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Homogeneous Complex Compounds of Calcium Nitrate with Amides

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ABSTRACT: Various amide complexes of calcium nitrate with some acid amides have been synthesized. The composition, individuality, methods of coordination of nitrate groups, acetamide and thiocarbamide molecules have been established. The methods of coordination of organic ligands, the environment of the central ion, and the thermal behavior of the synthesized compounds have been proved by vibrational spectroscopy and thermal analysis. Comparison of interplanar distances and relative intensities of calcium nitrate, ligand molecules, and synthesized complexes showed that new coordination compounds differ from each other, as well as from the initial components. Consequently, the compounds have an individual crystal lattice. Analysis of the IR absorption spectra of uncoordinated molecules of acetamide, thiocarbamide and their complex compounds with the nitrate of the selected metals showed that, with the transition to coordinated positions, the values of some frequencies of the amide molecules change significantly. Due to the complexity of the IR absorption spectra of complex compounds of the selected metals with amides, we were unable to assign all observed frequencies to the corresponding vibrations of bond groups.

KEY WORDS: Synthesis, composition, physicochemical methods of analysis, IR absorption spectra, X-ray phase analysis, individuality, thermal behaviour.

I.INTRODUCTION

At present, interest has increased in complex compounds of biogenic elements with organic ligands exhibiting different types of biological activity. Possible areas of application of the results obtained are shown on the basis of the research carried out. In particular, purification, separation and determination of metals and organic ligands based on the selective interaction of metal nitrates with amides; obtaining glaze components to improve the physical, mechanical and decorative properties of oxide materials and complex compounds with antianemic, anti-inflammatory, anti-atherosclerotic effects, as well as the creation of highly effective growth stimulants leading to an increase in the productivity of cotton, wheat, corn, sofler, izen, licorice, stock roses and basma.
The development of modern technologies using grinding devices has made the problem of obtaining crystalline materials due to intense mechanical influences very urgent. The use of mechanical energy in modern industrial technologies and its use in many cases is a necessary link in the preparation of substances for various technological operations. Various raw materials and materials are subjected to mechanical processing on a huge scale in chemical, metallurgical, food and other enterprises. The most common effective way of transferring energy in grinding processes is shock-shear action, since it is this effect that allows concentrating mechanical energy in certain areas of the crystal structure of the processed substance and in quantities necessary for its destruction [1].

II. SIGNIFICANCE OF THE SYSTEM

The synthesis of a complex compound of calcium nitrate with tiocarbamide and acetamide was carried out. The study of literature survey is presented in section III, methodology is explained in section IV, section V covers the experimental results of the study, and section VI discusses the future study and conclusion.

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III. METHODOLOGY

For the synthesis of coordination compounds, we used analytical grade Ca (NO3) 2 * 4H2O. Acetamide (CH3CONH2) (AA), thiocarbamide (CS (NH2) 2) (TC), analytical grade were used as ligands. The synthesis of coordination compounds of metal nitrates with amides was carried out by the mechanochemical (solid-phase) method. The synthesis procedure was carried out according to [3].

A complex compound of the composition Ca (NO3) 2 · 2CS (NH2) 2 was synthesized in a similar way by reacting 2.3612 g (0.01 mol) Ca (NO3) 2 · 4H2O with 1.5214 g (0.02 mol) thiocarbamide in a ball mill at room temperature for 0.15-0.20 hours. The product yield is 84.0%.

When synthesizing a complex compound of the composition Ca (NO3) 2 · 2CH3CONH2, 2.3612 g (0.01 mol) of Ca (NO3) 2 · 4H2O were ground with 1.1825 g (0.02 mol) acetamide in a ball mill at room temperature for 0.15-0.20 hours. The product yield is 83.0%.

The synthesis was carried out by a mechanochemical method, grinding a mixture of calcium nitrate: amide in a molar ratio of 1: 2 and 1: 1 for 20 minutes at room temperature in a ball mill with working bodies 2-5 (mill volume 1.0 l). All mixed amide complexes of calcium with amides were obtained according to the above method.

IV. EXPERIMENTAL RESULTS

The amount of metals in the synthesized compounds was determined on a novAA 300 atomic absorption spectrophotometer manufactured by Analitik JenaAG (Germany) [2]. Nitrogen was determined by the Kjeldahl method [3], carbon, hydrogen and sulfur by burning in a stream of oxygen. The results of elemental analysis of complex compounds of calcium nitrate with acetamide, thiocarbamide are shown in Table 1.

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 [4]. To calculate the interplanar distances, tables [5] were used, 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 in the range of 400-4000 cm-1 on an AVATAR-360 spectrometer (Nicolet) using the method of pressing samples with KBr.

Table 1. Results of elemental analysis of coordination compounds of calcium nitrate

Connection	Me, %		N, %		S, %		C, %		H, %		Gross formula
	Naid	Calc	Naid	Calc	Naid	Calc	Naid	Calc	Naid	Calc	
$Ca(NO_3)_2{\cdot}2CH_3CONH2$	14,10	14,18	19,77	19,86			16,93	17,02	3,62	3,55	$CaN_4C_4O_8H_{10}$
$Ca(NO_3)_2{\cdot}2CS(NH_2)_2$	12,58	12,66	26,49	26,58	20,16	20,25	7,51	7,59	2,60	2,53	$CaN_6C_2S_2O_6H_8$

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Thermal analysis was carried out on a Paulik-Paulik-Erdey system derivatograph [9] at a speed of 10 deg / min and a sample of 0.1 g at the sensitivity of the T-900, TG-100, DTA-1/10, DTG-1/10 galvanometers. The recording was carried out under atmospheric conditions with constant removal of the gaseous medium using a water-jet pump. A platinum crucible 7 mm in diameter without a lid served as a holder. Al2O3 was used as a reference.

Comparison of interplanar distances and relative intensities of acetamide, thiocarbamide, and new complex compounds of calcium nitrates showed that they differ significantly from each other, from similar and starting compounds. Consequently, the synthesized coordination compounds have individual crystal lattices. (Fig. 1, Fig. 2)

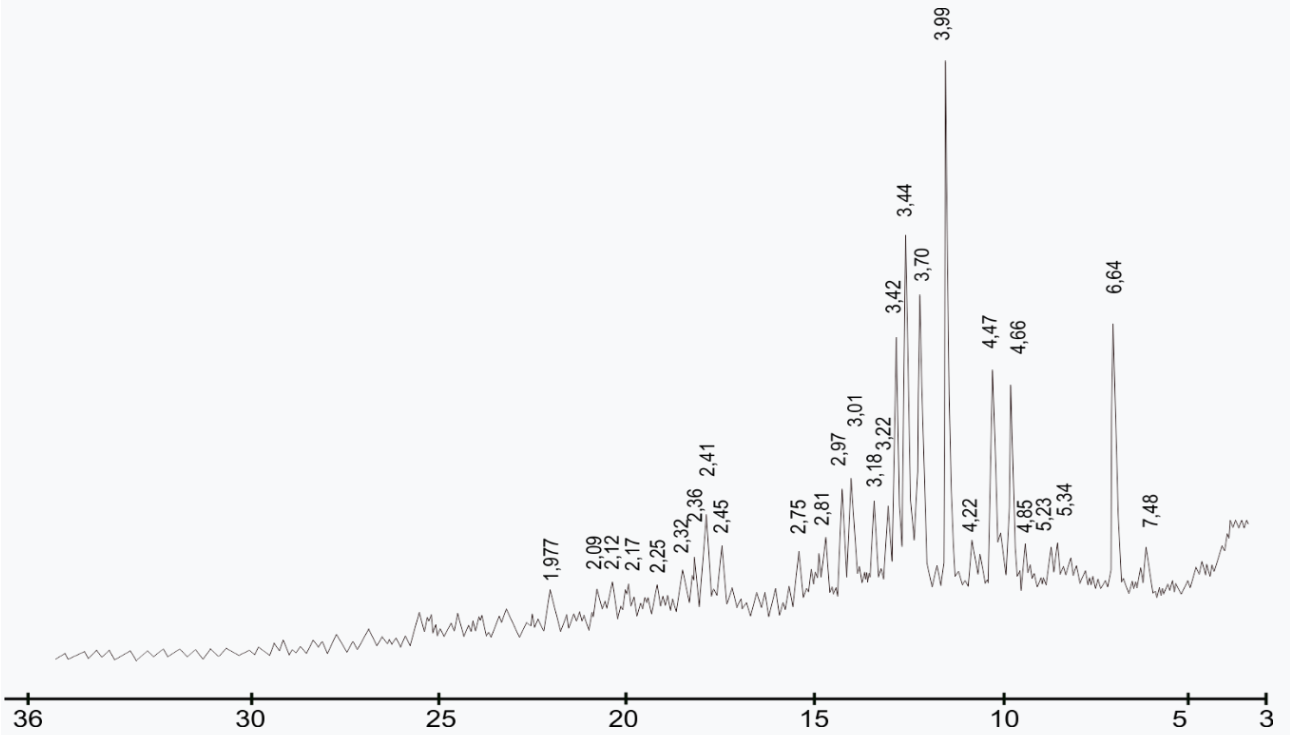

Fig.1. X-ray diffraction pattern: $Ca(NO_3)_2 \cdot 2CH_3CONH2$

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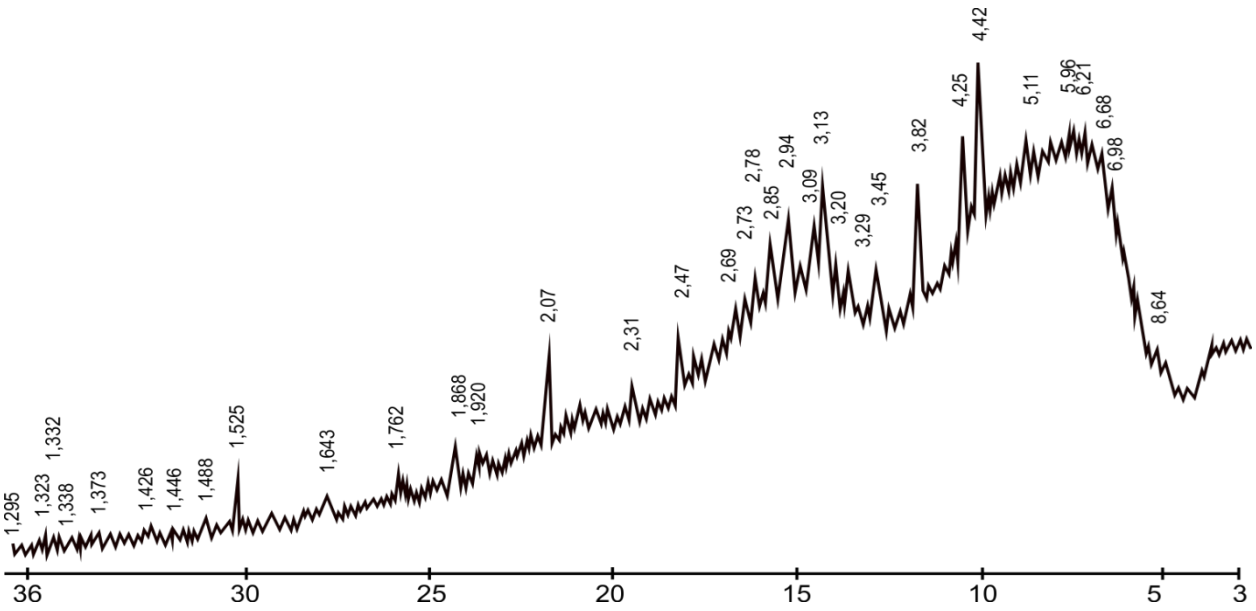

Fig.2. X-ray diffraction pattern: $Ca(NO_3)_2 \cdot 2CS(NH_2)_2$

Analysis of the IR absorption spectra of uncoordinated molecules of acetamide, thiocarbamide and their complex compounds with the nitrate of the selected metals showed that, with the transition to coordinated positions, the values of some frequencies of the amide molecules change significantly. Due to the complexity of the IR absorption spectra of the complex compounds of the selected metals with amides, we were unable to assign all the observed frequencies to the corresponding vibrations of the bond groups. (Tab 2, Fig 3, Fig 4)

Table 2. Characteristic frequencies of IR spectral absorption of free molecules of acetamide, thiocarbamide and their coordination compounds with calcium nitrates (cm-1)

Connection	**ν(C=O)**	**ν(C-N)**	**ν(CS), δ (CS)**	**νk, δ(C=O)**
CH_3CONH_2	1670	1390		
$CS(NH_2)_2$			730,632	
$Ca(NO_3)_2 \cdot CH_3CONH_2$	1660	1392		
$Ca(NO_3)_2 \cdot CS(NH_2)_2$			724, 632	

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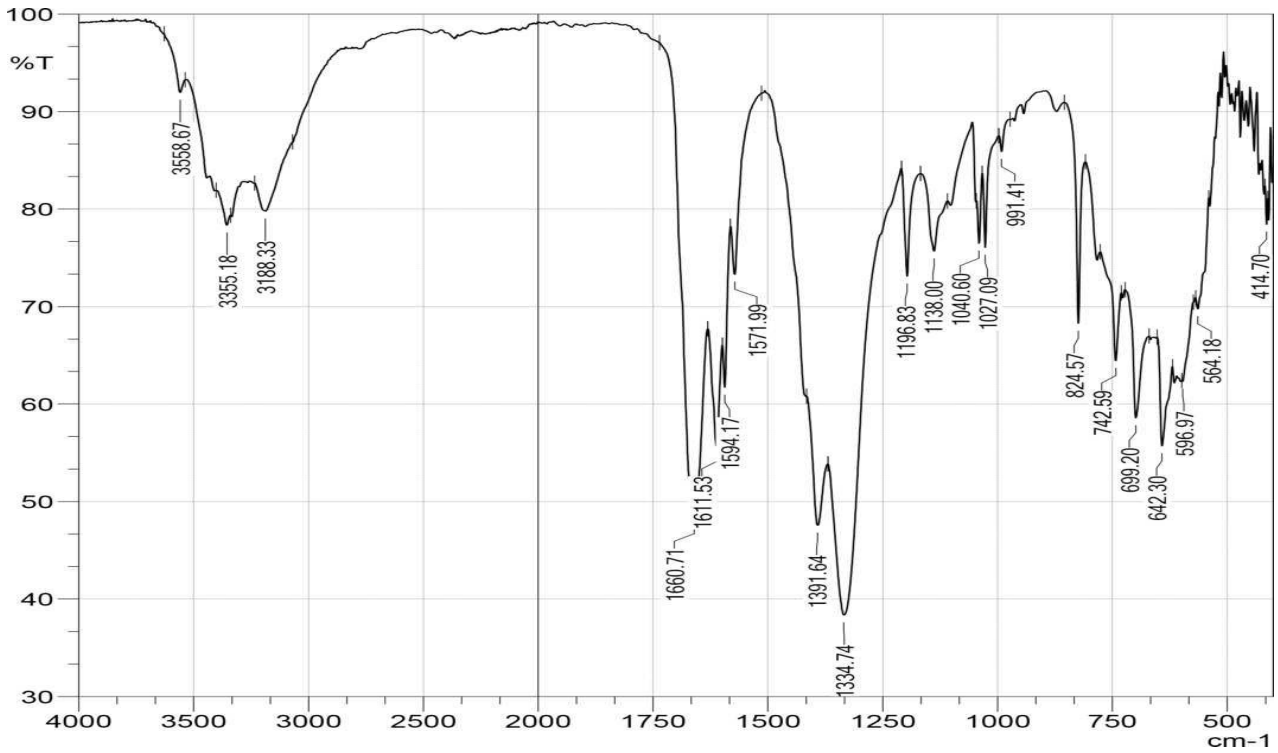

Fig.3. IR absorption spectrum: $Ca(NO_3)_2 \cdot CH_3CONH_2$

The presented complex compounds show the acetamide molecule coordinated through the oxygen atom of the carbonyl group. The thiocarbamide molecule is coordinated through the sulfur atom of the thio group.

The heating curve of the complex compound Ca (NO3) 2 · CS (NH2) 2 · H2O is characterized by seven endothermic effects at 108, 160, 200, 204, 689, 745.790 and three exothermic effects at 294, 332 and 602oC. The appearance of the first endothermic effect is due to the removal of one water molecule. The nature of other thermal effects is related to the stepwise decomposition of the complex. Weight loss in the temperature range 70-130 ° C is 0.4%, calculated 0.4%. In the temperature ranges 130-178, 178-202, 202-207, 207-230, 230-255, 255-283, 283-315, 315-570, 570-820oC, the weight loss is 6.64, respectively; 27.39; 1.66; 1.24; 1.49; 4.56; 14.11; 14.94; 2.49%. The total weight loss in the region of 70-760oC is 82.43%, which corresponds to the formation of CaO (Appendix, Table 5.).

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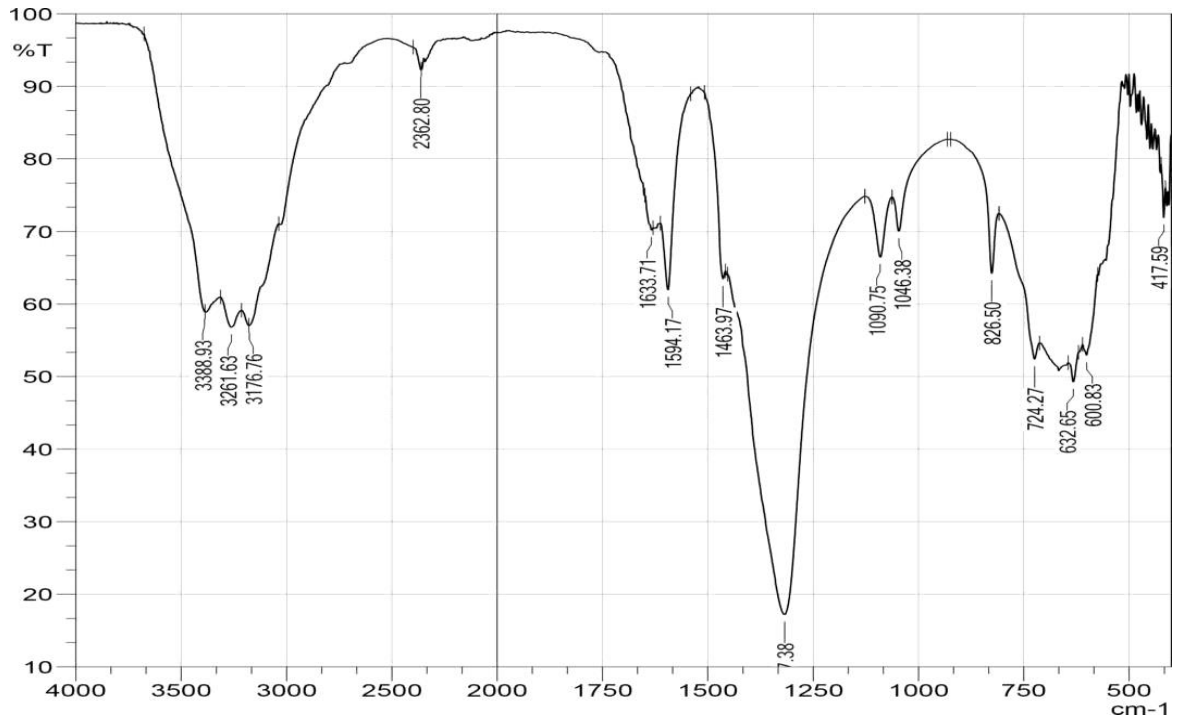

Fig.4. IR absorption spectrum: $Ca(NO_3)_2 \cdot CS(NH_2)_2$

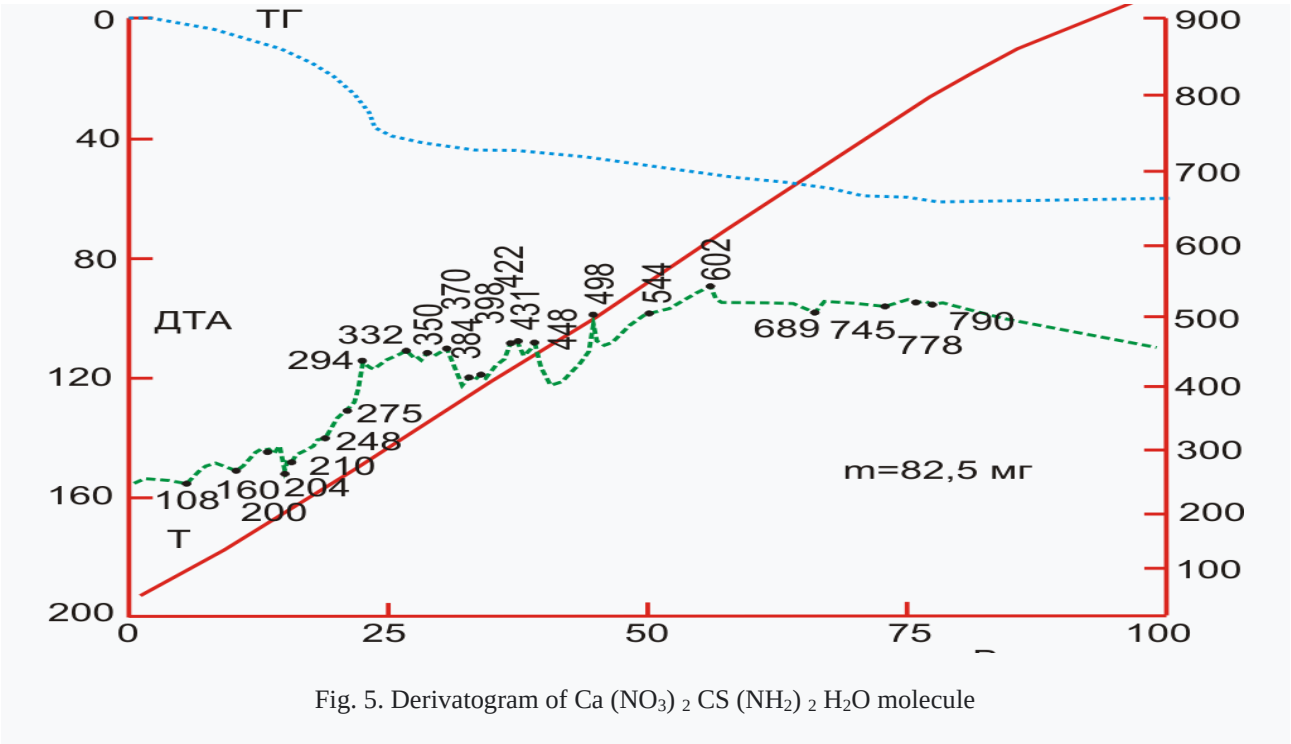

Fig. 5. Derivatogram of Ca $(NO_3)_2$ CS $(NH_2)_2$ H_2O molecule

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The DTA curve of the Ca (NO3) 2 · CH3CONH2 · H2O compound is characterized by eight endothermic effects at 62, 108, 168, 202, 310, 528, 570, 615 and three exothermic effects at 238, 328 and 517oC. The appearance of the first endothermic effect is due to the removal of one water molecule. The nature of other thermal effects is related to the stepwise decomposition of the complex. Weight loss in the temperature range 50-87 ° C is 0.6%, calculated 0.6%. In the temperature ranges 87-120, 120-185, 185-210, 210-244, 244-258, 258-265, 265-522, 522-642, 642-730, 730-860 ° C, the weight loss is, respectively, 1.64; 6.56; 34.43; 2.46; 0.41; 0.49; 4.92; 4.10; 13.74, 1.21%. The total weight loss in the range of 50-860oC is 74.64%, which corresponds to the formation of CaO

V. CONCLUSION AND FUTURE WORK

The conditions of synthesis were developed, the complex compound of calcium nitrate with thiocarbamide and acetamide in the solid state was isolated. With the help of X-ray phase, vibrational spectroscopy, derivatographic analyzes, the individuality, methods of

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