom is triclinically coordinated with the nitrogen atom of the isonicotinamide ring of two ligands and the oxygen atoms of two nitrate residues. In this, isonicotinamide molecules participated as monodentate ligands through nitrogen atom and two nitrate residues through oxygen atoms as bidentate ligands. The central zinc atom has a coordination number of 6 and is hybridized in the sp^3d^2 state (Fig. 4).

The parameters of the unit cell of the crystal are as follows: spatial group C1, a a=21.33(8) Å, b=21.33(13) Å, c=15.08(3)Å, α=90°, β=90°, γ=90°, V=6868.107Å^3, Z=2. Complex compound $[Zn(NO_3)_2 \cdot 2NC_5H_4CONH_2] \cdot H_2O$ is mononuclear, and the complex formed by Zn^{2+} ion with isonicotinamide, nitric acid anion and water molecules has a neutral nature (Table 1).

Table 1. Parameters crystallographic data and structure of complex compound

Formule	$C_{12}H_{12}N_6O_8Zn$, H_2O	Size of the crystal, [mm]	0.22×0.18×0.06
Molecular mass	451.68	T, °K	298
Syngonia	Triclinic	θ,° deg.	2,14; 34,10
Spatial group	C1	Interval h,k,l	999: 99; 999: 99; 999: 99
a, Å	21.33620	Reflex	2146
b, Å	21.33620	Refraction index	1568
c, Å	15.08700	R_{int}	0.71073
α, *β*, *γ*, deg	90(14);90(15);90(15)	$F^2 \geq 2\sigma(F^2)$ Criteria	R_1=0.054
V, Å^3	6868.107	Parameter	4162
Z	2	Eligibility Criteria (F^2)	460
D_x, g/cm^{-3}	0.218	R_1, $wR_2(I>2\sigma(I))$	R_1=0.0548, wR_2=0.1894
μ(CuK_α), mm^{-1}	0.187		

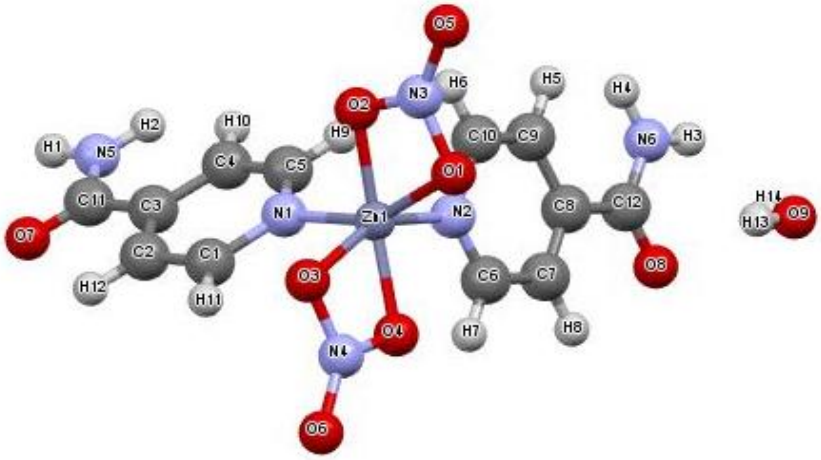

Figure 4. Molecular structure of the synthesized complex compound

Table 2. Valence bonds and bond lengths of complex compound

Bond	d, Å	Bond‘	d, Å
Zn(1)-O(1)	1.9737	C(3)-C(4)	1.4645
Zn(1)-O(2)	2.0082	C(3)-C(11)	1.4634
Zn(1)-O(3)	1.9751	C(4)-C(5)	1.3475
Zn(1)-O(4)	2.0088	N(5)-H(2)	1.0121
Zn(1)-N(1)	2.0013	N(5)-H(1)	1.0142
Zn(1)-N(2)	1.9984	N(6)-H(4)	1.0134
O(1)-N(3)	1.4505	C(6)-C(7)	1.4594
O(2)-N(3)	1.3581	N(6)-H(3)	1.0124
O(3)-N(4)	1.4513	C(7)-C(8)	1.3417
O(4)-N(4)	1.3573	C(8)-C(12)	1.4605
O(5)-N(3)	1.4872	C(8)-C(9)	1.4611
O(6)-N(4)	1.4799	C(9)-C(10)	1.3365
O(7)-C(11)	1.2200	C(1)-H(11)	1.0823
O(8)-C(12)	1.2199	C(2)-H(12)	1.0826
N(1)-C(1)	1.5152	C(4)-H(10)	1.0828
N(1)-C(5)	1.3227	C(5)-H(9)	1.0834
N(2)-C(6)	1.3191	C(6)-H(7)	1.0836
N(2)-C(10)	1.5192	C(7)-H(8)	1.0732
N(5)-C(11)	1.3197	C(9)-H(5)	1.0786
N(6)-C(12)	1.3200	O(9)-H(13)	0.9475
C(1)-C(2)	1.4548	O(9)-H(14)	0.9584
C(2)-C(3)	1.3388	C(10)-H(6)	1.0843

The value of the distance between the bonds Zn(1)−O(1), Zn(1)−O(2), Zn(1)−O(3), Zn(1)−O(4) va Zn(1)−N(1), Zn(1)−N(2) in the complex is corresponding to 1.9737Å, 20082Å, 1,9751Å, 2,0088Å va 2,0013Å, 1,9984Å accordingly (Fig.5., Tab.2.). It can be seen that the valence angles of the bonds O(1)−Zn(1)−O(2), O(1)−Zn(1)−O(3), O(1)−Zn(1)−O(4), O(2)−Zn(1)−O(3) and O(1)−Zn(1)−N(1), O(1)−Zn(1)−N(2) are equal to 64.61^0, 102.42^0, 106.12^0, 106.88^0 and 152.37^0, 90.56^0 , respectively (Fig.6., Tab.3.)

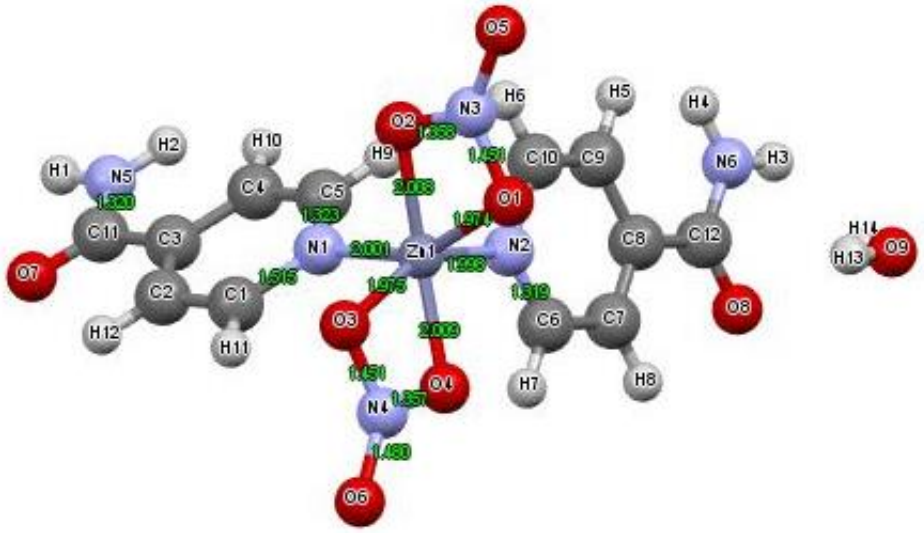

Figure 5. Interatomic bonds and bond length of complex compound

Table 3. Bond angles of a complex compound

Angle	ω, degree	Angle	ω, degree
O(1)-Zn(1)-O(2)	64.61	O(4)-N(4)-O(6)	130.72
O(1)-Zn(1)-O(3)	102.42	N(1)-C(1)-C(2)	118.02
O(1)-Zn(1)-O(4)	106.12	C(1)-C(2)-C(3)	118.46
O(1)-Zn(1)-N(1)	152.37	C(2)-C(3)-C(4)	120.64
O(1)-Zn(1)-N(2)	90.56	C(2)-C(3)-C(11)	119.69
O(2)-Zn(1)-O(3)	106.88	C(4)-C(3)-C(11)	119.68
O(2)-Zn(1)-O(4)	166.73	C(3)-C(4)-C(5)	121.88
O(2)-Zn(1)-N(1)	88.22	N(1)-C(5)-C(4)	121.23
O(2)-Zn(1)-N(2)	102.48	H(1)-N(5)-H(2)	124.21
O(3)-Zn(1)-O(4)	64.63	C(11)-N(5)-H(2)	123.24
O(3)-Zn(1)-N(1)	89.71	C(11)-N(5)-H(1)	126.32
O(3)-Zn(1)-N(2)	150.63	C(12)-N(6)-H(4)	126.34
O(4)-Zn(1)-N(1)	101.52	C(12)-N(6)-H(3)	120.36
O(4)-Zn(1)-N(2)	86.57	N(2)-C(6)-C(7)	119.72
N(1)-Zn(1)-N(2)	90.63	H(3)-N(6)-H(4)	120.23
Zn(1)-O(1)-N(3)	97.62	C(6)-C(7)-C(8)	123.63
Zn(1)-O(2)-N(3)	99.30	C(9)-C(8)-C(12)	119.68
Zn(1)-O(3)-N(4)	97.51	C(7)-C(8)-C(12)	119.67
Zn(1)-O(4)-N(4)	99.28	C(7)-C(8)-C(9)	120.65
Zn(1)-N(1)-C(1)	119.49	C(8)-C(9)-C(10)	119.61
Zn(1)-N(1)-C(5)	120.74	N(2)-C(10)-C(9)	119.27
C(1)-N(1)-C(5)	119.77	O(7)-C(11)-C(3)	120.01
Zn(1)-N(2)-C(6)	119.65	O(7)-C(11)-N(5)	119.18
Zn(1)-N(2)-C(10)	120.45	N(5)-C(11)-C(3)	126.12
C(6)-N(2)-C(10)	119.86	N(6)-C(12)-C(8)	127.16
O(1)-N(3)-O(2)	98.48	O(8)-C(12)-N(6)	119.24
O(1)-N(3)-O(5)	130.76	O(8)-C(12)-C(8)	120.01
O(2)-N(3)-O(5)	130.76	N(1)-C(1)-H(11)	122.32
O(3)-N(4)-O(4)	98.58	C(2)-C(1)-H(11)	122.34
O(3)-N(4)-O(6)	130.71	C(1)-C(2)-H(12)	121.76

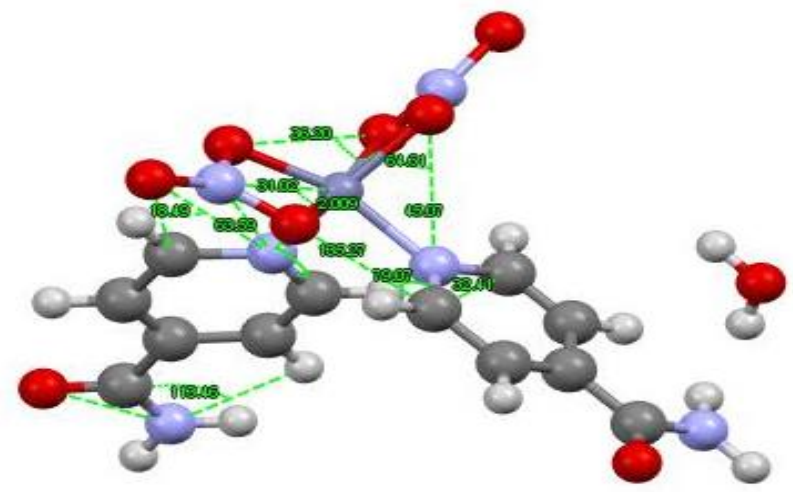

Figure. 6. Sizes of the bond angles of the complex compound

The structure of the complex compound was also previously studied by XAFS (X-ray absorption fine structure) analysis. According to the results of the analysis, Due to the hydrogen bonds C(6)--- H(7)... O(4), C(5)--- H(9)... N(2), C(1)---H(11)...O(3) with the participation of nitrate residues and nicotinamide molecules in the complex, It is stable due to the formation of a two-dimensional layer parallel to the *bc* plane (Fig.7).

Table 4. Hydrogen bonds in the crystal structure (A˚)

Bonds D−H···A	Distance, Å			Angle	Coordinates of the atom,
	D−H	N···A	D···A	D−H···A, deg.	A
			$[C_{12}H_{14}N_6O_9Zn]$		
C(6)--- H(7)... O(4)	1.0800	1.9900	2.6731	118.00	1-x,1-y,1-z
C(5)--- H(9)... N(2)	1.0800	2.5500	3.0045	105.00	2-x,2-y,-z
C(1)---H(11)...O(3)	1.0800	2.2500	2.8263	111.00	x,-1+y,z

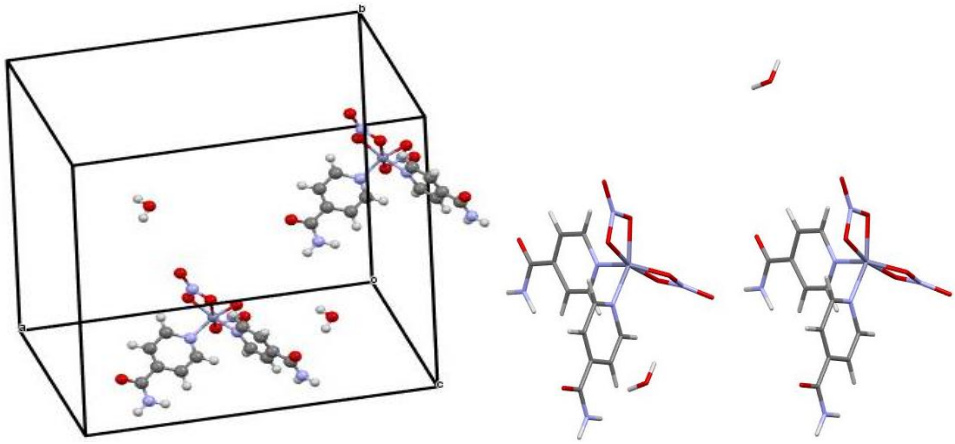

Figure. 7. Location projection of the complex compound in the crystal cell

Conclusion

Based on the changes in values of valence vibrations of functional groups and the values of chemical shifts of magnetic resonance signals by infrared and ^{1}N NMR spectroscopy methods, it was determined that isonicotinamide is coordinated monodentately through the nitrogen atom of the pyridine ring and bidentately coordinated through zinc nitrate oxygen atoms.

The structure proposed by IR and NMR was confirmed by X-ray. In the synthesized complex, it was found that the water molecule is located in the outer sphere.

Scientific Ethics Declaration

The authors declare that the scientific ethical and legal responsibility of this article published in EPSTEM journal belongs to the authors.

Acknowledgements or Notes

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