

**РАСТВОРИМОСТЬ И ФИЗИКО-ХИМИЧЕСКИЕ СВОЙСТВА НАСЫЩЕННЫХ РАСТВОРОВ  
В СИСТЕМЕ МОЛИБДАТ ЦЕЗИЯ–ИЗОБУТИЛОВЫЙ СПИРТ–ВОДА ПРИ 25 °С**

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*Впервые изучена растворимость в системе  $Cs_2MoO_4$ –изо- $C_4H_9OH$ – $H_2O$  при 25 °С.*

*Показано, что в данной системе не происходит образования новых соединений. Изобутиловый спирт обладает ограниченной растворимостью в воде. С увеличением количества молибдата цезия в системе происходит значительное уменьшение взаимной растворимости изобутилового спирта и воды: в нижнем водном слое растворимость  $C_4H_9OH$  в воде от 8,13 масс.% в чистой воде до 0,04 масс.%  $C_4H_9OH$  в насыщенном 79,91-%-м водном растворе  $Cs_2MoO_4$ , а в верхнем спиртовом слое растворимость воды в изобутиловом спирте уменьшается от 16,90 до 0,51 масс.%  $H_2O$  в присутствии 0,0069 масс.%  $Cs_2MoO_4$ . В нонвариантном состоянии насыщенные жидкие фазы ( $L_1$  и  $L_2$ ), находящиеся в равновесии с твердой фазой  $Cs_2MoO_4$ , имеют состав (масс. %):  $L_1$  – 79,58  $Cs_2MoO_4$ , 0,04  $C_4H_9OH$  и 20,38  $H_2O$ ;  $L_2$  – 0,069  $Cs_2MoO_4$ , 99,48  $C_4H_9OH$  и 0,51  $H_2O$ . Во всем концентрационном интервале наблюдается расслоение. В данной системе нет ни верхней, ни нижней критической точки растворимости и бинодальной поверхности растворимости тройной системы. Проявляются только два разомкнутых участка этой поверхности, которые на проекциях пространственной диаграммы отображаются как две изолированные кривые. Определены плотность, показатель преломления, динамическая вязкость насыщенных растворов системы и рассчитаны изотермы удельного и мольного объема. Результаты исследования физико-химических свойств насыщенных растворов системы показывают закономерное изменение всех свойств сопряженных жидких фаз в соответствии с изменением их состава и характера взаимодействия компонентов в системе.*

**Ключевые слова:** растворимость, молибдат цезия, одно-спиртовые смеси, изобутиловый спирт, фазовая диаграмма растворимости, физико-химические свойства растворов

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## SOLUBILITY AND PHYSICOCHEMICAL PROPERTIES OF SATURATED SOLUTIONS IN CESIUM MOLYBDATE – ISOBUTYL ALCOHOL – WATER SYSTEM AT 25 °C

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*The solubility in the  $Cs_2MoO_4$  – iso- $C_4H_9OH$  –  $H_2O$  system at 25 °C was studied for the first time. It is shown that in this system no formation of new compounds occurs. Isobutyl alcohol has limited solubility in water. This is confirmed by the fact that when cesium molybdate is added to the system, the mutual solubility of isobutyl alcohol and water decreases significantly. In the lower aqueous layer the solubility of  $C_4H_9OH$  in water is from 8.13 wt.%, in pure water to 0.04 wt.% of  $C_4H_9OH$  in a saturated 79.91% aqueous solution of  $Cs_2MoO_4$ ; in the upper alcohol layer the solubility of water in isobutyl alcohol decreases from 16.90 to 0.51 wt.% of  $H_2O$  in the presence of 0.0069 wt.% of  $Cs_2MoO_4$ . In the nonvariant state the saturated liquid phases (L1 and L2), which are in equilibrium with the solid phase of  $Cs_2MoO_4$ , have the composition (wt.%): L1 – 79.58  $Cs_2MoO_4$ , 0.04  $C_4H_9OH$  and 20.38  $H_2O$ ; L2 – 0.069  $Cs_2MoO_4$ , 99.48  $C_4H_9OH$  and 0.51  $H_2O$ . Stratification is observed throughout the concentration range. In this system there is neither upper nor lower critical point of solubility and there is no binodal solubility surface of the ternary system. Only two open sections of this surface appear which are displayed as two isolated curves on the projections of the spatial diagram. The density, refractive index, and dynamic viscosity of saturated solutions of the system were determined, and the isotherms of specific and molar volumes were calculated. The results of the study of physico-chemical properties of this system of saturated solutions show a regular change of all properties of conjugate liquid phases in accordance with the changes in their composition and character of interaction of components in the system.*

**Key words:** solubility, cesium molybdate, water-alcohol mixtures, isobutyl alcohol, phase solubility diagram, solutions physicochemical properties

### INTRODUCTION

The interest of scientists to the study of phase equilibria of salt water-organic systems is due to several reasons. On the one hand, the study of various physico-chemical properties of such systems solutions is an important and urgent task of the physical chemistry of solutions [1-3], the ultimate goal is the creation of empirical and semi empirical models to describe the thermodynamic properties of this type of systems [4-9]. On the other hand, the data obtained in the study of salt water-organic systems can be widely used in chemical technology for separation and puri-

fication of substances (salt distillation and extraction) [10-13]. In addition, experimental construction of phase diagrams of different saline water-organic systems solubility will allow to find regularities between the topological features of the graphs of solubility and change in the nature of solvation-hydration processes in saturated solutions. For several years, we have conducted systematic studies of the solubility and a number of physicochemical properties of saturated solutions in the systems based on molybdates (tungstates) of alkali metals and ammonium and mixed aqueous-organic solvents [14-16]. In the literature,

there are researches of the  $\text{Cs}_2\text{MoO}_4$  solubility in ethanol solution [16], other information about solubility of this salt in organic and mixed aqueous-organic solvents was not found.

Isobutyl alcohol and water have a limited solubility in each other: the solubility of isobutanol in pure water is 8.13 wt. %, and water – in alcohol-16.9 wt.%. The results obtained during the study of the addition effect of cesium molybdate on the solubility and properties of saturated solutions in the  $\text{Cs}_2\text{MoO}_4$ –iso- $\text{C}_4\text{H}_9\text{OH}$ – $\text{H}_2\text{O}$  system at 25 °C are present in this paper.

## EXPERIMENTAL PART

For solubility studies have taken twice recrystallized cesium molybdate  $\text{Cs}_2\text{MoO}_4$  (qualifications «chemically pure» containing 99.90 mas. %), bi-distillate water and isobutyl alcohol («pure for analysis»), dehydrated by heating in the presence of  $\text{CuSO}_4$  and obtained as distillate. Solubility in the system was studied by the isothermal saturation method in a typical installation, [14], used in the study of solubility in water-salt systems that uploaded pre-calculated amount of saturated (79.88%) aqueous solution of  $\text{Cs}_2\text{MoO}_4$ , isobutyl alcohol and water to achieve total volume of 200 ml mixture and intensively stirred. Full equilibrium in the system was installed within 2-3 days, depending on the composition of the load. To determine the composition of the liquid phase solutions of sample were analyzed directly from the vessel. The dry residue of the solution was obtained by evaporation of the solution at the beginning to wet salts at 90-100 °C and then calcinate at 130-150 °C till weight constant. In some cases, to control determined content of  $\text{MoO}_4^{2-}$  by gravimetric method in the form of  $\text{PbMoO}_4$  [17]. Thus, from the obtained

filtrate excess  $\text{Pb}^{2+}$  like deposition  $\text{PbSO}_4$  by sulfuric acid and was removed after evaporation and calcination to constant weight was carried out by quantitative determination of cesium ions in the form of sulphate. The content of cesium was controlled at some points by the weight tetraphenylborate method, as described in [18]. In samples with a low content of molybdenum the concentration was determined by the photometric method in the form of orange-red thiocyanat complex of molybdenum (V). Then stratification of the saturated solution in the samples selected for analysis of the lower aqueous layer, along with the concentration of  $\text{Cs}_2\text{MoO}_4$ , the content of the isobutyl alcohol was determined by method of acetylation, and in samples of the upper alcohol layer water content was determined by titration according to the method of Fischer and  $\text{Cs}_2\text{MoO}_4$  in the dry residue. Physicochemical properties of saturated solutions in the system were determined experimentally as described in [14-16].

## RESULTS AND DISCUSSION

Results of solubility studies in the system on Table 1 and Figure show that in the  $\text{Cs}_2\text{MoO}_4$ –iso- $\text{C}_4\text{H}_9\text{OH}$ – $\text{H}_2\text{O}$  system at 25 °C no formation of new chemical compounds occurs. With increasing number of  $\text{Cs}_2\text{MoO}_4$  in the system there is a significant reduction in the mutual solubility of isobutyl alcohol and water in the lower water layer, the solubility of iso- $\text{C}_4\text{H}_9\text{OH}$  in water from 8.13 wt.% in pure water up to 0.04 wt.%  $\text{C}_4\text{H}_9\text{OH}$  in the rich 79.91% aqueous solution of  $\text{Cs}_2\text{MoO}_4$ , and in the upper alcohol layer water solubility in isobutyl alcohol decreases from 16.90 to 0.51 mas. %  $\text{H}_2\text{O}$  in the presence of 0.0069 mas. %  $\text{Cs}_2\text{MoO}_4$ .

Table I

**Солubility in the  $\text{Cs}_2\text{MoO}_4$ -iso- $\text{C}_4\text{H}_9\text{OH}$ - $\text{H}_2\text{O}$  system at 25 °C**  
**Таблица 1. Растворимость в системе  $\text{Cs}_2\text{MoO}_4$ -изо- $\text{C}_4\text{H}_9\text{OH}$ - $\text{H}_2\text{O}$  при 25 °C**

Point number	The composition of the first liquid phase $L_1$ , weight %			The composition of the second liquid phase $L_2$ , weight %			Equilibrium phases
	$\text{Cs}_2\text{MoO}_4$	$\text{C}_2\text{H}_9\text{OH}$	$\text{H}_2\text{O}$	$\text{Cs}_2\text{MoO}_4$	$\text{C}_2\text{H}_9\text{OH}$	$\text{H}_2\text{O}$	
1	79.83	–	20.17	–	–	–	$L_1 + \text{Cs}_2\text{MoO}_4$
2	79.58	0.04	20.32	0.0069	99.48	0.51	$L_1 + L_2 + \text{Cs}_2\text{MoO}_4$
3	74.01	0.08	25.91	0.0063	98.14	1.85	$L_1 + L_2$
4	59.59	0.14	40.27	0.0061	95.00	4.99	the same
5	45.30	0.21	54.49	0.0075	91.23	8.74	the same
6	32.75	0.58	66.67	0.0081	88.83	11.16	the same
7	22.84	1.28	75.88	0.0043	88.17	11.83	the same
8	19.83	1.73	78.44	0.0034	87.25	12.75	the same
9	11.11	2.74	86.15	0.0057	85.47	14.52	the same
10	4.41	5.25	90.34	0.0038	84.05	15.95	the same
11	–	8.13	91.87	–	83.10	16.90	the same
12	–	–	–	0.0049	99.85	0.15	$L_2 + \text{Cs}_2\text{MoO}_4$
13	–	–	–	–	99.62	0.38	$L_2$

In nonvariant state saturated liquid phases ( $L_1$  and  $L_2$ ) in equilibrium with the solid phase  $\text{Cs}_2\text{MoO}_4$  contain (mas. %): the first liquid phase (lower water layer)  $L_1$  – 79/58  $\text{Cs}_2\text{MoO}_4$ , 0.04  $\text{C}_4\text{H}_9\text{OH}$  and  $\text{H}_2\text{O}$  20.38;  $L_2$  –  $\text{Cs}_2\text{MoO}_4$  to 0.069, 99.48  $\text{C}_4\text{H}_9\text{OH}$  and  $\text{H}_2\text{O}$  is 0.51. We can see it on the triangular diagram. The following fields are in the system: three-phase field  $E''\text{Cs}_2\text{MoO}_4$ , where the equilibrium variables are the number of  $L_1$ ,  $L_2$  and solid phase (crystals  $\text{Cs}_2\text{MoO}_4$ ); two-phase  $E'\text{AE}''$ , where in equilibrium there are conjugated liquid phases ( $L_1$  and  $L_2$ ), cesium molybdate unsaturated, but saturated with one of the other two components (water and isobutyl alcohol); a

minor two-phase field (almost merges with the axis of the  $\text{Cs}_2\text{MoO}_4 - \text{H}_2\text{O}$ ), where in equilibrium  $L_1$  and  $\text{Cs}_2\text{MoO}_4$ ; single-phase field  $E'\text{H}_2\text{O}$  ( $L_1$ ) of unsaturated aqueous solutions, and  $L_2$  (unsaturated alcohol solutions). Thus, this system differs from previously studied [6] by the fact that it has neither upper nor lower critical point of solubility, and on binoidal surface of solubility only two far open sections of that surface appear. It is noticed that the solutions were saturated with cesium molybdate and isobutyl alcohol, have (a lower layer solution) the characteristics of colloidal state.

**Table 2**

**Physico-chemical properties of the first liquid phase  $L_1$  (lower water layer) in the  $\text{Cs}_2\text{MoO}_4\text{-iso-C}_4\text{H}_9\text{OH}\text{-H}_2\text{O}$  system at 25 °C**

**Таблица 2. Физико-химические свойства первой жидкой фазы  $L_1$  (нижнего водного слоя) в системе  $\text{Cs}_2\text{MoO}_4\text{-изо-}\text{C}_4\text{H}_9\text{OH}\text{-H}_2\text{O}$  при 25 °C**

Point number	The composition of the first liquid phase $L_1$ mol, %			Properties		
	$\text{Cs}_2\text{MoO}_4$	iso- $\text{C}_4\text{H}_9\text{OH}$	$\text{H}_2\text{O}$	$\rho, \cdot 10^{-3}, \text{kg/m}^3$	$n_D$	$\eta, \cdot 10^{-3}, \text{Pa}\cdot\text{s}$
1	14.39	–	85.61	2.7424	1.5165	6.885
2	14.21	0.041	85.75	2.7217	1.5165	6.847
3	10.78	0.067	59.16	2.4309	1.4839	5.824
4	5.88	0.079	94.03	1.8987	1.4280	3.998
5	3.39	0.090	96.51	1.5601	1.3924	2.837
6	2.03	0.207	97.76	1.3496	1.3707	2.127
7	1.25	0.403	98.34	1.2166	1.3581	1.702
8	1.05	0.528	98.42	1.1810	1.3552	1.598
9	0.54	0.763	98.70	1.0889	1.3471	1.318
10	0.20	1.389	98.41	1.0247	1.3441	1.185
11	–	2.105	97.89	0.9845	1.3443	1.143

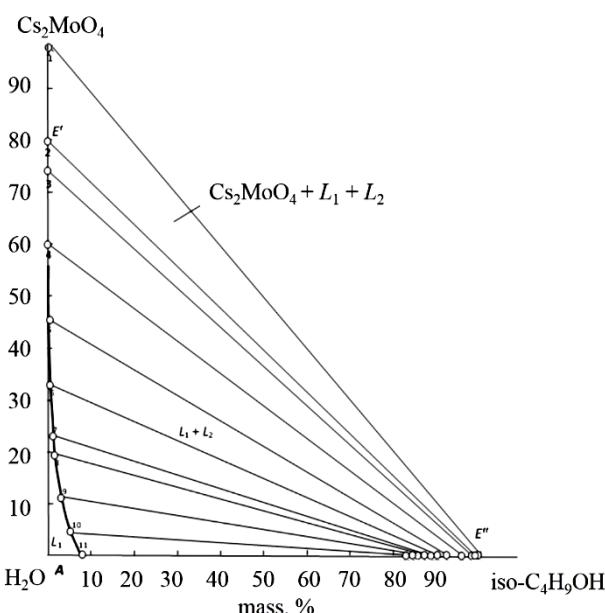


Fig. The solubility isotherm (25 °C) of the system  $\text{Cs}_2\text{MoO}_4\text{-iso-C}_4\text{H}_9\text{OH}\text{-H}_2\text{O}$

Рис. Изотерма (25 °C) растворимости системы  $\text{Cs}_2\text{MoO}_4\text{-изо-}\text{C}_4\text{H}_9\text{OH}\text{-H}_2\text{O}$

The results obtained for the solubility in binary systems  $\text{Cs}_2\text{MoO}_4\text{-H}_2\text{O}$   $\text{C}_4\text{H}_9\text{OH}$  and  $\text{H}_2\text{O}$  (table 1) close to literature data. We have not found literature data on the solubility of  $\text{Cs}_2\text{MoO}_4$ , iso- $\text{C}_4\text{H}_9\text{OH}$ .

Experimental determination and calculations of the properties of saturated solutions were carried out as described in [14-16]. We've experimentally determined density ( $\rho$ ), refractive index ( $n_D$ ) and dynamic viscosity ( $\eta$ ) (Table 2, 3) and on the basis of the obtained data we've calculated the specific volume ( $v_s$ ), molar volume ( $V_s$ ) and apparent molar volume of the amount of non-aqueous components ( $V_{\Sigma i}$ ) of saturated solution. In addition, calculations of the volumetric properties and refractive index were carried out according to the formulas of displacement, on the basis of perceived values of these properties of cesium molybdate and isobutyl alcohol in their saturated aqueous solutions and the properties of pure water. To determine the degree of deviation of experimental data from the additivity rule according to the offset we calculated additive values of all these properties of liquid phases [14] in the assumption that be-

tween them there is a chemical interaction.

It is established that the above mentioned properties of conjugate liquid phases ( $L_1$  and  $L_2$ ) change is quite natural in accordance with the changes in their composition and character of interaction of components in the system. The results obtained in the study of the first liquid phase  $L_1$  (lower water layer) of the system are slightly different from previously obtained data on the properties of the solutions of the

original dual system  $\text{Cs}_2\text{MoO}_4 - \text{H}_2\text{O}$  [16]. Noteworthy that the experimental data by the following properties ( $\rho$ ,  $v$ ,  $n_D$  and  $V$ ) of the first liquid phase deviate a little from their additive values calculated according to the rule of the offset of the apparent values of the properties of the components in the original binary (alcohol – water, cesium molybdate – water). The maximum deviation  $\rho_s$ ,  $v_s$ ,  $n_{D_s}$  and  $V_s$  from additivity does not exceed  $\pm 0.34$  rel. %.

**Table 3** Physico-chemical properties of the second liquid phase  $L_2$  (upper isobutyl layer) in the  $\text{Cs}_2\text{MoO}_4$ -iso- $\text{C}_4\text{H}_9\text{OH}-\text{H}_2\text{O}$  system at 25 °C

**Таблица 3. Физико-химические свойства второй жидкой фазы  $L_2$  (верхнего изобутилового слоя) в системе  $\text{Cs}_2\text{MoO}_4$  – изо- $\text{C}_4\text{H}_9\text{OH} - \text{H}_2\text{O}$  при 25 °C**

Point number	The composition of the second liquid phase $L_2$ , mol. %			Properties		
	$\text{Cs}_2\text{MoO}_4$	iso- $\text{C}_4\text{H}_9\text{OH}$	$\text{H}_2\text{O}$	$\rho \cdot 10^{-3}$ , kg/m <sup>3</sup>	$n_D$	$\eta \cdot 10^{-3}$ , Pa·sec
1	–	–	–	–	–	–
2	0.0012	97.93	2.07	0.8043	1.3940	2.808
3	0.0010	92.80	7.19	0.8074	1.3934	2.740
4	0.00095	82.23	17.77	0.8128	1.3927	2.382
5	0.0010	71.73	28.27	0.8181	1.3921	2.660
6	0.00104	65.92	34.08	0.8226	1.3915	2.644
7	0.00055	64.43	35.57	0.8231	1.3914	2.635
8	0.00042	62.45	37.55	0.8245	1.3912	2.625
9	0.00068	58.86	41.14	0.8233	1.3910	2.617
10	0.00044	56.15	43.84	0.8308	1.3907	2.612
11	–	54.44	45.56	0.8317	1.3906	2.615
12	0.000849	99.38	0.61	0.8031	1.3938	2.815
13	–	98.45	1.54	0.8035	1.3938	2.816

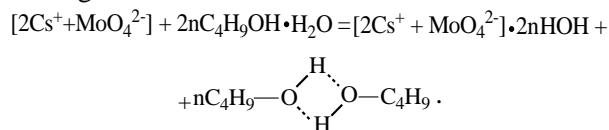
Depending on the composition of the solutions, the viscosity is most sharply changed, which is the most structurally sensitive property. In the entire concentration range, the degree of deviation of this quantity from additivity is more significant than that of all other properties, and is maximally  $\Delta\eta = -58.86$  rel. %.

The results of the properties study of the second liquid phase  $L_2$  (upper alcohol layer) show that the presence of trace amounts of  $\text{Cs}_2\text{MoO}_4$  does not significantly affect the magnitude and nature of the changes in the properties of this phase. The properties of the  $L_2$  phase deviate from additivity so little that these deviations are very close to the errors of the experimental determination of these properties.

#### CONCLUSIONS

Summarizing the obtained data on the solubility and physicochemical properties of saturated solutions of the  $\text{Cs}_2\text{MoO}_4$  – iso- $\text{C}_4\text{H}_9\text{OH} - \text{H}_2\text{O}$  system at 25 °C we can say that the main factor determining the nature of the change in solubility and, therefore, the properties of saturated solutions in the system, is the hydrophobization process of isobutyl alcohol in the presence of a hydrophilic electrolyte –  $\text{Cs}_2\text{MoO}_4$ .

Based on the analysis of some literature data [19-21], this process can be represented as a simplified diagram:



When this occurs, the rupture of weak hydrogen bonds between dissimilar molecules of isobutyl alcohol and water, the hydrophilic hydration of electrolyte ions ( $\text{Cs}^+$  and  $\text{MoO}_4^{2-}$ ), increases self-association of hydrophobic isobutyl alcohol molecules and their separation from the rest of the hydrophilic part of the system.

This leads to a significant reduction in the mutual solubility of alcohol and water, that is, a deeper splitting of the system into two liquid phases of very different composition and physicochemical properties of their saturated solutions.

All the studied and calculated properties of solutions established by the solubility method changed in full accordance with the variation of the composition of solutions and nature of components interaction in them.

## ЛИТЕРАТУРА

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