Coordination Compounds Of Calcium Acetate With Some Ligands

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Abstract. The synthesis of mixed-ligand coordination compounds of calcium magnesium acetates with formamide, acetamide, benzamide, benzoic and nicotinic acid was carried out. The composition, individuality, methods of coordination of amide molecules and fragments of anions of acetic and nicotinic acids have been established. The thermal behavior of the synthesized coordination compounds has been studied. The compositions, temperature ranges of thermal effects, quantitative changes in the mass of stages, and the nature of the final thermolysis product were determined. The most effective concentrations of complex compounds have been established in terms of their effect on the germination energy and germination of cotton seeds in a laboratory experiment.

Keywords: Coordination compounds, synthesis, composition, thermal, physicochemical methods of analysis, biological activity.

One of the urgent problems of modern chemistry is the synthesis of new chemical compounds with effective properties for use in agriculture. Complex compounds of s-, p-, d-metals, which have a number of specific properties, have found wide application in various sectors of the national economy. Electronic, stereochemical, kinetic and thermodynamic characteristics determine the scope and properties of coordination compounds.

Substances containing donor atoms, for example, amides of aliphatic, carboxylic, pyridinecarboxylic acids, in particular, formamide, acetamide, nicotinamide, nicotinic and benzoic acid, contribute to the formation of coordination compounds with metal ions. Anions of organic and inorganic acids (acetic, nicotinic, etc.), depending on the conditions of synthesis, the nature of the metals, and the composition of the complexes, exhibit diverse modes of coordination.

It should be noted that the electronic structures and properties of amide, as well as acetic, nicotinic, and benzoic acids are of interest for many reasons. First of all, a certain number of synthetic and natural biologically active compounds in their composition contain these groups as the main structural element. They actively participate in many biological and catalytic processes and are used as selective complexing agents and metal extractants. Consequently, the above representatives of the class of compounds have long attracted the attention of inorganic chemists as ligands [1–12].

Numerous works on the study of coordination compounds of p, d, and f-metals with acid amides are devoted to complexes with homogeneous ligands. However, by the beginning of our research, there were no data on homogeneous and mixed-ligand coordination compounds of metal carboxylates. The reasons for the competitive coordination of ligands, acid anions, and water molecules around the central atom are not shown.

To solve these problems, we chose magnesium acetate as complexing agents, since it is convenient to judge their ability to complex formation by changing the nature of acid anions. Formamide, acetamide, nicotinamide, benzamide, benzoic and nicotinic acids were used as organic ligands.

In connection with the above, the purpose of the study was to create the scientific foundations of synthesis, to establish the features of the composition, structure, crystal and electronic structures, reactivity, physicochemical properties of new coordination compounds, to identify and study new compounds with high biological activity to create new effective and environmentally friendly safe plant growth regulators.

In the process of performing this study, for the synthesis of complex compounds, magnesium acetate of the composition, Mg(CH₃COO)₂·4H₂O of the "analytical grade" or "chemically pure" brand was used. Formamide (HCONH₂), acetamide (CH₃CONH₂), nicotinamide (NC₅H₄CONH₂), benzamide (C₅H₆CONH₂), nicotinic acid (NC₅H₄COOH) of analytical grade were used as ligands. Nitrogen was determined by the Dumas micromethod, carbon and hydrogen were determined by combustion in an oxygen stream. To establish the individuality of the synthesized complex compounds, X-ray diffraction patterns were taken on a DRON-2.0 setup with a Cu anticathode. Tables were used to calculate the interplanar distances, and the relative intensity of the I/I1 line was determined as a percentage of the most pronounced reflection at the maximum.

IR absorption spectra were recorded on Specord-75 IR spectrophotometers (400-4000 cm-1 and PUE and NICAM (400-4000 cm-1) using the technique of pressing samples with KBr.

Quantum-chemical calculations of molecules were carried out by the MO LCAO method in the MNDO and AM-1 approximations with full optimization.

Thermal analysis was carried out on a derivatograph of the Paulik-Paulik-Erdey system [1] at a rate of 10 deg/min and a weight of 0.1 g at the sensitivity of T-900, TG-100, DTA-1/10, and DTG-1/10 galvanometers. Recording was carried out under atmospheric conditions with constant removal of the gaseous medium using a water jet pump. The holder was a platinum crucible 7 mm in diameter without a lid. Al₂O₃ was used as a reference.

To carry out the synthesis of complex compounds, we have chosen the most effective mechanochemical method, since it does not require scarce organic solvents.

The mechanochemical process of interaction of the initial components is carried out by intensive grinding at room temperature in a ball mill of the components taken in the molar ratios of acetates and nicotinates of magnesium, calcium and nickel, as well as ligands 1:1:1, respectively. All the following compounds were prepared similarly (Table 1).

А mixed-ligand complex compound of the composition Ca(CH₃COO)₂· ·HCONH₂·NC₅H₄COOH·H₂O was synthesized by intensive stirring of 0.1762 g (0.001 mol) of calcium acetate monohydrate with 0.045 g (0.001 mol) of formamide and 0.2464 g (0.001 mol) of nicotinic acid in an agate mortar at room temperature for three hours. The product yield is 92.5%.

A mixed amide compound of the composition Ca(CH₃COO)₂·HCONH₂··C₅H₆CONH₂·H₂O was obtained by mixing 0.1762 g (0.001 mol) of calcium acetate monohydrate with 0.045 g (0.001 mol) of formamide and 0.2423 g (0.001 mol) of benzamide in an agate mortar within three hours. The product yield is 93.6%.

A complex compound of the composition Ca(CH₃COO)₂·CH₃CONH₂··NC₅H₄COOH·2H₂O was synthesized by intensive stirring of 0.1762 g (0.001 mol) of calcium acetate monohydrate with 0.059 g (0.001 mol) of acetamide and 0.2464 g (0.001 mol) of nicotinic acid in agate mortar at room temperature for three hours. The product yield is 89.8%.

A mixed amide compound of the composition $Ca(CH_3COO)_2 \cdot CH_3CONH_2 \cdot \cdot C_5H_6CONH_2 \cdot H_2O$ was obtained by mixing 0.1762 g (0.001 mol) of calcium acetate monohydrate with 0.059 g (0.001 mol) of acetamide and 0.2423 g (0.001 mol) of benzamide in an agate mortar in within three hours. The product yield is 88.6%.

The coordination compound of the composition $Mg(CH_3COO)_2$ ··NC₅H₄CONH₂·HCONH₂·H₂O was synthesized by intensive stirring of 0.2144 g (0.001 mol) Mg(CH₃COO)₂·4H₂O with 0.1221 g (0.001 mol) of nicotinamide and 0.045 g (0.001 mol) formamide and in an agate mortar at room temperature for three hours. The product yield is 90.5%.

A mixed amide compound of the composition $Mg(CH_3COO)_2 \cdot HCONH_2 \cdot C_5H_6CONH_2 \cdot H_2O$ was obtained by mixing 0.2144 g (0.001 mol) of magnesium acetate tetrahydrate with 0.045 g (0.001 mol) of formamide and 0.2423 g (0.001 mol) of benzamide in an agate mortar within three hours. The product yield is 93.6%.

Results of elemental analysis of mixed-ligand coordination compounds of acetates magnesium and calcium												
		Me,%		N,%		C,%		H,%				
	Compounds	Found	Comp	Foun	Comp	Foun	Comp	Foun	Comp			
			•	d	•	d	•	d				

Table 1.

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Ca(CH ₃ COO) ₂ ·HCONH ₂	11,38	11,64	8,39	8,13	38,19	38,37	4,74	4,68
·NC5H4COOH·H2O								
Ca(CH ₃ COO) ₂ ·HCONH ₂ ·	11,54	11,71	8,47	8,18	41,92	42,10	5,36	5,30
·C ₅ H ₆ CONH ₂ ·H ₂ O								
Ca(CH ₃ COO) ₂ ·CH ₃ CONH ₂	10,79	10,65	7,28	7,44	39,01	38,29	5,28	5,36
··NC5H4COOH·2H2O								
Ca(CH ₃ COO) ₂ ·	12,04	11,85	8,41	8,28	46,27	46,15	5,33	5,26
·CH ₃ CONH ₂ ·C ₅ H ₆ CONH ₂ ·								
·H ₂ O								
Mg(CH ₃ COO) ₂ ·	7,28	7,42	13,04	12,83	40,44	40,33	5,29	5,23
·NC5H4CONH2·HCONH2·								
H ₂ O								
Mg(CH ₃ COO) ₂ ·	7,59	7,44	8,70	8,580	44,32	44,13	5,69	5,56
·HCONH ₂ ·C ₅ H ₆ CONH ₂ ·								
H ₂ O								

Comparison of interplanar distances and relative intensities of formamide, acetamide, carbamide, thiocarbamide, nitrocarbamide, nicotinamide and new calcium acetate complex compounds showed that they differ significantly from each other, from similar ones and from the original compounds. Therefore, the synthesized coordination compounds have individual crystal lattices (Table 7).

Table-7 Interplanar spacings and relative line intensities of some mixed-ligand coordination compounds of calcium acetate with amides.

calcium acetate with amides.											
Compounds	d, Å	I,	d, Å	I, %	d, Å	I, %	d, Å	I,	d, Å	I,	
		%						%		%	
Ca(CH ₃ COO) ₂ ·	13,52	31	4,97	4	2,82	8	1,967	21	1,542	8	
·HCONH ₂ ·	12,73	15	4,86	4	2,81	7	1,940	5	1,537	10	
·CH ₃ CONH ₂ ·	11,60	8	4,75	5	2,77	11	1,916	9	1,528	10	
$\cdot 2H_2O$	11,02	5	4,61	8	2,75	10	1,891	18	1,508	4	
	10,07	5	4,48	15	2,72	10	1,861	11	1,500	2	
	9,63	5	4,38	16	2,69	8	1,848	17	1,488	3	
	9,26	9	4,26	10	2,62	52	1,835	22	1,477	3	
	9,00	12	4,18	8	2,54	8	1,799	5	1,474	3	
	8,39	4	4,10	11	2,49	17	1,791	8	1,463	5	
	7,91	10	4,05	18	2,45	42	1,712	6	1,460	6	
	7,74	5	4,03	21	2,42	10	1,752	12	1,455	4	
	7,35	5	3,97	17	2,39	11	1,739	18	1,446	4	
	7,23	5	3,77	21	2,37	11	1,708	4	1,439	6	
	7,12	4	3,73	21	2,35	8	1,693	5	1,427	3	
	6,90	5	3,16	14	2,31	27	1,687	4	1,419	4	
	6,69	7	3,56	100	2,26	5	1,665	6	1,411	4	
	6,40	26	3,44	28	2,22	8	1,632	6	1,405	5	
	6,13	5	3,35	18	2,17	29	1,620	5	1,385	5	
	5,98	8	3,29	8	2,17	28	1,601	2	1,382	5	
	5,89	13	3,19	8	2,14	9	1,591	5	1,380	5	
	5,81	11	3,12	9	2,10	18	1,582	5			
	5,61	5	3,07	13	2,09	18	1,566	4			
	5,46	8	2,97	37	2,06	15	1,556	6			
	5,14	6	2,92	13	2,00	13	1,549	6			
Ca(CH ₃ COO) ₂ ·	12,59	17	5,35	7	2,97	25	1,920	10	1,506	8	
·HCONH ₂ ·	11,91	11	5,26	8	2,92	10	1,905	6	1,493	6	

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Compounds	d, Å	I, %	d, Å	I, %	d, Å	I, %	d, Å	I, %	d, Å	I, %
·H ₂ NCONHNO ₂ ·	11,36	11	5,19	6	2,81	100	1,886	8	1,488	4
·2H ₂ O	11,02	15	4,98	10	2,71	6	1,862	14	1,475	4
	10,91	8	4,89	7	2,69	7	1,833	7	1,471	7
	10,25	6	4,71	8	2,65	8	1,811	8	1,459	8
	10,02	7	4,61	7	2,61	10	1,792	15	1,451	6
	9,04	11	4,48	6	2,57	31	1,789	15	1,439	4
	8,51	7	4,32	34	2,50	7	1,778	10	1,435	6
	8,17	15	4,22	24	2,45	27	1,754	7	1,429	7
	7,91	51	4,15	6	2,44	24	1,730	8	1,421	4
	7,77	61	4,06	8	2,40	6	1,717	7	1,405	6
	7,28	7	3,99	10	2,36	6	1,707	10	1,396	6
	7,05	7	3,89	35	2,35	6	1,693	11	1,394	7
	6,94	6	3,81	28	2,33	7	1,674	8	1,389	4
	6,79	4	3,67	6	2,29	14	1,659	8	1,378	4
	6,69	7	3,58	18	2,27	14	1,648	14	1,371	6
	6,49	4	3,22	11	2,25	13	1,632	8	1,368	4
	6,31	6	3,38	21	2,22	10	1,624	7	1,361	6
	6,10	6	3,35	13	2,17	24	1,611	8	1,357	6
	5,97	6	3,32	10	2,12	14	1,593	6	1,355	4
	5,81	8	3,29	7	2,07	27	1,576	8	1,348	6
	5,71	10	3,25	17	2,06	25	1,566	6		
	5,61	8	3,21	18	2,03	6	1,548	6		
	5,52	6	3,09	10	1,967	23	1,530	7		
	5,42	6	2,99	23	1,951	10	1,519	6		
Ca(CH ₃ COO) ₂ ·	16,32	10	5,59	3	2,84	3	2,02	5	1,611	2
·HCONH ₂ ·	14,69	4	5,38	6	2,81	3	2,01	5	1,581	2
·NC ₅ H ₄ CONH ₂ ·	13,60	2	5,30	5	2,77	2	2,00	5	1,571	2
·H ₂ O	12,81	2	5,02	5	2,75	3	1,979	3	1,566	3
	12,17	4	4,66	6	2,72	3	1,948	3	1,554	2
	11,72	2	4,54	12	2,67	10	1,912	6	1,539	3
	10,40	2	4,47	7	2,60	12	1,893	3	1,528	2
	10,25	2	4,38	6	2,54	6	1,883	2	1,524	3
	10,02	2	4,21	5	2,51	8	1,862	3	1,515	2
	9,34	2	4,39	44	2,47	5	1,849	3	1,507	2
	8,96	3	3,90	13	2,43	22	1,833	4	1,496	2
	8,42	35	3,82	4	2,38	8	1,811	6	1,488	2
	8,02	8	3,69	2	2,33	11	1,793	4	1,479	2
	7,88	6	3,60	8	2,30	11	1,774	3	1,471	2
	7,33	2	3,55	8	2,27	4	1,749	3	1,465	2
	7,05	6	3,47	17	2,25	5	1,730	3	1,456	2
	6,81	4	3,40	21	2,20	9	1,700	3	1,415	2
	6,69	2	3,27	15	2,18	5	1,687	2	1,375	2
	6,53	2	3,14	8	2,16	5	1,676	2	1,370	2
	6,31	5	3,01	6	2,12	2	1,661	2	1,359	2
	5,98	100	2,97	11	2,08	3	1,631	2	1,350	2
	5,71	8	2,88	6	2,05	2	1,624	2	4	<u> </u>
Ca(CH ₃ COO) ₂ ·	13,60	6	4,56	13	2,93	5	2,15	17	1,687	4
$\cdot CH_3CONH_2$	13,35	4	4,48	10	2,88	18	2,11	14	1,661	4

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Compounds	d, Å	I, %	d, Å	I, %	d, Å	I, %	d, Å	I, %	d, Å	I, %
·H ₂ NCONHNO ₂	12,74	4	4,33	4	2,80	3	2,09	6	1,652	4
1121 (0 01 (111 (02	10,91	5	4,22	10	2,75	10	2,08	10	1,632	4
	10,16	3	4,03	45	2,69	12	2,00	5	1,621	4
	9,46	3	3,99	11	2,68	12	2,03	12	1,606	5
	8,78	13	3,94	13	2,66	5	1,976	8	1,579	5
	8,42	15	3,90	18	2,60	10	1,948	8	1,564	6
	7,69	5	3,77	4	2,57	14	1,917	7	1,558	4
	7,58	16	3,61	8	2,53	9	1,905	7	1,546	5
	7,03	6	3,57	15	2,47	5	1,884	6	1,524	5
	6,69	5	3,53	36	2,44	32	1,871	15	1,509	6
	6,49	10	3,50	24	2,40	18	1,840	5	1,494	4
	6,02	100	3,46	24	2,34	18	1,827	6	1,468	4
	5,69	9	3,36	21	2,33	28	1,818	10	1,446	5
	5,65	15	3,24	10	2,31	9	1,786	3	1,435	4
	5,33	6	3,13	3	2,28	12	1,769	12	1,408	4
	5,11	5	3,09	3	2,25	4	1,752	8	1,397	4
	1,91	4	3,03	10	2,21	13	1,720	9	1,392	4
	4,71	7	2,96	18	2,18	16	1,707	5	1,376	4
Ca(CH ₃ COO) ₂ ·	12,89	4	4,52	9	2,78	2	2,05	3	1,625	3
·CH ₃ CONH ₂ ·	12,31	2	4,43	6	2,74	7	2,03	2	1,615	2
·NC ₅ H ₄ CONH ₂ ·	11,79	23	4,34	3	2,68	8	2,03	5	1,604	3
·0,5H ₂ O	9,15	2	4,26	2	2,60	9	1,967	5	1,591	3
•,•2 -	8,55	13	4,16	5	2,56	11	1,936	3	1,567	3
	8,29	14	3,96	20	2,50	5	1,930	3	1,561	4
	7,74	5	3,88	11	2,30	3	1,852	9	1,538	2
	7,35	14	3,86	13	2,42	15	1,833	4	1,521	2
	6,90	5	3,51	31	2,39	11	1,820	4	1,504	3
	6,40	6	3,44	30	2,33	10	1,805	5	1,469	2
	5,90	61	3,33	15	2,33	7	1,764	6	1,441	3
	5,71	100	3,27	40	2,20	6	1,747	4	1,394	2
	5,52	11	3,27	6	2,17	9	1,713	5	1,388	2
	5,25	4	3,09	1	2,17	11	1,703	3	1,366	2
	5,06	2	3,01	6	2,10	9	1,678	3	1,500	-
	4,81	2	2,95	13	2,09	3	1,656	3		
	4,61	4	2,91	4	2,07	5	1,647	2		
	7,48	11	3,52	40	2,31	5	1,755	9	1,453	4
	7,09	6	3,42	93	2,31	8	1,739	5	1,447	4
	6,90	6	3,34	100	2,20	10	1,725	6	1,435	7
	6,59	5	3,14	7	2,24	10	1,723	6	1,433	4
	6,36	9	3,11	18	2,21	8	1,694	7	1,419	4
	6,08	6	3,05	4	2,15	12	1,681	4	1,407	3
	5,89	9	2,99	18	2,13	6	1,660	11	1,407	4
	5,54	58	2,93	14	2,14	21	1,654	6	1,393	4
	5,33	5	2,93	14	2,11	10	1,627	9	1,380	5
	5,33	24	2,89	36	2,09	8	1,615	9	1,370	7
	13,52	5	4,23	16	2,59	18	1,015	9 7	1,546	2
(`a((`H_(`()())_`	1 1 1. 1/.	1.2	1 T.4J	110	4,57	10	1,71	/	1,040	4
$Ca(CH_3COO)_2$ ··H ₂ NCONHNO ₂ ··	11,72	2	4,12	28	2,55	8	1,88	3	1,537	1

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Compounds	d, Å	I,	d, Å	I, %	d, Å	I, %	d, Å	I,	d, Å	I,
Compounds	u, 11	1, %	u, 11	1, 70	u, 11	1, 70	u, 11	1, %	u, 11	1, %
·0,5H ₂ O	10,02	1	3,82	14	2,49	14	1,85	9	1,519	1
	8,99	12	3,70	25	2,42	35	1,83	3	1,504	3
	8,16	5	3,62	17	2,39	3	1,81	2	1,482	3
	7,69	42	3,53	43	2,36	4	1,79	2	1,479	1
	7,07	1	3,39	100	2,32	3	1,79	4	1,465	1
	6,77	2	3,32	68	2,29	5	1,76	1	1,456	2
	6,57	2	3,23	26	2,27	6	1,75	3	1,445	2
	6,34	5	3,10	9	2,24	5	1,73	3	1,430	1
	6,22	2	3,07	7	2,21	7	1,71	2	1,421	1
	5,90	8	3,02	9	2,15	13	1,69	4	1,403	1
	5,87	8	2,95	22	2,13	6	1,66	3	1,396	1
	5,59	90	2,91	30	2,10	3	1,64	4	1,389	1
	5,20	19	2,86	5	2,06	6	1,62	4	1,379	2
	5,06	20	2,82	8	2,04	6	1,616	5	1,364	1
	4,85	16	2,77	38	2,03	5	1,602	3		
	4,67	6	2,75	18	2,01	2	1,581	2		
	4,56	5	2,69	5	1,96	10	1,573	2		
	4,37	9	2,64	15	1,95	11	1,552	1		

Synthesis conditions were developed, 6 mixed-ligand coordination compounds of calcium acetate with formamide, acetamide, nitrocarbamide, and nicotinamide were isolated in the solid state. The composition, individuality, physicochemical and biological properties of the synthesized compounds have been established. Methods of vibrational spectroscopy, thermal and quantum-chemical analyzes have been used to prove methods of coordination of organic ligands and environments of the central ion, thermal behavior and reactivity of the synthesized complexes.

Based on the data of IR spectroscopy, it was found that the molecules of formamide, acetamide, carbamide, nitrocarbamide, anions of acetic and nitric acids are coordinated through the oxygen atom. The thiocarbamide and nicotinamide molecules are coordinated, respectively, through the sulfur atom of the thioamide group and the nitrogen heteroatom of the pyridine ring. Acetate and nitrate anions, depending on the composition and geometric configuration of the coordination sites, exhibit mono- and bidentate-cyclic coordination. The results of studies by the IR spectroscopic method of analysis were translated into works [1-10]

The thermal behavior of the synthesized compounds was established by the method of derivatographic analysis. The intermediate products of thermolysis were obtained and the composition of the compounds was determined.

The electronic structure and reactivity of individual compounds depending on the nature of organic ligands and water molecules have been established. It is noted that most cases of stabilization of coordination compounds occur due to the formation of intramolecular hydrogen bonds.

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