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**POLYTHERMAL SOLUBILITY AND PHASE EQUILIBRIA IN THE ACETATE
CARBAMIDE–ETHANOLAMINE–WATER SYSTEMS AND THEIR POTENTIAL
APPLICATION IN AGROCHEMICAL SYNTHESIS.**

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Abstract. *The study investigates the polythermal solubility and phase equilibria of the ternary systems acetate carbamide–ethanolamine–water over a temperature range from –56.2 °C to 18 °C. The polythermal solubility diagrams were constructed based on multiple cross-sections, revealing the crystallization fields of ice, acetate carbamide, ethanolamines, and newly formed compounds–acetate carbamide–diethanolammonium and acetate carbamide–triethanolammonium. The position of secondary and ternary eutectic points was determined, indicating the complex phase behavior of these multicomponent aqueous systems. The phase boundaries and temperature–composition relationships were established experimentally. The obtained data provide valuable physicochemical insights for developing innovative agrochemical preparations, as the newly formed compounds exhibit promising potential as active ingredients in fertilizers and plant growth regulators. These results contribute to the rational design of efficient and environmentally safe agrochemical systems.*

Key words: *polythermal solubility, phase equilibria, acetate carbamide, ethanolamine, triethanolamine, ternary system, crystallization fields, agrochemical synthesis, eutectic point, physicochemical analysis.*

INTRODUCTION

The investigation of multicomponent aqueous systems plays a vital role in modern solution chemistry and chemical technology, particularly in developing new functional materials and agrochemical compounds. The phase behavior of such systems provides essential information on the mutual solubility of components, the formation of solid phases, and the identification of new compounds with potential industrial applications. Among these, systems containing carbamide derivatives and alkanolamines are of significant interest due to their ability to form stable hydrogen-bonded networks and to participate in acid–base and complex-forming reactions.

Acetate carbamide, a compound derived from acetic acid and urea, exhibits strong hydrogen-bonding interactions with alcohol amines such as monoethanolamine, diethanolamine, and triethanolamine. Studying the polythermal solubility and phase equilibria in such systems provides valuable insights into their physicochemical nature and thermal stability.

These investigations are also crucial for understanding the crystallization mechanisms, as the formation of new solid phases—such as acetate carbamide-diethanolammonium and acetate carbamide-triethanolammonium—can lead to the discovery of novel physiologically active substances. Previous studies have shown that temperature and concentration variations significantly influence the mutual solubility and phase composition of aqueous organic–inorganic systems.

However, systematic research covering wide temperature ranges for acetate carbamide–ethanolamine systems remains limited. Therefore, constructing detailed polythermal solubility diagrams and determining eutectic and peritectic points are essential for predicting the behavior of such systems under different conditions.

The present work aims to study the polythermal solubility and phase equilibria in the ternary systems acetate carbamide–diethanolamine–water and acetate carbamide–triethanolamine–water over a wide temperature range. The obtained data will provide a physicochemical foundation for developing new agrochemical agents and optimizing the synthesis of environmentally friendly fertilizers and plant growth stimulants based on urea derivatives and alkanolamines.

MATERIAL AND METHODS

The experimental investigation was carried out to study the polythermal solubility and phase equilibria in the ternary systems acetate carbamide–diethanolamine–water and acetate carbamide–triethanolamine–water. Chemically pure reagents of analytical grade were used: acetic acid, urea, diethanolamine, triethanolamine, and distilled water. All experiments were performed using sealed glass ampoules to minimize evaporation and contamination.

The solubility of the systems was determined by the polythermal method over a temperature range from $-56.2\text{ }^{\circ}\text{C}$ to $18.0\text{ }^{\circ}\text{C}$. Samples containing different component ratios were equilibrated in thermostatic baths at selected temperatures until equilibrium was reached.

The equilibrated mixtures were then quickly cooled and filtered to separate the liquid and solid phases. The concentration of each component in the liquid phase was analyzed gravimetrically, while the crystallization temperature was measured using a precision cryothermostat with an accuracy of $\pm 0.1\text{ }^{\circ}\text{C}$.

To construct the polythermal solubility diagrams, 7–10 internal cross-sections were studied for each ternary system, and the boundary points of phase crystallization fields were plotted. Phase compositions and eutectic points were established based on the mutual solubility data and the identification of solid phases. Phase diagrams and projections were developed to visualize the fields of ice, acetate carbamide, ethanolamines, and newly formed compounds.

The experimental data were processed using graphical interpolation and least-squares approximation to ensure the accuracy of the phase boundaries. The obtained solubility diagrams served as the basis for the physicochemical interpretation of the system behavior and for identifying new crystalline compounds with potential agrochemical applications.

RESULTS AND DISCUSSION

To provide a physicochemical rationale for the interactions between components in the $\text{CH}_3\text{COOH}\cdot\text{CO}(\text{NH}_2)_2 - \text{NH}(\text{C}_2\text{H}_4\text{OH})_2 - \text{H}_2\text{O}$ system, the system was studied over a wide range of temperatures and concentrations, and a polythermal solubility diagram was constructed.

In constructing the polythermal solubility diagram of the $\text{CH}_3\text{COOH}\cdot\text{CO}(\text{NH}_2)_2 - \text{NH}(\text{C}_2\text{H}_4\text{OH})_2 - \text{H}_2\text{O}$ system, ten internal cross-sections were used. Of these, sections I–V were drawn from the $\text{NH}(\text{C}_2\text{H}_4\text{OH})_2 - \text{H}_2\text{O}$ side toward the $\text{CH}_3\text{COOH}\cdot\text{CO}(\text{NH}_2)_2$ apex, while the

lateral sections VI–X were drawn from the $\text{CH}_3\text{COOH} \cdot \text{CO}(\text{NH}_2)_2 \cdot \text{H}_2\text{O}$ side toward the $\text{NH}(\text{C}_2\text{H}_4\text{OH})_2$ apex. Based on the binary systems and these internal sections, a polythermal solubility diagram of the $\text{CH}_3\text{COOH} \cdot \text{CO}(\text{NH}_2)_2 \cdot \text{NH}(\text{C}_2\text{H}_4\text{OH})_2 \cdot \text{H}_2\text{O}$ system was constructed for the temperature range from -56.2°C to 18.0°C (Fig. 1, Table 1).

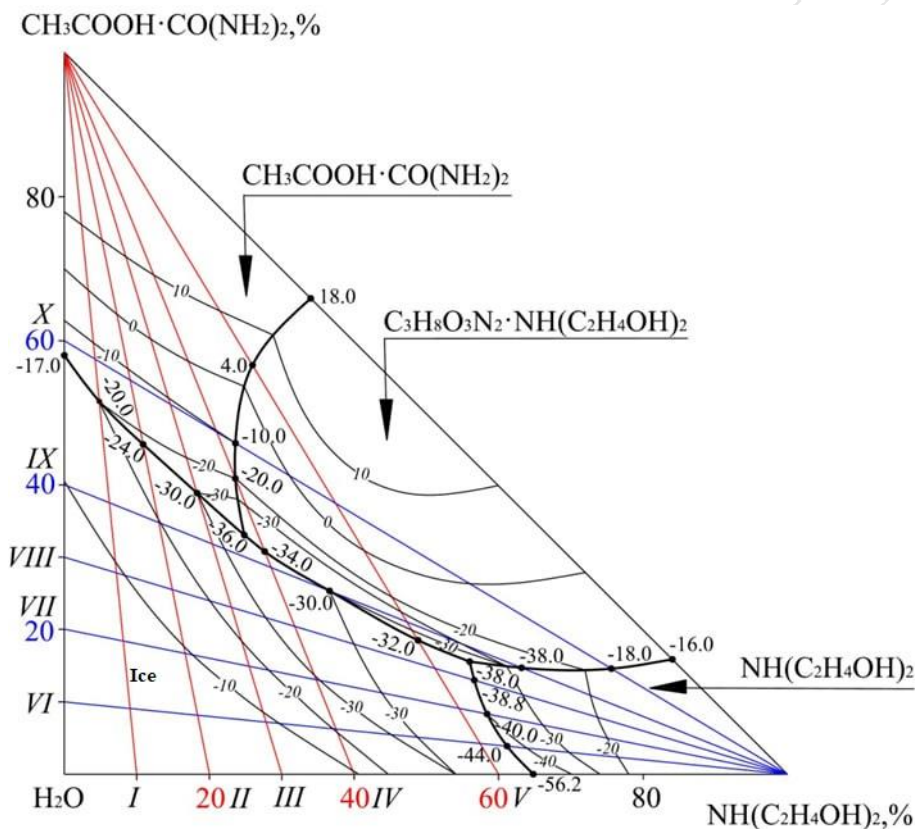


Figure 1. Polythermal solubility diagram of the acetate carbamide–diethanolamine–water system.

Table 1.

Classification of secondary and ternary invariant points in the acetate carbamide–diethanolamine–water system.

Composition of the liquid phase, %			Crystallization temperature, °C	Solid phase
$\text{CH}_3\text{COOH} \cdot \text{CO}(\text{NH}_2)_2$	$\text{NH}(\text{C}_2\text{H}_4\text{OH})_2$	H_2O		
58,0	-	42,0	-17,0	$\text{Ice} + \text{CH}_3\text{COOH} \cdot \text{CO}(\text{NH}_2)_2$
52,0	4,8	43,2	-20,0	-//-
46,0	10,8	43,2	-24,0	-//-
39,4	18,0	42,6	-30,0	-//-
33,4	24,8	41,8	-36,0	$\text{Ice} + \text{CH}_3\text{COOH} \cdot \text{CO}(\text{NH}_2)_2 + \text{C}_3\text{H}_8\text{O}_3\text{N}_2 \cdot \text{NH}(\text{C}_2\text{H}_4\text{OH})_2$
41,2	23,8	35,0	-20,0	$\text{CH}_3\text{COOH} \cdot \text{CO}(\text{NH}_2)_2 + \text{C}_3\text{H}_8\text{O}_3\text{N}_2 \cdot \text{NH}(\text{C}_2\text{H}_4\text{OH})_2$
46,2	23,0	30,8	-10,0	-//-
56,4	26,0	17,6	4,0	-//-
66,0	34,0	-	18,0	-//-
32,0	27,2	40,8	-34,0	$\text{Ice} + \text{C}_3\text{H}_8\text{O}_3\text{N}_2 \cdot \text{NH}(\text{C}_2\text{H}_4\text{OH})_2$

36,8	25,2	62,0	-30,0	-/-
18,8	48,8	32,4	-32,0	-/-
15,6	52,0	32,4	-38,0	Ice+C ₃ H ₈ O ₃ N ₂ ·NH(C ₂ H ₄ OH) ₂ + NH(C ₂ H ₄ OH) ₂
12,8	56,8	30,4	-38,6	Ice+NH(C ₂ H ₄ OH) ₂
8,4	58,4	33,2	-40,0	-/-
4,0	61,2	34,8	-44,0	-/-
-	64,8	35,2	-56,2	-/-
14,8	63,2	22,0	-38,0	C ₃ H ₈ O ₃ N ₂ ·NH(C ₂ H ₄ OH) ₂ + NH(C ₂ H ₄ OH) ₂
14,8	55,6	29,6	-18,0	-/-
16,0	84,0	-	-16,0	-/-

To substantiate the diagram, a two-dimensional projection of the system was constructed (Figure 2).

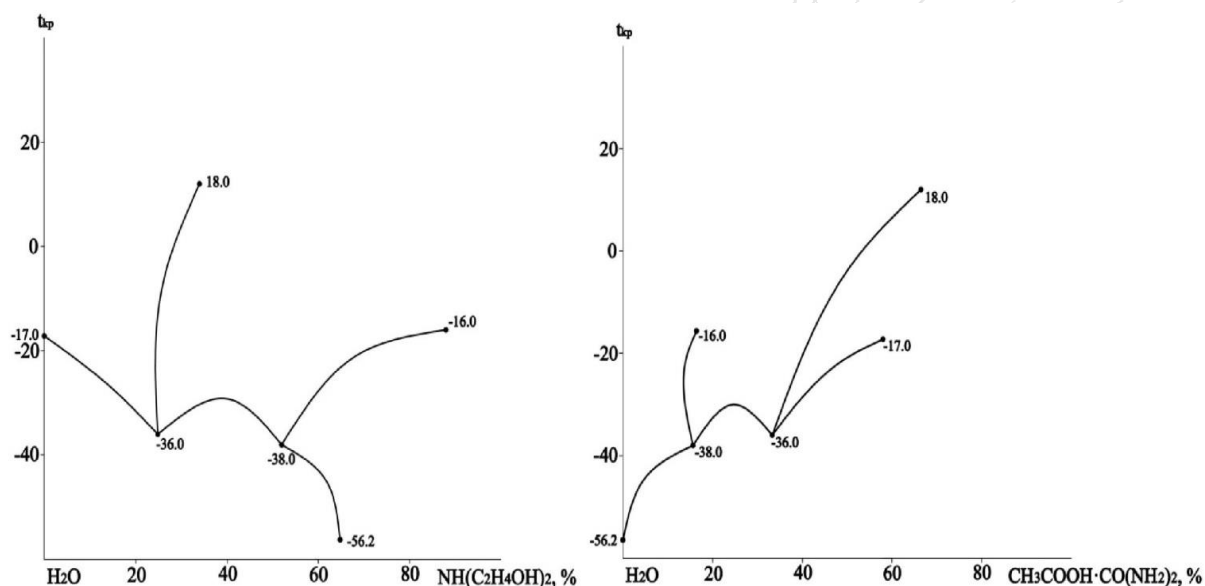


Figure 2. Projection of the acetate carbamide–diethanolamine–water solubility system.

In the phase diagram of this system, the crystallization fields of ice, acetate carbamide, diethanolamine, and the newly formed compound acetate carbamide-diethanolammonium were identified and delineated. These crystallization fields converge at two ternary invariant points. The first ternary point corresponds to a temperature of $-36.0\text{ }^{\circ}\text{C}$ and a composition of 33.4% acetate carbamide, 24.8% diethanolamine, and 41.8% water, while the second corresponds to $-38.0\text{ }^{\circ}\text{C}$ with 15.6% acetate carbamide, 52.0% diethanolamine, and 32.4% water.

Isothermal lines were drawn on the polythermal solubility diagram at every $10\text{ }^{\circ}\text{C}$ interval. From the constructed diagrams, it can be observed that ice crystallizes with acetate carbamide in the temperature range from $-17.0\text{ }^{\circ}\text{C}$ to $-30.0\text{ }^{\circ}\text{C}$; the new phase, acetate carbamide-diethanolammonium, crystallizes with ice between $-34.0\text{ }^{\circ}\text{C}$ and $-32.0\text{ }^{\circ}\text{C}$, and with diethanolamine between $-38.8\text{ }^{\circ}\text{C}$ and $-56.2\text{ }^{\circ}\text{C}$. Acetate carbamide-diethanolammonium also crystallizes together with acetate carbamide from $-20.0\text{ }^{\circ}\text{C}$ to $18.0\text{ }^{\circ}\text{C}$, and with diethanolamine from $-38.0\text{ }^{\circ}\text{C}$ to $-16.0\text{ }^{\circ}\text{C}$.

The solubility of the components in the complex acetate carbamide–triethanolamine–water system was studied over a wide concentration range and a temperature interval from -43°C to 18°C . When constructing the solubility diagram of the system, the binary subsystems of the initial substances were first examined. The study of the $\text{CH}_3\text{COOH}\cdot\text{CO}(\text{NH}_2)_2\text{--H}_2\text{O}$ binary system showed that its solubility diagram contains two crystallization branches corresponding to the solid phases of ice and acetate carbamide. The cryohydrate point of the system occurs at -17°C and corresponds to a composition containing 58.0% acetate carbamide and 42.0% water (Figure 3).

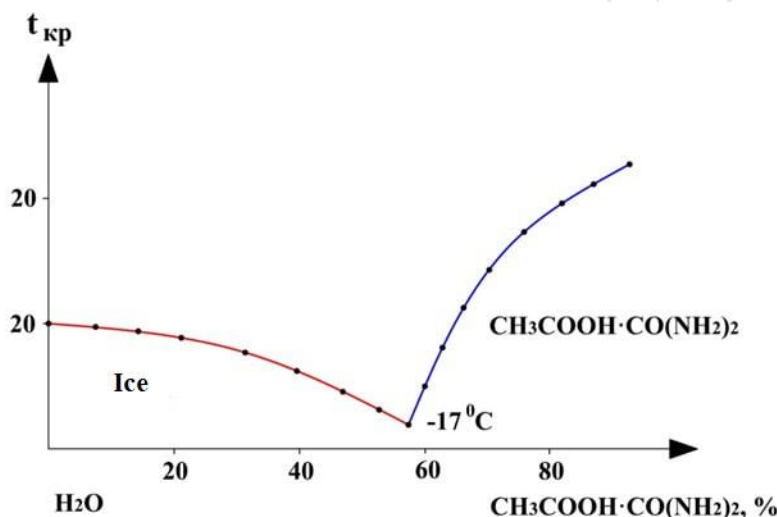


Figure 3. Binary solubility diagram of $\text{CH}_3\text{COOH}\cdot\text{CO}(\text{NH}_2)_2\text{--H}_2\text{O}$.

The solubility diagram of the $\text{CH}_3\text{COOH}\cdot\text{CO}(\text{NH}_2)_2\text{--N}(\text{C}_2\text{H}_4\text{OH})_3\text{--H}_2\text{O}$ system was constructed using seven internal cross-sections (Figure 4).

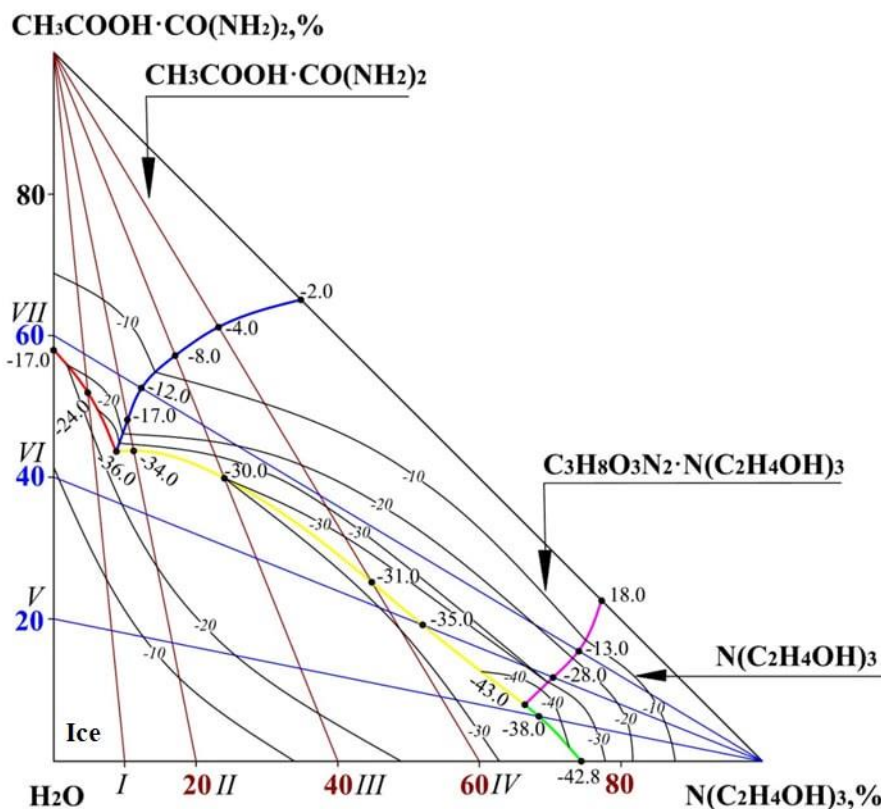


Figure 4. Polythermal solubility diagram of the acetate carbamide–triethanolamine–water system.

The internal cross-sections I–IV were directed from the triethanolamine–water side toward the acetate carbamide apex, whereas sections V–VII were directed from the acetate carbamide–water side toward the triethanolamine apex. The boundaries of the separated phases in the constructed system are also represented in the system projection (Figure 5). In the polythermal solubility diagram of the studied system, the crystallization fields of ice, acetate carbamide, the newly formed compound acetate carbamide–triethanolammonium, and triethanolamine were delineated.

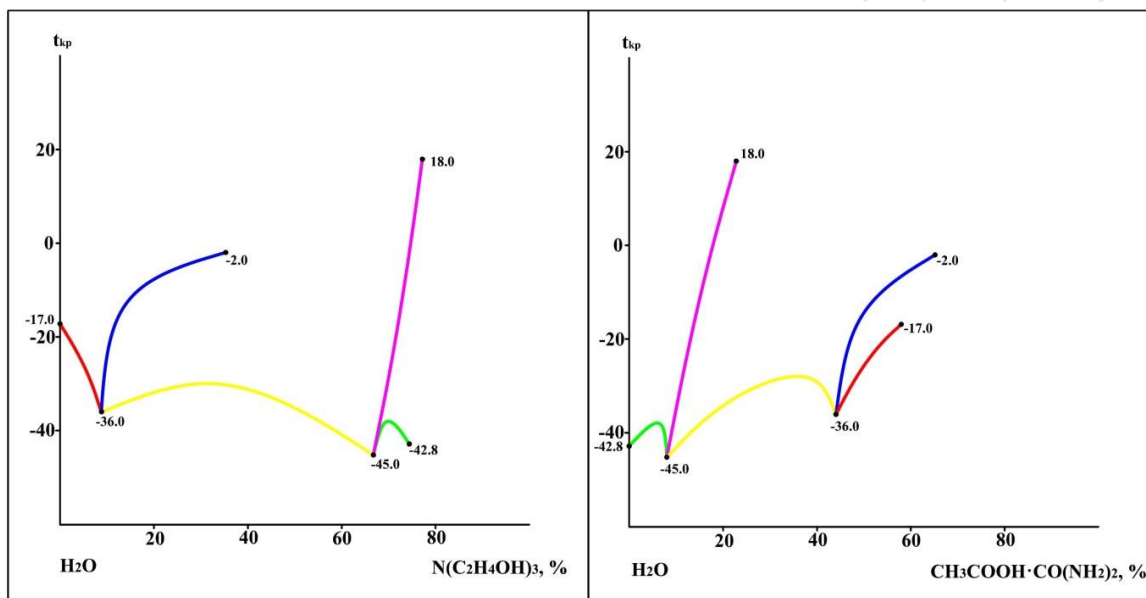


Figure 5. Projection diagram of the acetate carbamide–triethanolamine–water system.

From the constructed diagram, it can be seen that ice crystallizes with acetate carbamide in the temperature range from $-17.0\text{ }^{\circ}\text{C}$ to $-24.0\text{ }^{\circ}\text{C}$, with acetate carbamide–triethanolammonium in the range from $-34.0\text{ }^{\circ}\text{C}$ to $-35.0\text{ }^{\circ}\text{C}$, and with triethanolamine in the range from $-38.0\text{ }^{\circ}\text{C}$ to $-42.8\text{ }^{\circ}\text{C}$. The newly formed compound, acetate carbamide–triethanolammonium, crystallizes together with acetate carbamide between $-17.0\text{ }^{\circ}\text{C}$ and $-2.0\text{ }^{\circ}\text{C}$, and with triethanolamine between $-28.0\text{ }^{\circ}\text{C}$ and $18.0\text{ }^{\circ}\text{C}$ (Table 2).

The resulting phases merge at two ternary invariant points: at $-36.0\text{ }^{\circ}\text{C}$ with a composition of 43.6% acetate carbamide, 8.8% triethanolamine, and 47.6% water; and at $-43.0\text{ }^{\circ}\text{C}$ with a composition of 8.0% acetate carbamide, 66.6% triethanolamine, and 25.4% water.

Table 2.

Classification of secondary and ternary invariant points in the acetate carbamide–triethanolamine–water system.

Composition of the liquid phase, %			Crystallization temperature, $^{\circ}\text{C}$	Solid phase
$\text{CH}_3\text{COOH}\cdot\text{CO}(\text{NH}_2)_2$	$\text{N}(\text{C}_2\text{H}_4\text{OH})_3$	H_2O		
58,0	-	42,0	-17,0	Ice + $\text{CH}_3\text{COOH}\cdot\text{CO}(\text{NH}_2)_2$
52,0	2,8	39,2	-24,0	-//-
43,6	8,8	47,6	-36,0	Ice+ $\text{CH}_3\text{COOH}\cdot\text{CO}(\text{NH}_2)_2$ + $\text{C}_3\text{H}_8\text{O}_3\text{N}_2\cdot\text{N}(\text{C}_2\text{H}_4\text{OH})_3$
48,0	10,4	41,6	-17,0	$\text{CH}_3\text{COOH}\cdot\text{CO}(\text{NH}_2)_2$ + $\text{C}_3\text{H}_8\text{O}_3\text{N}_2\cdot\text{N}(\text{C}_2\text{H}_4\text{OH})_3$

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 ₃ H ₈ O ₃ N ₂ ·N(C ₂ H ₄ OH) ₃
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 ₃ H ₈ O ₃ N ₂ ·N(C ₂ H ₄ OH) ₃ + N(C ₂ H ₄ OH) ₃
6,4	68,2	25,4	-38,0	Ice+N(C ₂ H ₄ OH) ₃
-	74,4	25,6	42,8	-/-
12,0	70,6	17,4	-28,0	C ₃ H ₈ O ₃ N ₂ ·N(C ₂ H ₄ OH) ₃ + N(C ₂ H ₄ OH) ₃
15,8	74,0	10,2	-13,0	-/-
22,8	77,2	-	18,0	-/-

The physiologically active compound obtained—acetate carbamide–triethanolammonium—provides a scientific basis for synthesizing new preparations aimed at increasing agricultural crop yields.

CONCLUSION

This study established comprehensive polythermal solubility and phase-equilibrium relationships for the ternary systems acetate carbamide–diethanolamine–water and acetate carbamide–triethanolamine–water over -56.2 to 18 °C. Polythermal diagrams constructed from multiple internal cross-sections revealed and delineated the crystallization fields of ice, acetate carbamide, ethanolamines, and two previously unreported salts—acetate carbamide–diethanolammonium and acetate carbamide–triethanolammonium. The locations of secondary and ternary invariant points were determined with good precision, clarifying temperature–composition domains where solid phases coexist and enabling reliable interpolation of phase boundaries.

The identification of acetate carbamide–alkanolammonium salts is of particular significance. Their formation windows span technologically accessible temperatures (from subzero to near ambient), which suggests feasibility for scalable syntheses. Given the hydrogen-bonding capacity and acid–base character of these systems, the new salts constitute promising candidates for agrochemical applications (e.g., fertilizer additives or growth-regulating components), offering a physicochemical basis for developing efficient and potentially environmentally safer formulations.

Methodologically, the work validates the use of polythermal sectioning for complex aqueous organic systems and provides reproducible protocols for mapping phase fields.

Limitations include the absence of crystal-structure determination and comprehensive physicochemical profiling (e.g., thermal stability, dissolution kinetics, and bioefficacy). Future research should therefore couple XRD/FTIR/DSC analyses with agronomic performance testing and stability studies under storage and field conditions.

Overall, the results furnish robust reference data and actionable guidance for rational design and optimization of urea- and alkanolamine-based agrochemical systems.

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