BARIUM NITRATE MIXED-AMIDE COMPLEX COMPOUNDS

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Annotatsiya: Bariy nitratning formamid, atsetamid, karbamid bilan aralash amidli koordinatsion birikmalari sintezi amalga oshirildi. Formamid, karbamid, atsetamid va nitrat fragmenti molekulalarining tarkibi, individualligi, koordinatsiya usullari aniqlangan.Tebranish spektroskopiyasi va termik tahlil usullari yordamida organik ligandlarning koordinatsiya usullari, markaziy ionning atrof-muhiti va sintez qilingan birikmalarning termik xatti-harakatlari isbotlangan. Bariy nitrat, formamid, karbamid, atsetamid va $Ba(NO_3)_2 \cdot HCONH_2 \cdot CH_3CONH_2$, $Ba(NO_3)_2 \cdot HCONH_2 \cdot CO(NH_2)_2$ tarkibli kompleks birikmalarning tekisliklararo masofalari va nisbiy intensivliklarini taqqoslash shuni ko‘rsatdiki, yangi koordinatsion birikmalar bir-biridan, shuningdek, dastlabki komponentlardan farq qiladi, shuning uchun birikmalar individual kristall panjaraga ega.

Kalit so‘zlar: Aralash amidli kompleks birikmalar, koordinatsiya, markaziy atom, sintez, element tahlili, koordinatsiya usullari, termik xatti-harakatlar, individuallik, tarkib, fizik-kimyoviy tahlil usullari, IQ yutilish spektrlari, rentgen fazali tahlil.

Аннотация: Осуществлен синтез смешанноамидных координационных соединений нитрата бария с формамидом, ацетамидом, карбамидом. Установлены состав, индивидуальность, способы координации молекул формамида, карбамида, ацетамида и нитратного фрагмента. Методами колебательной спектроскопии и термического анализа доказаны способы координации органических лигандов, окружение центрального иона и термическое поведение синтезированных соединений.Сравнением межплоскостных расстояний и относительных интенсивностей нитрата бария, формамида, карбамида, ацетамида и комплексных соединений составов $Ba(NO_3)_2 \cdot HCONH_2 \cdot CH_3CONH_2$, $Ba(NO_3)_2 \cdot HCONH_2 \cdot CO(NH_2)_2$ показано, что новые координационные соединения отличаются между собой, а также от исходных компонентов, следовательно, соединения имеют индивидуальную кристаллическую решетку.

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Ключевые слова: Смешанноамидные комплексные соединения, координация, центральный атом, синтез, элементный анализ, способы координации, термическое поведение, индивидуальность, состав, физико-химические методы анализа, ИК-спектры поглощения, рентгенофазовый анализ.

Abstract: Mixed amide coordination compounds of barium nitrate with formamide, acetamide, and carbamide were synthesized. The composition, individuality, and coordination methods of formamide, carbamide, acetamide, and nitrate fragment molecules have been established. By comparing the interplanar distances and relative intensities of barium nitrate, formamide, carbamide, acetamide, and complex compounds of the compositions $Ba(NO_3)_2 \cdot HCONH_2 \cdot CH_3CONH_2$, $Ba(NO_3)_2 \cdot HCONH_2 \cdot CO(NH_2)_2$, it has been shown that the new coordination compounds differ from each other, as well as from the initial components, therefore, the compounds have an individual crystal lattice.

Keywords: Mixed amide complex compounds, coordination, central atom, synthesis, elemental analysis, coordination methods, thermal behavior, individuality, composition, physicochemical analysis methods, IR absorption spectra, X-ray phase analysis.

Introduction.

Formide, acetamide, carbamide molecules, and nitric acid anion are representatives of bioorganic compounds. Their coordination compounds with certain metals exhibit high biological activity for plant growth [1,2].

Therefore, conducting research on the synthesis of new coordination compounds with biologically active substances is highly relevant. There is no information in the literature about the mixed amide complex compounds of barium nitrate with formamide, acetamide, and carbamide.

Literature review. For the synthesis of coordination compounds, both $Ba(NO_3)_2$ and the brands "a.c.d." and "a.c.d." were used. The synthesis of the corresponding nitrates of metals was carried out according to [3,4]. Acetamide (CH_3CONH_2), formamide ($HCONH_2$), and urea ($CO(NH_2)_2$) were used as ligands. The quantity of metals in the synthesized compounds was determined on a new AA 300 atomic absorption spectrophotometer from Analytik Jena AG (Germany) [5]. Nitrogen, hydrogen, and carbon were determined using Carlo Erb's EA-1108 element analyzer [6].

Determine the individuality of the synthesized compounds, X-ray diffraction patterns were taken on a DRON-3M unit with a Su-anticathode [7]. Tables [7] were used for calculating interplanar distances, and the relative intensity of the I/I1 line was determined as a percentage of the most pronounced reflex at the maximum. The IR absorption spectra were recorded in the region of 400-4000 cm-1 on the IR

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Fourier System-2000 spectrophotometer of the company "Perkin Elmer" using the method of pressing samples with K.

Research methodology. Thermal analysis was carried out on a derivatograph of the F.Paulik-J.Paulik-L.Erdey [8] system at a speed of 9 degrees/min and a weight of 0.1 g on the sensitivity of galvanometers T-900, TG-200, DTA, DTG-1/10. Recording was carried out under atmospheric conditions. The holder was a 7 mm diameter platinum crucible without a lid. Al_2O_3 was used as the standard.

Synthesis of coordination compounds of barium nitrates with amides was carried out by mechanochemical (solid-phase) method. Mechanochemical interaction of the initial components was carried out by intensive grinding of a mixture of barium nitrate: amide 1: amide 2 in lightning ratios 1:1:1 for 30 minutes at room temperature in a ball mill with a working body (mill volume 100 ml). Duration of one stir 30 sec. Three such mixings constitute one cycle. After each cycle, the apparatus rests for 2-3 seconds. Periodically, after each cycle, samples were taken for X-ray phase and derivatographic analysis. This operation was repeated 18-20 times. After 17-19 repetitions, no changes were observed in the diffractograms and derivatograms of the samples, which indicates the individuality of the obtained compounds.

A mixed amide complex compound of the composition $Ba(NO_3)_2 \cdot HCONH_2 \cdot CH_3CONH_2 \cdot 0.5H_2O$ was synthesized by intensive mixing of 1.3056 g (0.005 mol) barium nitrate with 0.2252 g (0.005 mol) formamide and 0.2954 g (0.005 mol) acetamide in a ball mill at room temperature for 0.10-0.15 hours. Product yield is 92.5%.

A mixed amide complex compound of the composition $Ba(NO_3)_2 \cdot HCONH_2 \cdot CO(NH_2)_2 \cdot H_2O$ was obtained by mixing 1.3056 g (0.005 mol) $Ba(NO_3)_2$ with 0.9252 g (0.005 mol) formamide and 0.3004 g (0.005 mol) urea in a ball mill for 0.10-0.15 hours. Product yield is 93.2%.

Discussion and results. Comparison of interplanar distances and relative intensities of barium nitrate, formamide, acetamide, carbamide and complexes based on them showed that the new coordination compounds differ from each other, as well as from the initial components, consequently, the compounds have an individual crystal lattice. The results are presented in Table 1.

1-Table.

Interplanar distances and relative intensities of lines of formamide, acetamide, urea with magnesium succinate.

Compound	d,Å	I,%	d,Å	I,%	d,Å	I,%	d,Å	I,%	d,Å	I,%
	18,35	75	6,38	33	3,52	25	2,61	29	2,02	29
	16,94	92	6,24	33	3,49	46	2,58	54	2,01	29

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$HCONH_2$	15,73	67	5,97	33	3,46	50	2,57	54	2,00	50
	15,52	59	5,82	42	3,42	50	2,54	42	1,980	50
	15,30	79	5,71	58	3,36	46	2,53	38	1,975	42
	14,31	50	5,64	42	3,27	71	2,51	33	1,958	29
	13,68	71	5,46	46	3,21	50	2,48	38	1,951	42
	13,27	83	5,30	63	3,18	50	2,46	29	1,921	42
	12,74	88	5,19	67	3,14	33	2,44	29	1,904	58
	12,17	75	4,85	63	3,09	50	2,35	29	1,888	58
	12,11	63	4,77	83	3,03	63	2,34	29	1,882	71
	11,60	42	4,66	58	3,01	46	2,33	29	1,871	71
	11,21	67	4,22	83	2,96	33	2,27	33	1,861	38
	10,55	100	4,15	75	2,95	50	2,24	33	1,854	58
	10,06	25	4,07	75	2,89	33	2,21	33	1,843	54
	9,11	63	4,00	50	2,87	46	2,20	38	1,826	46
	8,82	58	3,96	67	2,84	33	2,16	42	1,819	50
	8,17	38	3,89	63	2,80	29	2,15	42	1,791	50
	8,02	67	3,86	42	2,79	46	2,14	58	1,774	38
	7,74	63	3,82	63	2,76	42	2,11	58	1,754	54
	7,48	33	3,76	71	2,71	42	2,10	50	1,728	50
	7,23	38	3,64	54	2,68	46	2,08	42	1,712	38
	6,90	46	3,58	63	2,66	46	2,06	42		
	6,55	38	3,56	67	2,64	38	2,03	58		
CH_3CONH_2	20,21	6	4,51	4	2,84	83	2,05	1	1,581	6
	18,05	8	4,26	2	2,67	11	2,03	1	1,490	1
	16,60	10	4,03	1	2,56	3	1,984	1	1,427	10
	14,69	9	3,95	1	2,52	2	1,942	5	1,390	1
	12,24	3	3,85	1	2,49	2	1,887	1	1,311	1
	11,42	2	3,70	1	2,36	1	1,849	1	1,259	4
	6,13	5	3,62	1	2,30	7	1,805	3	1,246	1
	5,58	100	3,55	3	2,26	2	1,753	45		
	5,26	8	3,49	13	2,22	3	1,707	2		
	5,01	6	3,25	13	2,15	49	1,611	1		
	4,78	5	3,14	4	2,10	1	1,591	2		
	17,21	2	4,37	2	3,02	12	2,20	4	1,770	2
	16,08	3	3,98	100	2,80	27	2,15	2	1,736	1

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$CO(NH_2)_2$	15,29	3	3,56	10	2,49	42	2,01	1	1,660	5
	13,86	2	3,25	2	2,46	5	1,980	18	1,557	1
	12,59	1	3,14	3	2,33	1	1,827	6		
$Ba(NO_3)_2$ ·$HCONH_2$· CH_3CONH_2·$0,5H_2O$	14,22	14	3,88	14	2,47	10	1,779	21	1,411	8
	12,59	11	3,74	13	2,36	100	1,759	10	1,399	9
	10,86	10	3,62	11	2,31	42	1,741	7	1,375	9
	9,80	8	3,55	11	2,22	13	1,723	7	1,369	7
	8,82	14	3,46	11	2,17	13	1,716	9	1,356	7
	7,61	11	3,42	11	2,11	11	1,689	9	1,337	6
	7,35	13	3,33	13	2,08	11	1,665	7	1,327	7
	6,03	10	3,21	17	2,05	10	1,643	7	1,311	11
	5,89	10	3,18	13	2,02	9	1,581	17	1,305	12
	5,66	9	3,02	22	1,989	8	1,576	55		
	5,30	13	2,93	17	1,948	12	1,567	58		
	5,02	14	2,89	14	1,925	11	1,529	10		
	4,91	12	2,77	11	1,890	8	1,510	11		
	4,68	14	2,72	14	1,851	9	1,492	19		
	4,38	12	2,61	9	1,831	14	1,487	16		
	4,21	12	2,58	8	1,819	21	1,471	9		
	3,97	12	2,49	12	1,792	21	1,417	9		
$Ba(NO_3)_2$ ·$HCONH_2$· ·$CO(NH_2)_2$·H_2O	15,19	31	5,68	22	3,01	47	1,875	16	1,515	16
	14,21	24	5,20	19	2,88	29	1,858	14	1,487	29
	13,60	31	4,97	22	2,76	59	1,831	12	1,451	14
	12,59	28	4,75	24	2,66	26	1,807	22	1,436	12
	11,02	21	4,27	35	2,54	35	1,786	24	1,431	16
	10,06	17	4,00	26	2,48	19	1,773	33	1.411	12
	9,80	14	3,90	22	2,44	16	1,729	16	1,400	16
	8,82	16	3,86	28	2,39	36	1,718	17	1,386	14
	8,33	17	3,77	19	2,35	100	1,657	14	1,378	12
	8,02	19	3,68	19	2,30	31	1,625	17	1,365	12
	7,69	19	3,52	22	2,27	26	1,617	28	1,351	12
	7,61	21	3,47	24	2,15	41	1,607	11	1,344	12
	7,35	16	3,42	19	2,08	59	1,602	17	1,340	12
	6,98	16	3,20	28	2,02	16	1,592	17	1,330	14
	6,55	14	3,18	29	1,967	22	1,561	64	1,301	14

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	6,22	16	3,07	19	1,903	14	1,537	14		

2-Table.

Results of elemental analysis of mixed amides coordination compounds of barium nitrate.

Compound	Me,%		N,%		S,%		C,%		H,%	
	Found.	Calc.	Found.	Calc.	Found.	Calc.	Found.	Calc.	Found.	Calc.
$Ba(NO_3)_2 \cdot HCONH_2 \cdot \cdot CH_3CONH_2 \cdot 0{,}5H_2O$	36,66	36,59	14,97	15,05	-	-	9,38	9,62	2,27	2,41
$Ba(NO_3)_2 \cdot HCONH_2 \cdot \cdot CO(NH_2)_2 \cdot H_2O$	35,94	35,67	18,87	18,23	-	-	6,04	6,25	2,93	2,34

As can be seen from Table 2, the composition of the obtained complex is the same as the reduced complexes.

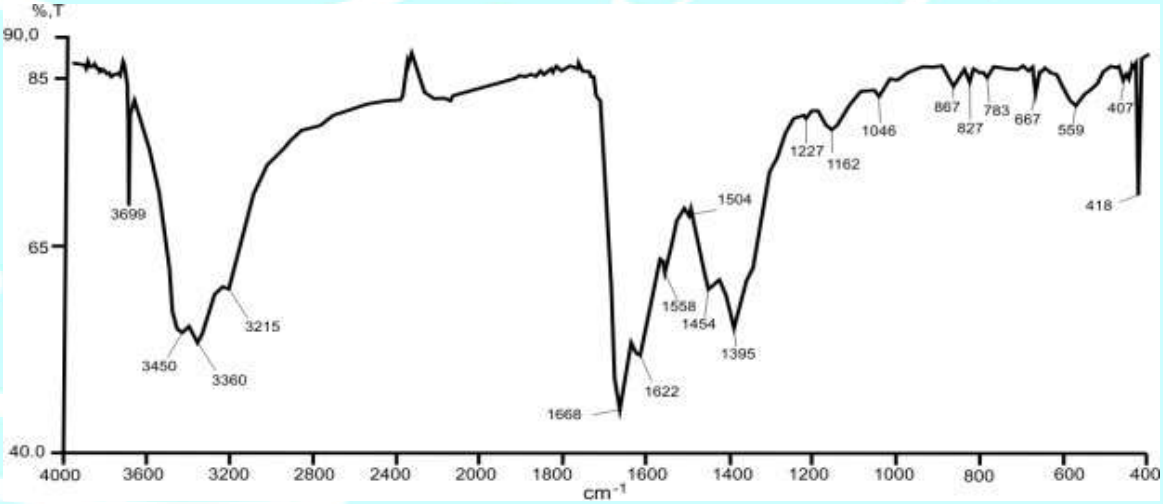


Fig.1. IR absorption spectrum of the mixed amide complex compound of barium nitrate with formamide and acetamide.

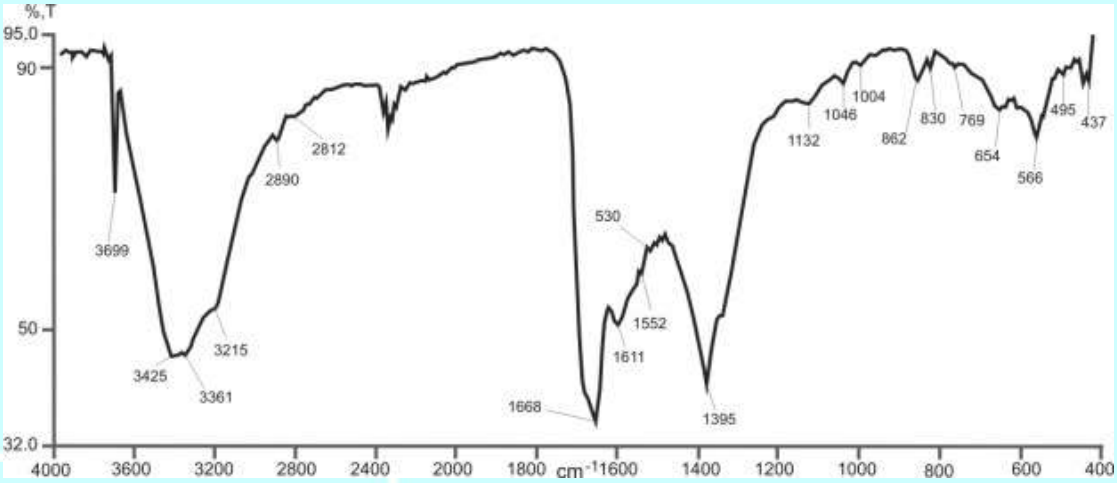


Fig.2. IR absorption spectrum of the mixed amide complex compound of barium nitrate with formamide and carbamide.

In the IR absorption spectrum of the free formamide molecule, two frequencies at 1692 and 1316 cm^{-1} correspond predominantly to the valence vibration of the C=O

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and C-H bonds. With the transition to a coordinated state, i.e., in mixed amide coordination compounds, the value of the C=O frequency decreases by 5-40 cm^{-1}. Meanwhile, the value of the C-H frequency increases by 15-30 cm^{-1}. Such a change in frequency indicates the coordination of formamide molecules with barium ions through the oxygen atom of the carbonyl group (Figure 1 and Figure 2).

Table 3.

Derivatographic data of thermolysis of coordination compounds of barium nitrate with formamide and acetamide.

Compound	Temperature interval of effect, °C	Peak effect, °C	Mass decrease,%	Total mass loss, %	Nature of the effect	Resulting compounds
$Ba(NO_3)_2 \cdot HCONH_2 \cdot CH_3CONH_2 \cdot 0{,}5H_2O$	60-123	120	3,55	3,55	endothermic	$Ba(NO_3)_2 \cdot HCONH_2 \cdot CH_3CONH_2$
	123-150	142	2,21	5,76	endothermic	Thermolysis product
	150-210	192	9,63	15,39	endothermic	Thermolysis product
	210-295	278	18,72	34,11	exothermic	Продукт термолиза
	295-340	308	5,88	39,99	exothermic	Thermolysis product
	340-380	376	6,95	46,94	exothermic	Thermolysis product
	380-420	397	12,16	59,10	exothermic	Thermolysis product
	420-660	620	3,14	62,24	exothermic	Thermolysis product
	660-780	709	1,00	63,24	exothermic	Thermolysis product
	780-890	850	0,93	64,17	exothermic	Thermolysis product
$Ba(NO_3)_2 \cdot HCONH_2 \cdot CO(NH_2)_2 \cdot H_2O$	60-124	97	6,82	6,82	endothermic	$Ba(NO_3)_2 \cdot HCONH_2 \cdot CO(NH_2$

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	124-147	126	2,61	9,43	endothermic	Thermolysis product
	147-188	175	6,52	15,95	endothermic	Thermolysis product
	188-220	200	11,59	27,54	exothermic	Thermolysis product
	220-305	285	9,42	36,96	exothermic	Thermolysis product
	305-358	340	6,52	43,48	exothermic	Thermolysis product
	358-395	390	13,77	57,25	exothermic	Thermolysis product
	395-422	405	0,43	57,78	endothermic	Thermolysis product
	422-478	458	3,35	61,13	endothermic	Thermolysis product
	478-496	488	1,35	62,48	endothermic	Thermolysis product
	496-520	500	2,00	64,48	endothermic	Thermolysis product
	520-720	658	0,40	64,88	endothermic	Thermolysis product
	720-890	803	0,34	65,22	endothermic	Thermolysis product

In Table 3, derivatographic data on the thermolysis of coordination compounds of barium nitrate with formamide and acetamide are presented. The heating curve of a complex compound of the composition $Ba(NO_3)_2 \cdot HCONH_2 \cdot CH_3CONH_2 \cdot 0.5H_2O$ is characterized by four endothermic effects at 120, 142, 192, 850 and six exothermic effects at 278, 308, 376, 397, 620, and 709°C. The appearance of the first of the three endoeffects is associated with the removal of half the water molecule. The nature of other thermal effects is accompanied by the stepwise decomposition of the anhydrous compound. In the temperature ranges of 60-123, 123-150, 150-210, 210-295, 295-340, 340-380, 380-420, 420-660, 660-780, 780-890°C, the mass loss is, respectively, 3.55; 2.21; 9.63; 18.72; 5.88; 6.95; 12.16; 3.14; 1.00; 0.93%. The total mass loss in the temperature range 60-8900C along the TG curve is 64.17%. On the DTA curve, compounds Ba $(NO_3)_2 \cdot HCONH_2 \cdot CO\ (NH_2)_2 \cdot H_2O$ exhibit eight endothermic effects at 97, 126, 175, 40, 5.458, 500, 658, 803°C, and five exothermic effects at 200, 285, 340, 390,

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and 488°C. The appearance of the first endoelectric effect is due to the removal of one water molecule. The nature of other thermal effects is related to the stepwise decomposition and combustion of thermolysis products. Mass loss in the temperature range of 60-124 °C is 6.82%, calculated 6.80%. In the temperature ranges of 124-147, 147-188, 188-220, 220-305, 305-358, 358-395, 395-422, 422-478, 478-496, 496-520, 520-720, 720-880°C, the mass loss is 2.61; 6.52; 11.59; 9.42; 6.52; 13.77; 0.43; 3.35; 1.35; 2.00; 0.40; 0.34%, respectively. The total mass loss in the temperature range of 60-880°C along the TG curve is 65.22%.

Conclusions and suggestions. Thus, a comparison of the interplanar distances and relative intensities of free molecules of formamide, carbamide, acetamide, and mixed amide coordination compounds of barium nitrates showed that they differ significantly from each other, from similar and initial compounds. Consequently, the synthesized coordination compounds have individual crystal lattices.The IR absorption spectra (400-4000 cm^{-1}) of all the obtained compounds were studied. As a result, it was established that in formamide, acetamide, and urea compounds, the coordination of ligands is carried out through the oxygen atom of the carbonyl group.Derivatographic studies of various amide complex compounds of barium nitrate with formamide, acetamide, and carbamide showed that the thermal behavior of the new studied compounds significantly depends on the composition, methods of coordination of amides and the nitrate fragment, and the relative positions of donor atoms around the coordination unit of the barium ion.

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