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## INTERACTION OF UREA WITH INORGANIC ACIDS

**Yulbarsova M. V.<sup>1</sup>, Kucharov B. Kh.<sup>1</sup>,  
Erkayev A. U.<sup>2</sup>, Zakirov B. S.<sup>1</sup>**

<sup>1</sup> Institute of General and Inorganic Chemistry Academy  
of Sciences of the Republic of Uzbekistan,

<sup>2</sup> Tashkent Institute of Chemical Technology, Tashkent, Uzbekistan

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### Abstract

This article presents research data on systems involving urea, nitrate, sulfuric, and phosphoric acids using the polythermal method in the production of complex fertilizers. Polythermal solubility diagrams were constructed, and the obtained data were confirmed by results from electron microscopy and X-ray structural analysis methods.

**Keywords:** *research, fertilizer, solution, crystallization, nitric, phosphoric, and sulfuric acids, water, solubility, polytherm, temperature, equilibrium*

### Introduction

Urea, also known as carbamide, is one of the most widely used fertilizers in agriculture and industry. Urea is a white crystalline powder that is easily soluble in water. Its excellent solubility and chemical stability make it widely utilized across various industrial sectors. The high nitrogen content of urea makes it a valuable source of nutrients for plants.

Therefore, studying the physicochemical properties of urea is crucial for optimizing its use in different fields. Understanding the characteristics of this substance allows for the development of new application methods, improvement of existing technologies, and enhancement of its effectiveness in various areas of activity (Dor-

meshkin O. B., Mumunov N. Sh., Khoshimov B. T., 2024).

The high chemical activity of urea facilitates its ability to easily form various compounds. Numerous urea compounds with metal salts and acids have been identified in the solid state, where it can act as a neutral molecule or exhibit positive or negative charges (Molodkin A. K., Ellert G. V., Ivanova O. M., Skotnikova G. A., 1967).

The literature has explored the interaction processes of urea with sulfuric, nitric, and phosphoric acids in solution using spectrophotometric methods. The compositions of the resulting compounds have been determined, and their stability constants have been calculated (Alimova G. A., Saibova M. T., Prisekina L. I., 1982).

### Methods and materials

Phase equilibrium studies in physico-chemical systems were conducted using the visual polythermal method (Kirgintsev A. N., Trushnikova L. N., Lavrentieva V. G., 1972). This method involves visually observing the temperature at which the first crystals appear during uniform and slow cooling, or the disappearance of the last crystals during uniform heating with continuous stirring of solutions. The equipment used includes a test tube sealed with a stopper, a glass stirrer, and a thermometer with a scale interval of 0.1 °C. For uniform cooling, the test tube is placed in an outer test tube – called a jacket – that is immersed in a cooling mixture. Heating is also performed through the jacket. Cooling is achieved using liquid nitrogen in Dewar vessels.

Phase characteristics of the samples were measured using a powder X-ray diffractometer, Panalytical Empyrean. Equipment control was managed via a computer with the Data Collector software, and X-ray diffraction patterns were analyzed using the High Score program with the PDF 2013 database. X-ray phase analysis of the samples was performed on a Panalytical Empyrean diffractometer equipped with a Cu tube ( $K\alpha_1 = 1.5406 \text{ \AA}$ ). Measurements were conducted at room temperature over a  $2\theta$  angle range from  $5^\circ$  to  $90^\circ$  in step scanning mode with a step size of  $0.013^\circ$  and a signal accumulation time of 5 seconds per point.

Scanning electron microscopy was performed using a scanning electron microscope EVO MA-10 (Carl Zeiss, Germany), equipped with an energy-dispersive X-ray (EDX) microanalysis system (Oxford Instruments, UK), which can detect all elements of the periodic table of D. I. Mendeleev starting from boron. The microscope has a resolution of up to 2.5 nm at an accelerating voltage of 30 kV (secondary electron imaging), with accelerating voltage ranging from 1.0 to 30 kV, magnification from  $\times 10$  to  $\times 500.000$ , and a beam current of up to 200 nA. Sample preparation was carried out using equipment from Jeol (Japan) and Gatan (USA). Results were analyzed using specialized programs, such as INCA Point & ID for qualitative and quantitative element analysis, Mapping and QuantMap for map-

ping, and Feature for quantitative phase distribution and inclusions.

### Results and discussion

To characterize the behavior of urea with inorganic acids in an aqueous environment, the solubility in the systems urea – nitric acid – water, urea – phosphoric acid – water, and urea – sulfuric acid – water was studied over a wide range of temperatures and concentrations using the visual polythermal method.

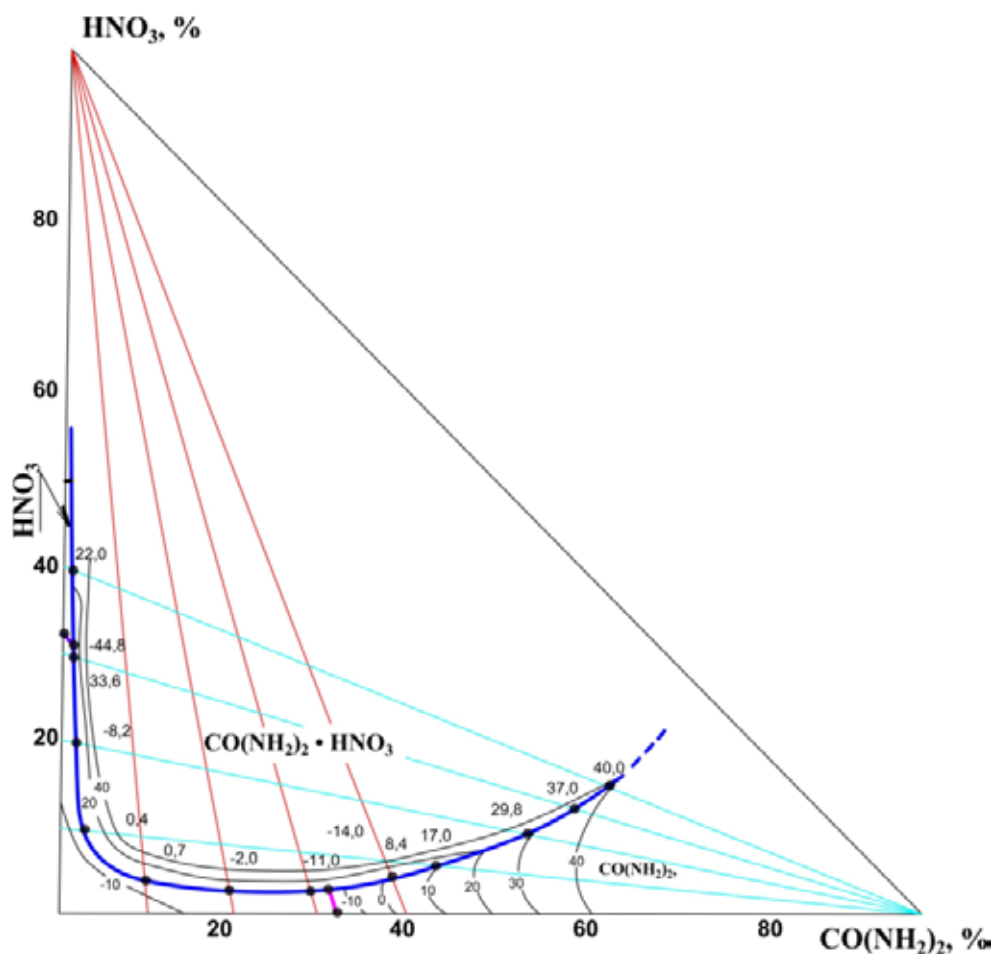
Binary systems of urea – water, nitric acid – water, phosphoric acid – water, and sulfuric acid – water, which are components of the studied system, have been examined by various authors (Kirgintsev A. N., Trushnikova L. N., Lavrentieva V. G., 1972).

The urea – nitric acid – water system was investigated through eight internal sections: I–IV from the side of  $\text{CO}(\text{NH}_2)_2\text{-H}_2\text{O}$  towards the  $\text{HNO}_3$  pole, and V–VIII from the  $\text{HNO}_3\text{-H}_2\text{O}$  side towards the  $\text{CO}(\text{NH}_2)_2$  vertex (Fig. 1).

Based on the polythermal solubility of the binary systems and internal sections, a polythermal solubility diagram for the  $\text{CO}(\text{NH}_2)_2\text{-HNO}_3\text{-H}_2\text{O}$  system was constructed, ranging from the complete freezing temperature ( $-44.8 \text{ }^\circ\text{C}$ ) to  $40 \text{ }^\circ\text{C}$ . This diagram delineates the crystallization fields of four solid phases: ice,  $\text{CO}(\text{NH}_2)_2$ ,  $\text{HNO}_3 \times 3\text{H}_2\text{O}$ , and a new phase  $\text{CO}(\text{NH}_2)_2 \times \text{HNO}_3$ . Two triple points of the system, corresponding to the simultaneous crystallization of three different solid phases, were identified (Table 1). Isotherms were drawn on the polythermal solubility diagram within the crystallization fields at intervals of  $10 \text{ }^\circ\text{C}$ . Projections of the polythermal curves on the urea – water and nitric acid – water sides were constructed.

The study revealed that the crystallization field of urea nitrate occupies a larger portion of the polythermal diagram compared to the crystallization fields of the initial components, indicating its low solubility in this system. The urea nitrate compound was isolated in crystalline form and identified using chemical and physicochemical analysis methods.

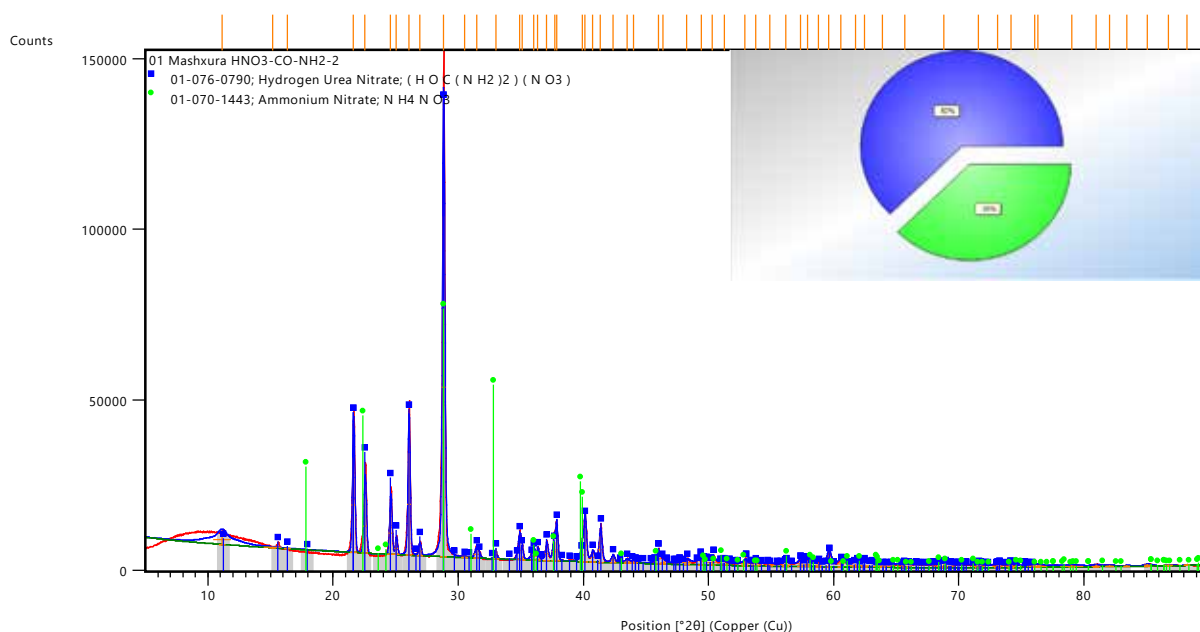
**Figure 1.** Polythermal solubility diagram of the urea – nitric acid – water system



**Table 1.** Double and triple points of the  $HNO_3$ - $CO(NH_2)_2$ - $H_2O$  system

Liquid phase composition, %			Temp. crystallization, °C	Solid phases
$HNO_3$	$CO(NH_2)_2$	$H_2O$		
32.8	–	67.2	–43.0	Ice+ $HNO_3 \times 3H_2O$
30.9	1.6	67.5	–44.8	Ice+ $HNO_3 \times 3H_2O$ + $HNO_3 \times CO(NH_2)_2$
29.6	1.5	68.9	–33.4	Ice+ $HNO_3 \times CO(NH_2)_2$
19.6	1.6	78.8	–8.2	Too
9.8	2.2	88.0	–0.4	Too
4.0	9.6	86.4	0.7	Too
3.0	19.6	77.4	–0.2	Too
3.0	29.8	67.2	–11.0	Too
–	32.0	68.0	–11.2	Ice+ $CO(NH_2)_2$
3.6	31.0	65.4	–14.0	Ice+ $CO(NH_2)_2$ + $HNO_3 \times CO(NH_2)_2$
4.8	38.2	57.0	8.4	$HNO_3 \times CO(NH_2)_2$ + $CO(NH_2)_2$
6.0	42.8	51.2	17.0	Too
9.8	53.0	37.2	29.8	Too
12.4	59.7	27.9	37.0	Too
15.0	63.8	21.2	40.0	Too
39.8	1.1	59.1	22.0	$HNO_3$ + $HNO_3 \times CO(NH_2)_2$

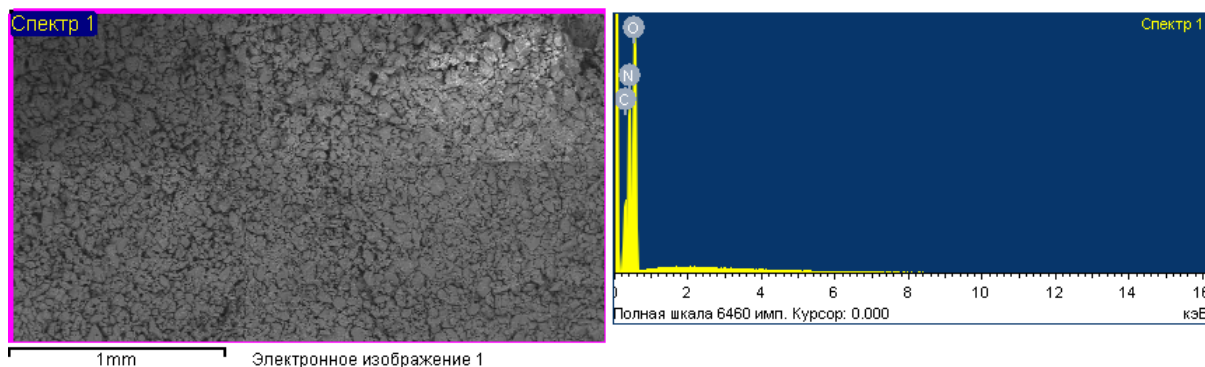
**Figure 2.** X-ray diffraction pattern of the compound:  $HNO_3 \times CO(NH_2)_2$



Based on the X-ray analysis, the compound contains 62% urea nitrate and 38% ammonium nitrate (Fig. 2). This is likely due to the partial decomposition of urea in nitric acid solutions. To study the ele-

mental and mineralogical composition, a microscopic analysis was conducted using scanning electron microscopy (SEM). The elemental composition of the product was determined.

**Figure 3.** Microstructure of urea nitrate



Based on the study (Fig. 3), the elemental composition of urea nitrate was determined as follows (in %): carbon (C) – 12.21, oxygen (O) – 54.46, nitrogen (N) – 33.33.

To characterize the interaction of components in the  $CO(NH_2)_2-H_3PO_4-H_2O$  system, the system was studied under polythermal conditions, ranging from the eutectic freezing point of  $-89.4\text{ }^\circ\text{C}$  to  $40\text{ }^\circ\text{C}$  (Figure 1). The components of this system are thoroughly discussed in the literature (Kirgintsev A.N., Trushnikova L.N., Lavrentieva V.G. 1972; Yulbarsova M.V., Kucharov B.K., Zakirov B.S., Erkaev A.U.,

2023; Yulbarsova M.V., Kucharov B.K., Zakirov B.S., Erkaev A.U., Nazirova R.M. 2024; Yulbarsova M.V., Kucharov B.K., Zakirov B.S., Erkaev A.U., Usmanova Z.D., 2023; Molodkin A.K., Ellert G.V., Ivanova O.M., Skotnikova G.A., 1967). Our data are consistent with the literature findings.

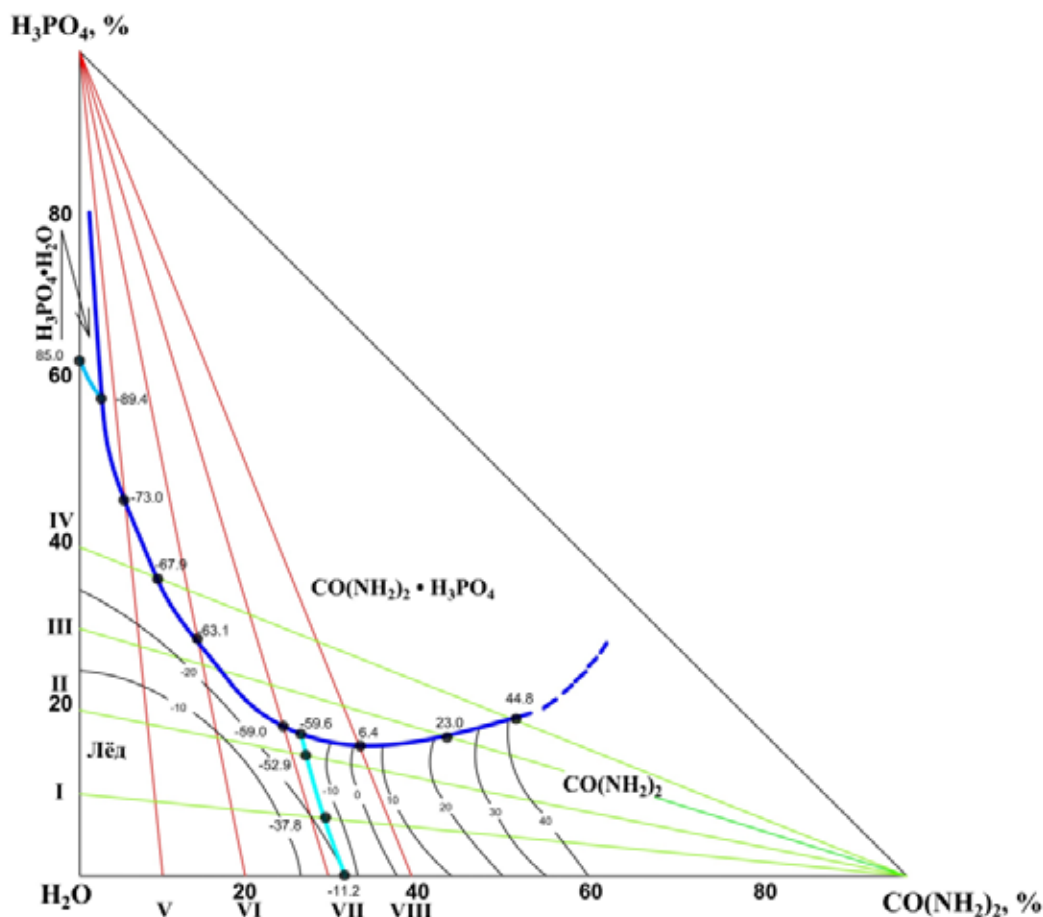
The authors of (Yulbarsova M.V., Kucharov B.K., Zakirov B.S., Erkaev A.U., 2023), in their study of the urea-phosphoric acid-water system, established that limited type solid solutions are formed in this system, and they also identified the eutectic point, which corresponds to a composi-

tion of 19.2%  $H_3PO_4$ , 34.3%  $CO(NH_2)_2$ , and 46.5%  $H_2O$ .

In our study of the urea-phosphoric acid-water system, eight internal sections were investigated, with sections I–IV ranging from

the phosphoric acid-water side to the  $H_3PO_4$  vertex, and sections V–VIII ranging from the  $CO(NH_2)_2$ - $H_2O$  side to the phosphoric acid vertex.

**Figure 4.** Polythermal Solubility diagram of the urea – phosphoric acid – water system



Based on the polythermal solubility data of the side systems and internal sections, a polythermal solubility diagram of the urea-phosphoric acid-water system was constructed,

delineating the crystallization fields of ice,  $CO(NH_2)_2$ ,  $H_3PO_4 \times H_2O$ , and the compound  $CO(NH_2)_2 \times H_3PO_4$ . Two nodal triple points of the system were identified (Table 2).

**Table 2.** Double and triple nodal points of the  $H_3PO_4$ - $CO(NH_2)_2$ - $H_2O$  system

Liquid phase composition, %			Temp. crystallization, °C	Solid phases
$H_3PO_4$	$CO(NH_2)_2$	$H_2O$		
–	32.0	68.0	–11.2	Ice+ $CO(NH_2)_2$
57.8	2.8	39.4	–89.4	Ice+ $H_3PO_4$ + $H_3PO_4 \times CO(NH_2)_2$
45.6	5.8	48.6	–73.0	Ice+ $H_3PO_4 \times CO(NH_2)_2$
36.2	9.1	54.7	–67.9	Too
29.0	14.0	57.0	–63.1	Too
18.1	24.2	57.7	–59.0	Too
14.8	27.4	42.2	–52.9	Ice+ $CO(NH_2)_2$
7.4	29.8	61.8	–37.8	Too
–	32.0	68.0	–11.2	Too



Liquid phase composition, %			Temp. crystallization, °C	Solid phases
H <sub>3</sub> PO <sub>4</sub>	CO(NH <sub>2</sub> ) <sub>2</sub>	H <sub>2</sub> O		
17.0	26.9	56.1	-59.6	Ice+CO(NH <sub>2</sub> ) <sub>2</sub> + H <sub>3</sub> PO <sub>4</sub> × CO(NH <sub>2</sub> ) <sub>2</sub>
16.0	33.8	50.2	6.4	H <sub>3</sub> PO <sub>4</sub> × CO(NH <sub>2</sub> ) <sub>2</sub> +CO(NH <sub>2</sub> ) <sub>2</sub>
17.0	44.2	38.8	23.0	Too
19.1	52.8	28.1	44.8	Too
62.6	–	37.4	-85.0	Ice+H <sub>3</sub> PO <sub>4</sub> × H <sub>2</sub> O

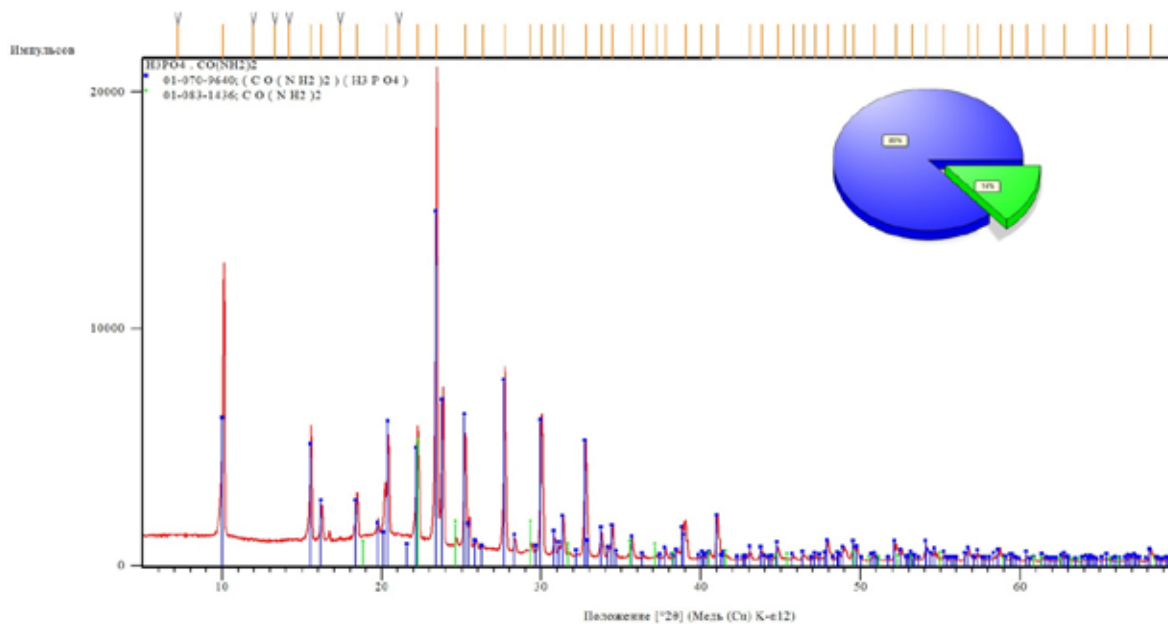
On the polythermal solubility diagram, isotherms of solubility are plotted within the crystallization fields at every 10 °C interval. Projections of the polythermal curves onto the sides of the urea-water and phosphoric acid-water systems are also constructed (Fig. 4).

In the studied system, an incongruently soluble compound of composition CO(NH<sub>2</sub>)<sub>2</sub>

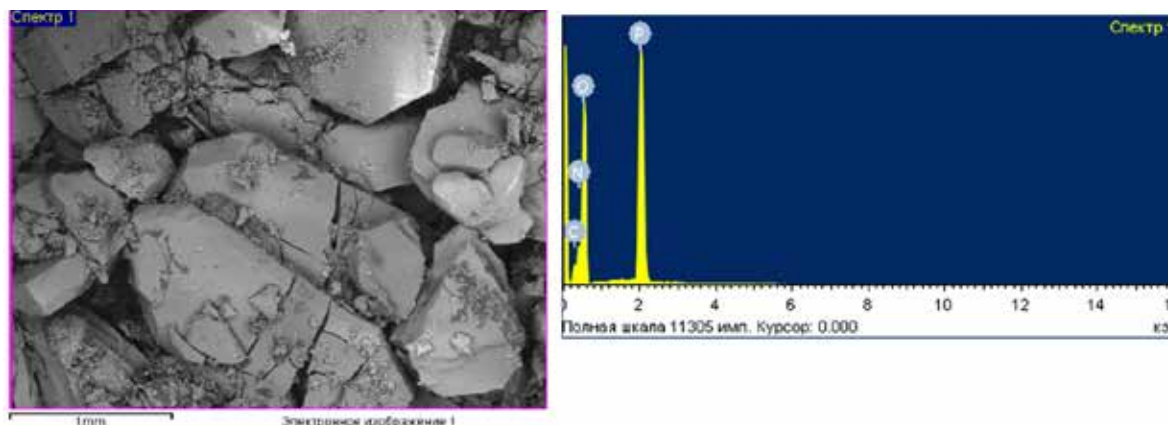
× H<sub>3</sub>PO<sub>4</sub> is formed, which occupies a large area of the diagram. This is due to its lower solubility in the system compared to other components.

The compound was isolated in crystalline form and identified using chemical and physico-chemical analysis methods.

**Figure 5.** X-ray diffraction pattern of the formed compound: H<sub>3</sub>PO<sub>4</sub> × CO(NH<sub>2</sub>)<sub>2</sub>



**Figure 6.** Microstructure of urea phosphate



According to the X-ray analysis, the compound contains 86% urea phosphate and 14% urea (Fig. 5).

A microscopic analysis was conducted using scanning electron microscopy (SEM) to study the elemental and mineralogical composition.

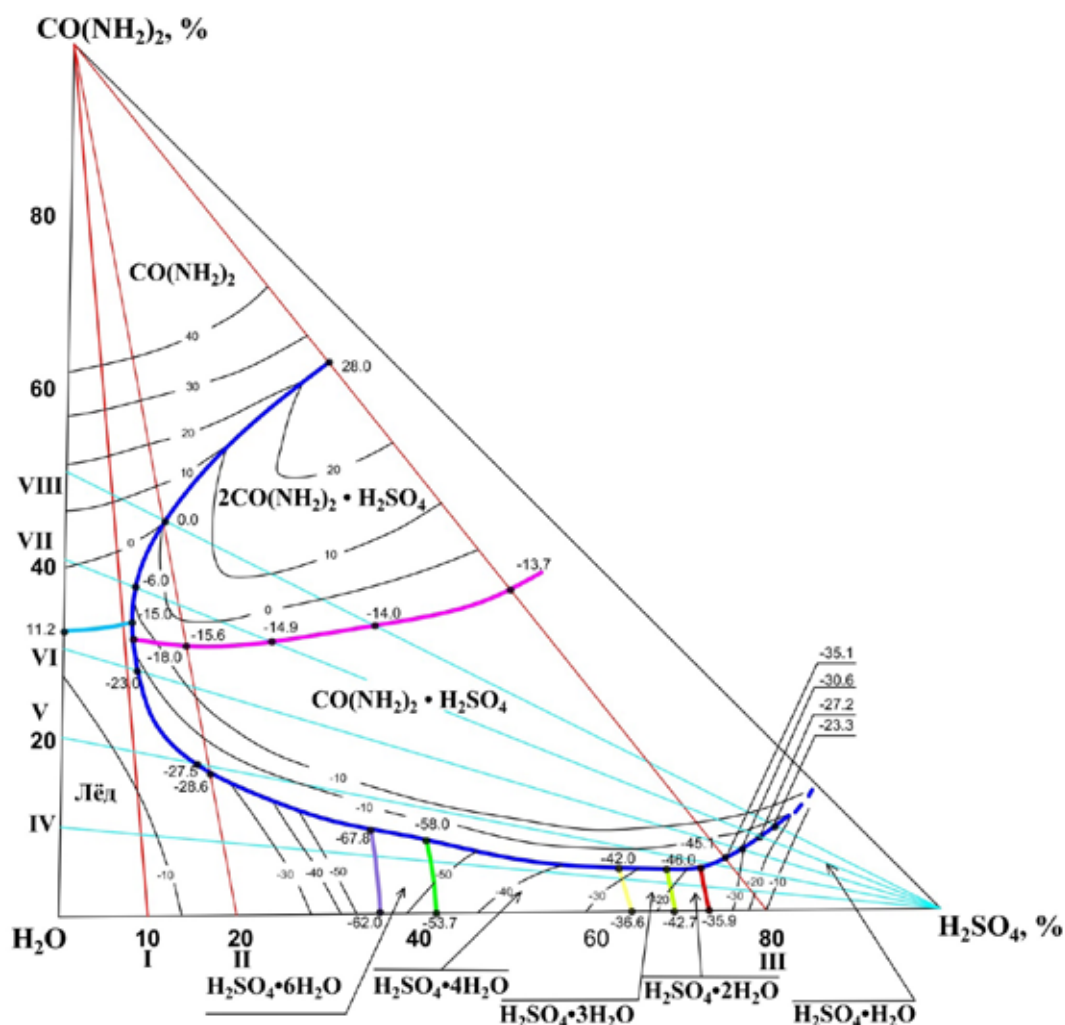
Based on the study (Fig. 6), the elemental composition of urea nitrate was determined as follows (in %): carbon (C) – 13.13, oxygen (O) – 51.67, nitrogen (N) – 19.59, phosphorus (P) – 15.61.

The polythermal solubility of the  $\text{CO}(\text{NH}_2)_2\text{-H}_2\text{SO}_4\text{-H}_2\text{O}$  system was studied using

eight internal sections (Fig. 7). Sections I–III were taken from the sulfuric acid-water side towards the urea pole, while sections IV–VIII were taken from the urea-water side towards the sulfuric acid vertex.

Based on literature data (Molodkin A.K., Ellert G.V., Ivanova O.M., Skotnikova G.A. 1967; Alimova G.A., Saibova M.T., Prisekina L.I. 1982; Nurakhmetov N.N., Beremzhano B.A., 1978) and the polythermal solubility of binary systems and internal sections, a solubility diagram of the urea-sulfuric acid-water system was constructed for temperatures ranging from  $-67.8\text{ }^\circ\text{C}$  to  $40\text{ }^\circ\text{C}$  (Fig. 7).

**Figure 7.** Polythermal solubility diagram of the urea – sulfuric acid – water system



This diagram delineates the crystallization fields of ice, urea, and the six, four, three, two, and monohydrated forms of sulfuric acid, as well as two new compounds:  $\text{CO}(\text{NH}_2)_2 \times \text{H}_2\text{SO}_4$  and  $2\text{CO}(\text{NH}_2)_2 \times \text{H}_2\text{SO}_4$ . These fields converge at seven triple points in the system, for which the crystalliza-

tion temperatures and compositions of the equilibrium solution were determined. The characteristics of the double and triple nodal points of the  $\text{CO}(\text{NH}_2)_2\text{-H}_2\text{SO}_4\text{-H}_2\text{O}$  system are provided in Table 3.

**Table 3.** Double and triple nodal points of the  $\text{CO}(\text{NH}_2)_2\text{-H}_2\text{SO}_4\text{-H}_2\text{O}$  system

Liquid phase composition, %			Temp. crystallization, °C	Solid Phases
$\text{CO}(\text{NH}_2)_2$	$\text{H}_2\text{SO}_4$	$\text{H}_2\text{O}$		
32.0	–	68.0	–11.2	Ice+ $\text{CO}(\text{NH}_2)_2$
32.3	8.2	59.5	–15.0	Ice+ $\text{CO}(\text{NH}_2)_2+2\text{CO}(\text{NH}_2)_2 \times \text{H}_2\text{SO}_4$
31.0	7.9	61.1	–18.0	Ice+ $2\text{CO}(\text{NH}_2)_2 \times \text{H}_2\text{SO}_4+\text{CO}(\text{NH}_2)_2 \times \text{H}_2\text{SO}_4$
27.8	8.8	63.4	–23.0	Ice+ $\text{CO}(\text{NH}_2)_2 \times \text{H}_2\text{SO}_4$
16.9	15.6	67.5	–27.5	Too
16.0	17.0	67.0	–28.6	Too
9.4	35.1	55.5	–67.8	Ice+ $\text{H}_2\text{SO}_4 \times 6\text{H}_2\text{O}+\text{CO}(\text{NH}_2)_2 \times \text{H}_2\text{SO}_4$
8.3	41.2	50.5	–58.0	$\text{H}_2\text{SO}_4 \times 6\text{H}_2\text{O}+\text{H}_2\text{SO}_4 \times 4\text{H}_2\text{O}+\text{CO}(\text{NH}_2)_2 \times \text{H}_2\text{SO}_4$
5.1	63.2	31.7	–42.0	$\text{H}_2\text{SO}_4 \times 4\text{H}_2\text{O}+\text{H}_2\text{SO}_4 \times 3\text{H}_2\text{O}+\text{CO}(\text{NH}_2)_2 \times \text{H}_2\text{SO}_4$
5.0	68.4	26.6	–46.0	$\text{H}_2\text{SO}_4 \times 3\text{H}_2\text{O}+\text{H}_2\text{SO}_4 \times 2\text{H}_2\text{O}+\text{CO}(\text{NH}_2)_2 \times \text{H}_2\text{SO}_4$
5.2	72.5	22.3	–45.1	$\text{H}_2\text{SO}_4 \times 2\text{H}_2\text{O}+\text{H}_2\text{SO}_4 \times \text{H}_2\text{O}+\text{CO}(\text{NH}_2)_2 \times \text{H}_2\text{SO}_4$
6.1	75.6	18.3	–35.1	$\text{H}_2\text{SO}_4 \times \text{H}_2\text{O}+\text{CO}(\text{NH}_2)_2 \times \text{H}_2\text{SO}_4$
6.9	77.1	16.0	–30.6	Too
8.1	79.2	12.7	–27.2	Too
9.6	81.3	9.1	–23.3	Too
30.1	9.8	60.1	–15.6	$2\text{CO}(\text{NH}_2)_2 \times \text{H}_2\text{SO}_4+\text{CO}(\text{NH}_2)_2 \times \text{H}_2\text{SO}_4$
30.8	23.6	45.6	–14.9	Too
32.2	35.8	32.0	–14.0	Too
36.4	51.1	12.1	–13.7	Too
36.9	8.4	54.7	–6.0	$\text{CO}(\text{NH}_2)_2+2\text{CO}(\text{NH}_2)_2 \times \text{H}_2\text{SO}_4$
63.2	29.9	6.9	28.0	Too
–	35.9	64.1	–62.0	Ice+ $\text{H}_2\text{SO}_4 \times 6\text{H}_2\text{O}$
–	42.6	57.4	–53.7	$\text{H}_2\text{SO}_4 \times 6\text{H}_2\text{O}+\text{H}_2\text{SO}_4 \times 4\text{H}_2\text{O}$
–	64.7	35.3	–36.6	$\text{H}_2\text{SO}_4 \times 4\text{H}_2\text{O}+\text{H}_2\text{SO}_4 \times 3\text{H}_2\text{O}$
–	69.5	30.5	–42.7	$\text{H}_2\text{SO}_4 \times 3\text{H}_2\text{O}+\text{H}_2\text{SO}_4 \times 2\text{H}_2\text{O}$
–	73.6	26.4	–39.9	$\text{H}_2\text{SO}_4 \times 2\text{H}_2\text{O}+\text{H}_2\text{SO}_4 \times \text{H}_2\text{O}$

On the polythermal solubility diagram, isotherms of solubility are plotted within the crystallization fields at every 10 °C interval. Projections of the polythermal curves onto the sides of the urea-water and sulfuric acid-water systems have also been constructed.

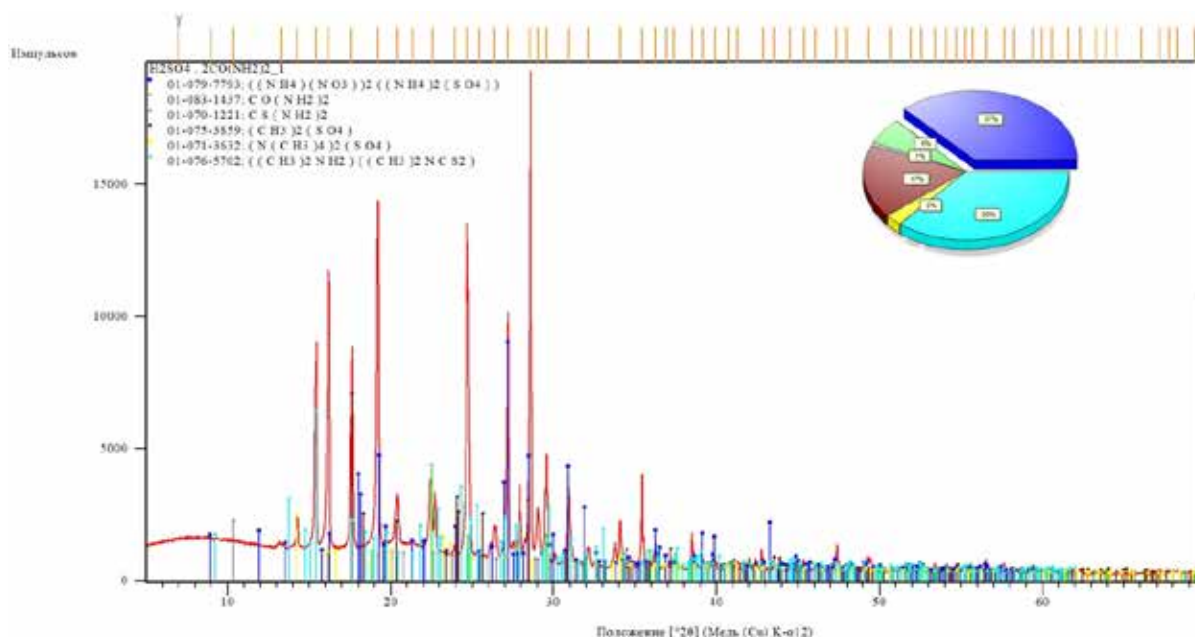
The formed compound with the composition  $\text{H}_2\text{SO}_4 \times 2\text{CO}(\text{NH}_2)_2$  was isolated in crystal-

line form and identified using chemical, X-ray, and physico-chemical analysis methods.

The chemical and physico-chemical analysis of the solid phase, isolated from the presumed area of existence of the formed compounds, confirmed the formation of this compound.



**Figure 8.** X-ray diffraction pattern of the formed compound:  $H_2SO_4 \times 2CO(NH_2)_2$

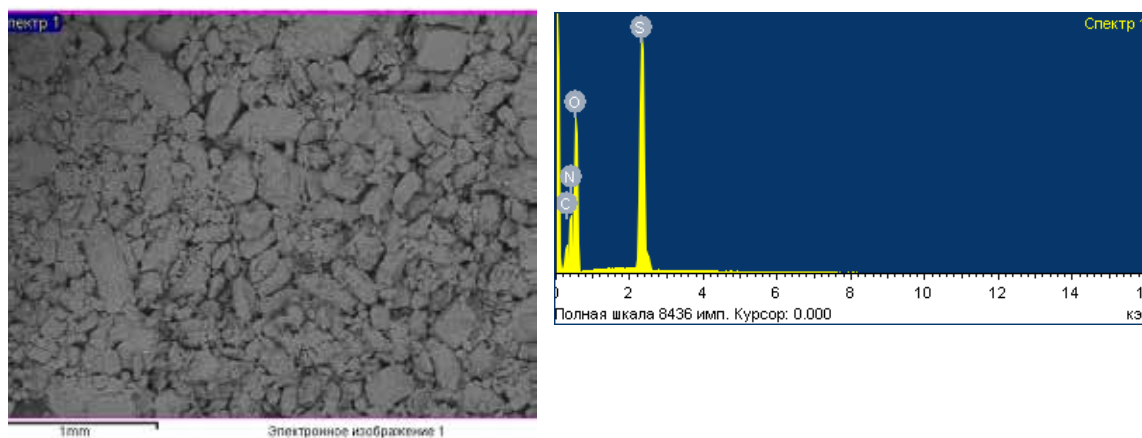


The X-ray phase analysis data of the urea-sulfuric acid compound indicate that all the reflections on the diffractograms are typically characterized by their specific reflection angles, sets of interplanar spacings, and intensities of diffraction lines (Fig. 8). This sug-

gests the uniqueness of the crystal lattices of the obtained compounds.

A microscopic analysis using scanning electron microscopy (SEM) was conducted to study the elemental and mineralogical composition of the compound.

**Figure 9.** Microstructure of urea sulfate



Based on the study (Fig. 9), the elemental composition of urea sulfate was determined as follows (in %): carbon (C) – 14.80, oxygen (O) – 45.69, nitrogen (N) – 26.32, sulfur (S) – 13.19.

### Conclusion

The solubility of components in systems composed of urea, nitric, phosphoric, and sulfuric acids was studied using the visual-polythermal method. Polythermal solubility diagrams were constructed based on

solubility data of binary systems and internal sections. In the studied systems, the formation of four new chemical compounds was established:  $CO(NH_2)_2 \times HNO_3$ ,  $CO(NH_2)_2 \times H_3PO_4$ ,  $CO(NH_2)_2 \times H_2SO_4$ , and  $2CO(NH_2)_2 \times H_2SO_4$  from the original components. The formed compounds were isolated from the presumed crystallization areas in crystalline form and identified using chemical and physico-chemical analysis methods.

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Contact: mashhura\_1909@mail.ru