

STUDY OF THE POLYTHERMAL SOLUBILITY AND RHEOLOGICAL PROPERTIES OF THE SULFOSALICYLIC ACID DIHYDRATE - MONOETHANOLAMINE - WATER SYSTEM

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ABSTRACT

In this work, the polythermal solubility of the three-component system containing $C_7H_6O_6S \cdot 2H_2O - NH_2C_2H_4OH - H_2O$ was studied in the visual polythermic method based on binary systems and internal sections in the temperature range from $-50.2^\circ C$ to $42.0^\circ C$, and the diagram was constructed. In the solubility diagram, based on the phase-separating binary and ternary points the table was created, and the projection was drawn. In the diagram, the crystallization areas of ice, $C_7H_6O_6S$, $C_7H_6O_6S \cdot 2H_2O$, $NH_2C_2H_4OH \cdot 2H_2O$, $NH_2C_2H_4OH \cdot H_2O$, $NH_2C_2H_4OH$, and a new chemical compound $C_7H_6O_6S \cdot NH_2C_2H_4OH$ were separated. A new phase formed during the study of the $C_7H_6O_6S \cdot 2H_2O - NH_2C_2H_4OH - H_2O$ system was separated, and the separated phase was analysed using chemical, physico-chemical methods and the presence of a new chemical compound ($C_7H_6O_6S \cdot NH_2C_2H_4OH$) was confirmed.

Keywords: monoethanolamine, sulfosalicylic acid, solubility diagram, rheological property, IR spectroscopy, NMR spectrum.

INTRODUCTION

Today, agricultural arable land is shrinking. Therefore, the demand for physiologically active substances important in increasing the yield of agricultural crops is considered high [1]. It is known from the literature that the treatment of pepper seedlings with salicylic and sulfosalicylic acid significantly increases the resistance of seedlings to cold climates [2].

In experiments, salicylic acid, thiourea, methionine, 2 % sucrose and 5-sulfosalicylic (5-SSA) acid, 5-SSA + 4 % sucrose (1:1 ratio) were found to be effective in prolonging the life of the sepal [3]. An experiment was conducted to study the effect of sulfosalicylic acid on the number of flowers of "Gladiolus Grandiflora"

belonging to the "Green Willow" type. The result of the experiment shows that the number of opened flowers increases significantly when the plant is treated with a solution containing sulfosalicylic acid [4].

Monoethanolamine is present in several food products such as cumin, grapes, radish, etc., and has significant activity in plant growth and fighting microbes [5, 6]. Monoethanolamine and its derivatives are also used to protect plants from abiotic and biotic stresses such as salinization [7, 8]. In the plant body, monoethanolamine actively participates in the oxidation-reduction process, enhances the synthesis of organic phosphorus compounds, increases protein metabolism and the activity of enzymatic processes [9, 10]. Monoethanolamine has important functions in

plants such as growth and development, stimulation and effective synergism [11-13]. Under the influence of monoethanolamine salts, ethylene is released in the plant and accumulates in the plant band, because of which the leaves of the plant fall prematurely, which allows to harvest of the cotton crop in agriculture [14 - 16].

In previous studies, several scientific works on the synthesis of physiologically active substances based on inorganic and organic acids and monoethanolamine and their use in agricultural crops were carried out, and effective results were achieved. In particular, the interaction of components in the monoethanolamine, acetic acid and water system was studied in a wide range of concentrations and temperatures from -72.1 to 0°C [17]. The solubility of the components in the acetic acid-triethanolamine-water system was studied in the temperature range from -44.6 to 33.8°C by visual polythermal method, and the new compound was confirmed by physico-chemical methods [18]. The interaction of monoethanolamine, citric acid and the water system has been studied in a wide range of concentrations and temperatures [19]. Optimal conditions for synthesizing monoethanol ammonium sulfate based on sulfuric acid and monoethanolamine were found [20]. Compounds of ethanolamine with inorganic and organic acids have been found to have physiological activity [21 - 23].

We studied the solubility of a three-component system consisting of sulfosalicylic acid dihydrate, monoethanolamine, and water by visual polythermal method, since the system of sulfosalicylic acid dihydrate, monoethanolamine, and water has not been studied before [24].

EXPERIMENTAL

Sulfosalicylic acid dihydrate (GOST 4478-68) and monoethanolamine were used in the experiment. Also, in the experiment, a TN-6 glass mercury thermometer with a detection limit of -30 to 70°C was used in the visual polythermic method. Physico-chemical analysis of the compound $C_7H_6O_6S \cdot NH_2C_2H_4OH$ was determined using a Perkin Elmer Spectrum IR (version 10.7.2), Jeol NMR spectrometer (JNM-ECZR 600 MHz) and elemental percentage composition was determined using a FlashSmart (CHNS/O) elemental analyser.

The pH value of the solution was determined using

an FE 20 METTLER TOLEDO pH meter developed by Mettler-Toledo International Inc. The refractive index of the solutions was measured using the digital refractometer (PAL-BX/RI model, Atago refractometer) at a temperature of 25°C. The viscosity of the solutions was determined by an HPV capillary viscometer with a diameter of 1.16 - 1.84 mm [25]. The viscosity of the solution was repeatedly measured three times, and the average value was recorded. The relative density of the solution was measured by the pycnometric method on a 10 mL capillary pycnometer [26].

RESULTS AND DISCUSSION

The solubility of the system consisting of $C_7H_6O_6S \cdot 2H_2O - NH_2C_2H_4OH - H_2O$ was studied in the temperature range from -50.2°C to 42.0°C by the visual polythermal method using binary systems and six internal cuts and the polythermal solubility diagram of the system was constructed (Fig. 1.). The polythermal solubility diagram of the system was studied at every 10°C temperature interval and the secondary and ternary points of the diagram are found.

The first ternary point corresponds to 4.40 % sulfosalicylic acid dihydrate, 78.4% monoethanolamine and 17.2 % water at -26.0°C. It was found that monoethanolamine monohydrate, monoethanamine, new phase monoethan ammonium sulphasalicylates co-crystallize at this ternary point (Table 1). The second ternary point corresponds to 6.80 % sulfosalicylic acid dihydrate, 63.2 % monoethanolamine, 30.0 % water at -47.0°C and at this ternary point, monoethanolamine dihydrate, monoethanolamine monohydrate and monoethanol ammonium sulfosalicylate crystallize together. The third ternary point was observed at a temperature of -50.2°C. This point corresponds to 10.4 % sulfosalicylic acid dihydrate, 48.0 % monoethanolamine, 41.6 % water. At this ternary point, ice, monoethanolamine dihydrate, and monoethanol ammonium sulfosalicylates co-crystallize. The fourth ternary point corresponds to 48.8 % sulfosalicylic acid dihydrate, 12.0 % monoethanolamine and 39.2 % water at -25.2°C. At this ternary point, ice, sulfosalicylic acid dihydrate, and monoethanol ammonium sulfosalicylates co-crystallize. The fifth ternary point corresponds to 59.2 % sulfosalicylic acid dihydrate, 22.4 %

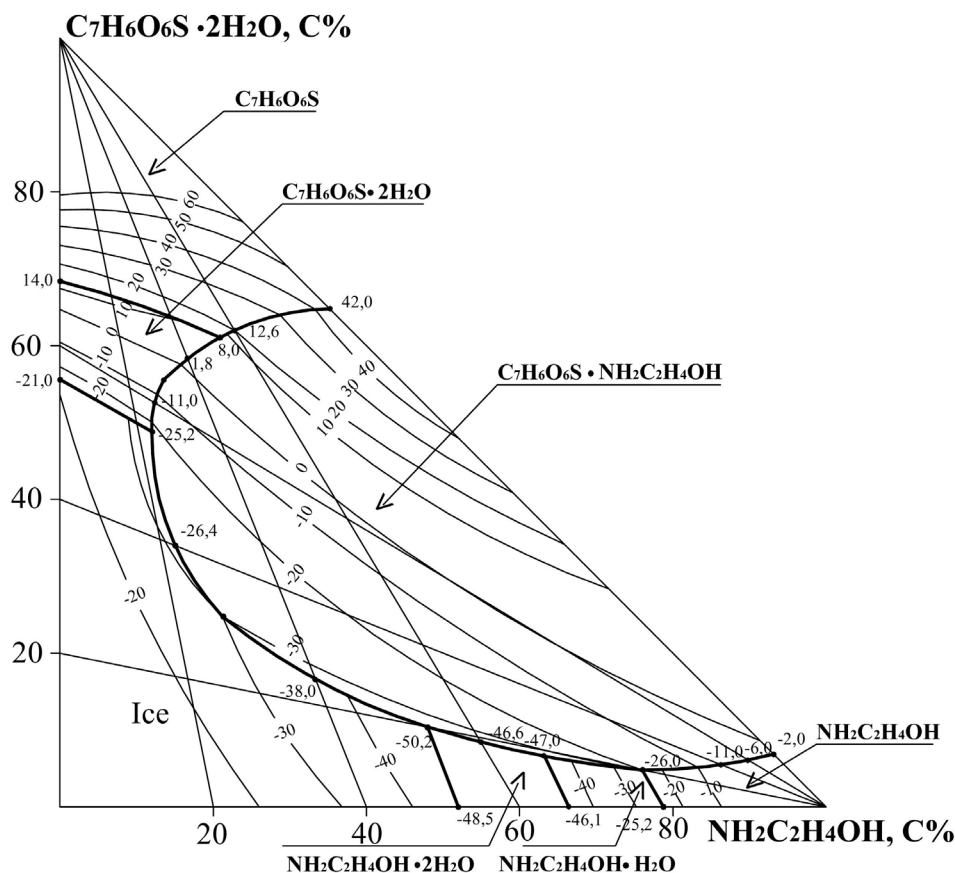


Fig. 1. Solubility diagram of the $C_7H_6O_6S \cdot 2H_2O - NH_2C_2H_4OH - H_2O$ system.

monoethanolamine and 18.4 % water at 8.00°C and at this point, sulfosalicylic acid dihydrate, sulfosalicylic acid, and monoethanolammonium sulfosalicylates were crystallized.

The crystallization areas of ice, sulfosalicylic acid, sulfosalicylic acid dihydrate, monoethanolamine dihydrate, monoethanolamine monohydrate, anhydrous monoethanolamine and the resulting chemical compound monoethanolammonium sulfosalicylate were separated in the spatial state diagram of the studied system. These crystallization areas connect at five ternary points (Figs. 2 and 3).

The new phase formed in the diagram was isolated from the solubility diagram, its composition was checked by chemical and physico-chemical research methods and the presence of a new compound monoethanolammonium sulfosalicylate was determined.

The elemental percentage composition of the compound $C_7H_6O_6S \cdot NH_2C_2H_4OH$ was determined on a FlashSmart (CHNS/O) elemental analyser.

The obtained results were compared practically and theoretically (Table 2).

To identify a compound based on sulfosalicylic acid dihydrate and monoethanolamine by IR spectroscopy, we studied the absorption in the IR spectrum of sulfosalicylic acid dihydrate and monoethanolammonium salicylate (Figs. 4 and 5).

Asymmetric stretch (ν_{as}) and symmetric stretch (ν_s) of the carboxyl group of sulfosalicylic acid were found at 1657.49 cm^{-1} and 1478.51 cm^{-1} , respectively. The characteristic vibration of the water hydroxyl group (OH) in sulfosalicylic acid crystallogite was recorded at 3008.37 cm^{-1} . The deformation oscillation of sulfosalicylic acid, specific for the phenolic (OH) group, was determined at 1365.12 cm^{-1} , and the asymmetric stretch (ν_{as}) and symmetric stretch (ν_s) oscillations characteristic of S-O were at 712.26 - 844.08 cm^{-1} (Fig. 4).

Characteristic asymmetric stretch (ν_{as}) and symmetric stretch (ν_s) of the carboxyl (COO-) group

Table 1. Binary and ternary points of the $C_7H_6O_6S \cdot 2H_2O$ - $NH_2C_2H_4OH$ - H_2O system.

Composition of the liquid phase, wt. %			Cryst, T °C	Solid phase
$C_7H_6O_6S \cdot 2H_2O$	$NH_2C_2H_4OH$	H_2O		
7.20	92.6	0.20	-2.00	$NH_2C_2H_4OH + C_7H_6O_6S \cdot NH_2C_2H_4OH$
6.00	90.4	3.60	-6.00	
5.20	86.8	8.00	-11.0	
4.40	78.4	17.2	-26.0	$NH_2C_2H_4OH \cdot H_2O + NH_2C_2H_4OH + C_7H_6O_6S \cdot NH_2C_2H_4OH$
-	78.4	21.6	-25.2	$NH_2C_2H_4OH \cdot H_2O + NH_2C_2H_4OH$
6.80	63.2	30.0	-47.0	$NH_2C_2H_4OH \cdot 2H_2O + NH_2C_2H_4OH \cdot H_2O + C_7H_6O_6S \cdot NH_2C_2H_4OH$
-	66.4	33.6	-46.1	$NH_2C_2H_4OH \cdot 2H_2O + NH_2C_2H_4OH \cdot H_2O$
8.40	54.8	36.8	-46.6	$NH_2C_2H_4OH \cdot 2H_2O + C_7H_6O_6S \cdot NH_2C_2H_4OH$
10.4	48.0	41.6	-50.2	Ice + $NH_2C_2H_4OH \cdot 2H_2O + C_7H_6O_6S \cdot NH_2C_2H_4OH$
-	52.0	48.0	-48.5	Ice + $NH_2C_2H_4OH \cdot 2H_2O$
15.2	34.0	50.8	-38.0	Ice + $C_7H_6O_6S \cdot NH_2C_2H_4OH$
33.6	16.0	50.4	-26.4	
48.8	12.0	39.2	-25.2	Ice + $C_7H_6O_6S \cdot 2H_2O + C_7H_6O_6S \cdot NH_2C_2H_4OH$
55.6	-	44.4	-21.0	Ice + $C_7H_6O_6S \cdot 2H_2O$
52.0	13.6	34.4	-11.0	$C_7H_6O_6S \cdot 2H_2O + C_7H_6O_6S \cdot NH_2C_2H_4OH$
55.6	17.6	26.8	1.80	
59.2	22.4	18.4	8.00	$C_7H_6O_6S \cdot 2H_2O + C_7H_6O_6S + C_7H_6O_6S \cdot NH_2C_2H_4OH$
60.0	24.2	15.8	12.6	$C_7H_6O_6S + C_7H_6O_6S \cdot NH_2C_2H_4OH$
64.8	35.2	-	42.0	

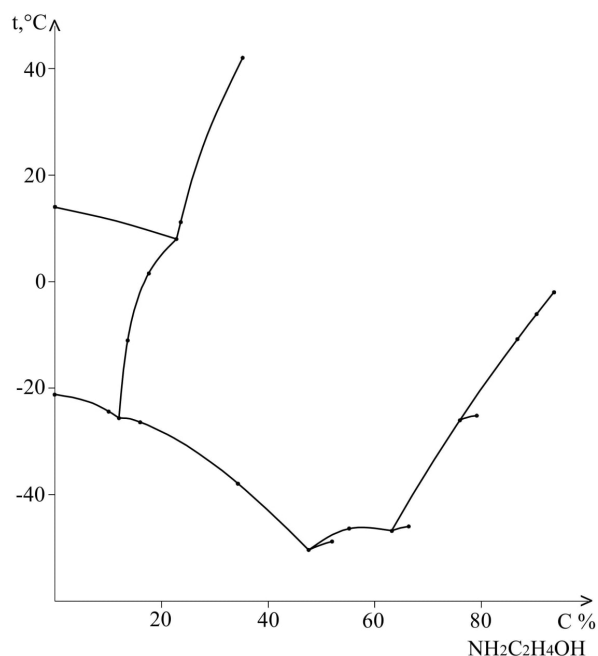
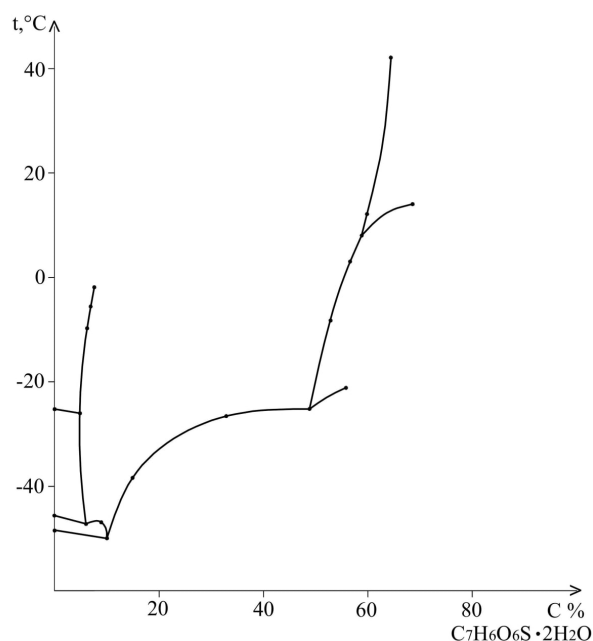
Fig. 2. Projection diagram of the $C_7H_6O_6S \cdot 2H_2O$ - $NH_2C_2H_4OH$ - H_2O system, monoethanolamine side.Fig. 3. Projection diagram of the system $C_7H_6O_6S \cdot 2H_2O$ - $NH_2C_2H_4OH$ - H_2O , sulfosalicylic acid dihydrate side.

Table 2. Elemental percentage composition of the compound $C_7H_6O_6S \cdot NH_2C_2H_4OH$.

Compound formula	Molecular mass	C %		H %		O %		S %		N %	
		calculated	found	calculated	found	calculated	found	calculated	found	calculated	found
$C_7H_6O_6S \cdot NH_2C_2H_4OH$	279 g mole ⁻¹	38.71	38.64	4.66	4.64	40.14	40.26	11.47	11.45	5.02	5.01

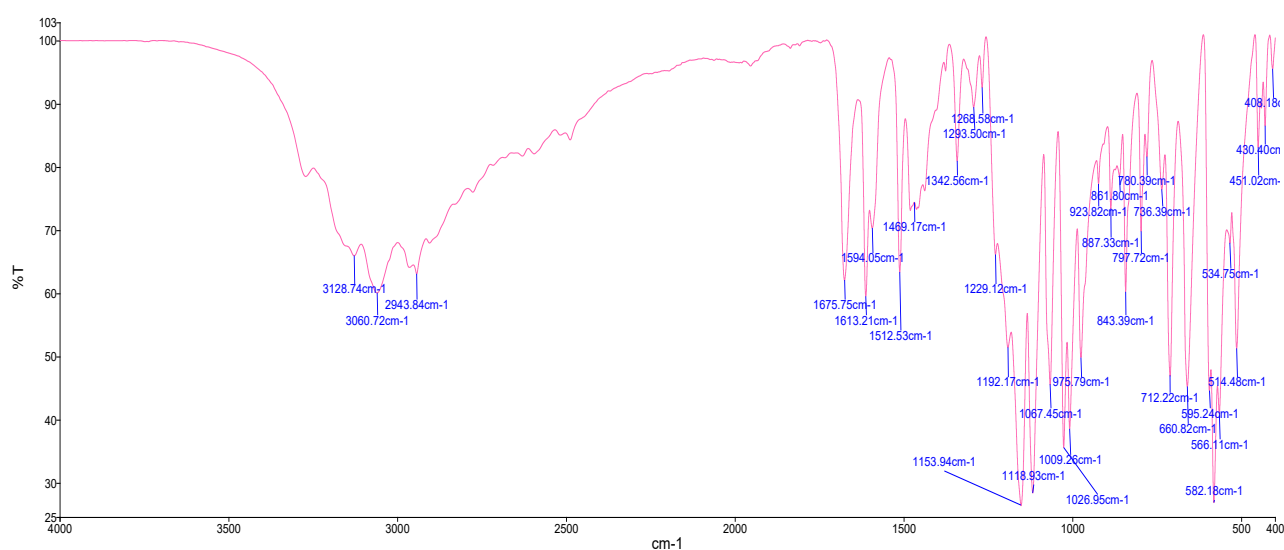


Fig. 4. The IR spectrum of sulfosalicylic acid dihydrate.

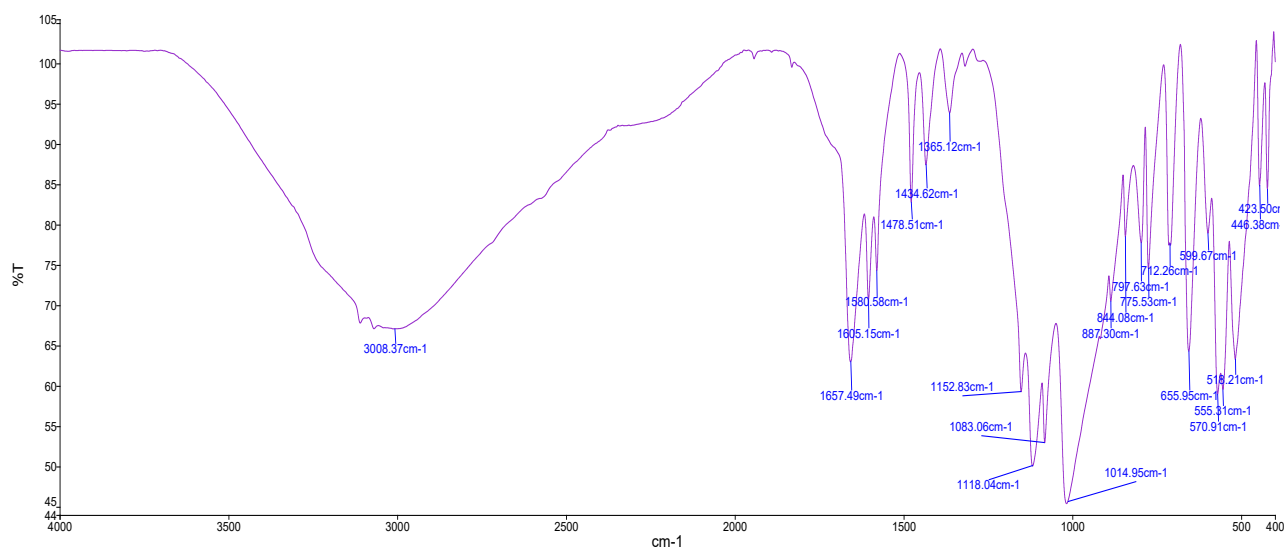


Fig. 5. The IR spectrum of monoethanolammonium sulfosalicylate.

of monoethanolammonium sulfosalicylate were found at 1675.75 cm^{-1} and 1594.05 cm^{-1} , respectively.

It was found that the characteristic deformation stretch of the phenolic (OH) group of monoethanolammonium sulfosalicylate remained at 1342.56 cm^{-1} .

However, the asymmetric valence (ν_{as}) and symmetric valence vibrations (ν_s) of sulfosalicylic acid shift to the high frequency region 975.79 and 1009.26 cm^{-1} , and the intensity changes were determined.

Also, in the IR spectrum of monoethanolammonium sulfosalicylate, the oscillation bands characteristic of the amino group was recorded at 3128.74 cm^{-1} (Fig. 5). These indicators indicate the chemical interaction of the sulfogroup and the amino group.

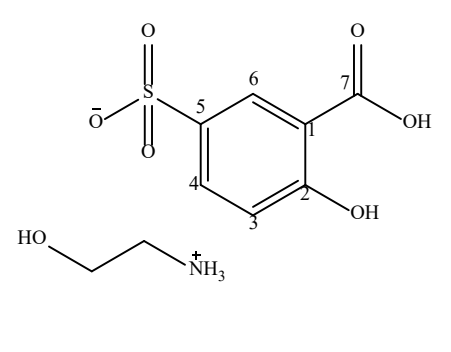
The new phase-separated compound formed in

the polythermal solubility diagram was dissolved in D_2O and ^1H and ^{13}C NMR spectra were obtained and analysed (Figs. 6 and 7). ^1H and ^{13}C NMR spectra of the synthesized compound are presented (Table 3).

It can be seen from the ^1H and ^{13}C NMR spectra (Fig. 5 and Fig. 6) of the sample that sulfosalicylic acid and monoethanolamine satisfy a 1:1 stoichiometric ratio and together form a salt-like compound.

The “composition-property” of the $[45.0\% \text{ NH}_2\text{C}_2\text{H}_4\text{OH} + 55.0\% \text{ H}_2\text{O}] - \text{C}_7\text{H}_6\text{O}_6\text{S} \cdot 2\text{H}_2\text{O}$ system were also checked. When sulfosalicylic acid dihydrate was added in different proportions to a 45.0 % monoethanol solution, it was found that the refractive index, viscosity, density, crystallization temperature and pH parameters of the solution changed (Table 4).

Table 3. Data of ^1H and ^{13}C NMR spectra of the synthesized compound.

	No	$\delta (^{13}\text{C} / \text{ppm})$	$\delta (^1\text{H} / \text{ppm})$
	C-1	112.80	-
	C-2	162.18	-
	C-3	117.80	7.05
	C-4	132.62	7.89
	C-5	133.90	-
	C-6	128.37	8.27
	C-7	171.52	-
	HO-CH ₂ -	57.54	3.83
	-CH ₂ -NH ₃ ⁺	41.22	3.16

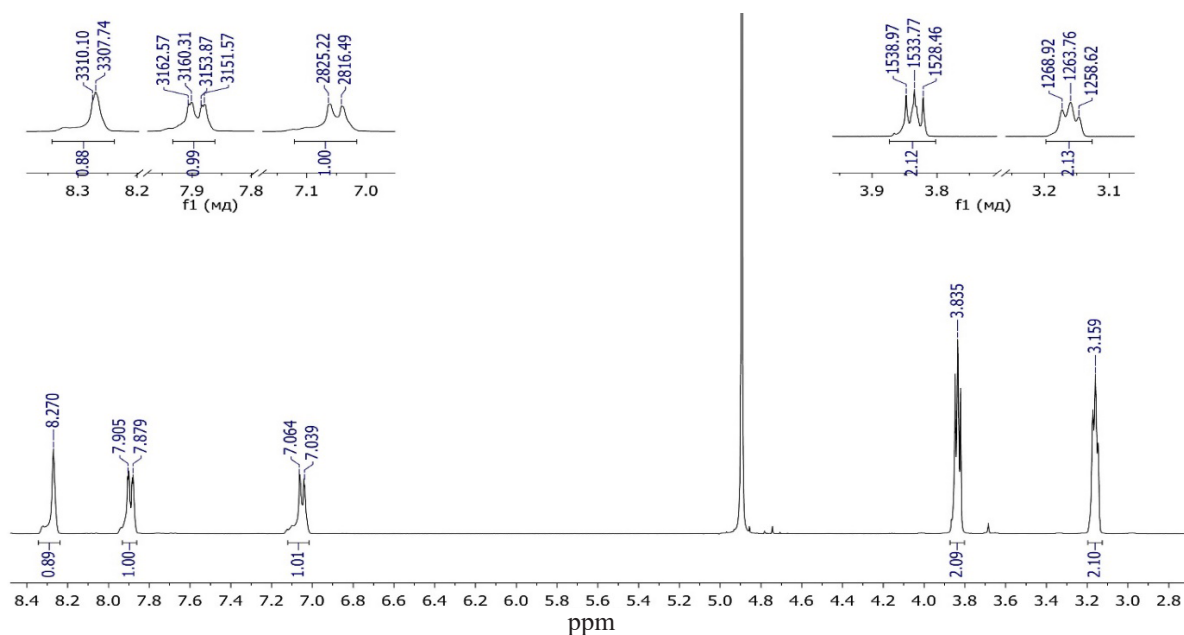


Fig. 6. ^1H NMR spectrum of monoethanolammonium sulfosalicylate.

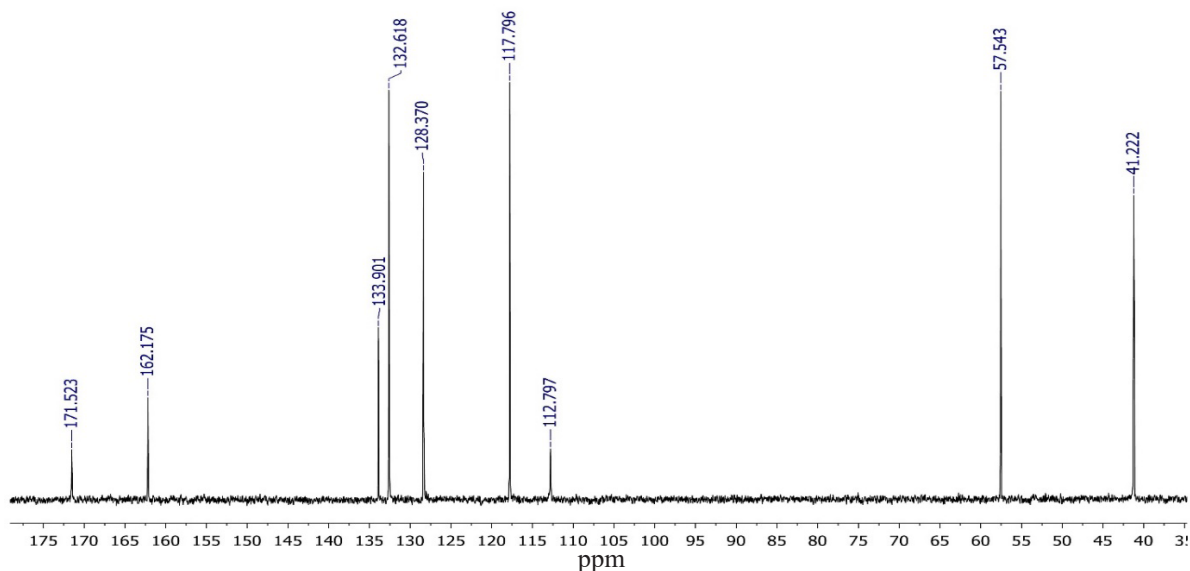


Fig.7. ^{13}C NMR spectrum of monoethanolammonium sulfosalicylate.

Table 4. Physico-chemical properties of solutions of the $[45.0\% \text{NH}_2\text{C}_2\text{H}_4\text{OH} + 55.0\% \text{H}_2\text{O}] - \text{C}_7\text{H}_6\text{O}_6\text{S} \cdot 2\text{H}_2\text{O}$ system.

The composition of the components, %		pH	Cryst, t, °C	Refractive index, n_D	Density d, g ml ⁻¹	Viscosity η , mm ² s ⁻¹
[45% $\text{NH}_2\text{C}_2\text{H}_4\text{OH} + 55\% \text{H}_2\text{O}$]	$\text{C}_7\text{H}_6\text{O}_6\text{S} \cdot 2\text{H}_2\text{O}$					
100.0	-	12.10	-39.0	1.3840	1.0210	3.3235
97.48	2.520	12.01	-35.2	1.3889	1.0354	3.3948
94.99	5.010	11.82	-31.6	1.3935	1.0495	3.4645
90.00	10.00	11.50	-26.2	1.3990	1.0556	3.7700
87.76	12.24	11.40	-23.5	1.4016	1.0715	3.8527
84.98	15.02	11.01	-21.2	1.4040	1.0870	3.9345
82.69	17.31	11.09	-14.7	1.4052	1.1058	4.2290
79.60	20.40	10.80	-12.1	1.4098	1.1245	4.5220
76.80	23.20	10.64	-6.80	1.4167	1.1388	4.7338
74.96	25.04	10.49	-4.80	1.4237	1.1530	4.9450
71.98	28.02	10.25	-1.20	1.4275	1.1659	5.1449
67.58	32.42	10.08	2.40	1.4325	1.1786	5.3445
64.97	35.03	9.60	4.80	1.4360	1.1891	5.3978
61.98	38.02	9.35	7.20	1.4390	1.1996	5.6505
58.48	41.52	8.90	9.60	1.4420	1.2093	5.8145
54.99	45.01	8.30	12.2	1.4470	1.2190	6.0025
52.96	47.04	8.02	13.8	1.4492	1.2267	6.1084
49.99	50.01	7.50	16.0	1.4510	1.2340	6.2140
47.41	52.59	7.10	17.2	1.4532	1.2415	6.2610
44.17	55.83	6.40	19.6	1.4550	1.2490	6.3080
42.20	57.80	6.01	20.2	1.4570	1.2535	6.4960
39.72	60.28	5.10	24.4	1.4635	1.2640	6.8015
37.99	62.01	3.62	26.8	1.4672	1.2745	7.1072

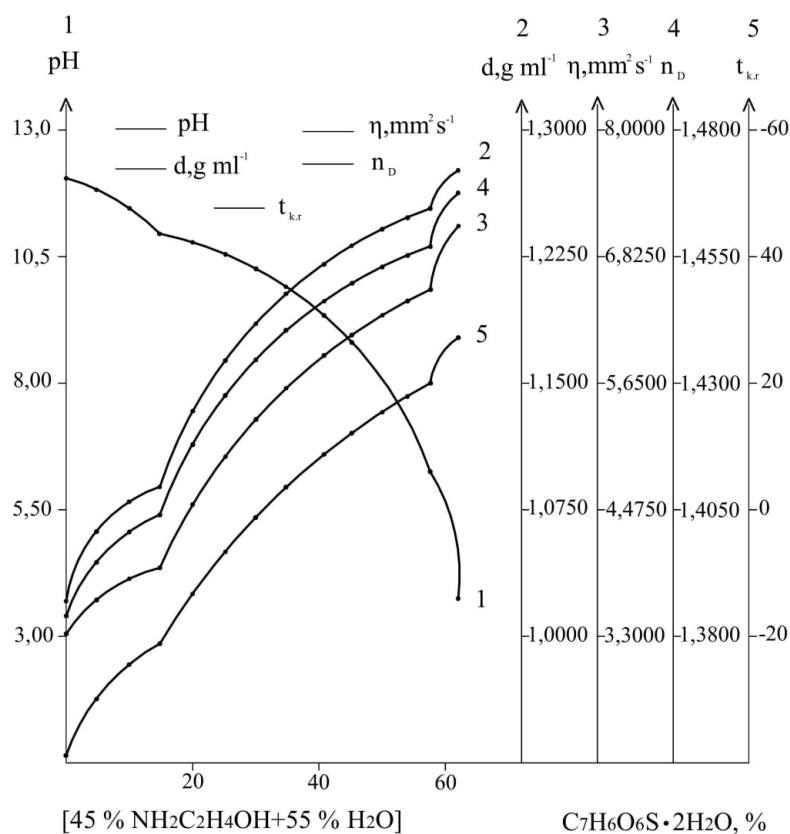


Fig. 8. Physico-chemical and rheological properties of the [45.0 % NH₂C₂H₄OH + 55.0 % H₂O] - C₇H₆O₆S · 2H₂O system at a temperature of 25°C: 1 - pH indicator, 2 - density, 3 - viscosity, 4 - refractive index, 5 - crystallization temperature.

Based on the results obtained, the “composition-property” diagram of the system was constructed (Fig. 8). In the diagram “composition-property” of the [45 % NH₂C₂H₄OH + 55 % H₂O] - C₇H₆O₆S · 2H₂O system, a change in pH was observed (curve 1), with an increase in the concentration of sulfosalicylic acid dihydrate, a change in the pH value of the solution from 12.10 to 3.62. Curve 2 shows a graph of the density dependence on the concentration of the solution, the density of the solution increases from 1.0210 to 1.2745 g ml⁻¹. The viscosity (curve 3) increases from 3.3235 to 7.1072 mm² s⁻¹ with an increase in the concentration of sulfosalicylic acid. The composition-property diagram shows the refractive index graph of the system when sulfosalicylic acid dihydrate is added to 62.01 % on the fourth line.

The fifth line shows the graph of crystallization temperature as a function of concentration. In the [45.0 %

NH₂C₂H₄OH + 55.0 % H₂O] - C₇H₆O₆S · 2H₂O system, when the percentage of sulfosalicylic acid dihydrate was added to 15.02 %, the ice crystallization area was separated. When the percentage of sulfosalicylic acid dihydrate increased from 15.02 % to 57.80 %, the crystallization area of monoethanolammonium sulfosalicylate of the new compound in the system was separated.

From the studied composition-property diagram (viscosity, density, refractive index, pH and crystallization temperature), we can see that the new compound was formed when 15.02 % to 57.80 % sulfosalicylic acid dihydrate was added to this system.

CONCLUSIONS

The polythermal solubility of a three-component system consisting of C₇H₆O₆S · 2H₂O - NH₂C₂H₄OH -

H₂O was studied by the authors for the first time, and the polythermal solubility diagram was constructed based on binary systems and internal cuts. The crystallization areas of the components were delimited in the polythermal solubility diagram. The system belongs to the complex eutonic type, and the formation of a new phase was observed.

The compound formed in the new phase was isolated, its composition was analysed by chemical and physico-chemical methods and the presence of monoethanolammonium sulfosalicylates in the new compound was confirmed.

By adding sulfosalicylic acid dihydrate to a 45.0 % monoethanolamine solution, the rheological properties of the solution were studied, and the formation of the new phase was confirmed in the polythermal solubility diagram.

Authors' contribution: M.I., A.S.: Study conception and design; M.I.: data collection; M.I., A.S., A.B and R.K.: analysis and interpretation of results; M.I., A.S.: draft manuscript preparation. All authors reviewed the results and approved the final version of the manuscript. The M.I. confirms sole responsibility for the following: study conception and design, data collection, analysis and interpretation of results, and manuscript preparation.

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