The solubility in monoetanolfmine–3–oxyhiridazone-6-water and sodium trikarbamidochlorate-3-oxyhiridazone-6-water

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ABSTRACT: The solubility in the ternary monoethanolamine-3-oxypyridazone-6-water systems and sodium tricarbamidochlorate-monoethanolamine-3-oxypyridazone-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(C₂H₄OH)₃ ∙ C₄H₄N₂O₂, 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 defoliants that enhance their activity [1-5]. With respect to the bases, 3-hydroxypyridazone-6 (C₄H₄N₂O₂) behaves as a monobasic acid [5]. Therefore, it is of interest to study heterogeneous phase equilibrium in the ternary system N(C₂H₄OH)-C₄H₄N₂O₂-H₂O 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 NH₂C₂H₄OH ∙ C₄H₄N₂O₂. 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 NH₂C₂H₄OH-C₄H₄N₂O₂-H₂O 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 NH₂C₂H₄OH ∙ C₄H₄N₂O₂ 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 NH$_2$C$_2$H$_4$OH · C$_6$H$_4$N$_2$O$_2$ gave the following results: found in %: C - 41.57; H$_2$ - 6.38; N$_2$ - 24.31. For NH$_2$C$_2$H$_4$OH · C$_6$H$_4$N$_2$O$_2$ is calculated, in %: C – 41.61; H$_2$ - 6.35; N$_2$ - 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; 3.448; 3.326; 3.227; 3.129; 2.970; 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).

![X-ray patterns](image)

**Fig. 2.** X-ray patterns of 3-hydroxypyridazone-6 (1) and 3-hydroxypyridazinate-6 monoethanolammonium (2).

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

In this regard, the solubility with the NaClO$_3$ · 3CO (NH$_2$)$_2$ · HOC$_2$H$_4$NH$_2$ · C$_6$H$_4$O$_2$N$_2$-H$_2$O system was studied visually for the physicochemical justification of the process for obtaining the defoliant based on sodium tricarbamidochlorate and - 3-hydroxypyridazinate-6 monoethanolammonium, as well as establishing the chemical compatibility of the initial components - polythermic method [6] (Figure 3).

The NaClO$_3$ · 3CO (NH$_2$)$_2$ · HOC$_2$H$_4$NH$_2$ · C$_6$H$_4$O$_2$N$_2$-H$_2$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 NaClO$_3$ · 3CO(NH$_2$)$_2$ · HOC$_2$H$_4$NH$_2$ · C$_6$H$_4$O$_2$N$_2$·H$_2$O system was constructed at temperatures from -34.4 to 80 °C, on which crystallization fields of ice, CO(NH$_2$)$_2$, NaClO$_3$; NaClO$_3$ · 3CONH$_2$ and HOC$_2$H$_4$NH$_2$ · C$_6$H$_4$O$_2$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
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 NaClO₃ • 3CO(NH₂)₂ - HOC₂H₄NH₂ • C₄H₄O₂N₂ - H₂O 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-hydroxyypyridazonate-6 monoethanolammonium, the solubility of sodium tricarbamidochlorate decreases, while the latter, on the contrary, exerts a salting out effect on 3-hydroxyypyridazonate-6 monoethanolammonium. Therefore, its solubility in the presence of sodium tricarbamidochlorate is increased.

IV. SCOPE

From the results of the study of the NaClO₃ • 3CO(NH₂)₂ - HOC₂H₄NH₂ • C₄H₄O₂N₂ - H₂O 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-hydroxyypyridazonate-6 monoethanolammonium at relatively low temperature conditions.

REFERENCES