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The solubility in monoethanolamine-3- oxyhyridazone-6-water and sodium trikarbamidochlorate-3-oxyhyridazone-6- water

Fazlidin Khudoyberdiev Isroilovich, Farxod Umirov Ergashovich, Saidaxror Tukhtaev

Senior Lecturer, department of Chemical Technology of the Navoi State Mining Institute, Navoi city, Uzbekistan
Head of department of Metallurgy of the Navoi State Mining Institute, Navoi city, Uzbekistan
Academician, Institute of General and Inorganic Chemistry of the Academy of Sciences
Tashkent, Republic of the Uzbekistan

ABSTRACT: The solubility in the ternary monoethanolamine-3-oxyhyridazone-6-water systems and sodium tricarbamidochlorate-monoethanolamine-3-oxyhyridazone-6-water was studied visually-by the polythermic method. Their polythermic diagrams of solubility have been constructed from freezing to 800 °C. The formation of a compound of the composition $N(C_2H_4OH)_3 \cdot C_4H_4N_2O_2$, identified by the methods of chemical and physicochemical analysis, is established

I. INTRODUCTION

Ethanolamines, 3-hydroxypyridazone-6 and their derivatives are effective regulators of plant growth, inhibitors of secondary leaf regrowth after defoliation and additives to defoliant that enhance their activity [1-5]. With respect to the bases, 3-hydroxypyridazone-6 ($C_4H_4N_2O_2$) behaves as a monobasic acid [5]. Therefore, it is of interest to study heterogeneous phase equilibrium in the ternary system $N(C_2H_4OH)_3 \cdot C_4H_4N_2O_2 \cdot H_2O$ in order to justify the conditions for the synthesis of a new compound on the basis of initial components with high physiological activity.

II. ESSENTIALS OF PERFORMANCE

The triple monoethanolamine-3-hydroxypyridazone-6-water system was examined by six internal cuts; of which I-II were conducted by 3-hydroxypyridazone-6-water to the top of monoethanolamine, and III-IV from the side of monoethanolamine-water to the top of 3-hydroxypyridazone-6.

Based on the polyterm of the side systems and internal sections, a polythermic solubility diagram of the monoethanolamine-3-hydroxypyridazone-6-water system was constructed at temperatures from -53.2 to 60 °C (Fig. 1).

The phase diagram of the state of the system demarcates the crystallization fields of ice, monoethanolamine, 3-hydroxypyridazone-6 and a new compound of the composition $NH_2C_2H_4OH \cdot C_4H_4N_2O_2$. Four triple non-invariant points of the system are established.

The diagram of polythermic solubility shows isotherms every 10 °C in the temperature range - 40-60 °C. According to the data obtained in the presence of monoethanolamine, the solubility of 3-hydroxypyridazone-6 is increased, which also indicates complexation in the $NH_2C_2H_4OH \cdot C_4H_4N_2O_2 \cdot H_2O$ system. Indeed, in the system studied, the formation of a new compound of composition takes place.

III. DIAGRAMMATIC REPRESENTATION

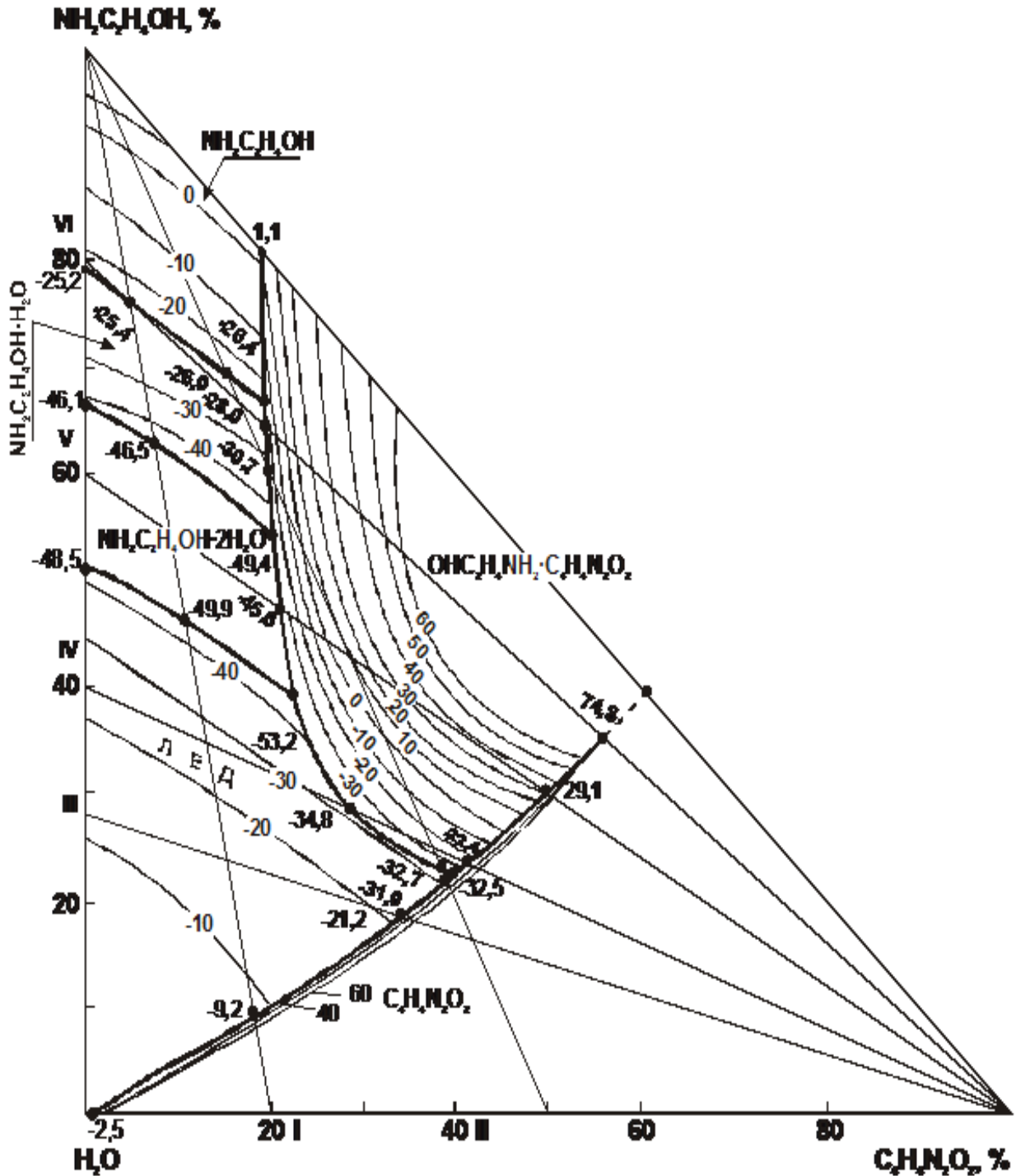


Fig. 1. Diagram of solubility of the monoethanolamine-3-hydroxypyridazone-6-water $\text{NH}_2\text{C}_2\text{H}_4\text{OH} \cdot \text{C}_4\text{H}_4\text{N}_2\text{O}_2$ in the temperature range 53.2-60 °C with a content of 22.3-81.2% 3-hydroxypyridazone-6.

The compound is isolated in a crystalline form and identified by chemical and physicochemical analyzes. Chemical analysis of the phase separated from the expected crystallization region of $\text{NH}_2\text{C}_2\text{H}_4\text{OH} \cdot \text{C}_4\text{H}_4\text{N}_2\text{O}_2$ gave the following results: found in %: C - 41,57; H₂ - 6,38; N₂ - 24,31. For $\text{NH}_2\text{C}_2\text{H}_4\text{OH} \cdot \text{C}_4\text{H}_4\text{N}_2\text{O}_2$ iscalculated, in %: C - 41,61; H₂ - 6,35; N₂ - 24,27.

X-ray analysis confirms the formation of this compound, characterized by its own diffraction reflexes with values of interplanar distances: 4,657; 4,426; 4,315; 3,990; 3,695; 3,448; 3,326; 3,227; 3,129; 2,290; 2,876; 2,765; 2,720; 2,610; 2,540; 2,420; 2,270; 2,205; 2,097; 1,970; 1,810; 1,677 Å°, not typical for the original components (Figure 2).

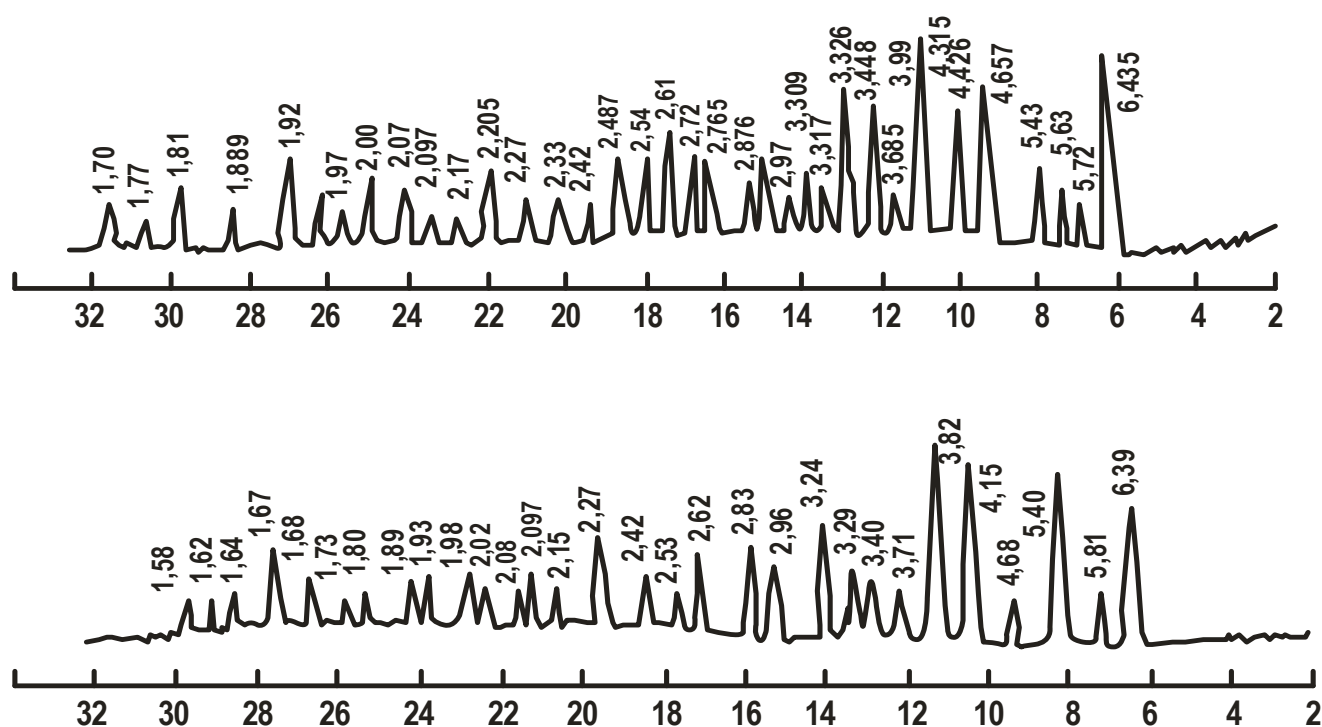


Fig. 2. X-ray patterns of 3-hydroxypyridazine-6 (1) and 3-hydroxypyridazonate-6 monoethanolammonium (2).

Sodium tricarbamidochlorate is the active ingredient of a number of domestic defoliant - Sihat, Sadaf-70, Sadaf-96, ethanolamines, 3-hydroxypyridazine-6 and their derivatives - effective additives to chlorate-containing defoliant, enhancing their effectiveness and preventing the secondary growth of leaves after defoliation.

In this regard, the solubility with the $\text{NaClO}_3 \cdot 3\text{CO}(\text{NH}_2)_2 - \text{HOC}_2\text{H}_4\text{NH}_2 \cdot \text{C}_4\text{H}_4\text{O}_2\text{N}_2 - \text{H}_2\text{O}$ system was studied visually for the physicochemical justification of the process for obtaining the defoliant based on sodium tricarbamidochlorate and - 3-hydroxypyridazonate-6 monoethanolammonium, as well as establishing the chemical compatibility of the initial components - polythermic method [6] (Figure 3).

The $\text{NaClO}_3 \cdot 3\text{CO}(\text{NH}_2)_2 - \text{HOC}_2\text{H}_4\text{NH}_2 \cdot \text{C}_4\text{H}_4\text{O}_2\text{N}_2 - \text{H}_2\text{O}$ system was studied by five internal polythermal sections. Based on the data on the solubility of the side binary system and the internal sections, a polythermic diagram of the solubility of the $\text{NaClO}_3 \cdot 3\text{CO}(\text{NH}_2)_2 - \text{HOC}_2\text{H}_4\text{NH}_2 \cdot \text{C}_4\text{H}_4\text{O}_2\text{N}_2 - \text{H}_2\text{O}$ system was constructed at temperatures from - 34,4 to 80 °C, on which crystallization fields of ice, $\text{CO}(\text{NH}_2)_2$, $\text{NaClO}_3 \cdot \text{NaClO}_3 \cdot 3\text{CONH}_2)_2$ and $\text{HOC}_2\text{H}_4\text{NH}_2 \cdot \text{C}_4\text{H}_4\text{O}_2\text{N}_2$, and two invariant points of the system are established.

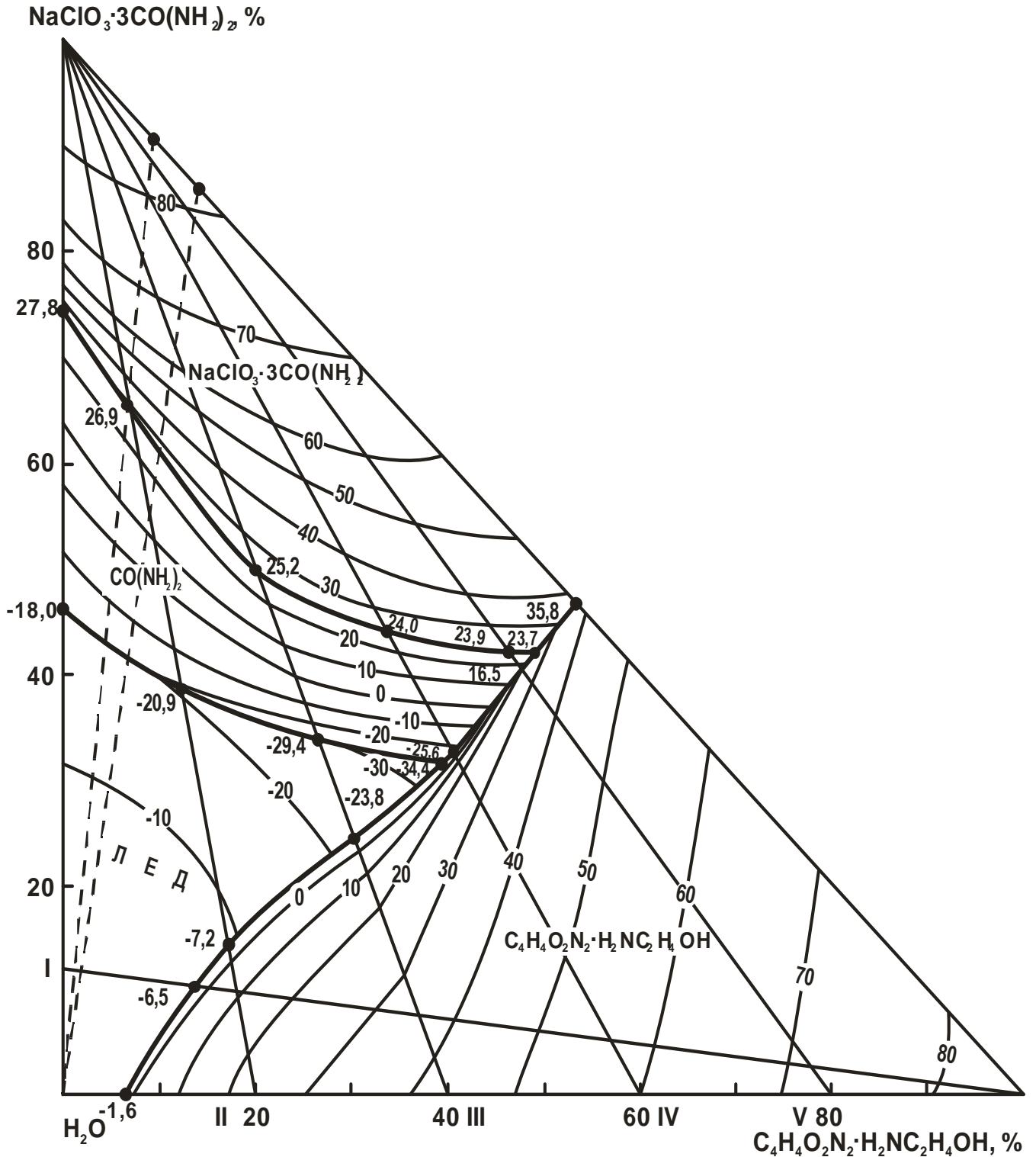


Fig. 3. Diagram of the solubility of the tricarbamidochlorate system sodium - 3-hydroxypyridazonate-6 monoethanolammonium-water



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The polythermic solubility diagram shows isotherms every 10 ° C in the temperature range from -30 to 80 ° C. From the given data it is visible. That in the system studied, neither new chemical compounds nor solid solutions are formed on the basis of the original components. The components of the $\text{NaClO}_3 \cdot 3\text{CO}(\text{NH}_2)_2 - \text{HOC}_2\text{H}_4\text{NH}_2 \cdot \text{C}_4\text{H}_4\text{O}_2\text{N}_2 - \text{H}_2\text{O}$ system retain their individuality. And consequently, the necessary physiological activity. The specificity of the polytherm of solubility of the system studied is that in the presence of 3-hydroxypyridazone-6 monoethanolammonium, the solubility of sodium tricarbamidochlorate decreases, while the latter, on the contrary, exerts a salting out effect on 3-hydroxypyridazone-6 monoethanolammonium. Therefore, its solubility in the presence of sodium tricarbamidochlorate is increased.

IV. SCOPE

From the results of the study of the $\text{NaClO}_3 \cdot 3\text{CO}(\text{NH}_2)_2 - \text{HOC}_2\text{H}_4\text{NH}_2 \cdot \text{C}_4\text{H}_4\text{O}_2\text{N}_2 - \text{H}_2\text{O}$ system it follows that with the simultaneous presence of the initial components, the formation of their concentrated solutions or melts with a low crystallization temperature is observed. This indicates the possibility of obtaining liquid and solid defoliants based on sodium tricarbamidochlorate-3-hydroxypyridazone-6 monoethanolammonium at relatively low temperature conditions.

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