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Synthesis and Spectroscopic (IR) Studies of Complex Compounds of Some 3D Metals with N,N-dimethylformamide

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Abstract. Complex compounds of sulphates and chlorides of cobalt (II), nickel (II), copper (II), manganese (II), cadmium (II) and zinc with N, N-dimethylformamide, as well as mixed acidoligand complex compounds of cobalt (II), nickel (II), manganese (II) and copper (II) with N,N-dimethylformamide were synthesized.

In order to study the rules of coordination of N,N-dimethylformamide and thiocyanate groups, IR spectra of synthesized complex compounds were studied. Based on the study of IR spectra, it was found that in all synthesized complex compounds, the coordination of the N,N-dimethylformamide molecule with complexing metals is carried out by the oxygen atom of the carbonyl group.

For sulfate and chloride compounds of cobalt (II), nickel (II) and manganese (II) with N, N-dimethylformamide, an octahedral structure of synthesized complexes is proposed, and in the case of copper (II) and cadmium, a tetrahedral structure.

Keywords: bimetallic complex compounds; N,N-dimethylformamide (DMF); spectroscopy (IR).

Introduction

In the middle of the 20th century, methods were developed for the synthesis of thiocyanate and seleno-

cyanate complex compounds using non-aqueous solvents. The use of non-aqueous solvents (methanol, ethanol) makes it possible to carry out such reactions that would be impossible in aqueous solutions.

A procedure was developed for the synthesis of complex compounds containing both thiocyanate and selenocyanate groups, as well as ligands of the 2,2'-dipyridyl (α -Dipy), 1,10-phenanthroline (Phen), ammonia, pyridine (Py), 2-aminopyridine (Ampy), 4,4'-dipyridyl (γ -Dipy), formamide (FA), methylformamide (MFA), N,N-dimethylformamide (DMF), dimethylsulfoxide (DMSO), benzoylhydrazide (BH), urotropine (Ur).

Complex compounds have been synthesized and studied: $M(NCS)_2 \cdot 4Py$ ($M=Mn, Fe, Co, Cd, X=O, S, Se$); $M(NCS)_2 \cdot 2Py$ ($M=Co, Cu, Zn, X=O, S, Se$).

In complex compounds $[M(NCS)_2 \cdot 4DMF]$ (where $M = Mn, Fe, Co, Ni, X = S, Se, DMF = N,N$ -dimethylformamide), the structure of the XCN groups is linear. The coordination of DMF molecules is carried out by the oxygen atom of the carbonyl group. Complex compounds have an octahedral structure. Coordination of NCS groups with metals is carried out by the nitrogen atom, and they are in the trans position with respect to each other [1].

The authors found that the use of non-aqueous solvents in the synthesis of complex compounds increases the stability of pseudohalide compounds in solutions and facilitates the isolation of mixed-ligand complex compounds in the solid state.

Main Part

EXPERIMENTAL

Chemicals

Chlorides of cobalt (II), nickel (II), copper (II) and manganese (II), rhodanide potassium and N,N-dimethylformamide were used as the starting reagents. Reagents correspond to the trademark "ch.d.a". Ethanol, methanol, dimethylformamide, dimethyl sulfoxide, acetone and benzene were used as high quality solvents. Solvents were dried by standard methods and distilled under an inert atmosphere.

Analytical methods

The Infrared spectra of the prepared complexes were obtained by FTIR-Fur-spectrometer TENSOR II (range 400-4000 cm^{-1} , accuracy of measurements 0.5 cm^{-1} .) using KBr as the matrix in the range 400-4000 cm^{-1} . Polystyrene was used as the standard to calibrate the spectrophotometer. Contribution of the elements determined with Scanning electron microscopy (SEM) Tescan Vega 3. An electro thermal melting point apparatus was used for the determination of the melting or decomposition points of the complexes.

Results and discussion

Synthesis Coordination compounds of metals with N, N-dimethylformamide (DMF)

Complex compounds synthesized: $[CoSO_4 \cdot 4DMF] \cdot 0.5H_2O$ (pink powder), $NiSO_4 \cdot 4DMF$ (green powder), $[ZnSO_4 \cdot 4DMF] \cdot 2H_2O$ (white powder), $CuSO_4 \cdot 4DMF$ (yellow powder), $[CoCl_2 \cdot 4DMF] \cdot 2H_2O$ (pink crystals), $[NiCl_2 \cdot 4DMF] \cdot 2H_2O$ (green crystals), $CdCl_2 \cdot 2DMF$ (white crystals) $[MnCl_2 \cdot 4DMF] \cdot 2H_2O$ (light pink crystals), $[CuCl_2 \cdot 4DMF] \cdot 2.5H_2O$ (yellow crystals).

The synthesized compounds are stable in air, can be stored in a closed vessel for a long time, readily soluble in water, ethanol, acetone; insoluble in ether [2-5].

Synthesis of mixed acidoligand coordination compounds of metals with N,N-dimethylformamide (DMF)

The mixed acidoligand coordination compounds of cobalt (II), nickel (II), copper (II), manganese (II) and cadmium (II) with N, N-dimethylformamide were synthesized. Chlorides of the corresponding metals and potassium thiocyanate, all compounds of the analytical grade were used as starting materials. Ethyl alcohol was chosen as the solvent.

Ethanol solutions of chlorides of the corresponding metals $MeCl_2$ ($Me = Co$ (II), Ni (II), Mn (II), Cu (II), and Cd (II)) were mixed with an ethanolic solution of potassium thiocyanate at a 1:1 ratio. After separation of the precipitated precipitate, purified N,N-dimethylfor-

amide was added dropwise to the filtrate in a molar ratio of 1:4. The solution was slightly evaporated and allowed to stand for several days until the precipitate was isolated.

A few days later, a precipitate formed from the solutions, which was washed with small portions of cold ethanol and dried in a vacuum desiccator over CaCl_2 .

Complex compounds synthesized: $\text{Mn}(\text{SCN})\text{Cl}\cdot 4\text{DMF}$ (light pink powder), $\text{Cu}(\text{SCN})\text{Cl}\cdot 4\text{DMF}$ (gray powder), $\text{Ni}(\text{SCN})\text{Cl}\cdot 4\text{DMF}$ (greenish yellow powder), $\text{Cd}(\text{SCN})\text{Cl}\cdot 4\text{DMF}$ (white powder).

The synthesized compounds are stable in air. Stored in a closed container for a long time. Well soluble in water, ethanol, acetone; insoluble in ether.

Infrared spectra

Infrared absorption spectra of metal complexes with N, N-dimethylformamide (DMF)

In order to establish the coordination rule of N,N-dimethylformamide (DMF) molecules, H_2O molecules, and SO_4^{2-} and Cl^- groups, the IR absorption spectra of the synthesized compounds were studied. The assignment of the found basic vibration frequencies of coordinated molecules of N,N-dimethylformamide (DMF) was established on the basis of a theoretical analysis of the normal vibration frequencies of the corresponding uncoordinated molecules.

As is known from the literature, in the IR spectra of tertiary amides, in contrast to primary or secondary amides, only one absorption band of the $\text{C}=\text{O}$ group (the so-called “Amide-I”) appears in the region of $1670\text{-}1630\text{ cm}^{-1}$ [6-8].

The spectra of the complexes with N,N-dimethylformamide show low-intensity splitting into several components in the range of $2850\text{-}3000\text{ cm}^{-1}$. They refer to stretching vibrations of the methyl group $\nu(\text{CH}_3)$.

In the spectra of all studied complexes, intense bands were found in the range of $\sim 1640.98\text{-}1642.28\text{ cm}^{-1}$. In the spectra of the synthesized complexes, the vibration frequencies of the carbonyl group are reduced by $\sim 21.7\text{-}25.3\text{ cm}^{-1}$ in comparison with the non-associated ligand.

A medium-intensity singlet band corresponding to the “amide III” vibration in the spectra of complexes with N, N-dimethylformamide appears at $\sim 1431.09\text{-}1450.05\text{ cm}^{-1}$ both for the uncoordinated molecule and in the synthesized complexes.

Separately, it should be noted that, upon coordination of the N,N-dimethylformamide molecule, a slight increase in vibration frequencies is observed, which, presumably, refers to bending vibrations with the participation of the —N—C=O group. For a free, uncoordinated molecule of N,N-dimethylformamide, these frequencies were found in the $\sim 657\text{ cm}^{-1}$ region, and upon coordination they are shifted to region $\sim 675\text{-}682\text{ cm}^{-1}$, as, for example, in the case of a $\text{trans- Me}(\text{NCX})_2\cdot 4\text{DMF}$], where $\text{Me} = \text{Mn, Fe, Co, Ni,}$ and $\text{X} = \text{S, Se}$.

All the observed spectral changes in accordance with indicate that, in the complexes under study, the coordination of the N, N-dimethylformamide molecule with the complexing atom occurs via the oxygen atom, which is in full agreement with the data of quantum chemical studies [9].

Taking into account the inner sphericity of the N,N-dimethylformamide molecules, one can assume the octahedral structure of the complexes under study in the case of cobalt (II) and nickel (II) compounds and the tetrahedral structure for the copper (II) complex.

In the IR spectra of the synthesized complexes in the range of $1050\text{-}1060\text{ cm}^{-1}$ and 1170 cm^{-1} , identification of the absorption frequencies of SO_4^{2-} groups is difficult, which is caused by the existence of vibration frequencies $\sim 1100\text{-}1150\text{ cm}^{-1}$. As for the SO_4^{2-} group, the existence of vibration frequencies in the range of $\sim 560, \sim 600, \sim 980$ and $\sim 110\text{-}1120\text{ cm}^{-1}$ ($[\text{CoSO}_4 \cdot 4\text{DMF}] \cdot 0.5\text{H}_2\text{O} - 542.29, 606.60$ and 1140.91 cm^{-1} , $\text{NiSO}_4 \cdot 4\text{DMF} - 607.39\text{ cm}^{-1}$, $[\text{ZnSO}_4 \cdot 4\text{DMF}] \cdot 2\text{H}_2\text{O} - 607.66\text{ cm}^{-1}$) makes it possible to judge about the bidentate nature of sulfato groups.

If we take into account the bidentate character of sulfate groups and the intrasphericity of N, N-dimethylformamide molecules, we can assume the octahedral structure of the synthesized complex compounds [10].

Table 1.

Some vibrational frequencies (cm⁻¹) found in the IR spectra of metal complexes with N,N-dimethylformamide

Complexes	$\nu_{as}(\text{CH}_3)$	$\nu(\text{CO})$	$\nu(\text{CN})$	$\nu_{as}(\text{CN})$	$\nu(\text{CH}_3)$	$\nu_s(\text{CN})$
DMF	3070.27m 2931.41m	1666.28 vs	1496.56m 1442.56	1257.42s	1095.42s	864.00w
[CoSO ₄ ·4DMF]·0.5H ₂ O	3182.91w	1506.00m		1140.91s	1019.08s	879.51m
NiSO ₄ ·4 DMF	3188.93m	1635.14m			1083.78vs	832.60s
CuSO ₄ ·4 DMF	3145.50w	1665.70m			1066.88vs	870.58s
[ZnSO ₄ ·4 DMF]·2H ₂ O	3234.60s	1654.03w 1625.29w			1074.93vs	

* vs = very strong; s = strong; ms = medium strong; m = medium; w = weak; vw = very weak

IR absorption spectra of mixed acidoligand coordination compounds of metals with N, N-dimethylformamide

As the analysis of the IR spectra of the mixed-ligand complex compounds synthesized by us shows, the vibration frequencies of the DMF molecules in comparison with the uncoordinated molecule lie in the range of ~1643.78 - 1649.22 cm⁻¹. That is, they decrease by ~17.06-22.5 cm⁻¹, which indicates the coordination of N, N-dimethylformamide molecules with the metal atom through the oxygen atom of the carbonyl group.

When studying the IR spectra of the synthesized complexes, the frequencies of stretching vibrations of the thiocyanate group SCN⁻ were determined: $\nu(\text{CN})$ ~2056.9-2151.7 cm⁻¹, $\nu(\text{CS})$ ~ 740.5-789.3 cm⁻¹, and $\nu(\text{NCS})$ ~ 469.7-477.1 cm⁻¹ respectively.

The presence of the stretching vibration frequencies of the $\nu(\text{C}\equiv\text{N})$ group in the range 2076.40-2152.21 cm⁻¹ indicates the coordination of thiocyanate ions with the nickel atom via the nitrogen atom (the isocyanate structure M←NCS is realized).

As for the complex compound Cu(SCN)Cl·4DMF, the vibration frequency of the $\nu(\text{CN})$ group was found in the region of 2152.21 cm⁻¹, which indicates the presence of the bridging function of the thiocyanate ion. A simultaneous increase in the stretching vibration frequencies of the $\nu(\text{CS})$ group to 820.0 cm⁻¹ and a decrease in the frequency of the (NCS) stretching vibrations to 426.72 cm⁻¹ also confirms the assumption of the presence of thiocyanate bridges.

Table 2.

Some characteristics of vibration frequencies of mixed-ligand complex compounds of metals with N,N-dimethylformamide

complexes	$\nu(\text{CO})$	$\nu(\text{CN})$	$\nu_{as}(\text{CN})$	$\nu(\text{CN})$	$\nu(\text{CS})$	$\delta(\text{SCN})$
Mn(SCN)Cl·4DMF	1643.78s	1443.0 s	1257.42w	2076.40vs		486.36w
Cu(SCN)Cl·4DMF	1649.22vs	1405.64vs	1250.30m	2152.21s	820.0m	426.72w

* vs = very strong; s = strong; ms = medium strong; m = medium; w = weak; vw = very weak

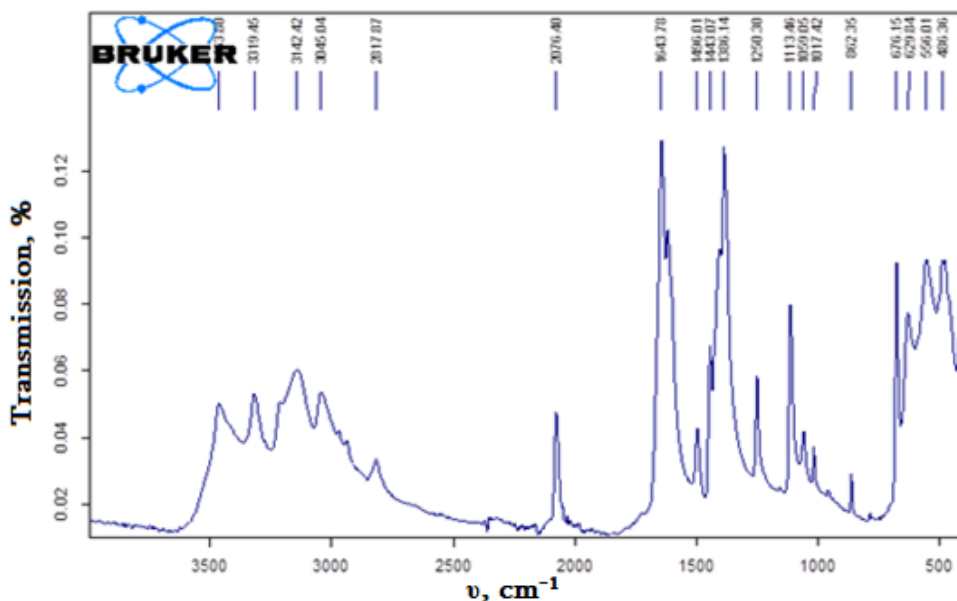


Figure 1. IR absorption spectrum of Mn(SCN)Cl·4DMF in vaseline oil (400-4000 cm⁻¹)

Thermogravimetric Analysis

Thermogravimetric and calorimetric studies of the thermal stability of the synthesized complex metal compounds with N,N-dimethylformamide have been carried out. Analysis of the data obtained showed that the main thermal transformations of the studied compounds occur up to 500 °C.

Exothermic processes of oxidation of thermolysis products, completing the thermal dissociation of the complexes, begin below 500°C and end at 600°C. On the whole, the thermolysis of the studied complex compounds is a rather complex multistep process. The decomposition of some complexes begins already at a temperature of ~60.6-110.7°C. Thermal analysis shows that in this temperature range, the elimination of outer-sphere water molecules occurs.

Thermograms of the complexes, as a rule, have a similar character and, with a few exceptions, proceed in 3-4 stages, while the decomposition of some of the complexes proceeds in a series of difficult-to-separate stages.

Thermal decomposition of complex compounds [CoCl₂·4DMF]·2H₂O, CdCl₂·2DMF, NiSO₄·4DMF, [CoSO₄·4DMF]·0.5H₂O, [ZnSO₄·4DMF]·2H₂O and

CuSO₄·4DMF occurs, as a rule, in three - four stages and has a similar character. The onset of decomposition at a temperature of 60–166° C is accompanied by small endothermic effects, which corresponds to the removal of outer-sphere water molecules. Weight loss 7.21-10%. In some cases, weight loss corresponds to the removal of 1-2 moles of DMF. At a temperature of 238.7-314.9°C, a maximum weight loss of 16.93-60.06% is observed. The organic ligand is degraded. In the temperature range 460.0-495.30°C, the oxidation of thermolysis products occurs. At a temperature of 542.8-565.2°C, the destruction of complex compounds is completed. Thermolysis products are oxides of the corresponding metals.

As for the complex compounds [NiCl₂·4DMF]·2H₂O, [MnCl₂·4DMF]·2H₂O and [CuCl₂·2DMF]·2.5H₂O, the decomposition of these complexes is accompanied by a number of endo- and exo-effects. In the temperature range 97.3-110.70 °C, the outer-sphere water molecules are removed and the organic ligand is destroyed. Above a temperature of 251.5-460.0 °C, the thermolysis products are oxidized. At temperatures around 460.0 °C, the destruction of complex compounds is completed. Thermolysis products are oxides of the corresponding metals.

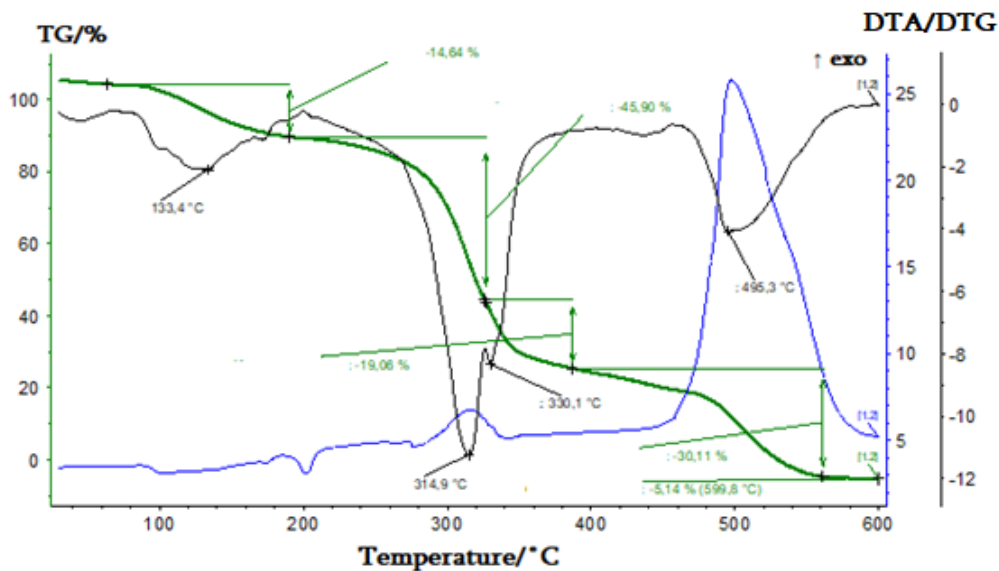


Figure 2. Thermogram curve of the complex compound $[CoCl_2 \cdot 4DMF] \cdot 2H_2O$

Conclusion

Complex compounds of sulfates and chlorides of cobalt (II), nickel (II), copper (II), manganese (II), cadmium (II) and zinc with N, N-dimethylformamide have been synthesized, mixed acidoligande and double thiocyanate complex compounds of cobalt (II), nickel (II), manganese (II) and copper (II) with N,N-dimethylformamide.

In order to study the coordination rules of N, N-dimethylformamide and thiocyanate groups, the IR spectra of the synthesized complex compounds were

studied. Based on the study of IR spectra, it was found that in all synthesized complex compounds, the coordination of the N, N-dimethylformamide molecule with the complexing metals is carried out by the oxygen atom of the carbonyl group, which is in full agreement with the data of the quantum chemical calculations.

For sulfate and chloride compounds of cobalt (II), nickel (II), and manganese (II) with N, N-dimethylformamide, one can assume the octahedral structure of the synthesized complexes, and in the case of copper (II) and cadmium, tetrahedral.

Table 3.

Thermoanalytical results (TG,DTG,DSC) of the synthesized complex metal compounds with N,N-dimethylformamide

Complex	Stage	Peak temperature (°C)	Peak nature/ kJ/mol H ₂ O	Mass loss, %	Loss	Final residue
$[CoCl_2 \cdot 4DMF] \cdot 2H_2O$	I	133.4°C	endo	14.64	Removal of 1 moles of DMF	CoO, Co ₂ O ₃
	II	314.9°C	exo	45.90	Removal of 3 moles of DMF	
	III	495.3°C	exo	30.11	Oxidation of thermolysis	

[NiCl ₂ ·4DMF]·2H ₂ O	I	60.6 °C	endo	1.08	Removal of outer-sphere water molecules and destruction of organic ligand	NiO, Ni ₂ O ₃
	II	115.7 °C	endo	16.36		
	III	137.5 °C	endo	10.24		
	IV	190.8 °C	endo	11.28		
	V	251.5 °C	exo	5.39	Oxidation of thermolysis	
[MnCl ₂ ·4DMF]·2H ₂ O	I	110.7 °C	endo	2.12	Removal of outer-sphere water molecules and destruction of organic ligand	Mn ₂ O ₃
	II	136.5 °C	endo	12.39		
	III	147.6 °C	endo	2.79		
	IV	165.5 °C	exo	4.88		
	V	205.8 °C	endo	14.91	Oxidation of thermolysis	
	VI	315.6 °C	endo	12.30		
[CuCl ₂ ·2DMF]·2.5H ₂ O	I	97.3°C	endo	17.05	Removal of outer-sphere water molecules and destruction of organic ligand	Cu ₂ O CuO
	II	178.5°C	endo	7.14		
	III	261.4°C	endo	1692		
	IV	460.0°C	exo	11.82	Oxidation of thermolysis	
CdCl ₂ ·2DMF	I	166.6 °C	endo	10.95	Removal of 1 moles of DMF	CdO
	II	238.4 °C	endo	12.54	Removal of 3 moles of DMF	
	III	565.2 °C	exo	1.47	Oxidation of thermolysis	
[CoSO ₄ ·4DMF]·0.5H ₂ O	I	71.1 °C	endo	1.18	Removal of water molecules	CoO, Co ₂ O ₃ , CoS
	II	136.0°C	endo	3.15	Organic ligand destruction	
	III	361.4 °C	endo	12.34	Oxidation of thermolysis	
NiSO ₄ ·4DMF	I	151.8 °C	endo	16.09	Removal of 2 moles of DMF	NiO Ni ₂ O ₃
	II	187.1°C	endo	15.50	Organic ligand destruction	
	III	361.4 °C	экзо	7.15	Oxidation of thermolysis	
[ZnSO ₄ ·4DMF]·2H ₂ O	I	111.3 °C	endo	22.94	Organic ligand destruction	ZnO
	II	289.6 °C	endo	9.38	Oxidation of thermolysis	
	III	129.1°C	endo	13.58	DMF destruction	
	IV	260.7 °C	endo	8.39	Oxidation of thermolysis	

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ზოგიერთი 3d - მეტალის კოორდინაციული ნაერთების სინთეზი N, N-დიმეთილფორმამიდთან და იწ სპექტროსკოპული კვლევა

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ანოტაცია. სინთეზირებულია კობალტის (II), ნიკელის (II), სპილენძის (II), მანგანუმის (II), კადმიუმის (II) და თუთიის სულფატური და ქლორიდული კომპლექსური ნაერთები N,N-დიმეთილფორმამიდთან. აგრეთვე სინთეზირებულია კობალტის (II), ნიკელის (II), სპილენძის (II) და მანგანუმის (II) შერეულიგანდიანი კოორდინაციული ნაერთები N,N-დიმეთილფორმამიდთან.

N,N-დიმეთილფორმამიდისა და თიოცინატ ჯგუფების კოორდინაციის წესის დადგენის მიზნით შესწავლილია სინთეზირებული ნაერთების იწ სპექტრები. დადგენილია, რომ ყველა სინთეზირებულ ნაერთში N,N-დიმეთილფორმამიდის მოლეკულის კოორდინაცია ხორციელდება კარბონილის ჯგუფის ჟანგბადის ატომის საშუალებით.

N,N-დიმეთილფორმამიდთან კობალტის(II), ნიკელის(II) და მანგანუმის(II) სულფატური და ქლორიდული კომპლექსების შემთხვევაში აგებულია ოქტაედრულია, ხოლო სპილენძის(II)-ისა და კადმიუმის შემთხვევაში ტეტრაედრული.

საკვანძო სიტყვები: ბიმეტალური კომპლექსური ნაერთები; N,N-დიმეთილფორმამიდი (DMF); იწ სპექტროსკოპია.

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