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The solubility of the $\text{NaClO}_3 \cdot \text{CO}(\text{NH}_2)_2 \cdot \text{N}(\text{C}_2\text{H}_4\text{OH})_3 \cdot \text{HNO}_3 \cdot \text{H}_2\text{O}$ system

Sidikov A.A., Toghsharov A.S., Shukurov J.S., Tukhtaev S.

PhD doctoral student of The Institute of General and Inorganic Chemistry of the Academy of Sciences of the Republic of Uzbekistan, Mirzo Ulugbek 77A, 100071 Tashkent, Uzbekistan,

Chief researcher of The Institute of General and Inorganic Chemistry of the Academy of Sciences of the Republic of Uzbekistan,

Chief researcher of The Institute of General and Inorganic Chemistry of the Academy of Sciences of the Republic of Uzbekistan

Leading researcher, academician of The Institute of General and Inorganic Chemistry of the Academy of Sciences of the Republic of Uzbekistan

ABSTRACT: The solubility of the $\text{NaClO}_3 \cdot \text{CO}(\text{NH}_2)_2 \cdot \text{N}(\text{C}_2\text{H}_4\text{OH})_3 \cdot \text{HNO}_3 \cdot \text{H}_2\text{O}$ system from the freezing point (-44.2) to 60.0°C was studied. A polythermal solubility diagram was constructed, on which the crystallization fields of ice, $\text{NaClO}_3 \cdot \text{CO}(\text{NH}_2)_2$, $\text{CO}(\text{NH}_2)_2$, and $\text{N}(\text{C}_2\text{H}_4\text{OH})_3 \cdot \text{HNO}_3$, are demarcated. The system belongs to a simple eutonic type. The physicochemical properties of the system [60% $\text{NaClO}_3 \cdot \text{CO}(\text{NH}_2)_2 + 40\% \text{H}_2\text{O}$] - $\text{N}(\text{C}_2\text{H}_4\text{OH})_3 \cdot \text{HNO}_3$ were also studied and a “composition-properties” diagram was constructed based on the obtained data.

KEY WORDS: solubility, system, diagram, concentration, crystallization, temperature, viscosity, density, pH of the medium, refractive index

I. INTRODUCTION

The main factor in growing high and high-quality crops is the rational use of chemicals. Especially defoliation is one of the important conditions for successful and high-quality harvesting of raw cotton in the pre-breeding season [1]. In this regard, special attention is paid to the production of highly effective, low-toxic and physiologically active defoliant. Existing chlorate-containing defoliant based on chlorates do not meet modern requirements for defoliant. It is known that the defoliating effect of chlorates is always to one degree or another accompanied by a desiccation effect [2, 3]. In the synthesis of new effective defoliant, the use of ethanol ammonium nitrate, which is a plant growth stimulator, is of considerable interest. Therefore, as a result of adding this substance to the composition of the defoliant, the drug acquires physiological activity [4].

The aim of this study was to obtain a new effective, physiological active cotton defoliant based on mono carbamidochlorate, produced in the domestic industry and ethanol ammonium nitrate, which is an effective additive to chlorate-containing defoliant.

In the present work, we present new data obtained on heterogeneous phase equilibrium in a system with the participation of water, sodium chlorate, urea, and ethanol ammonium nitrate; its polythermal solubility diagrams are constructed. To justify the process of obtaining a complex acting defoliant containing physiologically active substances, the “composition-properties” of the system [60% $\text{NaClO}_3 \cdot \text{CO}(\text{NH}_2)_2 + 40\% \text{H}_2\text{O}$] - $\text{N}(\text{C}_2\text{H}_4\text{OH})_3 \cdot \text{HNO}_3$ were studied.

II. LITERATURE SURVEY

Kucharov X. et al [5] Clarified the interaction of components in the system of sodium chlorate, triethanolamine and water. The system has been studied over a wide temperature range. It is established that the system belongs to a simple eutonic type.

Authors Hamdamova Sh.Sh. and Mirzaev N.A. [6] investigated the solubility of the magnesium chlorate-tetranolamine-water system using the visual-polythermal method at temperatures from -56.0 to 31.2 °C. The polythermic diagram of solubility was built, on which bordered the fields of crystallization of an ice, sixteen, twelve and six-aqua

magnesium chlorate, treethanolamin and new substances with the structure $\text{MgOHClO}_3 \cdot \text{N}(\text{C}_2\text{H}_4\text{OH})_3 \cdot 2\text{H}_2\text{O}$ and $(\text{C}_2\text{H}_4\text{OH})_3 \cdot \text{HClO}_3$ are established. The compounds were identified by chemical and physical chemical methods of analysis.

Khudoyberdiyev F.I. [7] studied the solubility in the $\text{NaClO}_3 \cdot 3\text{CO}(\text{NH}_2)_2 \cdot \text{N}(\text{C}_2\text{H}_4\text{OH})_3 \cdot \text{C}_4\text{H}_4\text{N}_2\text{O}_2 \cdot \text{H}_2\text{O}$ system by using a visual polythermal method. The solubility diagram of the system is constructed in the temperature range (-23.9) to 60°C in order to justify the conditions for the synthesis of a new compound based on the starting components.

III.METHODOLOGY

The solubility polytherm of the $\text{NaClO}_3 \cdot \text{CO}(\text{NH}_2)_2 \cdot \text{N}(\text{C}_2\text{H}_4\text{OH})_3 \cdot \text{HNO}_3 \cdot \text{H}_2\text{O}$ system was studied by the visual polythermal method [8]. To identify the components of the system, chemical and physicochemical methods of analysis were used. Quantitative chemical analysis of sodium solid phases was carried out by flame photometry [9], the chlorate-ion content was determined by the volume permanganometric method [10] and carbon, nitrogen, and hydrogen by elemental analysis [11]. The relative density was determined by the pycnometric method [12] using a capillary pycnometer of 5 and 10 ml. The pH of the solutions was measured according to the procedure [13] with a FE20 METTLER TOLEDO pH meter. The kinematic viscosity of the solutions was determined on a VPZ capillary viscometer [14] with a capillary diameter of 1.16-1.84 mm. The refractive index was determined on an IRF 454 refractometer of the BM model in the range from 1.2 to 1.7 with one refractometric block [15].

IV.MATERIALS

In our studies, we used $\text{NaClO}_3 \cdot \text{CO}(\text{NH}_2)_2$, synthesized by alloying carbamide with sodium chlorate at a molar ratio of 1: 1. After the formation of a homogeneous melt of the starting components, crystals of the compound $\text{NaClO}_3 \cdot \text{CO}(\text{NH}_2)_2$ were isolated by cooling.

The binary $\text{NaClO}_3 \cdot \text{CO}(\text{NH}_2)_2 \cdot \text{H}_2\text{O}$ system was studied by us in the temperature range from -33.0 to 100.0 °C. On the solubility curve of the system, the crystallization branches of ice, urea, sodium monocarbamidochlorate were established. The results obtained are consistent with the literature data presented in [16].

Triethanolammonium nitrate was synthesized based on nitric acid and triethanolamine, taken at a molar ratio of 1: 1. It is a brown colored fluid solution at 25 °C with a pH of 6.08.

V. RESULTS AND DISCUSSION

The solubility in the $\text{N}(\text{C}_2\text{H}_4\text{OH})_3 \cdot \text{HNO}_3 \cdot \text{H}_2\text{O}$ system was studied by us in the temperature range from -19.0 to 1.0 °C (Fig. 1). The solubility diagram of the $\text{N}(\text{C}_2\text{H}_4\text{OH})_3 \cdot \text{HNO}_3 \cdot \text{H}_2\text{O}$ system is characterized by the presence of two branches of ice crystallization and $\text{N}(\text{C}_2\text{H}_4\text{OH})_3 \cdot \text{HNO}_3$. The eutectic point of the system corresponds to 81.75% $\text{N}(\text{C}_2\text{H}_4\text{OH})_3 \cdot \text{HNO}_3$ and 18.25% H_2O , at a temperature of -19.0 °C.

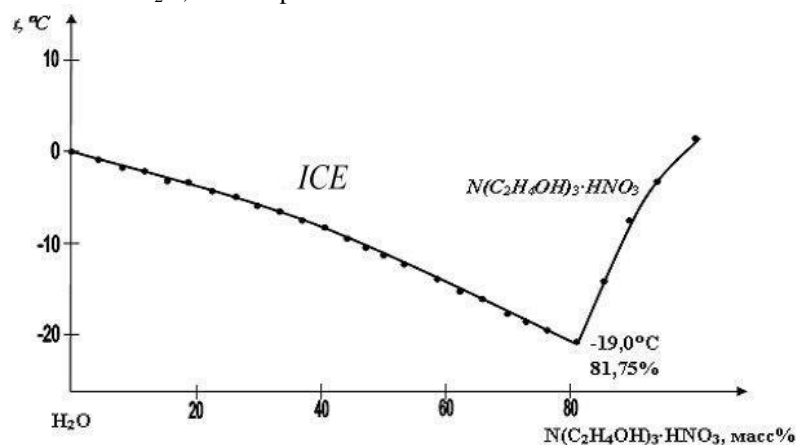


Fig. 1. The solubility diagram of the $\text{N}(\text{C}_2\text{H}_4\text{OH})_3 \cdot \text{HNO}_3 \cdot \text{H}_2\text{O}$ system

The $\text{NaClO}_3 \cdot \text{CO}(\text{NH}_2)_2 - \text{N}(\text{C}_2\text{H}_4\text{OH})_3 \cdot \text{HNO}_3 - \text{H}_2\text{O}$ system was studied using seven internal sections from -44.2 to 60 °C. On the polythermal solubility diagram, the fields of crystallization are distinguished: ice, $\text{NaClO}_3 \cdot \text{CO}(\text{NH}_2)_2$, $\text{CO}(\text{NH}_2)_2$ and $\text{N}(\text{C}_2\text{H}_4\text{OH})_3 \cdot \text{HNO}_3$ (Fig. 2.).

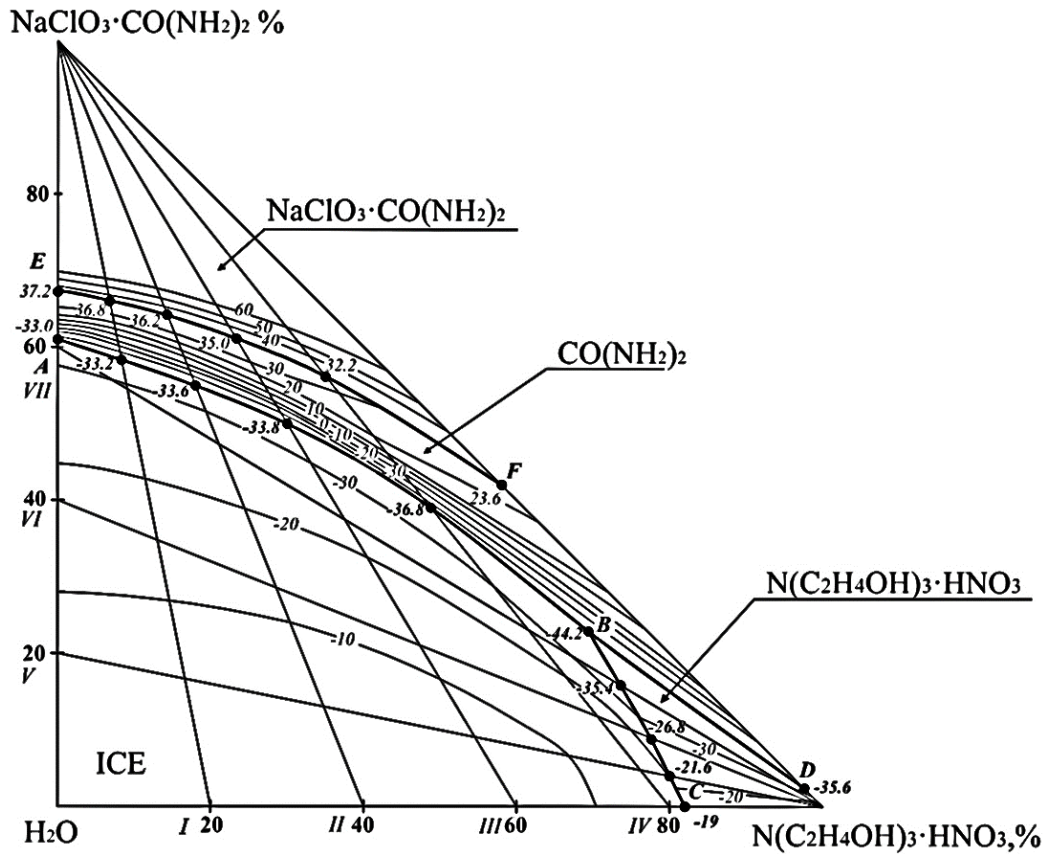


Fig. 2. Polythermal solubility diagram of the $\text{NaClO}_3 \cdot \text{CO}(\text{NH}_2)_2 - \text{N}(\text{C}_2\text{H}_4\text{OH})_3 \cdot \text{HNO}_3 - \text{H}_2\text{O}$ system

The ice and urea crystallization fields in the diagram are delimited by a curved line connecting points A and B. At the eutectic point of the system, the concentration of components is 22.2% $\text{NaClO}_3 \cdot \text{CO}(\text{NH}_2)_2$, 69.6% $\text{N}(\text{C}_2\text{H}_4\text{OH})_3 \cdot \text{HNO}_3$ and 8.2% H_2O . The crystallization temperature of point C (-19.0 °C), the concentration of triethanolammonium nitrate is 81.75%, and water is 18.25%. The fields of urea and triethanolammonium nitrate are delimited by a curved line between points B and D. The crystallization temperature of point D is (-35.6 °C), the concentration of $\text{NaClO}_3 \cdot \text{CO}(\text{NH}_2)_2$ and $\text{N}(\text{C}_2\text{H}_4\text{OH})_3 \cdot \text{HNO}_3$ is 2.4% and 97.6%, respectively. The crystallization fields of sodium and urea monocarbamidochlorate are delimited by a curve connecting the points of the diagrams E and F. The double and triple points of the $\text{NaClO}_3 \cdot \text{CO}(\text{NH}_2)_2 - \text{N}(\text{C}_2\text{H}_4\text{OH})_3 \cdot \text{HNO}_3 - \text{H}_2\text{O}$ system are shown in Table 1.

Table 1. Double and triple points of the $\text{NaClO}_3 \cdot \text{CO}(\text{NH}_2)_2 - \text{N}(\text{C}_2\text{H}_4\text{OH})_3 \cdot \text{HNO}_3 - \text{H}_2\text{O}$ system

Composition of the liquid phase (%)			T _c (°C)	Solid phase
$\text{NaClO}_3 \cdot \text{CO}(\text{NH}_2)_2$	$\text{HNO}_3 \cdot \text{N}(\text{C}_2\text{H}_4\text{OH})_3$	H_2O		
61.2	-	38.8	-33.0	Ice + $\text{CO}(\text{NH}_2)_2$
58.4	8.2	33.4	-33.2	The same

55.2	17.8	27	-33.6	-/-
50.0	29.8	20.2	-33.8	-/-
38.2	49.0	12.8	-36.8	-/-
22.2	69.6	8.2	-44.2	Ice + CO(NH ₂) ₂ + HNO ₃ · N(C ₂ H ₄ OH) ₃
16.0	73.4	10.6	-35.4	Ice + HNO ₃ · N(C ₂ H ₄ OH) ₃
8.8	77.8	13.4	-26.8	The same
4.0	80.0	16	-21.6	-/-
-	81.6	18.4	-19.0	-/-
2.4	97.6	-	-35.6	CO(NH ₂) ₂ + HNO ₃ · N(C ₂ H ₄ OH) ₃
67.4	-	32	37.2	NaClO ₃ · CO(NH ₂) ₂ + CO(NH ₂) ₂
66.2	6.8	27	36.8	The same
64.2	14.2	21.6	36.2	-/-
56.4	34.6	9	32.2	-/-
42.6	57.4	-	23.6	-/-

Projections of the system were constructed on the sides of sodium monocarbamidochlorate - water A and on the sides of triethanolammonium nitrate - water B (Fig. 3.).

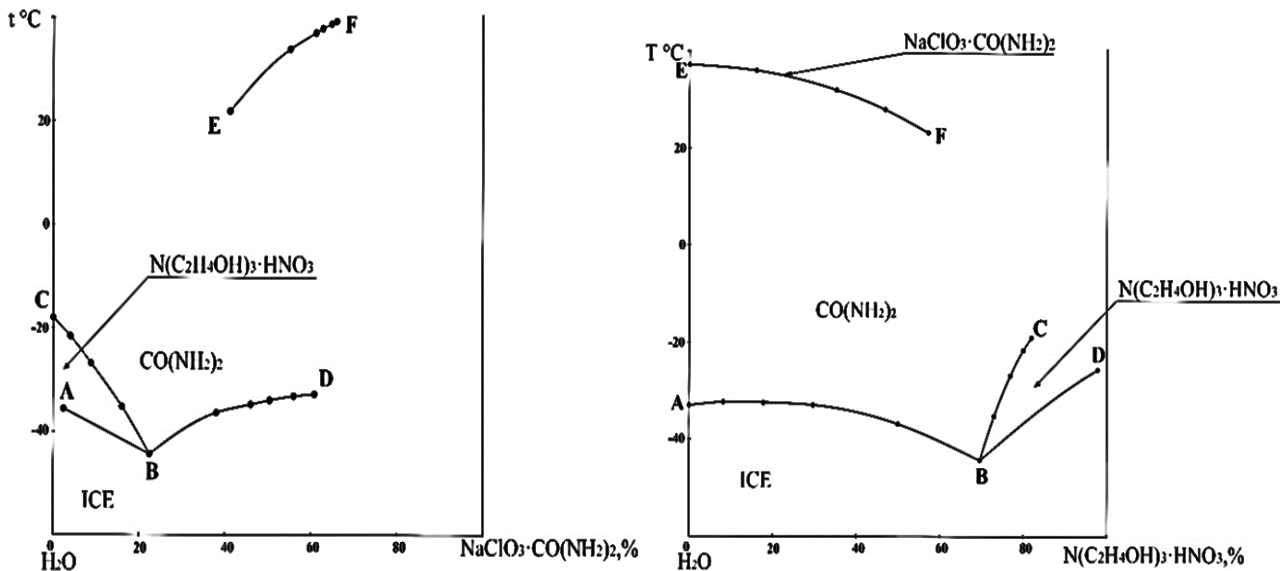


Fig. 3. Projections of the NaClO₃ · CO(NH₂)₂ · N(C₂H₄OH)₃ · HNO₃ · H₂O system:
A-NaClO₃ · CO(NH₂)₂ · H₂O; B-N(C₂H₄OH)₃ · HNO₃ · H₂O

In order to justify the process of obtaining a defoliant based on sodium monocarbamidochlorate and triethanolammonium nitrate, we studied the solubility and rheological properties of the components in the system [60%NaClO₃ · CO(NH₂)₂ + 40% H₂O] · N(C₂H₄OH)₃ · HNO₃.

The system [60%NaClO₃ · CO(NH₂)₂ + 40% H₂O] · N(C₂H₄OH)₃ · HNO₃ was studied by the solubility method, measuring

density, viscosity, pH and refractive index, the results are shown in Table 2. The composition – properties diagram is constructed »Of this system (Fig. 4).

**Physico-chemical and rheological properties of the system
[60%NaClO₃·CO(NH₂)₂+40%H₂O]-N(C₂H₄OH)₃·HNO₃**

Components content, %		Cryst. temperature, t, °C	Density, d, g/cm ³	Viscosity, η, mm ² /s	pH	Refractive index, n	Solid phase
60%NaClO ₃ ·CO(NH ₂) ₂ +40%H ₂ O	NH(C ₂ H ₄ OH) ₃ ·HNO ₃						
100	-	13.0	1.425	1.82	6.01	1.4153	Ice + CO(NH ₂) ₂
93.3	6.7	9.2	1.422	1.94	6.02	1.4172	The same
85.2	14.8	4.5	1.412	2.06	6.02	1.4206	-/-
79.0	21.0	0	1.402	2.25	6.03	1.4232	-/-
73.5	26.5	-3.0	1.392	2.44	6.03	1.4252	-/-
68.1	31.9	-6.5	1.383	2.56	6.04	1.4274	-/-
61.6	38.4	-11.0	1.372	2.84	6.04	1.4308	-/-
56.6	43.4	-14.0	1.363	3.11	6.05	1.4357	-/-
50.7	49.3	-18.5	1.354	3.48	6.05	1.4353	-/-
45.0	55.0	-22.0	1.345	3.77	6.06	1.4370	-/-
40.0	60.0	-25.5	1.348	4.03	6.07	1.4397	-/-
26.6	73.4	-35.4	1.332	4.38	6.07	1.4415	-/-
18.3	81.7	-21.2	1.307	5.55	6.07	1.4472	CO(NH ₂) ₂ + N(C ₂ H ₄ OH) ₃ ·HNO ₃
8.2	91.8	-7.5	1.297	6.18	6.08	1.4498	Ice + HNO ₃ ·N(C ₂ H ₄ OH) ₃
-	100	1.0	1.292	6.59	6.08	1.4525	The same

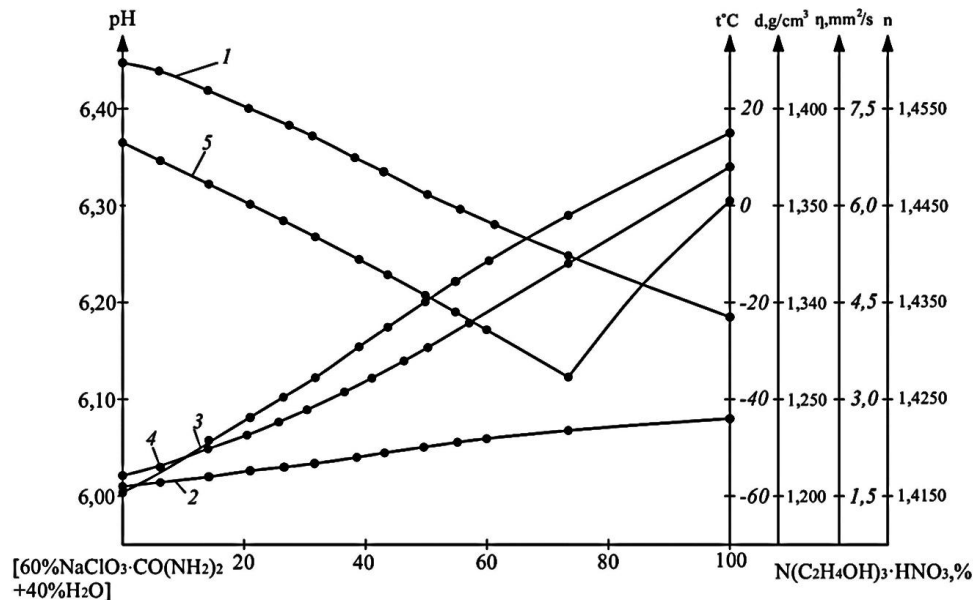


Fig. 4. The composition-property diagram of the system $[60\%NaClO_3 \cdot CO(NH_2)_2 + 40\%H_2O] - N(C_2H_4OH)_3 \cdot HNO_3$ at $25^\circ C$ depending on the density change (1); pH of the medium (2); viscosity (3); refractive index (4); crystallization temperature (5).

Studies have shown that when (74.67%) triethanolammonium nitrate is added to the studied saturated sodium monocarbamidochlorate solution, a decrease in the crystallization temperature of the solution is observed from 13.0 to $(-35.4)^\circ C$, density from 1.425 to 1.292 g/cm^3 and viscosity increase from 1.82 up to 6.59 mm^2/s , medium pH from 6.01 to 6.08, refractive index from 1.4153 to 1.4525. At the eutectic point, two solid phases crystallize - urea and triethanolammonium nitrate. As the solubility study showed, a further increase in the concentration of triethanolammonium nitrate in a saturated solution of systems from 73.6 to 100% leads to an increase in the crystallization temperature from (-35.4) to $-1.0^\circ C$.

VI. CONCLUSION

The $NaClO_3 \cdot CO(NH_2)_2 - N(C_2H_4OH)_3 \cdot HNO_3 - H_2O$ system under study belongs to the simple eutonic type, and no new compounds were found.

Since this system has not been previously studied, the data we have obtained are a scientific reference for masters and doctoral students working in this field.

Based on the results of the study, a physicochemical substantiations of the processes for obtaining a new defoliant was established.



With the aim to develop technologies for the production of new effective defoliant and determine their composition, studied the solubility of the $[60\%NaClO_3 \cdot CO(NH_2)_2 + 40\% H_2O] - N(C_2H_4OH)_3 \cdot HNO_3$ system, «composition-property». On the basis of the data obtained, a polythermal solubility diagram and a «composition-property» diagram of the system was constructed. The data obtained are used for fundamental investigation into the analysis of salt systems as reference data.

REFERENCES

- [1] Teshaev F., Khaitov B. Effect of defoliant and fertilizers on yield and quality of upland cotton (*Gossypium hirsutum* L.) // Journal of Cotton Research and Development (CRDA). - India, 2015. - №1. pp. 57-60.
- [2] Umarov A.A., Kutyanin L.I. New defoliant: search, properties, applications. M.: Chemistry. 2000. - 87s.
- [3] Shukurov J.S., Askarova M.K., Tukhtaev S. Solubility in systems comprising sodium dicarbamidochlorate, monoethanolamine acetate, ethanol and acetamidiprid // Russian Journal of Inorganic Chemistry. - Moscow, 2018 Vol. 63.-№ 2. pp. 260-264.

- [4] Turaev K.A., Togasharov A.S., ShukurovZh.S., Tukhtaev S. Study of the solubility and rheological properties of solutions of the system sodium tricarbamidochlorate - monoethanolammonium nitrate - water // Universum: Engineering: electron. scientific journal 2019.No.10 (67) S. 66-72. <http://7universum.com/en/tech/archive/category/1067>.
- [5] KhamdamovaSh.Sh., Kucharov X., Tukhtaev S. Solubility polytherm of sodium chlorate-triethanolamine-water system // Uzbek Chemical Chem. - 2003.-№5. S.29-32.
- [6] KhamdamovaSh.Sh., Mirzaev N.A. The interaction of components in the Mg (ClO₃)₂ - N (C₂H₄OH)₃ - H₂O system // Universum: Chemistry and Biology: Electron. scientific journal 2020. No. 1 (67). <http://7universum.com/en/nature/archive/item/8606>
- [7] Khudoyberdiyev F.I. Solubility study in the NaClO₃ · 3CO (NH₂)₂ - N (C₂H₄OH)₃ · C₄H₄O₂N₂ - H₂O system // Universum: Chemistry and Biology: Electron. scientific journal 2018.No 9 (51). <http://7universum.com/en/nature/archive/item/6288>
- [8] Trunin A.S. Petrova D.G. Visual polythermal method / Kuibyshev Polytechnic. Inst. - Kuibyshev: 1977, 94 p. Dep. at VINITI No. 584-78.
- [9] Poluektov N.S. Methods of analysis by flame photometry. - M.: Chemistry, 1967.- 307 p.
- [10] GOST 12257 - 77 Sodium chlorate. Technical conditions - M.: Publishing house of standards. -1987. No. 548 - 78.
- [11] Klimova V.A. The main micromethods for the analysis of organic compounds. - M.: Chemistry, 1975.- 224 p.
- [12] Zdanovsky A.B. Gallurgy. - L.: Chemistry, 1972.- 572 p.
- [13] Gorbachev S.V. Workshop on physical chemistry. - M.: Higher School, 1974. - 310 p.
- [14] Frolov Yu.G. The course of colloid chemistry // Surface phenomenon and dispersed systems. - M. 1982.P. 117 - 124.
- [15] TO 34. 15.051 Technical description and instruction manual.

AUTHOR'S BIOGRAPHY

№	Full name place of work, position, academic degree and rank	Photo
1	<p>Sidikov Abdulaziz Abdumanopogli,</p> <p>PhD doctoral student of The Institute of General and Inorganic Chemistry of the Academy of Sciences of the Republic of Uzbekistan, Tashkent, Uzbekistan.</p>	
2	<p>Togasharov AhadSalimovich,</p> <p>Chief researcher of The Institute of General and Inorganic Chemistry of the Academy of Sciences of the Republic of Uzbekistan, DSc., Tashkent, Uzbekistan.</p>	
3	<p>Shukurov Jamshid Sulstonovich,</p> <p>Chief researcher of The Institute of General and Inorganic Chemistry of the Academy of Sciences of the Republic of Uzbekistan, DSc., Tashkent, Uzbekistan.</p>	
4	<p>Tukhtaev Saidakhral,</p> <p>Leading researcher, Professor, Academician of The Institute of General and Inorganic Chemistry of the Academy of Sciences of the Republic of Uzbekistan, Tashkent, Uzbekistan.</p>	