

## STUDY OF SOLUBILITY PROPERTIES OF COMPONENTS IN ACETATE UREA - TRIETHANOLAMINE - WATER SYSTEM

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### ABSTRACT

*In this work, in order to synthesize new physiologically active substances, the solubility properties of the components in the acetate urea triethanolamine water ternary system were studied by observational polythermal method at a wide range of concentration and a temperature range from -43°C to 18°C. The solubility diagram of the studied system was constructed and the crystallization areas of all substances acetateurea, triethanolamine, ice and the new compound  $C_3H_8O_3N_2 \cdot N(C_2H_4OH)_3$  acetate ureatriethanolammonium were separated. The composition of the new substance  $C_3H_8O_3N_2 \cdot N(C_2H_4OH)_3$  was determined using the chemical analysis method. In order to determine optimal conditions for the synthesis of  $C_3H_8O_3N_2 \cdot N(C_2H_4OH)_3$  acetate ureatriethanolammonium, which has a new physiologically active property, the rheological properties of the solution were studied by adding triethanolamine to 60, 70 and 80 % acetateurea. A "composition-property" diagram was constructed based on the rheological properties of the studied system, the pH indicator, the density and viscosity of the solution, and the refractive index. The optimal conditions for the synthesis of the new substance  $C_3H_8O_3N_2 \cdot N(C_2H_4OH)_3$  acetate ureatriethanolammonium, obtained by the ratio of components, were determined by adding triethanolamine to a 60 % acetate urea solution.*

*Keywords:* visual polythermy, system, diagram, refractive index, viscosity, solution density, pH indicator.

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### INTRODUCTION

The role of physiologically active substances in ripening of agricultural crops and increasing their productivity is extremely large. Physiologically active substances increase the activity of enzymatic processes in plants and ensure their growth and development [1, 2]. The use of such substances as additives to defoliant is highly effective.

One of the main factors of increasing the quality of productivity and growing a high yield is the rational use of chemical and physiologically active substances [3, 4]. In this regard, special attention is paid to the production of highly effective, low-toxic and physiologically active defoliant.

In addition to the use of agrotechnical methods of

growing crops, the use of chemical preparations for drying or drying plant leaves accelerates the delivery of sufficient amounts of nutritional and physiologically active substances to their fruit. These processes allow for faster ripening of plant fruits, an increase in productivity and wide use of technology in harvesting [5].

Organic substances amino acids, carbonic acids, ethanolamines and their derivatives have high physiological activity. Ethanolamines are highly reactive. Ethanolamines have been found to activate oxidation-reduction processes in plants and increase the activity of protein metabolism and enzymatic systems [6 - 8].

In previous studies, the authors have studied those compounds of ethanolamines with phosphate urea [9], vinegar [10, 11], lemon [12], apple [13] nitrate [14]

and sulfate [15] have physiologically active properties. The properties of the components in the acetateurea - triethanolamine - water system have not been studied before. Part of the novelty of our scientific research work is studying the properties of substances in this complex system in a wide range of temperatures and concentrations.

## EXPERIMENTAL

The object of research is acetate urea and triethanolamine. In order to synthesize acetate urea, urea and 98 % acetic acid were added in a 1:1 molar ratio. For the synthesis, triethanolamine purified by the vacuum distillation method was used [16].

The solubility of the system was studied using the visual polythermal method [17 - 22]. A TN-6 glass mercury thermometer with a detection limit of  $-30^{\circ}\text{C}$  to  $60^{\circ}\text{C}$  and a TL-15 alcohol glass thermometer with a detection limit of  $-100$  to  $20^{\circ}\text{C}$  was used for solubility determination. Nitrogen in the imine group was determined by the spectrophotometric method (GOST 20851), carbon and hydrogen by elemental analysis (Zeiss EVO MA10) methods [23]. The viscosity of the solution was studied on a VPJ viscometer, the pH indicator on a FE20 METTLERTOLEDO pH meter, and the refractive index on an IRF 454 refractometer. The composition of the new substance formed was determined using the chemical analysis method.

## RESULTS AND DISCUSSION

Acetate urea-triethanolamine-water system was studied in a wide range of temperature and concentration for the physicochemical justification of the synthesis of ethanolamine derivatives that produce additional ethylene, added to defoliants containing chlorates.

A study of the binary system  $\text{CH}_3\text{COOH}\cdot\text{CO}(\text{NH}_2)_2\text{-H}_2\text{O}$  shows that its solubility diagram has two crystallization zones corresponding to the limits of ice and acetateurea as the solid phase. The critical point of the system corresponds to 58.0 % acetateurea and 42.0% water at  $-17^{\circ}\text{C}$  (Fig. 1).

Solubility of components  $\text{CH}_3\text{COOH}\cdot\text{CO}(\text{NH}_2)_2\text{-N}(\text{C}_2\text{H}_4\text{OH})_3\text{-H}_2\text{O}$  system was studied in a wide concentration and a temperature range from  $-43^{\circ}\text{C}$  to  $18^{\circ}\text{C}$ , and solubility diagram was drawn through seven internal lines.

Internal lines I-IV are directed from the triethanolamine and waterside to the acetateurea side, and lines V-VII are directed to the triethanolamine side from the acetateurea and waterside. The boundaries of separated substances in the constructed system are also reflected in the projection of the system (Fig. 2).

In the polythermal solubility diagram of the studied system, crystallization areas such as ice, acetateurea, new compound acetateurea triethanolammonium, triethanolamine were distinguished (Fig. 3).

It can be seen from the constructed diagram that

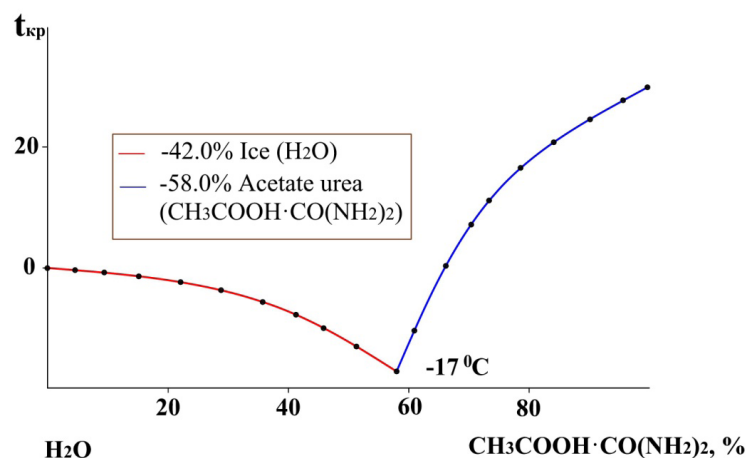


Fig. 1.  $\text{CH}_3\text{COOH}\cdot\text{CO}(\text{NH}_2)_2\text{-H}_2\text{O}$  binary diagram.

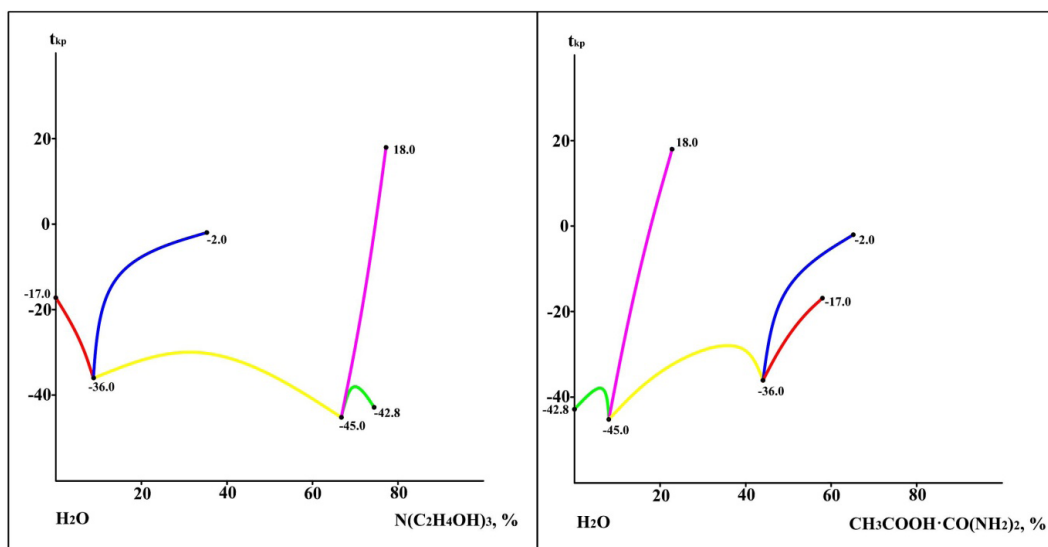


Fig. 2. Projection diagram of acetateurea - monoethanolamine - water system.

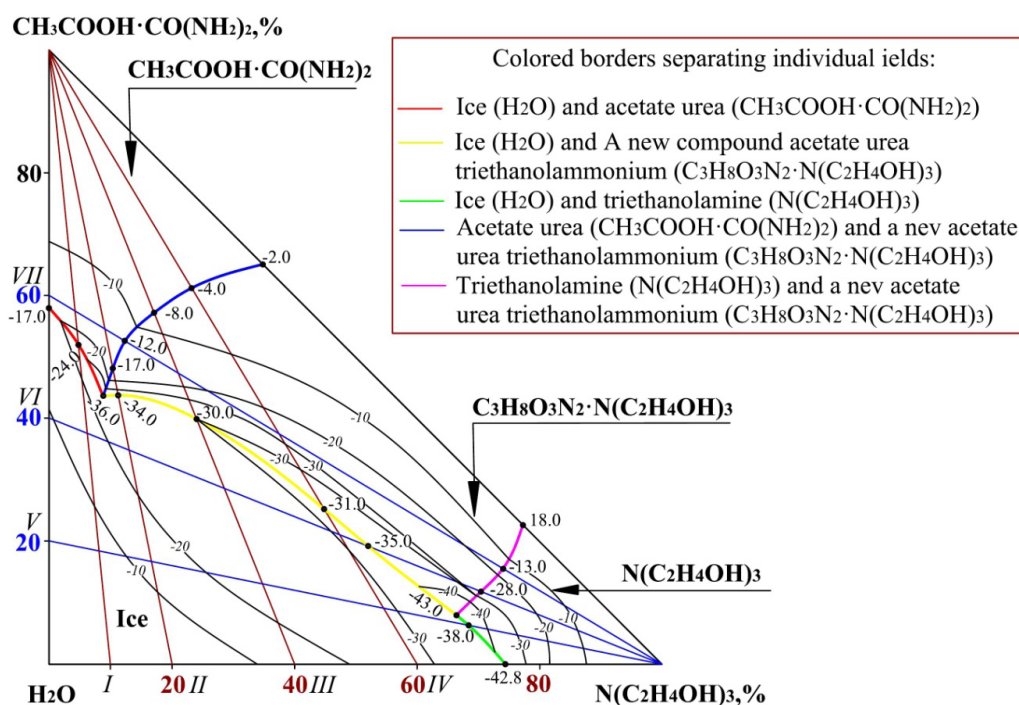


Fig. 3. Polythermal diagram of the acetateurea - triethanolamine - water solubility system.

ice co-crystallizes with acetateurea in the temperature range from  $-17.0^{\circ}\text{C}$  to  $-24.0^{\circ}\text{C}$ , with acetateurea triethanolammonium in the temperature range from  $34.0^{\circ}\text{C}$  to  $-35.0^{\circ}\text{C}$ , and with triethanolamine in the temperature range from  $-38.0^{\circ}\text{C}$  to  $-42.8^{\circ}\text{C}$ .

The areas defined below converge at two ternary points, respectively: 43.6 % acetateurea 8.8 %

triethanolamine and 47.6 % water at  $-36.0^{\circ}\text{C}$ . At a temperature of  $-43.0^{\circ}\text{C}$ , 8.0 % acetateurea corresponds to 66.6 % triethanolamine and 25.4 % water (Table 1).

In order to physico-chemically justify the formation of a new physiologically active substance in the studied system, the components were taken in different proportions [60 %  $\text{CH}_3\text{COOH} \cdot \text{CO}(\text{NH}_2)_2$  + 40 %

Table 1. Classification of binary and triple points in the acetate urea - triethanolamine - water system.

Liquid phase composition, %			Crystallization temperature, °C	Solid phase
CH <sub>3</sub> COOH·CO(NH <sub>2</sub> ) <sub>2</sub>	N(C <sub>2</sub> H <sub>4</sub> OH) <sub>3</sub>	H <sub>2</sub> O		
58	-	42	- 17.0	Ice + CH <sub>3</sub> COOH·CO(NH <sub>2</sub> ) <sub>2</sub>
52	2.8	39.2	- 24.0	
43.6	8.8	47.6	- 36.0	Ice+CH <sub>3</sub> COOH·CO(NH <sub>2</sub> ) <sub>2</sub> + C <sub>3</sub> H <sub>8</sub> O <sub>3</sub> N <sub>2</sub> ·N(C <sub>2</sub> H <sub>4</sub> OH) <sub>3</sub>
48.0	10.4	41.6	- 17.0	CH <sub>3</sub> COOH·CO(NH <sub>2</sub> ) <sub>2</sub> + C <sub>3</sub> H <sub>8</sub> O <sub>3</sub> N <sub>2</sub> ·N(C <sub>2</sub> H <sub>4</sub> OH) <sub>3</sub>
52.4	12.4	35.2	- 12.0	
57.0	17.0	26.0	- 8.0	
61.2	23.4	15.4	- 4.0	
65.2	34.8	-	- 2.0	
43.6	11.2	45.2	- 34.0	Ice + C <sub>3</sub> H <sub>8</sub> O <sub>3</sub> N <sub>2</sub> ·N(C <sub>2</sub> H <sub>4</sub> OH) <sub>3</sub>
39.4	23.8	36.8	- 30.0	
25.6	44.8	29.6	- 31.0	
17.4	55.8	26.8	- 35.0	
8.0	66.6	25.4	- 43.0	Ice + C <sub>3</sub> H <sub>8</sub> O <sub>3</sub> N <sub>2</sub> ·N(C <sub>2</sub> H <sub>4</sub> OH) <sub>3</sub> + N(C <sub>2</sub> H <sub>4</sub> OH) <sub>3</sub>
6.4	68.2	25.4	- 38.0	Ice+N(C <sub>2</sub> H <sub>4</sub> OH) <sub>3</sub>
-	74.4	25.6	- 42.8	
12.0	70.6	17.4	- 28.0	C <sub>3</sub> H <sub>8</sub> O <sub>3</sub> N <sub>2</sub> ·N(C <sub>2</sub> H <sub>4</sub> OH) <sub>3</sub> + N(C <sub>2</sub> H <sub>4</sub> OH) <sub>3</sub>
15.8	74.0	10.2	- 13.0	
22.8	77.2	-	18.0	

H<sub>2</sub>O] - N(C<sub>2</sub>H<sub>4</sub>OH)<sub>3</sub>, [70 % CH<sub>3</sub>COOH·CO(NH<sub>2</sub>)<sub>2</sub> + 30 % H<sub>2</sub>O] - N(C<sub>2</sub>H<sub>4</sub>OH)<sub>3</sub> and [80 % CH<sub>3</sub>COOH · CO(NH<sub>2</sub>)<sub>2</sub> + 20 % H<sub>2</sub>O] - N(C<sub>2</sub>H<sub>4</sub>OH)<sub>3</sub> solutions at 25°C temperature, viscosity, density, pH index and refractive index change was studied. Based on the obtained results, “composition-property” diagrams of the system were created (Fig. 4 - 7).

The fourth figure shows a diagram of the change of solution pH when triethanolamine is added to 60, 70 and 80 % urea acetate solutions. The diagram shows that with an increase in the concentration of triethanolamine, the value of pH also increases in solutions with three concentrations, and depending on the concentration of triethanolamine, three phases are separated: the first phase corresponds to ice, the second phase corresponds to the new compound acetateurea triethanolammonium, and the third phase corresponds to triethanolamine.

Fig. 5 shows a diagram of the change in density of

the solution when triethanolamine is added to 60, 70 and 80 % acetic acid solutions. We can see in the constructed diagram that when triethanolamine is added to a solution of acetateurea with three different concentrations, the density of the solution first increases, and then gradually decreases.

When triethanolamine is added to a 60, 70 and 80 % solution of acetateurea, the viscosity of the solution increases sharply, and three phases are mixed depending on the concentration of triethanolamine. The separated intermediate phase corresponds to the new compound acetateurea triethanolammonium (Fig. 6).

When triethanolamine is added to three different 60, 70 and 80 % solutions of acetateurea, the refractive index increases maximally in the triethanolamine concentration range of 48 % to 55 %, and gradually decreases in the range of 72 % to 75 % (Fig. 7). Then, when the concentration of triethanolamine in the solution

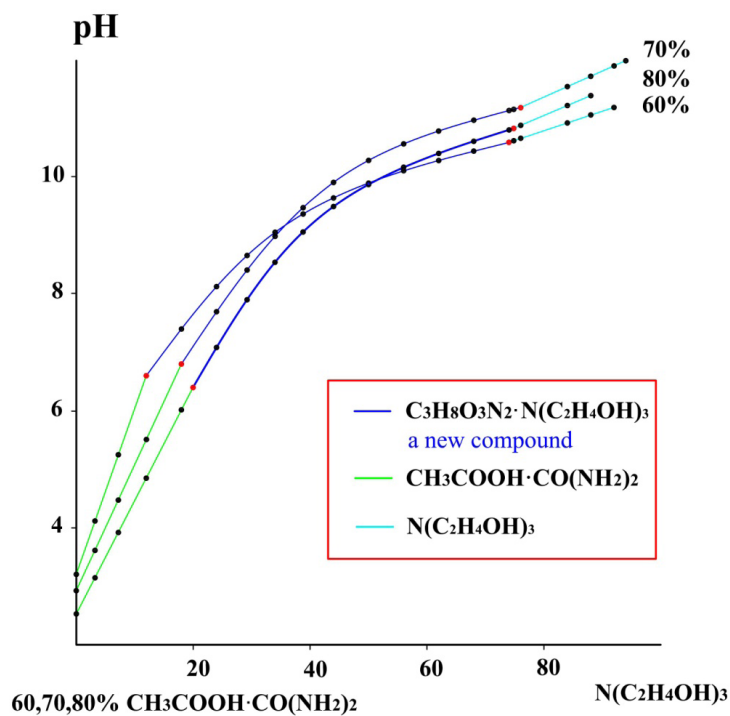


Fig. 4. Diagram of the change in solution pH when triethanolamine is added to 60, 70 and 80 % acetateurea.

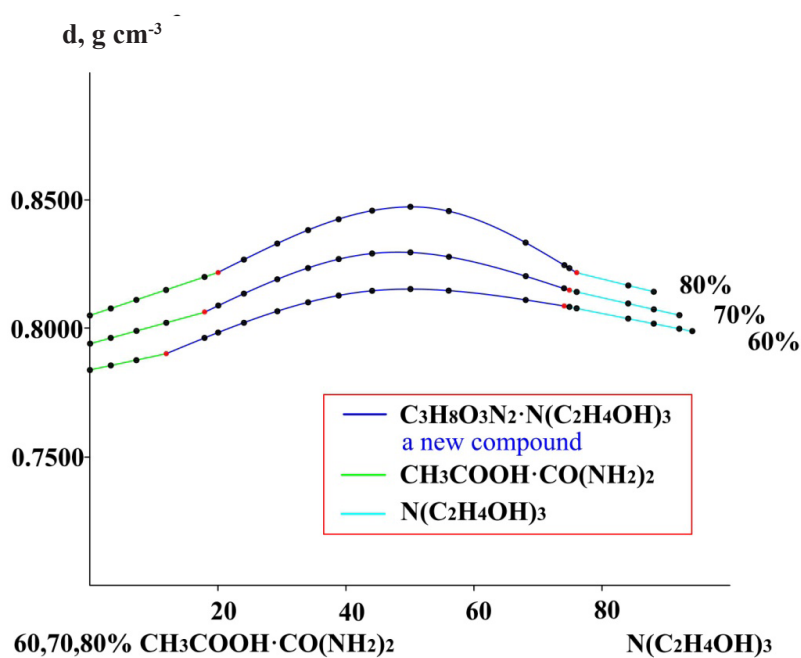


Fig. 5. Diagram of the change in solution density when triethanolamine is added to 60, 70 and 80 % acetateurea.

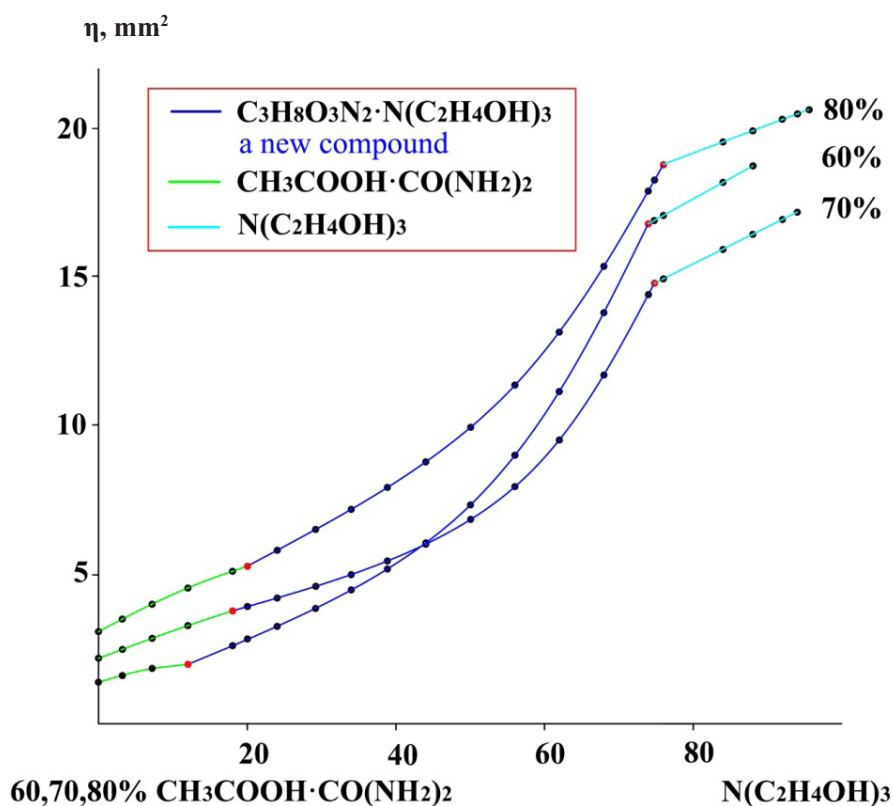


Fig. 6. Diagram of the change in solution viscosity when adding triethanolamine to 60, 70 and 80 % acetateurea.

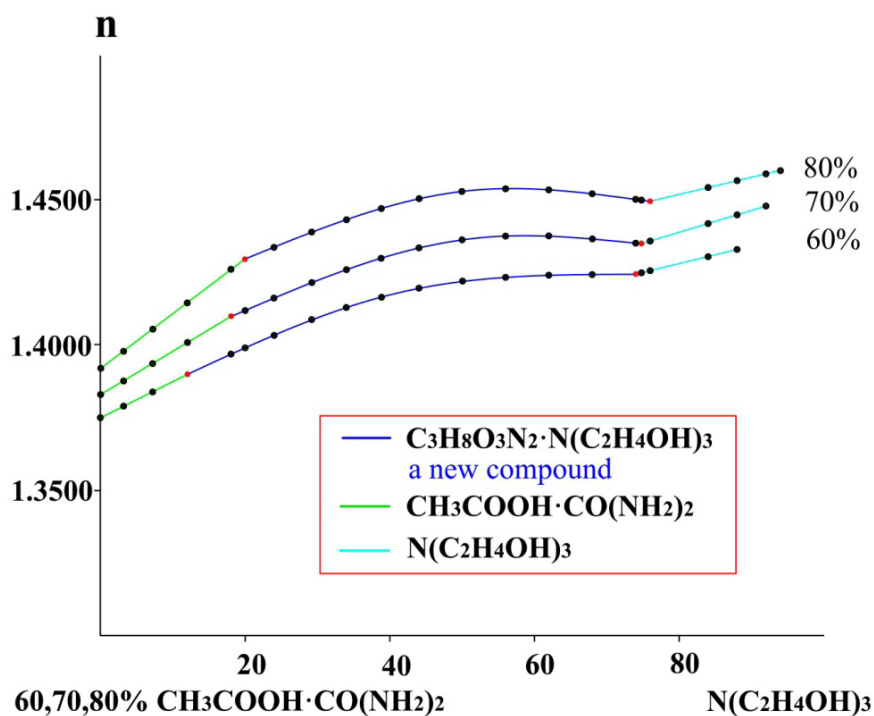


Fig. 7. A diagram of the change in the refractive index of a solution when triethanolamine is added to 60, 70 and 80 % acetateurea.



starts to increase from 75 %, the refractive index of the solution increases sharply again.

Chemical analysis of the synthesized compound showed the following results: mass. %: N = 15.97; C = 41.06; H = 8.74; O = 36.50. When calculated for  $C_3H_8O_3N_2 \cdot N(C_2H_4OH)_3$ , mass. %: N = 15.82; C = 40.99; H = 8.68; O = 36.41

## CONCLUSIONS

Solubility of components in  $CH_3COOH \cdot CO(NH_2)_2 - N(C_2H_4OH)_3 - H_2O$  system was studied in a wide range of temperature and concentration. A solubility diagram of the system was constructed and the diagram separated ice, acetateurea, triethanolamine and the new compound acetateurea triethanolammonium. The composition of the new compound  $C_3H_8O_3N_2 \cdot N(C_2H_4OH)_3$  formed in the system was determined by chemical analysis.

The physico-chemical parameters of the synthesis of acetateurea triethanolammonium, which is considered as a new physiologically active substance, were studied by adding triethanolamine to acetateurea solutions containing 60, 70 and 80 %. It was found that the optimal condition of synthesis consists of adding triethanolamine to 60 % acetateurea solutions  $[60\% CH_3COOH \cdot O(NH_2)_2 + 40\% H_2O] \cdot NH_2C_2H_4OH$  composition's physicochemical parameters: average pH= 6.9, density 0.7850 g cm<sup>-3</sup>, viscosity 3.8 mm<sup>2</sup>/s and refractive index 1.3840 (n) indicated that.

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