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## ВЗАИМОДЕЙСТВИЕ КОМПОНЕНТОВ В ВОДНОЙ СИСТЕМЕ С УЧАСТИЕМ ДИКАРБАМИДОХЛОРАТА КАЛЬЦИЯ И ФОСФАТА КАРБАМИДА

© Ш.Ш. Хамдамова

Институт общей и неорганической химии Академии наук Республики Узбекистан,  
Республика Узбекистан, 100170, г.Ташкент, ул. Мирзо-Улугбека, 77-а.

*Рассмотрены вопросы взаимного поведения компонентов в тройной водной системе с участием дикарбамидохлората кальция и фосфата мочевины визуально-политермическим методом в широком интервале температур и концентраций. На основе полученных данных построена политермическая диаграмма растворимости системы на прямоугольном треугольнике от эвтектической точки замерзания до 30 °С. Поверхность ликвидуса разделена на четыре части, которые соответствуют полям кристаллизации льда, дикарбамидохлората кальция, фосфата мочевины и соединения состава  $\text{Ca}(\text{H}_2\text{PO}_4)(\text{ClO}_3) \cdot \text{CO}(\text{NH}_2)_2$ . Соединение было выделено из предполагаемой области кристаллизации и идентифицировано методами химического, термографического и ИК-спектроскопического анализа. Благодаря хорошей растворимости исходных компонентов в системе наблюдается незначительное высаливающее действие дикарбамидохлората кальция и фосфата мочевины на соединение состава  $\text{Ca}(\text{H}_2\text{PO}_4)(\text{ClO}_3) \cdot \text{CO}(\text{NH}_2)_2$ .*

*Ключевые слова: растворимость, тройная система, дикарбамидохлорат кальция, фосфат мочевины, политерма, идентификация, высаливающее действие.*

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## INTERACTION OF COMPONENTS IN WATER SYSTEM WITH CALCIUM DI-UREA-CHLORATE AND UREA PHOSPHATE

© Sh.Sh. Khamdamova

Institute of General and Inorganic Chemistry of Uzbek Academy of Science,  
77a, Mirzo-Ulugbek St., Tashkent, 100170, Uzbekistan

*In this article the mutual behavior of components in triple water system with participation of calcium di-urea-chlorate and urea phosphate in wide interval of the temperature and concentration have been studied by the visual-polythermyc method. Based on data obtained, the polythermic diagram of solubility of the system on right-angled triangle from eutectic freezing point till 30 °C has built. Surface of liquidus is divided into four parts, which correspond to the fields of crystallization of ice, calcium di- urea-chlorate, urea phosphate and compound with  $\text{Ca}(\text{H}_2\text{PO}_4)(\text{ClO}_3) \cdot \text{CO}(\text{NH}_2)_2$  composition. The compound was precipitated from supposed area of crystallization and was identified by the methods of chemical, thermographic and IR-spectroscopic analysis. Due to good solubility of initial components the insignificant salting-out effect of calcium di-urea-calcium chlorate and urea phosphate on compound containing  $\text{Ca}(\text{H}_2\text{PO}_4)(\text{ClO}_3) \cdot \text{CO}(\text{NH}_2)_2$  is observed in the system.*

*Keywords: solubility, triple system, calcium di-urea-chlorate, urea phosphate, polytherma, identification, salt action*

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## INTRODUCTION

The development of a multifunctional and effective preparation for agriculture based on local raw materials is an important issue. Calcium chlorate is an inorganic defoliant that is effective in dewy and not dewy regions. Urea phosphate is a high concentrated complex fertilizer containing about 17.8% of the nitrogen and 44.9% of the phosphate anhydride. An alternative way of obtaining a polyfunctional defoliant with nutrients is a combination of defoliant with different mineral fertilizers [1].

Previously, the interaction of the above components in water systems has not been studied. For physico-chemical justification and further development of technology of calcium chlorate defoliant with active additives, we have studied the interaction of components in the system calcium di-urea-chlorate – urea phosphate – water in a wide interval of temperatures and concentrations by the visual-polythermal method [2].

## MATERIALS AND METHODS

For research used calcium di-urea-chlorate was used, which was obtained by interaction of the melt of urea with calcium chlorate at a molar ratio  $2:1 = \text{CO}(\text{NH}_2)_2 : \text{Ca}(\text{ClO}_3)_2$ . Urea phosphate is synthesized by reacting urea and concentrated phosphoric acid in a molar ratio 1:1 [3]. The solid phase was identified by chemical and various methods of physico-chemical analysis. To clarify the nature of the interaction between the constituent components of the synthesized compounds was carried out infrared spectroscopic analysis. The IR absorption spectra of initial components and the studied compounds were recorded on FTIR spectrophotometer, System 2000 of the company Perkin-Elmer in the frequency range  $4000\text{--}400\text{ cm}^{-1}$ . Samples were prepared by pressing tablets with potassium bromide [4]. Thermal analysis was recorded on derivatograph system Paulik – Paulik – Erdey [5] with heating rate  $10\text{ deg/min}$ , the batches of the substance 179, 197 mg, with the galvanometer sensitivity,  $1/5$  of the DTA, DTG  $1/10$ , TG-200, T –  $900\text{ }^\circ\text{C}$ . Recording was carried out under atmospheric conditions. A corundum crucible with a diameter of 10 mm without lid was served as a holder. As calcined aluminum oxide was etalon. In the quantitative analysis, well-known methods of analytical chemistry were applied. Calcium was determined by volumetric complexometric method [6]; chlorine-ion by argentometric method [7].

The system  $\text{Ca}(\text{ClO}_3)_2 \cdot 2\text{CO}(\text{NH}_2)_2 - \text{CO}(\text{NH}_2)_2 \cdot$

$\text{H}_3\text{PO}_4 - \text{H}_2\text{O}$  studied with six internal sections, of which I-III, conducted from the side of  $\text{Ca}(\text{ClO}_3)_2 \cdot 2\text{CO}(\text{NH}_2)_2 - \text{H}_2\text{O}$  to pole of  $\text{CO}(\text{NH}_2)_2 \cdot \text{H}_3\text{PO}_4$ , and sections IV-VI is to the contrary from the  $\text{CO}(\text{NH}_2)_2 \cdot \text{H}_3\text{PO}_4 - \text{H}_2\text{O}$  to the pole  $\text{Ca}(\text{ClO}_3)_2 \cdot 2\text{CO}(\text{NH}_2)_2$ . Binary system  $\text{Ca}(\text{ClO}_3)_2 \cdot 2\text{CO}(\text{NH}_2)_2 - \text{H}_2\text{O}$  is characterized by the branches of crystallization of ice and calcium di-urea-chlorate with a transition point at  $15\text{ }^\circ\text{C}$ , in which the concentration of  $\text{Ca}(\text{ClO}_3)_2 \cdot 2\text{CO}(\text{NH}_2)_2$  is 46.1%. Data for the solubility of the binary system  $\text{CO}(\text{NH}_2)_2 \cdot \text{H}_3\text{PO}_4 - \text{H}_2\text{O}$  fully correspond with the data of authors [8].

Based on the data of the binary systems and polythermal sections built polythermal diagram of solubility on a right triangle and polythermal projection system from eutectic freezing point of the system ( $-22,7\text{ }^\circ\text{C}$ ) to  $30\text{ }^\circ\text{C}$  (Fig. 1–2 a, b). The surface of the polythermal liquidus of diagram is divided into four fields corresponding to the fields of crystallization of ice, urea phosphate, calcium di-urea-chlorate and compound of composition of  $\text{Ca}(\text{H}_2\text{PO}_4)(\text{ClO}_3) \cdot \text{CO}(\text{NH}_2)_2$ . There have been found the two of triple points of the system are defined the temperature of crystallization and the compositions of equilibrium solutions (table).

## RESULTS AND DISCUSSION

It is seen from the diagram of solubility that in the system as a new phase formed the compound  $\text{Ca}(\text{H}_2\text{PO}_4)(\text{ClO}_3) \cdot \text{CO}(\text{NH}_2)_2$ . It takes up a significant portion of the solubility diagram, which indicates its lower solubility relative to other components of the system. The minimum concentration of calcium di urea chlorate leading to the formation of compounds is 10.0%, and urea phosphate is 10.8%. It is possible that the mechanism of the reaction is similar to interaction in the system  $\text{Ca}(\text{ClO}_3)_2 - \text{CO}(\text{NH}_2)_2 \cdot \text{H}_3\text{PO}_4 - \text{H}_2\text{O}$  [9]. The compound  $\text{Ca}(\text{H}_2\text{PO}_4)(\text{ClO}_3) \cdot \text{CO}(\text{NH}_2)_2$  was isolated in the crystalline state and identified by the methods of chemical and physico-chemical analysis.

According to the thermal analysis curve of heating double water of calcium di-urea-chlorate is characterized by the presence of ten endothermic effects at 110, 190, 243, 325, 350, 360, 370, 400, 460,  $780\text{ }^\circ$ , with five exothermic effects at 285, 315, 446, 492 and  $610\text{ }^\circ\text{C}$ . Effects at  $110\text{--}190\text{ }^\circ\text{C}$  are characterized by removal surface sphere molecule of water. Calcium chlorate being in the urea compound is decomposed in  $\text{NH}_3$  and  $\text{CO}_2$  at  $243\text{ }^\circ\text{C}$  resulting decomposition of carbamide. Moreover, exothermic peaks at  $310\text{--}$

325°C indicate on decomposition process of calcium chlorate, which continues till 400 °C. Since 400 °C and more combustion action of thermolysis with generation of CaO takes place. The total mass loss in the temperature range 60–900°C by thermogravimetry curve is 86,21% [Fig.3 a].

Curve on heating connection  $\text{Ca}(\text{H}_2\text{PO}_4)_2$  ( $\text{ClO}_3$ ) $\cdot\text{CO}(\text{NH}_2)_2$  there are the following exothermic effects at 100, 215, 311, 420, 585 and 730 °C and endothermic effects at 85, 123, 150 °C.

Thermoeffects at 85–100°C corresponds emission of fragments of chlorates that loss of mass is 28-30%. Effects at 215–311°C are character for decomposition compound urea with emission ammonia and carbon dioxide. Following that, heating stepped decomposition of  $\text{Ca}(\text{H}_2\text{PO}_4)_2$  and combustion of thermolysis products takes place that thermoeffects testify increase temperature above 420°C. The total mass loss in TG is 43,26% [Fig.3 b].

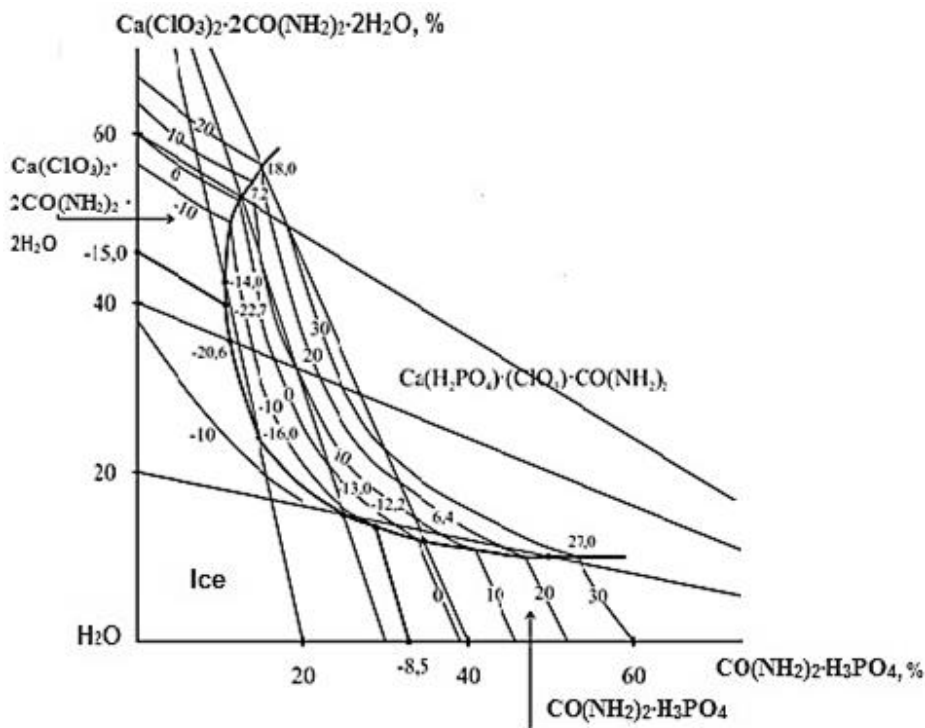


Fig. 1. Polythermal solubility diagram of the system calcium di-urea-chlorate - urea phosphate – water

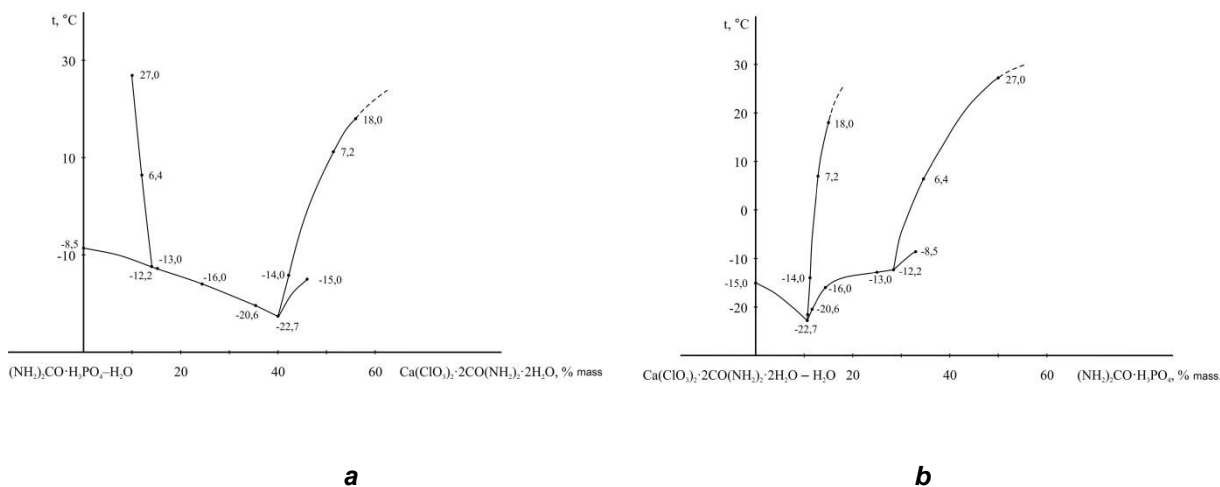


Fig. 2. Polythermal projection of the system of calcium di-urea-chlorate – urea phosphate – water on the sides of urea phosphate – water (a) and calcium di-urea-chlorate – water (b)

Double and triple points of the system  $\text{Ca}(\text{ClO}_3)_2 \cdot 2\text{CO}(\text{NH}_2)_2 - \text{CO}(\text{NH}_2)_2 \cdot \text{H}_3\text{PO}_4 - \text{H}_2\text{O}$ 

Composition of the liquid phase, %			temp. of cryst. °C	Solid phase
$\text{Ca}(\text{ClO}_3)_2 \cdot 2\text{CO}(\text{NH}_2)_2$	$\text{CO}(\text{NH}_2)_2 \cdot \text{H}_3\text{PO}_4$	$\text{H}_2\text{O}$		
46,1	–	53,9	-15,0	Ice + $\text{Ca}(\text{ClO}_3)_2 \cdot 2\text{CO}(\text{NH}_2)_2$
56,0	15,2	28,8	18,0	$\text{Ca}(\text{ClO}_3)_2 \cdot 2\text{CO}(\text{NH}_2)_2 + \text{Ca}(\text{H}_2\text{PO}_4) \cdot (\text{ClO}_3) \cdot \text{CO}(\text{NH}_2)_2$
52,8	12,8	34,4	7,2	The same
42,0	10,7	47,3	-14,0	The same
40,0	10,9	49,1	-22,7	Ice + $\text{Ca}(\text{ClO}_3)_2 \cdot 2\text{CO}(\text{NH}_2)_2 + \text{Ca}(\text{H}_2\text{PO}_4) \cdot (\text{ClO}_3) \cdot \text{CO}(\text{NH}_2)_2$
35,5	13,2	51,3	-20,6	Лед + $\text{Ca}(\text{H}_2\text{PO}_4) \cdot (\text{ClO}_3) \cdot \text{CO}(\text{NH}_2)_2$
24,2	14,8	60,8	-16,0	The same
15,0	25,0	60,0	-13,0	The same
13,9	18,9	67,2	-12,0	Ice + $\text{CO}(\text{NH}_2)_2 \cdot \text{H}_3\text{PO}_4 + \text{Ca}(\text{H}_2\text{PO}_4) \cdot (\text{ClO}_3) \cdot \text{CO}(\text{NH}_2)_2$
12,0	34,6	53,4	6,4	$\text{CO}(\text{NH}_2)_2 \cdot \text{H}_3\text{PO}_4 + \text{Ca}(\text{H}_2\text{PO}_4) \cdot (\text{ClO}_3) \cdot \text{CO}(\text{NH}_2)_2$
10,0	50,0	40,0	27,0	$\text{CO}(\text{NH}_2)_2 \cdot \text{H}_3\text{PO}_4 + \text{Ca}(\text{H}_2\text{PO}_4) \cdot (\text{ClO}_3) \cdot \text{CO}(\text{NH}_2)_2$

To identify ways and places the coordination of the molecules of the compounds studied the IR spectra of it and the source components [Fig. 4 a, b].

The IR spectrum of the urea phosphate absorption band at  $3456\text{--}2927\text{ cm}^{-1}$  attributed to the valent and deformation vibrations of  $\text{NH}_2$  group. The band at  $2340$  and  $1247\text{ cm}^{-1}$  relate to stretching and deformation vibrations (P-O-H) bands.  $\nu_s(\text{H}_2\text{PO}_4)$  and  $\nu_{as}(\text{H}_2\text{PO}_4)$  are observed in the absorption bands and  $1079\text{--}1174\text{--}996\text{ cm}^{-1}$ . The valent and deformation vibrations of groups observed in  $553\text{--}482$  and  $1669\text{ cm}^{-1}$  respectively [10].

The IR spectrum sodium di-urea-chlorate is characterized by  $\nu_s(\text{NH}_2^+)$  and  $\nu_{as}(\text{NH}_2^+)$  at  $3389$  and  $3467\text{ cm}^{-1}$ . The bands at  $1633$  and  $773\text{ cm}^{-1}$  corresponds to valent and deformation vibrations

of CO group. Deformation vibrations of  $\text{NH}_2$  group are observed at  $1471\text{ cm}^{-1}$ . The bands at  $1155$ ,  $967$ ,  $613\text{--}474\text{ cm}^{-1}$  correspond to the valent asymmetric, symmetric and deformation vibrations ( $\text{ClO}_3$ ) group [11].

The IR spectrum of the formed compound characterized by the presence of absorption bands at  $2324$  and  $1239\text{ cm}^{-1}$ , characteristic for valent and deformation vibrations of (P-O-H) group. The bands at  $1635$  and  $567\text{ cm}^{-1}$  correspond to  $\nu(\text{CO})$  and  $\delta(\text{CO})$ , and the band at  $1466\text{ cm}^{-1}$  and  $670\text{--}619\text{ cm}^{-1}$  corresponds to vibrations of  $\delta(\text{NH}_2^+)$ . The band at  $1091\text{ cm}^{-1}$  attributed to the  $\nu_{as}(\text{H}_2\text{PO}_4^-)$ . To the valent symmetric, asymmetric and deformation vibrations of  $\text{ClO}_3^-$  are belonged to the bands at  $970$ ,  $1152$ , and  $491\text{ cm}^{-1}$ .

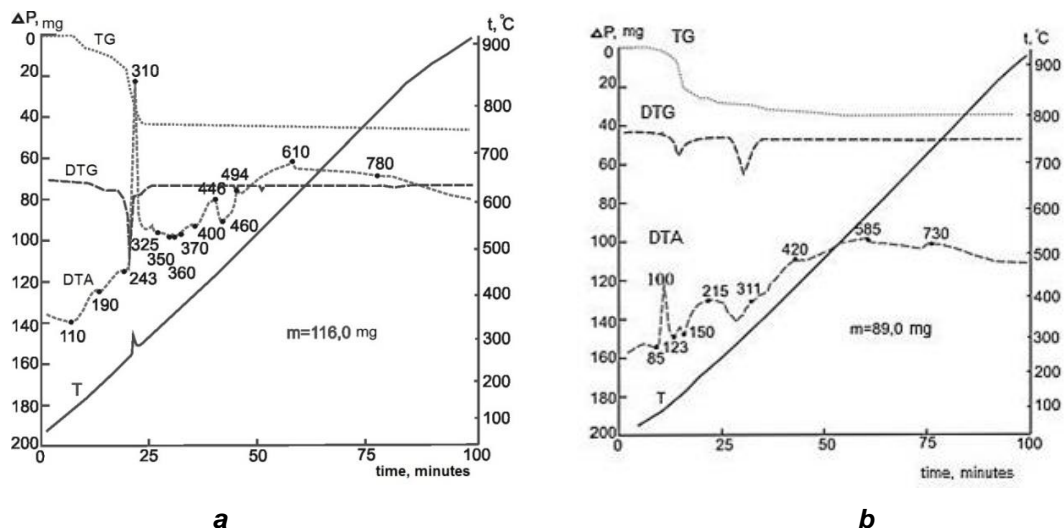


Fig. 3. Derivatograms: a –  $\text{Ca}(\text{ClO}_3)_2 \cdot 2\text{CO}(\text{NH}_2)_2$ ; b –  $\text{Ca}(\text{H}_2\text{PO}_4)(\text{ClO}_3) \cdot \text{CO}(\text{NH}_2)_2$

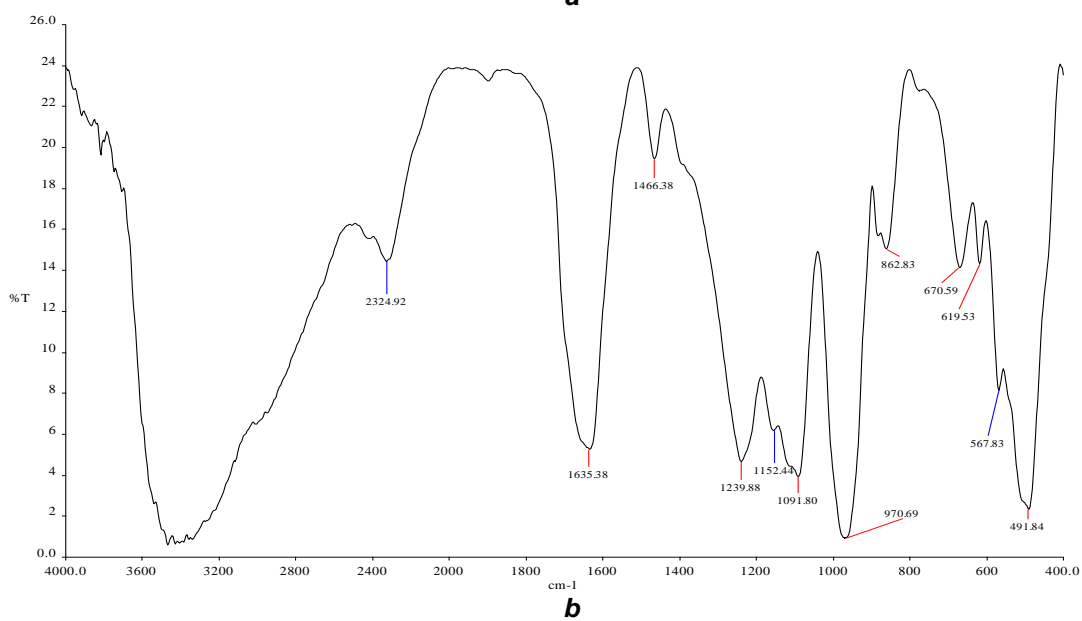
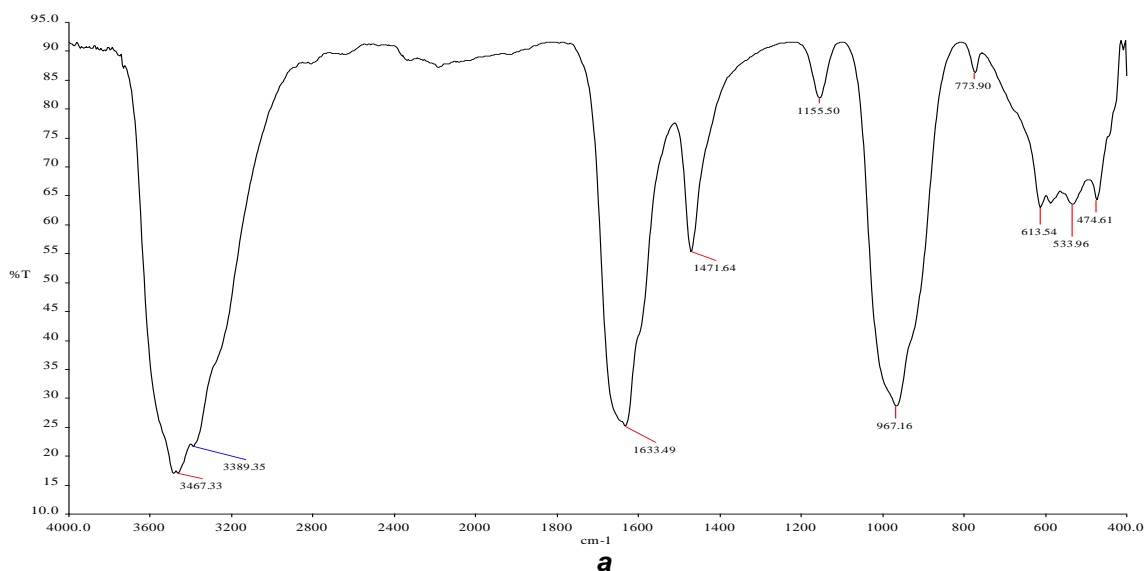


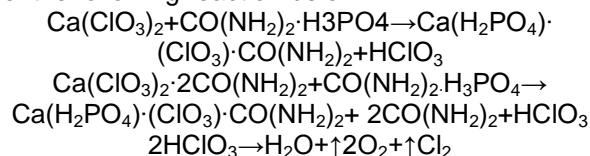
Fig.4. IR- spectra: a –  $\text{Ca}(\text{ClO}_3)_2 \cdot 2\text{CO}(\text{NH}_2)_2$ ; b –  $\text{Ca}(\text{H}_2\text{PO}_4)(\text{ClO}_3) \cdot \text{CO}(\text{NH}_2)_2$

In IR compound  $\text{Ca}(\text{H}_2\text{PO}_4)(\text{ClO}_3)\text{CO}(\text{NH}_2)_2$  composition relatively spectrum of starting components it is observed that fringe shift characterizing P-O-H, CO,  $\text{NH}_2^+$  and  $\text{ClO}_3^-$  groups apparently it is said on concern of these groups for formation of coordinating bonds of complex.

### CONCLUSION

Thus, it was studied that interaction calcium di-urea-chlorate with urea phosphate in water system in a wide range of temperature and concentration. There had been revealed temperature and concentration bounds of existence of solid phases of system components and generation of complex compound  $\text{Ca}(\text{H}_2\text{PO}_4)(\text{ClO}_3) \cdot \text{CO}(\text{NH}_2)_2$ , which was identified by chemical and physic-chemical analysis. As seen from results conducted by we study, above mentioned compound  $\text{Ca}(\text{H}_2\text{PO}_4)(\text{ClO}_3) \cdot \text{CO}(\text{NH}_2)_2$  was formed at interaction components in triple system:  $\text{Ca}(\text{ClO}_3)_2 - \text{CO}(\text{NH}_2)_2 \cdot \text{H}_3\text{PO}_4 - \text{H}_2\text{O}$  [9] и  $\text{Ca}(\text{ClO}_3)_2 \cdot$

$2\text{CO}(\text{NH}_2)_2 - \text{CO}(\text{NH}_2)_2 \cdot \text{H}_3\text{PO}_4 - \text{H}_2\text{O}$  as a result of the following reaction below



The IR spectrum of the compound relative to the spectra of the source components is observed shift of the bands describing P-O-N, CO,  $\text{NH}_2^+$  and  $\text{ClO}_3^-$  groups that, apparently, speaks about the participation of these groups in the formation of coordination bonds of the complex.

The data obtained based on study of the interaction of components in the water system with the participation of calcium di-urea-chlorate and urea phosphate, are of interest for subsequent development of technology for complex active defoliant of cotton due to the content in it of some physiologically active groups.

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#### **Критерии авторства**

Хамдамова Ш.Ш. выполнила экспериментальную работу, на основании полученных результатов провела обобщение и написала рукопись. Хамдамова Ш.Ш. имеет на статью авторские права и несет ответственность за плагиат.

#### **Конфликт интересов**

Автор заявляет об отсутствии конфликта интересов.

#### **СВЕДЕНИЯ ОБ АВТОРАХ** **Принадлежность к организации**

**Шохида Ш. Хамдамова**  
Институт общей и неорганической химии Академии наук Республики Узбекистан, К.т.н., с.н.с. лаборатории «Дефолиантов»  
hamdamova79@mail.ru

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#### **Contribution**

Khamdamova Sh.Sh. carried out the experimental work, on the basis of the results summarized the material and wrote the manuscript. Khamdamova Sh.Sh. have author's rights and bear responsibility for plagiarism.

#### **Conflict of interests**

The author declare no conflict of interests regarding the publication of this article.

#### **AUTHORS' INDEX** **Affiliations**

**Shokhida Sh. Khamdamova**  
Institute of General and Inorganic Chemistry of Uzbek Academy of Science  
Ph.D. (Engineering), Senior Researcher  
Defoliants Laboratory  
hamdamova79@mail.ru

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