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Complex compounds of magnesium stearate with thiocarbamide

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Abstract
The homogeneous complex compounds of magnesium stearate with thiocarbamide were synthesized. The composition, individuality, methods of coordination of the stearate thiocarbamide molecule, fragments of complex compounds are established, the thermal behavior of the new compounds is studied.

Keywords: complex compounds, composition, synthesis, analysis methods, IR absorption spectra, X-ray phase analysis, thermolysis

Introduction

The synthesis of new chemical compounds, which has effective properties for use in agriculture, is one of the urgent tasks of modern chemistry. Homogeneous complex compounds of metals, possessing a number of specific properties, are widely used in various sectors of the national economy. The use as substances of ligands containing donor atoms of carboxylic amides, thiamides of thiocarboxylic acids, in particular, urea and thiocarbamide, promotes the formation of coordination compounds with macronutrient content.

Numerous works by the authors are devoted to the synthesis and study of different amide complex compounds of metal carboxylates: Parpieva N.A., Shabilalova. A.A., Yunusova. D.Kh., Palkina. K.K., Azizova. T.A., Mukimova. G.Zh., Azizova. O.T and others [1-6]. There are no data in the literature on homogeneous coordination compounds of magnesium stearate with thiocarbamide.

Objects and research methods

To carry out the synthesis of coordination compounds, we have chosen the most effective mechanochemical method using a ball mill, since it does not require scarce organic solvents. The synthesis procedure was carried out according to [7, 8].

Complex compounds of the composition $Mg(C_{17}H_{35}COO)_2 \cdot 2CS(NH_2)_2 \cdot 2H_2O$ were obtained by vigorous stirring of 0.6273 g (0.002 mol) of magnesium dihydrate stearate with 0.1522 g (0.002 mol) of thiocarbamide in a 100 ml ball mill at room temperature within 33 minutes.

The analysis of synthesized compounds for magnesium content was carried out according to [9]. Nitrogen was determined by the Dumas method [10], carbon and hydrogen by burning in a stream of oxygen (table 1). To establish the individuality of the synthesized compounds, X-ray diffraction patterns were recorded on a DRON-2.0 apparatus with a Cu anticathode [11]. IR spectroscopies were recorded in the range 400–4000 cm – 1 on an AVATAP – 360 spectrometer (Nicolet). Thermal analysis was performed on a Paulik–Paulik–Erdey derivatograph [12].

Table 1: The results of elemental analysis of complex compounds of magnesium stearate with thiocarbamide $Mg(C_{17}H_{35}COO)_2 \cdot 2CS(NH_2)_2 \cdot 2H_2O$

Mg,%		S,%		N,%		C,%		H,%		Брутто формула
Found	Calc.	Found	Calc.	Found	Calc.	Found	Calc.	Found	Calc.	
3,15	3,12	8,27	8,23	7,24	7,19	58,39	58,55	10,52	10,60	$MgC_{38}H_{82}O_6S_2N_4$

Results and its discussion. Based on the above data, to identify the synthesized compounds, we performed an x-ray phase analysis of the free molecules of thiocarbamide and magnesium stearate, as well as the synthesized compounds.

Comparison of the main interplanar distances (d, Å) and relative intensities (I,%) of the lines of free thiocarbamide molecules – 4.30 (100) and new homogeneous complexes of magnesium stearate of the compositions $Mg(C_{17}H_{35}COO)_2 \cdot 2CS(NH_2)_2 \cdot 2H_2O$ – 3,77 (100) shows that they significantly differ from each other and from similar initial components. Therefore, homogeneous complex compounds are individual chemicals (Table 2).

Table 2: Interplanar distances and relative intensities of lines of free thiocarbamide molecules and their complex compounds with magnesium stearate

d,Å	I,%	d,Å	I,%	d,Å	I,%	d,Å	I,%	d,Å	I,%
$CS(NH_2)_2$									
4,76	1	3,00	37	2,27	5	1,835	8	1,602	8
4,44	6	2,88	13	2,17	2	1,799	15	1,546	6
4,30	100	2,78	14	2,12	8	1,773	8	1,486	3
4,13	17	2,69	9	2,07	3	1,745	11	1,411	2
3,70	54	2,48	8	2,00	2	1,725	6	1,357	3
3,39	59	2,42	33	1,894	2	1,665	2	1,316	
3,06	52	2,35	15	1,884	4	1,623	5		
$Mg(C_{17}H_{35}COO)_2 \cdot 2CS(NH_2)_2 \cdot 2H_2O$									
15,96	4	5,82	7	3,06	42	2,15	5	1,676	2
15,09	7	5,74	5	3,01	10	2,11	3	1,665	2
14,22	7	5,68	5	2,93	17	2,07	2	1,641	4
13,86	7	5,46	4	2,90	3	2,02	6	1,632	4
12,59	7	5,20	6	2,83	24	1,991	2	1,611	2
12,16	7	4,91	3	2,72	8	1,967	4	1,566	2
11,02	4	4,78	3	2,63	2	1,926	9	1,556	3
10,50	5	4,59	4	2,61	2	1,881	4	1,491	2
8,82	4	4,43	77	2,51	20	1,854	6	1,459	2
8,02	3	4,22	92	2,46	17	1,841	2	1,419	4
7,85	3	3,92	8	2,41	11	1,816	3	1,375	2
7,48	3	3,77	100	2,35	2	1,774	5	1,361	2
7,35	3	3,46	31	2,30	8	1,760	8	1,354	2
7,01	3	3,37	4	2,27	7	1,753	5	1,330	2
6,44	6	3,24	4	2,21	2	1,695	2	1,318	2
5,97	5	3,11	33	2,18	12	1,687	3		

The IR spectra of free ligand molecules and synthesized compounds were studied. The frequencies were found in the IR spectrum of thiocarbamide at 3380–$\nu_{as}(NH_2)$, 3276–$\nu s(NH_2)$, 3178–$2\delta(NH_2)$, 1619–$\delta(NH_2)$, δ(HNC), 1474–ν(CN), 1413–ν(CS), 1084–ν(CN), 739–ν(CS), 740–δ(CS), δ(NCS), 487–δ(NCN) and 413–δ(NCS).

Table 3 shows the characteristic frequencies (cm^{-1}) of the above compounds.

Table 3: The values of the characteristic frequencies (cm^{-1}) in the IR absorption spectra of the free molecule of thiocarbamide and its coordination compounds with magnesium stearate

Compound	$\nu_{(CS)}$, $см^{-1}$	$\delta_{(CS)}$, $см^{-1}$	$\nu_{as}(COO)^-$, $\nu_s(COO)^-$	H_2O, $см^{-1}$
$CS(NH_2)_2$	739	640		
$Mg(C_{17}H_{35}COO)_2 \cdot 2CS(NH_2)_2 \cdot 2H_2O$	729	632	1562, 1471	3221, 3254

In the IR spectroscopy of the free thiocarbamide molecule, three characteristic frequencies are observed at 1413–ν(CS), 739–ν(CS) and 640 cm^{-1}–δ(CS). In complex compounds of thiocarbamide, it is not possible to observe a change in the frequency value of 1413 cm^{-1}–ν(CS), since it overlaps with a wide band ν(COO) of palmitate, stearate groups. Upon transition to a coordinated state in the low-frequency region of the spectrum, the frequencies of thiocarbamide molecules at 739 and 640 cm^{-1} decrease by 10 cm^{-1} and 8 cm^{-1}, respectively. This is evidence of the coordination of the central atom through the sulfur atom.

The frequency difference $\nu_{as}(COO)$–vs(COO) in stearate complexes is 63 cm^{-1}, respectively, which is typical for bidentant coordination of the stearate group. Thus, the complex compounds have six coordination sites of the magnesium ion.

The bands at 3221–3254 cm^{-1} confirm the presence of crystallization water in the molecule.

Using the method of derivatographic analysis, the thermal behavior of the synthesized compounds was established. Intermediate thermolysis products were obtained and the composition of the compounds was established.

On the heating curve of the complex compound $Mg(C_{17}H_{35}COO)_2 \cdot 2CS(NH_2)_2 \cdot 2H_2O$, eight endothermic effects were detected at 100, 119, 198, 223, 613, 654, 685, 860°C and seven exothermic effects at 285, 346, 370, 410 444, 528 and 810°C. The appearance of the first and second endothermic effect is due to the removal of two water molecules. The mass loss in the temperature range 65–150 is 4,62%. The nature of the subsequent thermal effects is accompanied by a stepwise decomposition of the anhydrous compound. In the temperature ranges 150–218, 218–240, 240–309, 309–358, 358–400, 400–428, 428–467, 467–540, 540–638, 638–670, 670–737, 738–815 and 815–870°C the mass loss is 1.26, 2.89, 8.55, 6.29, 19.50, 12.20, 19.50, 15.09, 0.25, 0.83, 0, 90, 0.15, 0.12%. The total mass loss in the temperature range 65–900° C according to the TG curve is 92.25% (Table 4).

Table 4: Dervatographic data on thermolysis of homogeneous coordination compounds of magnesium stearate with thiocarbamidomy

Effect temperature °C	Peak effect, °C	Mass decline, %	Total mass decline	The nature of effects	The process
65–105	100	2,31	2,31	*Endothermic*	$-2H_2O$
105–150	119	2,31	4,62	*Endothermic*	$-2CS(NH_2)_2$

150–218	198	1,26	5,88	*Endothermic*	melting, evaporation, or chemical reactions of dehydration, dissociation, oxidation, and some structural transformations
218–240	223	2,89	8,77	*Endothermic*	
240–309	285	8,55	17,32	*Exothermic*	
309–358	346	6,29	23,61	*Exothermic*	
358–400	370	19,50	43,11	*Exothermic*	
400–428	410	12,20	55,31	*Exothermic*	
428–467	444	19,50	74,81	*Exothermic*	Decomposition and fading, change in crystal structure. Getting MgO
467–540	528	15,09	89,90	*Exothermic*	
540–638	613	0,25	90,15	*Endothermic*	
638–670	654	0,83	90,98	*Endothermic*	
670–737	685	0,90	91,98	*Endothermic*	
738–815	810	0,15	92,13	*Exothermic*	
815–870	860	0,12	92,25	*Endothermic*	

The test drug was administered to experimental animals once orally in the form of a 25% aqueous suspension (with the addition of Tween-80), in doses: 3000 mg/kg (0.24 ml/20 g), 4500 mg/kg (0.36 ml/20 g), 6000 mg/kg (0.48 ml/20 g), 7500 mg/kg (0.6 ml/20 g) and 9000 mg/kg (0.72 ml/20 g).

However, according to published data, the maximum volume for a single oral administration is 0.5 ml/20 g [13, 14]; therefore, we used the fractional administration method for a dose of 7500 mg / kg and 9000 mg/kg. When a dose of 7500 mg/kg was administered, a volume of 0.5 ml/20 g was first administered, then 0.1 ml/20 g was administered after 10 minutes. When a dose of 9000 mg/kg was administered, a volume of 0.5 ml/20 g was first introduced, then after 10 minutes 0.22 ml/20 g was administered.

The animals were placed in separate cages in groups and were continuously monitored for the first hour, then they were monitored hourly for the first day, and once a day, for the next 13 days of the experiment (total observation period was 14 days). At the same time, the general condition of the animals, the features of their behavior were taken into account. The intensity and nature of motor activity, the presence and nature of seizures, coordination of movements, skeletal muscle tone, reaction to tactile, pain, sound and light stimuli, the frequency and depth of respiratory movements, heart rate, the condition of the coat and skin, the color of the mucous membranes, position tail, the amount and texture of fecal matter, feed and water consumption, as well as other indicators characterizing the toxic effect. The timing of the development of intoxication and death of animals were also recorded [13, 14]. The calculation of the average lethal dose (LD50), as well as LD84 (the dose causing the death of 84% of the animals in the group), LD16 (the dose causing the death of 16% of the animals in the group) [15].

During the experiment, all animals were kept under standard vivarium conditions and were on a complete diet and water diet.

After oral administration of the drug, a number of symptoms of intoxication, changes in the general condition and other effects characterizing the toxic effect of the drug were observed (Table 5).

Table 5: The results of the toxic effects of the drug

Dose	Results
3000 мг/кг	After administration of the drug in animals, there were no significant changes in the general condition, as well as death of animals.
4500 мг/кг	An hour after administration of the drug in animals, a decrease in motor activity was observed within an hour. Onthefifthday, onemousedied.
6000 мг/кг	Fifteen to twenty minutes after the administration of the drug, animals showed a decrease in motor activity and bunching for five hours. On the second day, the death of one mouse was observed, and on the third day, the death of two more mice was observed.
7500 мг/кг	10-15 minutes after the administration of the drug, animals were observed to cluster and decrease in motor activity during the day. On the second day, the death of three mice was observed, and on the third day, the death of two more mice was observed.
9000 мг/кг	10 minutes after the administration of the drug in animals, bunching and a decrease in motor activity during the day were observed. The death of four mice was observed on the first day, and the death of two more mice was observed on the second day.

Based on the results of the death of experimental animals, we calculated LD50, as well as LD84 and LD16 of the test drug (Table 6).

Table 6: The results of the study of indicators of acute toxicity of the drug (p = 0.05)

Doses	Number of animals/dead/total
3000 mg/kg	0/6
4500 mg/kg	1/6
6000 mg/kg	3/6
7500 mg/kg	5/6
9000 mg/kg	6/6
$ЛД_{50}$= 6000 (4980÷7229) mg/kg 980–7229) мг/кг	
$ЛД_{84}$= 4337 mg/kg	
$ЛД_{16}$= 7663 mg/kg	

Based on the obtained data on the average lethal dose indicator, we determined toxicity by the toxicity classifier (the classifier contains six levels of toxicity classification), described in the methodological manual for preclinical drug research edited by A.Stefanov [fifteen]. According to this classifier, when administered orally, the drug belongs to the fifth class of toxicity (Almost non-toxic).

When studying the clinical picture of intoxication, after oral administration of the drug, it was found that the central nervous system is the main target organ of the drug.

Conclusion

The homogeneous coordination compounds of magnesium stearate with thiocarbamide were synthesized. The composition, personality, methods of coordination of

stearate groups and thiocarbamide molecules are established. The thermal behavior of new complex compounds has been studied. As a result, it was found that the drug belongs to the fifth class of toxicity.

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