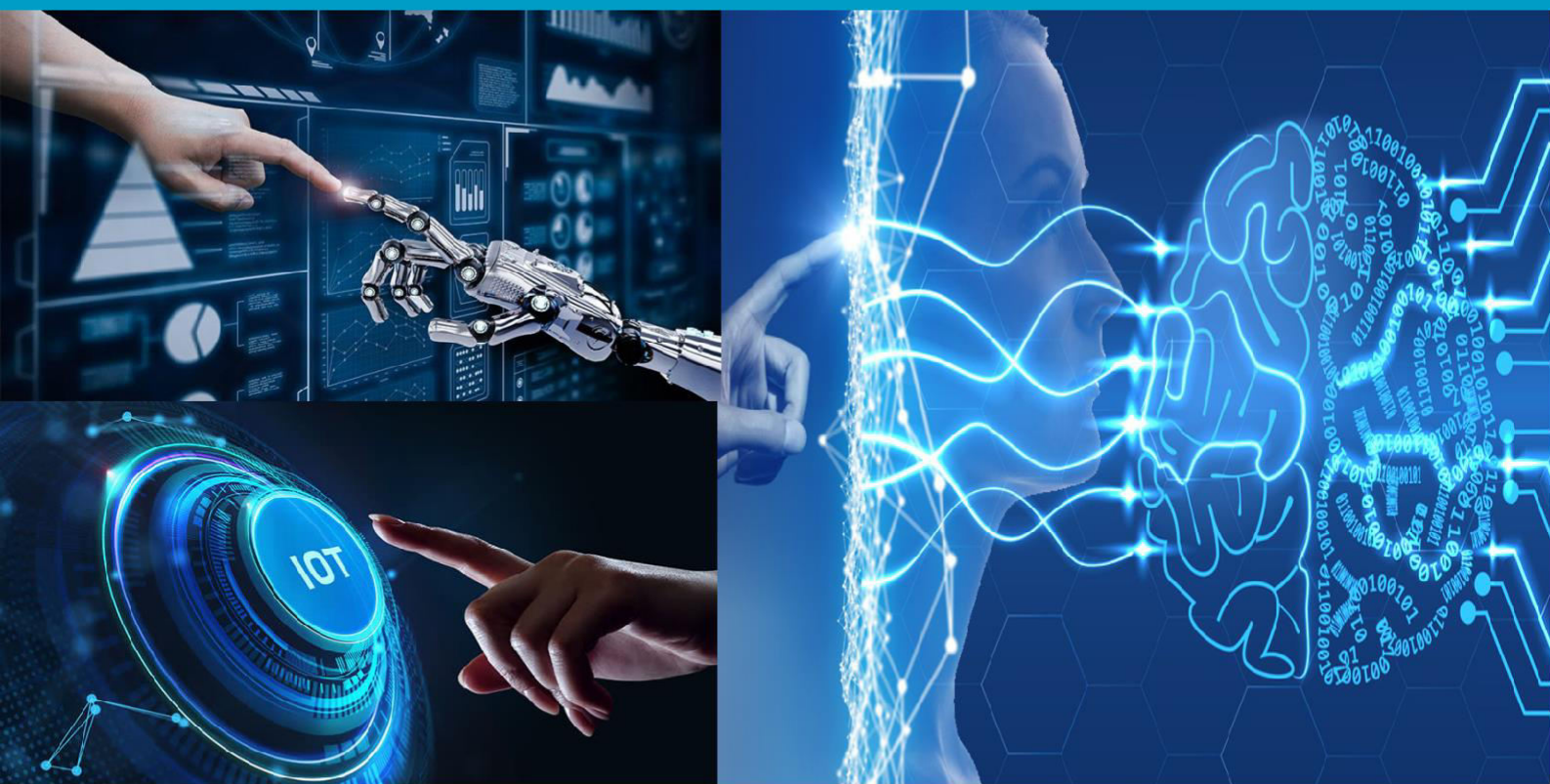




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Study of the Interaction of Components in the System Urea – Nitric Acid – Water

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Annotation. In order to obtain slow-acting fertilizers, we studied the solubility of the components in the $\text{CO}(\text{NH}_2)_2\text{-HNO}_3\text{-H}_2\text{O}$ system using the visual-polythermal method. Based on the data of solubility polytherms of binary systems and internal sections, a polythermal solubility diagram of the $\text{CO}(\text{NH}_2)_2\text{-HNO}_3\text{-H}_2\text{O}$ system was constructed, which delineates the crystallization fields of four solid phases: ice, $\text{CO}(\text{NH}_2)_2$, HNO_3 and the new phase $\text{CO}(\text{NH}_2)_2 \cdot \text{HNO}_3$. These fields converge at two triple invariant points of coexistence of three solid phases, for which the crystallization temperatures and compositions of the equilibrium solution have been established.

Keywords: System, component, solution, crystallization, urea, nitric acid, water, solubility, solid and liquid phases.

I. INTRODUCTION

In a set of measures aimed at increasing crop production, the use of mineral fertilizers is important. In almost all soil and climatic zones, mineral nitrogen fertilizers have a significant impact on the production process. Rational use of fertilizers dramatically increases crop yields and soil fertility, improves product quality [1-6].

Among the nitrogen fertilizers used, the most promising is urea, a highly concentrated ballast-free fertilizer.

The growth in the production and consumption of urea is explained by a number of its advantages - high nitrogen content, explosion safety, significantly lower hygroscopicity and caking compared to other nitrogen fertilizers. Urea nitrogen is easily absorbed by plants. The advantage of urea is its proximity to physiologically neutral fats [7].

Recently, there has been increased interest in the production of slow-release nitrogen fertilizers based on urea and urea nitrate. There are a number of patents and scientific works [8-15] for the production of slow-acting nitrogen fertilizers based on urea, as well as for use as an ethylene-producing additive to chlorate-containing defoliant [16].

For the production of slow-release fertilizers, certain crystalline compounds of urea with mineral acids are of particular interest. Among them, compounds of urea with nitric and oxalic acids - urea nitrate and oxalate, which are less soluble than other nitrogen fertilizers and contain the main plant nutrient - nitrogen in amide and nitrate forms, are of particular importance. When dissolved in water or soil solutions, they slowly decompose into their original components with the release of free acid, due to which one should expect the conversion of calcium, magnesium and sesquioxide forms and phosphates fixed in the soil into digestible forms [17].

To determine the parameters of the technological regime of the process of coating urea granules with nitric acid, data characterizing the interaction of urea with nitric acid in wide ranges of temperatures and concentrations is required.

Methods and materials. The study of phase equilibria in physicochemical systems was carried out visually using the polythermal method [18]. The essence of the visual-polythermic method is to visually observe the temperature of the appearance of the first crystals with uniform and slow cooling or the disappearance of the last crystals with uniform heating and continuous stirring of the solutions. The device for determining solubility is a test tube closed with a stopper, a glass stirrer, and a thermometer with a division value of 0.10C. For uniform cooling, the test tube is placed in an outer test tube - a coupling, located in the cooling mixture. Heating is also carried out through the coupling. Cooling is carried out in dewar flasks with liquid nitrogen.

Measurements of the phase characteristics of the samples under study were carried out using a Panalytical Empyrean powder X-ray diffractometer. All control over the operation of the equipment is carried out via a computer using the Data Collector program, and the analysis of x-ray diffraction patterns was carried out using the High Score program with the PDF 2013 database. The measurements were carried out at room temperature in the range of angles 2θ , in the range from 5° to 90° in step-by-step scanning mode with a step of 0.013 degrees and a signal accumulation time at a point of 5 s.

Results and discussion. The authors of [17] studied the solubility in the $\text{CO}(\text{NH}_2)_2\text{-HNO}_3\text{-H}_2\text{O}$ system in the temperature range from complete freezing -43.3 to 50°C using 12 internal sections. At the same time, the authors found that the constructed polythermal diagram outlines six crystallization fields: urea nitrate, α -, β -, γ -modifications of urea, trihydrate nitric acid and ice.

A number of works are devoted to the study of solubility in binary systems: urea – water and nitric acid – water [19].

Solubility in the urea – nitric acid system That water we studied from the temperature of complete freezing of the system (-44.80°C) to 40°C using eight internal sections (Fig. 1).

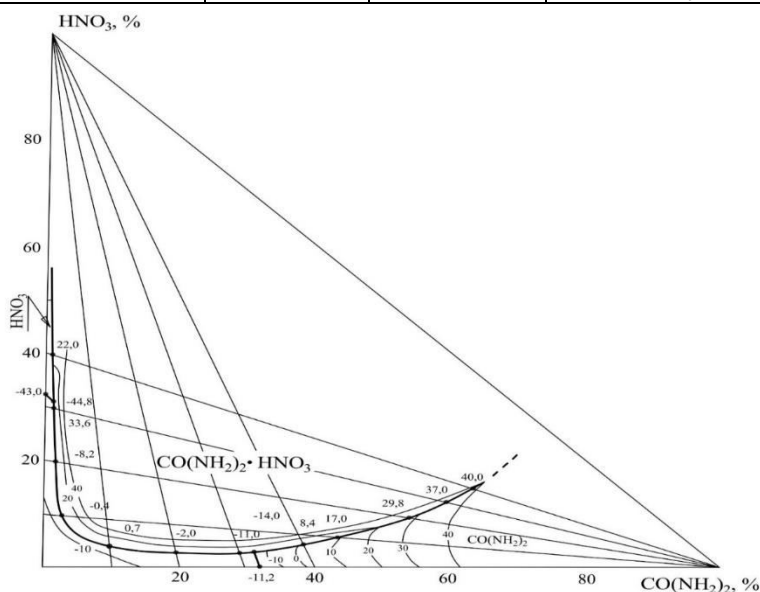
Based on literature data [20-23] and solubility polytherms of binary systems and internal sections, we constructed a polythermal solubility diagram for the $\text{CO}(\text{NH}_2)_2\text{-HNO}_3\text{-H}_2\text{O}$ system, which delineates the crystallization fields of four solid phases: ice, $\text{CO}(\text{NH}_2)_2$, HNO_3 and the new phase $\text{CO}(\text{NH}_2)_2\cdot\text{HNO}_3$. These fields converge at two triple invariant points of coexistence of three solid phases, for which the crystallization temperatures and compositions of the equilibrium solution have been established (Table 1).

Rice. 1. Polythermal solubility diagram of the system urea – nitric acid – water

Table 1

Double and triple node points of the $\text{HNO}_3\text{-CO}(\text{NH}_2)_2\text{-H}_2\text{O}$ system

Liquid phase composition, %			Pace. Chris,	Solid phase
HNO_3	$\text{CO}(\text{NH}_2)_2$	H_2O		
32,8	-	67,2	-43,0	$\text{HNO}_3\cdot 3\text{H}_2\text{O}$
30,9	1,6	67,5	-44,8	$\text{HNO}_3\cdot 3\text{H}_2\text{O} + \text{HNO}_3\cdot \text{CO}(\text{NH}_2)_2$



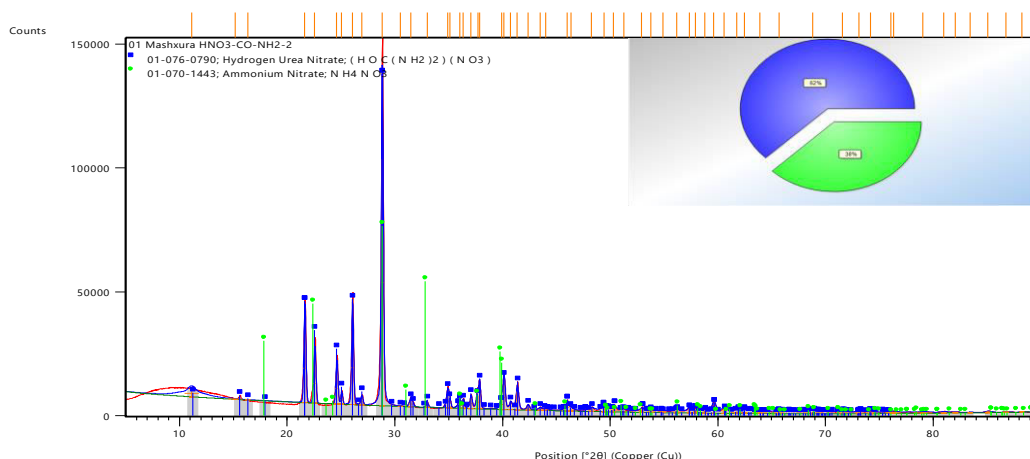
29,6	1,5	68,9	-33,4	$\text{Лед} + \text{HNO}_3\cdot \text{CO}(\text{NH}_2)_2$
19,6	1,6	78,8	-8,2	Same
9,8	2,2	88,0	-0,4	Same
4,0	9,6	86,4	0,7	Same
3,0	19,6	77,4	-0,2	Same
3,0	29,8	67,2	-11,0	Same
-	32,0	68,0	-11,2	$\text{Лед} + \text{CO}(\text{NH}_2)_2$
3,6	31,0	65,4	-14,0	$\text{Лед} + \text{CO}(\text{NH}_2)_2 + \text{HNO}_3\cdot \text{CO}(\text{NH}_2)_2$
4,8	38,2	57,0	8,4	$\text{HNO}_3\cdot \text{CO}(\text{NH}_2)_2 + \text{CO}(\text{NH}_2)_2$
6,0	42,8	51,2	17,0	Same
9,8	53,0	37,2	29,8	Same
12,4	59,7	27,9	37,0	Same
15,0	63,8	21,2	40,0	Same
39,8	1,1	59,1	22,0	$\text{HNO}_3 + \text{HNO}_3\cdot \text{CO}(\text{NH}_2)_2$

The polythermal diagram shows solubility isotherms every 100C. Projections of polythermal solubility curves onto the corresponding water lateral sides of the system were constructed.

According to the data obtained in our studies, the formation of crystallization fields of α -, β -, γ -modifications of urea and trihydrate nitric acid is not observed, in contrast to the data obtained in [17].

Rice. 2. X-ray diffraction pattern of the formed compound with the composition: $\text{HNO}_3 \cdot \text{CO}(\text{NH}_2)_2$

With the study of the system, it was established that the crystallization field of urea nitrate occupies a larger part of the polythermal diagram than the crystallization fields of the initial components. This indicates its low solubility in



this system.

The compound (urea nitrate) was isolated in crystalline form and identified by X-ray phase physicochemical analysis.

As follows from the x-ray diffraction pattern, the compound contains 62% urea nitrate, 38% ammonium nitrate. This is obviously due to the partial decomposition of urea in nitric acid solutions.

Conclusion. Thus, the solubility of the components in the $\text{CO}(\text{NH}_2)_2\text{-HNO}_3\text{-H}_2\text{O}$ system was studied using the visual-polythermal method. Based on the data of solubility polytherms of binary systems and internal sections, a polythermal solubility diagram of the $\text{CO}(\text{NH}_2)_2\text{-HNO}_3\text{-H}_2\text{O}$ system was constructed, which delineates the crystallization fields of four solid phases: ice, $\text{CO}(\text{NH}_2)_2$, HNO_3 and the new phase $\text{CO}(\text{NH}_2)_2 \cdot \text{HNO}_3$. These fields converge at two triple invariant points of coexistence of three solid phases, for which the crystallization temperatures and compositions of the equilibrium solution have been established.

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