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Study of Solubility of the $Ca(ClO_3)_2$-$3NH_2C_2H_4OH \cdot H_3C_6H_5O_7$-$H_2O$ System

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ABSTRACT: The solubility of the system $Ca(ClO_3)_2$-$3NH_2C_2H_4OH \cdot H_3C_6H_5O_7$-$H_2O$ was studied from the temperature of complete freezing (-45.6) to 63.8°C. A polythermal solubility diagram was constructed, on which the crystallization fields of ice, calcium chlorate hexa-, tetra- and dihydrate, trimonoethanolamine citrate monohydrate, trimonoethanolamine citrate and, as a new phase, the compound $(C_6H_5O_7)_2Ca_3 \cdot 4H_2O$, which was identified by chemical and physicochemical methods of analysis, were delineated.

KEY WORDS: solubility, system, diagram, concentration, defoliants, crystallization temperatures.

I.INTRODUCTION

This report presents the results of a study of the solubility of an aqueous system consisting of calcium chlorate and trimonoethanolamine citrate, which are absent from the literature but have a certain scientific and practical interest in the field of obtaining polyfunctional defoliants. Calcium chlorate, the active substance of a calcium chlorate-chloride defoliant of inorganic origin, was used for the studies. However, its effect on plants when the hectare consumption rate is exceeded leads to drying of leaves and burns of young unopened cotton bolls. In addition, the preparation does not have a polyfunctional effect. In the synthesis of new effective defoliants, the use of monoethanolamine salt of citric acid, which is a plant growth stimulator, is of considerable interest. It has biological activity, enhances oxidation-reduction processes, carbohydrate biosynthesis and the activity of enzymatic actions [1-4].

II. METHODOLOGY

To provide a physicochemical justification for the process of obtaining an effective defoliant based on calcium chlorate and trimonoethanolamine citrate, the solubility of the components in the $Ca(ClO_3)_2$-$3NH_2C_2H_4OH \cdot H_3C_6H_5O_7$-$H_2O$ system was studied in a wide temperature and concentration range.
Trimonoethanolamine citrate was synthesized based on citric acid and monoethanolamine taken at a molar ratio of 3:1.
Calcium chlorate dihydrate was obtained as a result of the interaction of stoichiometric mixtures of sodium chlorate and fused calcium chloride of grade "ch" in an acetone medium, followed by recrystallization from an aqueous solution of the solid product isolated from the acetone extract [5]. The binary system calcium chlorate - water, which is part of the system under study, was previously studied by the authors [6]. The data we obtained are in good agreement with the literature.
The solubility polytherm of the $Ca(ClO_3)_2$-$3NH_2C_2H_4OH \cdot H_3C_6H_5O_7$-$H_2O$ system was studied by the visual-polythermal method [7]. Chemical and physicochemical methods of analysis were used to identify the new phase obtained. In the quantitative chemical analysis of liquid and solid phases, calcium was determined by the volumetric complexometric method [8], the content of chlorate ion was determined by the volumetric permanganometric method [9], elemental analysis for carbon, nitrogen, and hydrogen was carried out according to the technique [10]. Thermal analysis was carried out on a Paulik-Paulik-Erdey derivatograph [11] at a rate of 10 deg/min and a sample of 0.1125 g with a sensitivity of T-

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900, TG-200, DTA-1/10, DTG-1/10 galvonometers. Recording was carried out under atmospheric conditions. A platinum crucible with a diameter of 10 mm without a lid served as a holder. Al_2O_3 was used as a standard. X-ray diffraction patterns of the compounds under study were recorded on a Dron-2.0 diffractometer with filtered copper radiation at a voltage of 40 kV, a current of 20 mA, and a counter speed of 2 deg/min [12]. The IR absorption spectra of the starting components and the compounds under study were recorded on a Specord IR-75 spectrophotometer in the frequency range of 4000 – 400 cm–1. The samples were prepared by pressing with KBr and by grinding in vaseline oil [13].

III. EXPERIMENTAL RESULTS

The $Ca(ClO_3)_2$-$3NH_2C_2H_4OH{\cdot}H_3C_6H_5O_7$-$H_2O$ system was studied using eight internal sections. Of these, I-V were drawn from the side of trimonoethanolamine citrate – water to the top of Ca(ClO3)2·2H2O, and VI-VIII from the side of calcium chlorate – water to the pole of $3NH_2C_2H_4OH{\cdot}H_3C_6H_5O_7$.

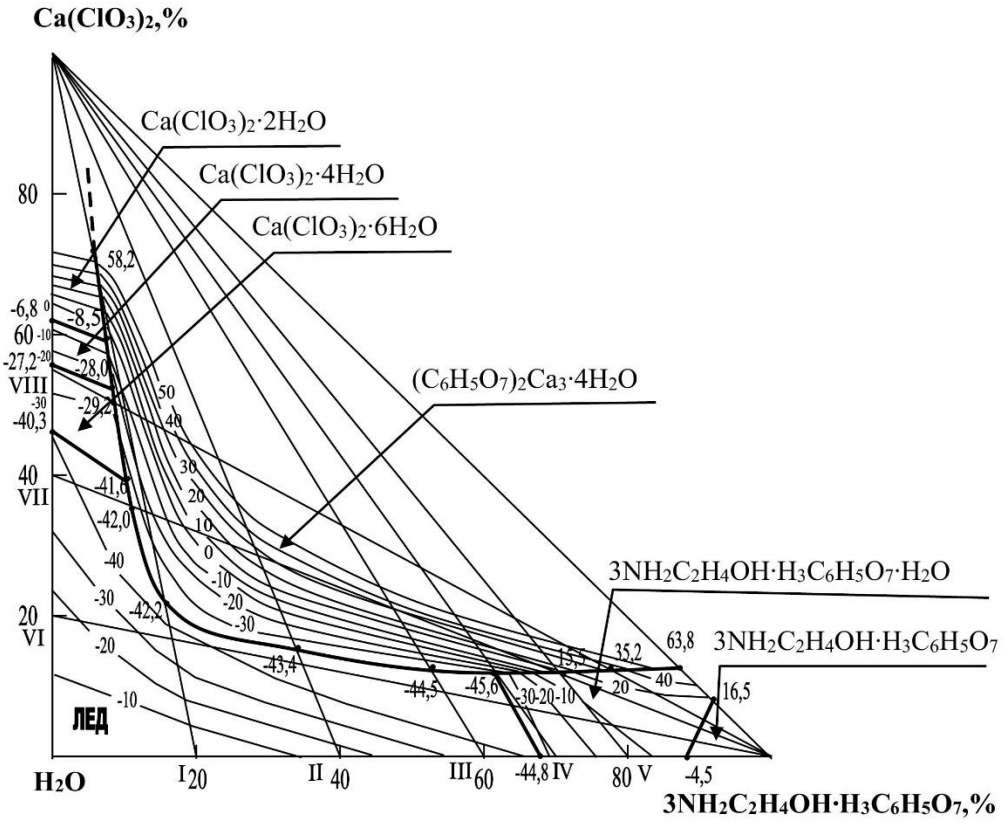

Fig 1: Solubility diagram of the $Ca(ClO_3)_2$-$3NH_2C_2H_4OH{\cdot}H_3C_6H_5O_7$-$H_2O$ system

We studied the solubility in the $3H_2NC_2H_4OH{\cdot}H_3C_6H_5O_7$-$H_2O$ system in the temperature range from -44.8 to 70.0°C. The polythermal diagram of its solubility is characterized by the presence of ice crystallization branches, $3H_2NC_2H_4OH{\cdot}H_3C_6H_5O_7{\cdot}H_2O$ and $3H_2NC_2H_4OH{\cdot}H_3C_6H_5O_7$, which intersect at two double points of joint existence of two solid phases. The first double point corresponds to the joint crystallization of ice and trimonoethanolamine citrate monohydrate at a temperature of -44.8°C and a concentration of 67.0% $2H_2NC_2H_4OH{\cdot}H_3C_6H_5O_7$ and 33.0 % H_2O. The second double point corresponds to the joint crystallization of monohydrate and anhydrous trimonoethanolamine citrate at a temperature of -4.5°C and a concentration of 89.2% dimonoethanolamine citrate and 10.2% water. Based on the solubility data for binary systems and internal sections, a polythermal diagram of the solubility of the calcium chlorate – trimonoethanolamine citrate – water system from -45.6 to 63.8 °C was constructed, in which the crystallization fields of ice, hexa-, tetra- and dihydrate calcium chlorate, monohydrate and anhydrous trimonoethanolamine citrate and the new phase $(C_6H_5O_7)_2Ca_3{\cdot}4H_2O$ were delimited (Fig. 1, Table).

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Table 1. Double and triple points of the system $Ca(ClO_3)_2$-$3NH_2C_2H_4OH{\cdot}H_3C_6H_5O_7$-$H_2O$

Composition of liquid phase, %			$T_{cr.}$, °C	Solid phase
$Ca(ClO_3)_2$	$3NH_2C_2H_4OH{\cdot}H_3C_6H_5O_7$	H_2O		
46,1	-	53.9	-40,3	$Ice+Ca(ClO_3)_2{\cdot}6H_2O$
39.4	11.8	48.8	-41.6	$Ice +Ca(ClO_3)_2{\cdot}6H_2O+ (C_6H_5O_7)_2Ca_3{\cdot}4H_2O$
37.2	12.0	50.8	-42.0	$Ice +(C_6H_5O_7)_2Ca_3{\cdot}4H_2O$
22.6	17.2	60.2	-42.2	The same
18.6	37.2	44.2	-43.4	-//-
17.4	55.8	26.8	-44.5	-//-
13.8	62.4	23.8	-45.6	$Ice+(C_6H_5O_7)_2Ca_3{\cdot}4H_2O+ 3NH_2C_2H_4OH{\cdot}H_3C_6H_5O_7{\cdot}H_2O$
-	67.0	33.0	-44.8	$Ice+3NH_2C_2H_4OH{\cdot}H_3C_6H_5O_7{\cdot}H_2O$
13.9	71.6	14.5	15.5	$(C_6H_5O_7)_2Ca_3{\cdot}4H_2O+ 3NH_2C_2H_4OH{\cdot}H_3C_6H_5O_7{\cdot}H_2O$
14.0	78.5	7.5	35.2	The same
14.2	85.8	-	63.8	-//-
-	89.2	10.8	-4.5	$3NH_2C_2H_4OH{\cdot}H_3C_6H_5O_7{\cdot}H_2O+ 3NH_2C_2H_4OH{\cdot}H_3C_6H_5O_7$
9.6	90.4	-	16.5	The same
55.0	-	45.0	-27.2	$Ca(ClO_3)_2{\cdot}6H_2O+Ca(ClO_3)_2{\cdot}4H_2O$
54.2	8.8	37.0	-28.0	$Ca(ClO_3)_2{\cdot}6H_2O+Ca(ClO_3)_2{\cdot}4H_2O+ (C_6H_5O_7)_2Ca_3{\cdot}4H_2O$
52.0	8.9	39.1	-29.2	$Ca(ClO_3)_2{\cdot}6H_2O+(C_6H_5O_7)_2Ca_3{\cdot}4H_2O$
62.0	-	38.0	-6.8	$Ca(ClO_3)_2{\cdot}4H_2O+Ca(ClO_3)_2{\cdot}2H_2O$
59.6	7.4	33.0	-8.5	$Ca(ClO_3)_2{\cdot}4H_2O+Ca(ClO_3)_2{\cdot}2H_2O+ (C_6H_5O_7)_2Ca_3{\cdot}4H_2O$
71.6	6.2	22.2	58.2	$Ca(ClO_3)_2{\cdot}2H_2O+ (C_6H_5O_7)_2Ca_3{\cdot}4H_2O$

Analysis of the polythermal diagram shows that within the studied concentration and temperature ranges, the formation of a new phase $(C_6H_5O_7)_2Ca_3{\cdot}4H_2O$ occurs in the system, which is isolated in crystalline form and identified by chemical and physicochemical analysis methods.

Chemical analysis yielded the following results: found, %:

C – 26.9; H – 3.15; CaO – 29.50.

for $(C_6H_5O_7)_2Ca_3{\cdot}4H_2O$ calculated, mass %

C – 27.0; H – 3.16; CaO – 29.47.

It is poorly soluble in hot water than in cold water, is well soluble in ammonia, and does not dissolve in organic solvents in acetone, alcohol and benzene.

X-ray phase analysis showed that the compound of the composition $(C_6H_5O_7)_2Ca_3{\cdot}4H_2O$ is characterized by its own values of interplanar distances, which confirms its individuality (Fig. 2). Analysis of the X-ray diffraction pattern of the isolated new phase showed that it does not contain impurities of the original products.

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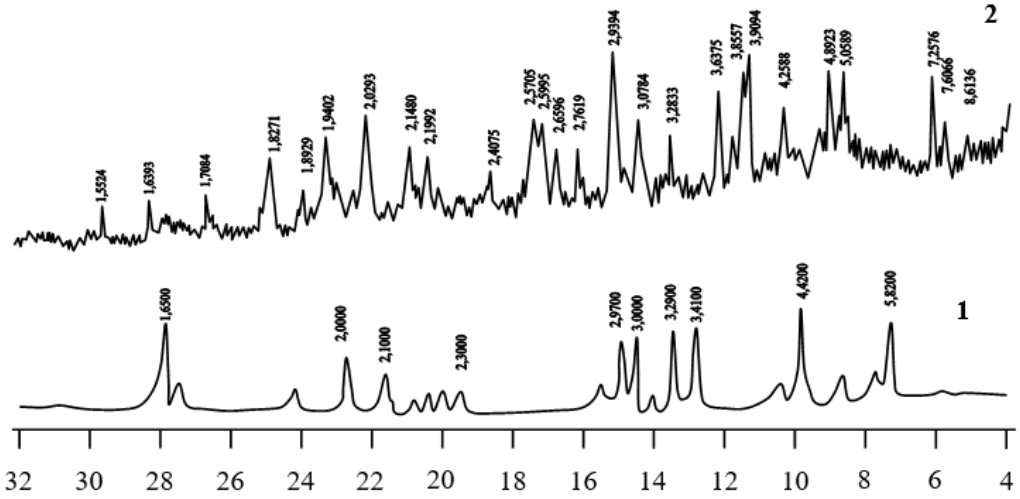

Fig 2: X-ray diffraction patterns 1 - $Ca(ClO_3)_2 \cdot 2H_2O$, 2 - compound $(C_6H_5O_7)_2Ca_3 \cdot 4H_2O$.

On the heating curve of Ca(ClO3)2·2H2O, six endothermic effects are observed at 138, 188, 370, 702, 848, 871 and six exothermic effects at 269, 315, 423, 502, 551 and 618 °C. The endothermic effect, at a temperature range of 138-188 °C, is accompanied by a mass loss of 0.2-16.8% of the substance, which corresponds to the loss of four water molecules. The total mass loss in the temperature range of 60-900 °C according to the thermogravimetric curve is 69.78% (Fig. 3). For the specified compounds, the change in the absorption bands in the IR spectrum is similar to the observed patterns for the compound (C6H5O7)2Ca3·4H2O. The absorption bands of Non are observed in the region of 3434.63 cm-1, which are broadened due to the formation of hydrogen bonds with water molecules. In the metallic carboxyl groups of COO-, symmetrical stretching vibrations are observed at 1360-1450, and asymmetrical stretching vibrations between 1540-1650 cm-1 [14]. In the obtained results, the symmetrical stretching vibrations of the COO- group are 1388-1437 cm-1, and asymmetrical stretching vibrations are obtained on the bands of 1541-1618 cm-1 (Fig. 4).

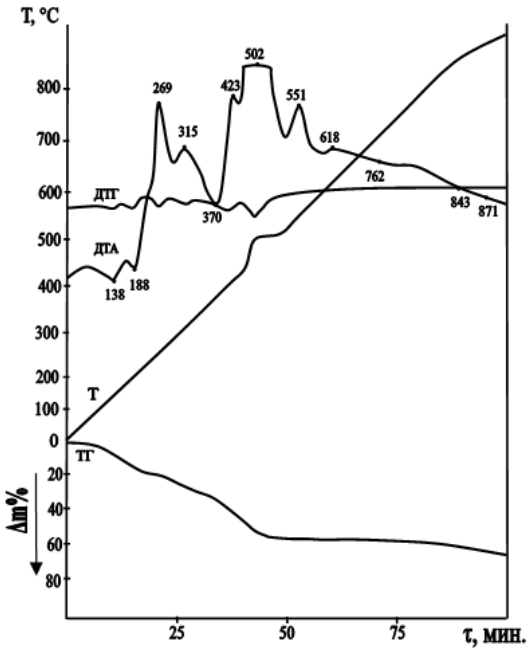

Fig 3: Derivatogram of the compound $(C_6H_5O_7)_2Ca_3 \cdot 4H_2O$.

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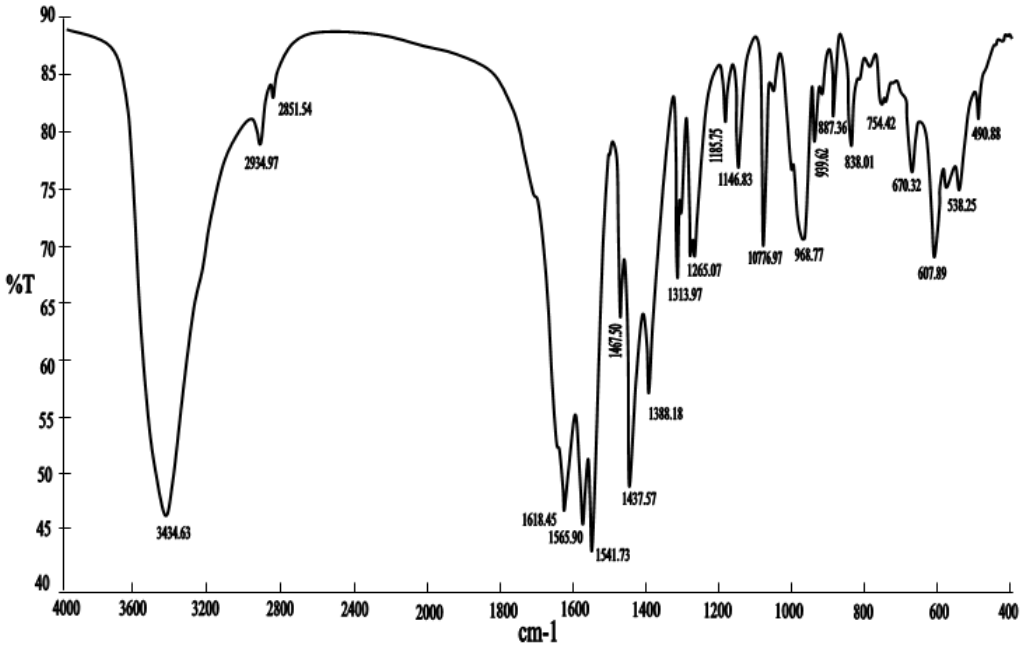

Fig 3: IR spectrum of the compound $(C_6H_5O_7)_2Ca_3 \cdot 4H_2O$.

IV. CONCLUSION AND FUTURE WORK

Thus, a complete polythermal diagram of solubility of the system $Ca(ClO_3)_2$-$3NH_2C_2H_4OH \cdot H_3C_6H_5O_7$-$H_2O$ has been constructed. On the polythermal diagram, the fields of crystallization of the initial components of the system and the new compound, which was identified by chemical and physicochemical analysis and its salt composition was established, are delimited. The obtained data indicate the reliability of the existence of a new compound $(C_6H_5O_7)_2Ca_3 \cdot 4H_2O$. Analysis of the polythermal diagram of solubility shows that the studied system belongs to a complex eutonic type, mutual in nature. Data on the solubility of components in the studied system have been obtained, which can serve as a scientific basis for obtaining a liquid defoliant based on calcium chlorate and trimonoethanolamine citrate.

REFERENCES

[1] Saibova M.T. Application of ethanolamines in agriculture // Uzb. chemical journal. - 1983. - № 1. - P. 58 - 64.
[2] Turayev Q.A., Togasharov A.S., Tukhtaev S. An effective defoliant based on calcium chlorate and sodium salt of monochloroacetic acid. // Journal of Chemical Technology and Metallurgy, 57, 5, 2022 pp. 977-983
[3] Narkhodjaev A.Kh., Adilova M.Sh., Isakova D., Tukhtaev S. Scientific basis for the synthesis of plant growth stimulants from non-returnable waste of primary processing of raw cotton // Proceedings of the International scientific and practical conference on the topic: "Scientific and practical foundations for improving soil fertility." - Tashkent, 2007. part I P. 353-355.
[4] M.I.Khushvaktov,A.S.Togasharov Study of the polythermal solubility and rheological properties of the $C_6H_4(OH)COOH$ • $NH_2C_2H_4OH$ – $CO(NH_2)_2$ – H_2O system // Узб. хим. журн., 2024, №5 С. 42-47.
[5] Auth. certificate. USSR No. 143691. Method for obtaining calcium defoliant chlorate-chloride // M.N. Nabiev, R. Shammasov, S. Tukhtayev et al. // Discoveries, inventions. 1985. No. 9. p. 84.
[6] Zh.Sh. Bobozhonov. Solubility of the Ca(ClO3)2·6H2O – CH3COONH4 –H2O system // Universum: technical sciences: electron. scientific journal. 2022.7(97).
[7] Trunin A.S., Petrova D.G. Visual-polythermal method. Kuibyshev: Kuibyshev Polytechnic Institute. 1977. 94 p. Dep. in VINITI No. 584-78.
[8] Schwarzenbach G., Flaschka G. Complexometric titration. - M.: Chemistry, 1970. - 360 p.
[9] GOST 12257 - 77. Sodium chlorate. Specifications. - M.: Publishing House of Standards, 1987. - 19 p.
[10] Klimova V.A. Basic micromethods for the analysis of organic compounds. - M.: Chemistry, 1975. - 224 p.
[11] Paulik F., Paulik J., Erdey L. The derivatograph I. Mitteilun E. in automatic recording apparatus for scanning. Analysis of differential-thermogravimetric processes // Z. Anal. Chem. - 1958. -V. 160. -№ 4. - P. 241.
[12] Kovba L.M., Trunov V.K. X-ray phase analysis. - Moscow: Moscow State University Press, 1969. - 160 p.
[13] Nakamoto K. IR spectra and Raman spectra of inorganic and coordination compounds. - Moscow: Mir, 1991. - 536 p.
[14] A. Smith // Applied IR spectroscopy. Moscow. "Mir", 1982, 319 p.