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STUDY OF PROCESSE OF OBTAINING MONOPOTASSIUM PHOSPHATE BASED ON MONOSODIUM PHOSPHATE AND POTASSIUM CHLORIDE¹M.A. Shaymardanova, ²Kh.Ch. Mirzakulov, ²G. Melikulova, ¹S.Z. Khodjamkulov, ¹A.K. Nomozov, ¹Kh.S. Shaymardanova¹Termiz Institute of Engineering and Technology,
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Abstract: Results of studies of the process of conversion of the flotation potassium chloride of the Tyubegatan deposit with solutions of sodium dihydrogen phosphate obtained on the basis of purified extraction phosphoric acid from phosphorites of the Central Kyzylkum to potassium dihydrogen phosphate are presented. The effect of the ratio of the components, S:L on the formation of potassium dihydrogen phosphate at temperatures of 25°C and 100°C was studied. Optimal technological parameters for isolation of potassium dihydrogen phosphate were determined. Rheological properties of solutions formed in the process of obtaining potassium dihydrogen phosphate were identified. The obtained results were confirmed by X-ray phase and IR-spectroscopic methods of analysis. Elemental composition of potassium dihydrogen phosphate was established by electron microscopy.

Keywords: monopotassium phosphate, flotation potassium chloride, solubility diagram, conversion, sodium and potassium dihydrogen phosphates.

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1. Introduction

Climate changes and shortages of mineral fertilizer supplies for the future of agriculture are raising fears of global food crisis. There is a need for proactive innovations in fertilizer research to maintain soil fertility. Using granules with special properties, particularly organic-mineral fertilizers with the nanoporous structure of the shell (coating), is a promising solution to the possibility of controlled dissolution in the soil [1]. Worldwide, it is estimated that over 820 million people are hungry or undernourished [2]. Furthermore, one of today's most serious challenges is effectively treating hunger, which has a variety of causes and manifestations, such as growing food demands, changing diets, and harsh weather conditions [3]. Moreover, in the not-too-distant future, the global food system is projected to

encounter growing demand. Food insecurity exists in many regions of the world, regardless of the fact that global food resources are now sufficient to supply the world's population's calorie demands [4-6]. As a result, the output of various critical products has significantly increased. To maintain food security, the annual output of cereals, especially wheat, must be increased by over one billion tons [7].

Phosphorus (P) is one of the most important nutrients for all living organisms [8]. The controlled release fertilizers, such as phosphate glass fertilizers (GF), constitute the future of the chemical fertilizers industry [9].

Based on the results of industrial monitoring of the extraction of phosphoric acid and mineral fertilizers, the operating range of concentrations of fluorine ions (0.6–4.0 g/dm³)

and phosphorus in terms of P_2O_5 (10–15 g/dm³) in industrial effluents was obtained [10].

The effect of impurities on the thermal decomposition kinetics of mineral fertilizers based on $(NH_4)_2HPO_4$ in self-generated atmosphere was studied by means of thermogravimetry and differential thermal analysis. Results are presented of isothermal measurements made in the temperature range 100–110°C [11].

Alimov et al. studied ammophosphate treatment technology with poor in phosphorus highly carbonized ordinary phosphorite flour,

washed dried concentrate, dust-like fraction, and mineralized phosphorite mass of Central Kyzylkum. As a result, it became possible to prepare concentrated phosphorus fertilizers of prolonged effect with the high content of assimilable form of P_2O_5 [12].

Results of research into physicochemical properties of carbamide containing NPK fertilizers conditioned by magnesium salts are presented based on the example of the 16-16-16 brand. It showed that the presence of magnesium salts in granular product reduces hygroscopicity and caking [13].

2. Experimental part

2.1. Analysis of the NaH_2PO_4 - KH_2PO_4 - $NaCl$ - KCl - H_2O system in relation to the production of monopotassium phosphate.

To select the optimal conditions for conversion of monosodium phosphate with potassium chloride and the preparation of potassium dihydrogen phosphate, comparative

studies were carried out on the solubility in the four-component water-salt system K^+ , $Na^+//Cl^-$, $H_2PO_4^-$ - H_2O , obtained at 25 and 100°C. Data on the solubility diagram of a four-component system in the form of a trapezoid, and in the form of a square at temperatures of 25 and 100 °C are shown in Figures 1-3.

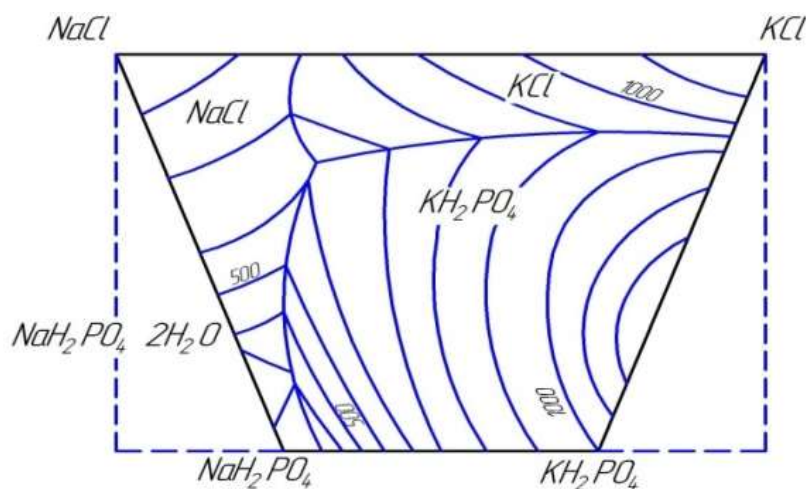


Fig. 1. Solubility diagram of the system K^+ , $Na^+//Cl^-$, $H_2PO_4^-$ - H_2O at 25°C

Four fields of crystallization $NaCl$, KCl , NaH_2PO_4 and KH_2PO_4 are distinguished on the solubility diagram. E_1 and E_2 are eutonic points at temperatures of 25 and 100°C, where the solution is in equilibrium with three salts. These points are located outside the composition triangle, which is in the solid phase of salts, and are incongruently saturated.

The field of crystallization of KH_2PO_4 on the solubility diagram of the system K^+ , $Na^+//Cl^-$, $H_2PO_4^-$ - H_2O at 25°C occupies a greater part of the square area than at 100°C. When

considering the isothermal solubility diagram, it can be seen that when the temperature rises, the crystallization fields of KH_2PO_4 sharply decrease, therefore, their solubility grows significantly. The saturation region of KH_2PO_4 at a temperature of 100°C is several times smaller than at a temperature of 25°C.

The crystallization field of $NaCl$ at a temperature of 100°C significantly increases compared to the crystallization field at 25°C due to a decrease in the KH_2PO_4 field.

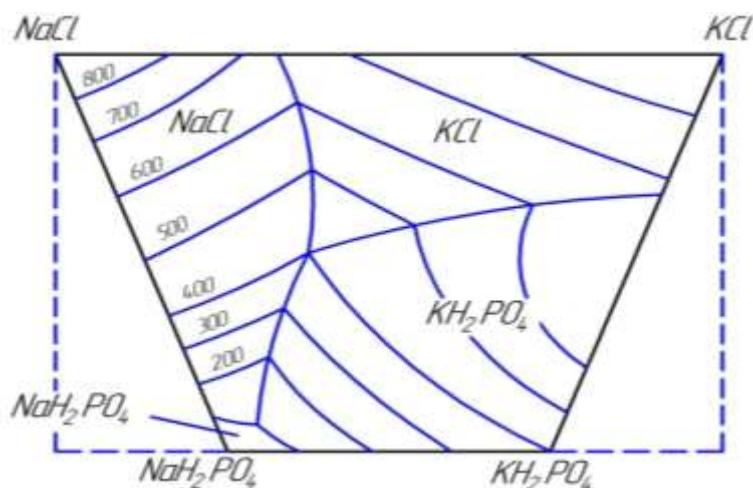


Fig. 2. Solubility diagram of the system K^+ , $Na^+//Cl^-$, $H_2PO_4^-$ - H_2O at $100^\circ C$.

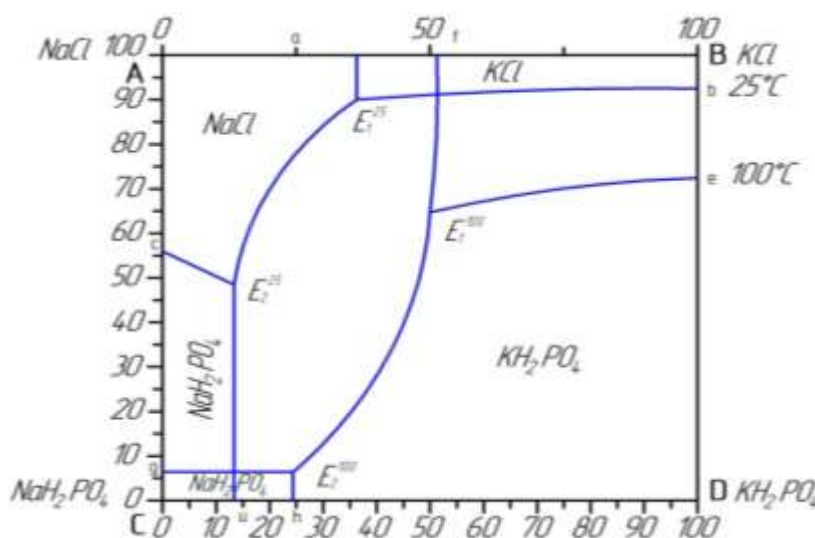


Fig. 3. Solubility diagrams of the K^+ , Na^+ system // Cl^- , $H_2PO_4^-$ - H_2O at 25 and $100^\circ C$.

Consequently, the solubility of KH_2PO_4 increases several times, and such a solution, when cooled, is able to precipitate this salt.

The KH_2PO_4 - NaH_2PO_4 - $NaCl$ - KCl - H_2O system was used to analyze the phase composition of industrial processes.

Before starting the analysis of the cyclic processes for the production of potassium dihydrogen phosphate, we carried out preliminary studies of the production of potassium dihydrogen phosphate at various temperatures. To our thinking, results of this analysis show that the most favorable conditions arise during the process of obtaining potassium dihydrogen phosphate with the release of sodium chloride at $100^\circ C$ and crystallization of potassium dihydrogen phosphate with suspension cooling to $25^\circ C$.

Based on the phase diagrams of the solubility of the mutual salt system K^+ , $Na^+ // Cl^-$, $H_2PO_4^-$ - H_2O , comparing a number of technological cycles, a scheme for the cycle Q_3 - M_3 - P_3 - S_3 - Q_3 was chosen (Fig. 4).

In the first variant, sodium dihydrogen phosphate, which is an intermediate product obtained by neutralizing phosphoric acid with sodium carbonate, and potassium chloride, was used as the initial component. The technological process proceeds as follows (Fig. 4): sodium dihydrogen phosphate is added to the circulation solution at point P_0 , potassium chloride is further added at point S_0 , then the solution is evaporated to the saturation point Q_0 , which lies on a line drawn from point D to point M_0 , which refers to the saturation point of sodium chloride at $100^\circ C$.

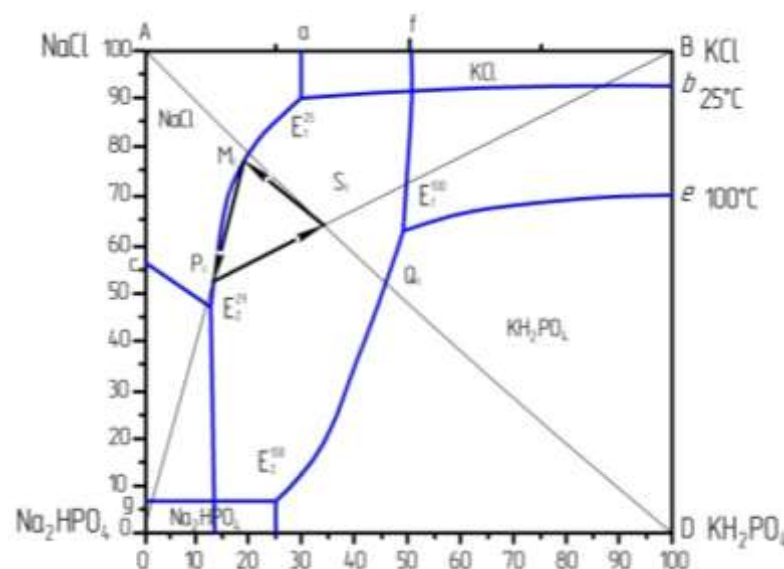


Fig. 4. Diagram of obtaining potassium dihydrogen phosphate.

After separating the precipitate, the mother liquor is cooled to a temperature of 25°C to the point M_1 . When the temperature is reached, the product is separated by filtration, and then dried at a temperature of 115-120°C, the remaining mother liquor is sent to the cycle. Thus, the process is closed.

In this case, the crystallization of KH_2PO_4 will take place from a solution with a lower content of chlorides, which will allow the production of KH_2PO_4 with lower NaCl content and relatively.

2.2. Study of the process of conversion of monosodium phosphate solutions by potassium chloride

The authors [14-16] studied the solubility diagrams for the theoretical justification for the production of potassium dihydrogen phosphate suitable for food production by the conversion method from sodium dihydrophosphate obtained by neutralizing thermal phosphoric acid with soda ash and chemically pure potassium chloride.

To select the optimal conditions for the conversion of a solution of monosodium phosphate obtained on the basis of EPA from phosphorites with flotation potassium chloride to obtain potassium dihydrophosphate, previously studied systems were analyzed and comparative studies were carried out on the solubility in the four-component water-salt system $K^+, Na^+ // Cl^-, H_2PO_4^- - H_2O$ at 25 and

100°C, in relation to the proposed compositions of the feedstock.

Analysis of the $NaH_2PO_4-KH_2PO_4-NaCl-KCl-H_2O$ system showed the possibility of obtaining monopotassium phosphate by converting a solution of monosodium phosphate with flotation potassium chloride with preliminary isolation of sodium chloride. Therefore, further studies were aimed at obtaining monopotassium phosphate by converting a solution of monosodium phosphate with flotation potassium chloride [17].

Purified solutions of monosodium phosphate and flotation potassium chloride produced by UE "Dekhkanabad Potash Plant" were used as initial components.

The technological process proceeds as follows (Fig. 1): sodium dihydrogen phosphate is added to the circulation solution at point P_0 , then potassium chloride is added at point S_0 , and the solution is evaporated to the saturation point Q_0 , which lies on the line drawn from point D to point M_0 , which refers to the saturation point of sodium chloride at 100°C.

After separating the precipitate, the mother liquor is cooled to a temperature of 25°C to point M_1 , at the temperature reached, the product is separated by filtration, then dried at a temperature of 115-120°C, the remaining mother liquor is sent to the cycle. Thus, the process is closed.

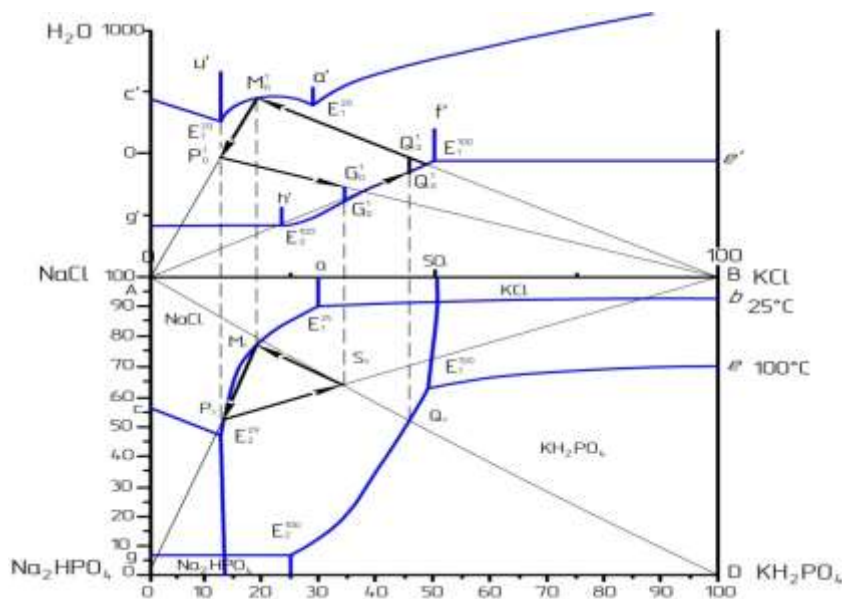


Fig. 5. Diagram $K^+, Na^+ // Cl^-, H_2PO_4^- - H_2O$ in relation to the process of obtaining potassium dihydrogen phosphate.

Table 1 shows the characteristics of sodium dihydrogen phosphate and potassium suspensions in the preparation of potassium chloride. [18].

Table 1. Characteristics of the suspension in the preparation of potassium dihydrogen phosphate from a solution of sodium dihydrogen phosphate and potassium chloride

№ Bap.	Q ₀				M ₀				P ₀	S ₀	
	Suspension, g.	Liquid phase.	Solid phase	Liquid: Solid	Suspension, g.	Liquid phase.	Solid phase	Liquid: Solid	Suspension, g.	Suspension, g.	Liquid phase.
1	189.35	170.73	18.62	9.17:1	183.33	142.49	40.84	3.49:1	323.68	300.21	189.35
2	189.74	169.93	19.71	8.62:1	180.76	137.12	43.64	3.15:1	330.38	305.35	189.74
3	191.31	170.84	20.47	8.35:1	181.67	136.71	44.96	3.04:1	336.01	310.20	191.31
4	190.63	170.15	20.48	8.31:1	184.58	139.62	44.95	3.11:1	338.93	313.11	190.63
5	189.35	170.74	18.61	9.17:1	183.96	143.09	40.87	3.50:1	324.28	300.81	189.35
6	182.77	167.26	15.51	10.79:1	196.09	162.03	34.06	4.76:1	313.0	293.45	182.77

Table 2 shows the results of experiments in the preparation of potassium dihydrogen phosphate according to six variants of the cyclic method.

Table 2. Chemical composition of precipitates and mother liquors formed during the conversion of NaH_2PO_4 solution with KCl

№	Precipitation composition of KH_2PO_4 , %				Precipitation composition of NaCl, %				Composition of mother liquor (M ₀), %			
	P ₂ O ₅	K ₂ O	Na ₂ O	Cl	P ₂ O ₅	K ₂ O	Na ₂ O	Cl	P ₂ O ₅	K ₂ O	Na ₂ O	Cl
1	42.05	29.15	0.89	0.99	0.64	0.95	52.24	43.71	4.18	6.04	10.88	6.13
2	43.76	28.87	0.12	0.81	0.73	0.14	52.75	44.42	4.93	10.44	11.41	6.85
3	48.85	33.02	1.66	0.62	1.99	1.70	60.23	56.83	6.42	4.41	12.69	7.99
4	43.95	28.30	8.99	3.49	3.90	3.04	52.74	44.61	5.77	5.53	13.08	8.70
5	39.52	25.29	10.62	4.54	6.35	3.11	57.82	42.26	11.15	4.08	14.22	7.60
6	43.72	22.40	8.66	3.41	6.93	2.71	41.87	40.34	17.17	3.10	11.59	7.20

After several cycles, potassium dihydrogen phosphate was isolated containing 48.85% P₂O₅, 33.02% K₂O, 1.66% Na₂O, and 0.6% Cl⁻. The NaCl precipitate obtained by evaporating water from the solution contains 1.99% P₂O₅, 1.70% K₂O, 60.23% Na₂O and 56.83% Cl⁻. The mother liquor, which contains 6.42% P₂O₅, 4.41% K₂O, 12.69% Na₂O, is returned to the cycle. Next, the precipitate of monopotassium phosphate was washed with a saturated solution of monopotassium phosphate and dried at 100-110°C.

The analyzes and calculations performed using the solubility diagrams in the K⁺,Na⁺//Cl⁻, H₂PO₄⁻-H₂O quaternary system and the obtained experimental data showed the fundamental possibility of obtaining potassium dihydrogen phosphate by the conversion method from solutions of monosodium phosphate obtained on the basis of EPA from phosphorites of the flotation potassium chloride.

2.3. Influence of technological parameters on the process of crystallization of monopotassium phosphate

The influence of technological parameters such as solution concentration, temperature and solution cooling rate on the process of crystallization of monopotassium phosphate from a conversion solution obtained by the conversion method from solutions of monosodium phosphate obtained on the basis of EPA from phosphorites and flotation potassium chloride was studied. The results obtained are shown in Table 3.

The slow cooling rate contributes to the precipitation of fewer impurities in the sediment. With a decrease in the cooling rate, the amount of Na₂O and Cl⁻ impurities in the product decreases by about a factor of two. Rise in the concentration of the solution over 30% leads to increase in the content of impurities in the finished product.

Table 3. Degree of transition of components of the solution into the product and the chemical composition of the mother liquor after crystallization of potassium dihydrogen phosphate

Temperature, °C	Cooling rate, °C/hour	Degree of transition of components into product, %				Chemical composition of mother liquor, mass. %			
		K ₂ O	P ₂ O ₅	Na ₂ O	Cl ⁻	K ₂ O	P ₂ O ₅	Na ₂ O	Cl ⁻
Concentration of monopotassium phosphate 27%									
31,0	10.0	68.48	69.72	4.28	2.59	3.98	5.72	11.42	7.20
	7.0	68.45	68.68	4.26	2.57	3.97	5.70	11.40	7.19
	5.0	68.43	68.66	4.25	2.56	3.97	5.70	11.40	7.18
25,0	10.0	79.62	81.06	4.60	2.78	3.93	5.72	10.15	7.11
	7.0	79.58	81.01	4.57	2.75	3.92	5.72	10.17	7.12
	5.0	79,56	80.99	4.55	2.74	3.92	5.72	10.18	7.13
Concentration of monopotassium phosphate 24%									
31,0	10.0	61.14	62.25	3.82	2.31	4.47	6.50	12.83	8.08
	7.0	61.12	61.23	3.81	2.29	4.46	6.49	12.82	8.07
	5.0	61.11	61.21	3.79	2.28	4.46	6.49	12.82	8.07
25,0	10.0	71.09	72.38	4.11	2.48	4.41	6.42	12.69	7.99
	7.0	71.06	72.34	4.07	2.46	4.41	6.42	12.71	8.01
	5,0	71.04	72.32	4.05	2.44	4.41	6.42	12.72	8.02

The mother liquors after crystallization of potassium dihydrogen phosphate are returned for reuse at the initial stage in the process of conversion of potassium chloride from sodium dihydrophosphate.

Thus, the optimal conditions for

obtaining potassium dihydrogen phosphate by converting a solution of monosodium phosphate with flotation potassium chloride with preliminary isolation of sodium chloride are temperature - no more than 25.0 ° C, solution concentration - at least 24-27%,

The chemical composition of the resulting product meets the requirements of GOST.

2.4. Studies of the process of washing monopotassium phosphate from impurities and chlorine

Table 4 shows the chemical and salt compositions of potassium dihydrogen phosphate obtained by washing once and twice with saturated solutions of monosodium and monopotassium phosphates.

Table 4. Chemical composition of potassium dihydrogen phosphate after washing

№	Ratio KH ₂ PO ₄ :Wash	Ionic composition, %				Salt composition, %				Output, %
		H ₂ PO ₄ ⁻	K ⁺	Na ⁺	Cl ⁻	KH ₂ PO ₄	NaH ₂ PO ₄	KCl	NaCl	
solution NaH₂PO₄										
1	1:1 _{NaH₂PO₄}	70.92	27.08	1.30	0.70	94.42	4.43	–	1.15	95.79
2	1:1 _{NaH₂PO₄}	70.82	27.62	0.92	0.64	96.33	2.62	–	1.05	95.46
3	1:1 _{NaH₂PO₄}	70.82	27.62	0.92	0.64	96.33	2.62	–	1.05	95.46
1	1:2 _{NaH₂PO₄}	70.87	26.97	1.40	0.76	94.07	4.67	–	1.26	87.30
2	1:2 _{NaH₂PO₄}	70.78	27.52	1.00	0.70	95.97	2.88	–	1.15	87.00
3	1:2 _{NaH₂PO₄}	70.78	27.52	1.00	0.70	95.97	2.88	–	1.15	87.00
solution KH₂PO₄										
1	1:1 _{KH₂PO₄}	70.47	28.08	0.66	0.79	97.91	0.79	–	1.30	100.0
2	1:1 _{KH₂PO₄}	70.43	28.32	0.49	0.76	98.75	–	–	1.25	100.0
3	1:1 _{KH₂PO₄}	70.43	28.32	0.49	0.76	98.75	–	–	1.25	100.0
1	1:2 _{KH₂PO₄}	70.41	28.14	0.63	0.82	98.13	0.52	–	1.35	98.00
2	1:2 _{KH₂PO₄}	70.39	28.31	0.51	0.79	98.70	–	–	1.30	97.88
3	1:2 _{KH₂PO₄}	70.39	28.31	0.51	0.79	98.70	–	–	1.30	97.88

When KH₂PO₄ crystals are washed with a saturated solution of NaH₂PO₄, a decrease in the yield of potassium phosphate for samples 1-3 is observed to 95.46% at a ratio of KH₂PO₄:solid = 1:1 and to 87.00% at a ratio of 1:2, which indicates the dissolution of KH₂PO₄ in a saturated solution of NaH₂PO₄ and the greater, the lower the ratio.

Washing samples 1-3 with saturated solutions of KH₂PO₄ practically does not lead to a decrease in the yield of KH₂PO₄ at a ratio of

1:1 and slightly decreases to 97.88-98.00% at a ratio of 1:2. It follows from the data obtained that washing is desirable to be carried out with a saturated solution of KH₂PO₄ at a ratio of 1:1.

2.5. Rheological properties of initial suspensions and mother liquors

The rheological properties of a monosodium phosphate solution were studied at a temperature of 20–80°C and pH = 3.8–4.5. The research results are shown in Table 5.

Table 5. Density and viscosity of solutions of monosodium phosphate obtained by neutralization of EPA from phosphorites of CK with soda ash

№	pH	Density, g/cm ³				Viscosity, mPa·c			
		20°C	40°C	60°C	80°C	20°C	40°C	60°C	80°C
Sodium dihydrogen phosphate stock solution									
1	3.8	1.240	1.234	1.230	1.228	3.41	2.17	1.42	1.10
2	4.1	1.242	1.236	1.232	1.230	3.50	2.23	1.49	1.14
3	4.5	1.246	1.240	1.236	1.234	3.96	2.55	1.68	1.29
One stripped off solution of sodium dihydrogen phosphate									
4	3.8	1.389	1.382	1.378	1.375	3.82	2.43	1.59	1.23
5	4.1	1.391	1.384	1.380	1.378	3.92	2.50	1.67	1.28

6	4.5	1.396	1.389	1.384	1.382	4.39	2.86	1.88	1.45
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When pH grows from 3.8 to 4.5, the densities of the initial and stripped off solutions grow to make up 1.240-1.246 and 1.389-1.396 g/cm³ at 20°C and 1.228-1.234 and 1.375-1.382 at 80°C, respectively.

With a growth in pH, the viscosities of the solutions also grow to make up 3.41-3.96 and 3.82-4.39 mPa s at 20 ° C and decrease to

1.10-1.29 and 1.23-1.45 mPa s at 80°C, respectively. The initial and stripped off solutions of monosodium phosphate have good rheological properties.

Table 6 shows the density and viscosity of the mother liquors that formed after the separation of potassium dihydrogen phosphate in three options [19].

Table 6. Density and viscosity of mother liquors formed after filtration of potassium dihydrogen phosphate

№ sample from Table. 2	Density, g/cm ³				Viscosity, mPa·c			
	20°C	40°C	60°C	80°C	20°C	40°C	60°C	80°C
1	1.285	1.277	1.271	1.266	2.718	1.867	1.364	1.205
2	1.295	1.287	1.281	1.277	2.787	1.973	1.462	1.284
3	1.291	1.283	1.277	1.273	2.786	1.972	1.461	1.282

In all cases, with rising temperature, the density and viscosity of the mother liquors drop as well. The study of the rheological properties

of mother liquors shows that they have satisfactory mobility and can be easily transported.

3. Results and Discussion

3.1. Results obtained using IR-infrared, scanning electron microscope and X-ray analysis

In order to establish the salt and chemical composition of potassium dihydrogen phosphate, X-ray, IR spectroscopic and scanning electron microscopic studies were carried out. X-ray analysis was performed on an XRD-6100 diffractometer (Shimadzu, Japan). We used CuK α radiation (β filter, Ni, tube current and voltage 30 mA, 30 kV) and a constant detector rotation speed of 4 deg/min. When removing the sample, a chamber with rotation was used, where the rotation speed is 30 rpm. The radiographs were interpreted using the American Mineralogist crystal structure database and Mikheev's X-ray determinant of minerals.

The IR absorption spectrum of the test substance, bischofite, was recorded on a SPECORD-751R in the frequency range of 400-4000 cm⁻¹. Samples were prepared by pressing tablets with KBr.

Fig. 6 shows a radiograph, and Fig. 7 IR spectrum of potassium dihydrogen phosphate synthesized under laboratory conditions.

On the IR spectrum (Fig. 7) there are

vibration frequencies characterizing vibrations related to PO₄ 429.11-1065.79 cm⁻¹ and crystalline water - 1277.42-2325.55 cm⁻¹. The data of IR spectroscopy of potassium dihydrogen phosphate confirm the data of chemical and X-ray studies.

Table 7 and Figure 8 show the main components of the obtained potassium dihydrogen phosphate based on sodium dihydrophosphate and flotation potassium chloride with the conversion method. Scanning electron microscopic analysis of potassium dihydrogen phosphate shows the following content of the elements of the composition: O-43.31%, Na-0.78%, P-24.96%, Cl-0.48%, K-30.47%, which corresponds to their content in potassium dihydrogen phosphate.

Thus, the effect of crystallization time and temperature on the elemental, basic chemical and salt composition of potassium dihydrogen phosphate was studied. The results obtained indicate the possibility of synthesizing potassium dihydrogen phosphate with the required performance properties through a solution of sodium dihydrogen phosphate and flotation potassium chloride.

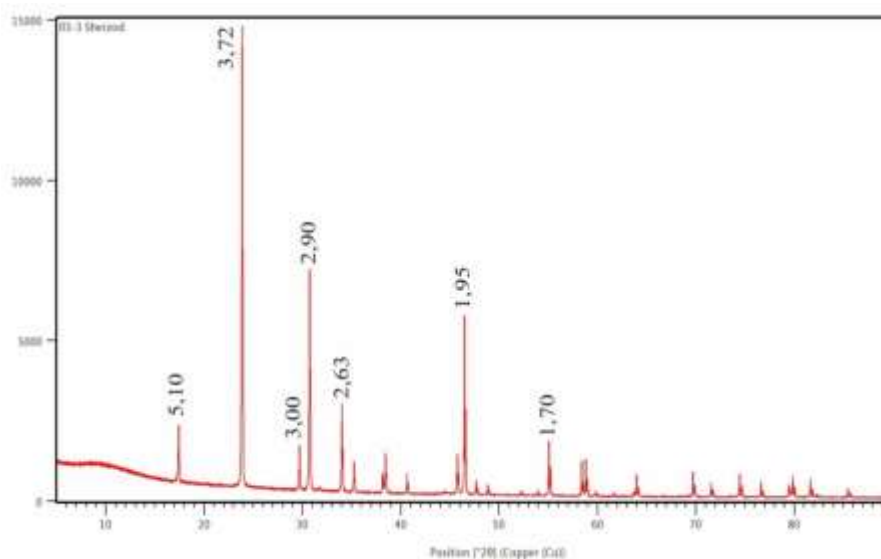


Fig. 6. X-ray of potassium dihydrogen phosphate 5.1; 3.72; 3.00; 2.90; 2.63 Å, which belong to KH_2PO_4 , as well as 1.70 Å NaCl

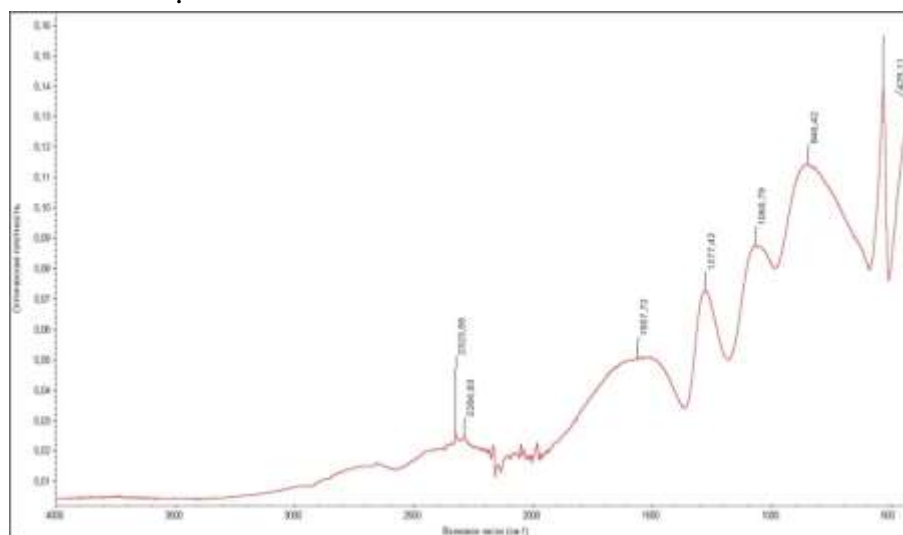


Fig. 7. IR spectrum of potassium dihydrogen phosphate

Table 7. Results of elemental chemical analysis of monopotassium phosphate

Element	Weight. %	Sigma, Bec. %
O	43.31	0.23
Na	0.78	0.05
P	24.96	0.14
Cl	0.48	0.04
K	30.47	0.16
Total	100.00	

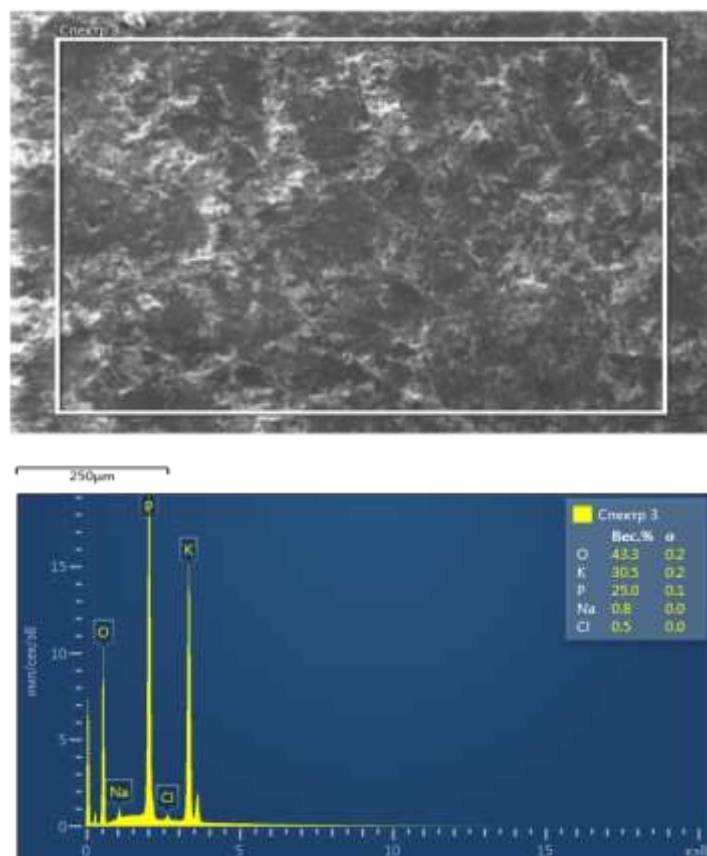


Fig. 8. Scanning microscopic analysis of potassium dihydrogen phosphate

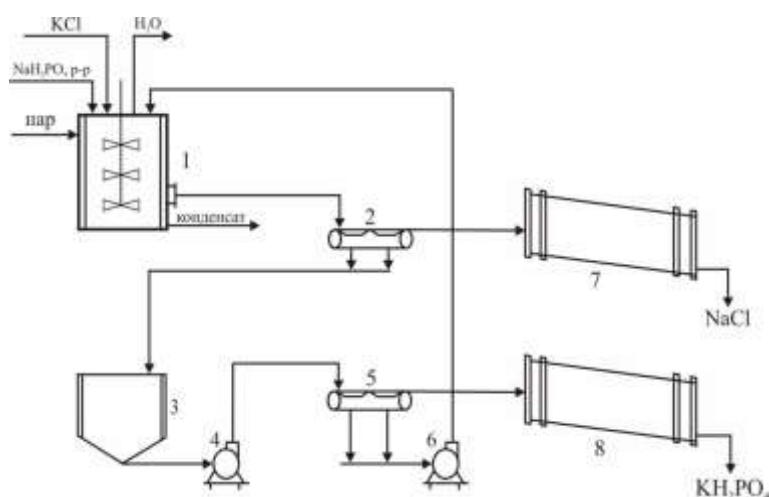


Fig. 9. Technological scheme for obtaining fodder-grade monopotassium phosphate based on extraction phosphoric acid obtained from phosphorites of the Central Kyzylkum by the conversion method: 1 – jacketed reactor; 2, 5 - filters; 3 – crystallizer.

3.2. Technological scheme for the production of monopotassium phosphate

The technological scheme and the material balance for obtaining chlorine-free feed-grade monopotassium phosphate based on

EPA and obtained from phosphorites are shown in Fig. 9 and Fig. 10.

A solution of monosodium phosphate and finely crystalline potassium chloride are simultaneously fed into the reactor (pos. 1) with

stirring and at a temperature of 95-100°C to evaporate moisture and concentrate the mixture.

The reactor also receives the mother liquor after separation of monopotassium phosphate. The processes of conversion and evaporation proceed for 2 hours, after which the mixture is fed to hot filtration to a vacuum filter (pos. 2) to separate precipitated sodium chloride crystals, which is sent to a drying drum (pos. 7) for drying. The filtrate from the filter (pos. 2) enters the mold (pos. 3) and then by pump (pos. 4) to the filter (pos. 5) to separate monopotassium phosphate crystals, which then enters the drum dryer (pos. 8) for drying the mother liquor is used as a circulation and fed

into the reactor (pos.1).

3.3 Approbation of technology for obtaining monopotassium phosphate

The conversion method for obtaining monopotassium phosphate is a new product for the chemical industry of the republic, requiring equipment that is different from those available at enterprises. Therefore, the tests of the developed technology were carried out on a model unit in the joint-stock company "Elektrokimyozavod".

The technological process for the production of monopotassium phosphate consists of stages as follows:

- conversion of a solution of monosodium phosphate with potassium chloride;
- evaporation of the conversion solution;
- separation of precipitation of sodium chloride;
- cooling and crystallization of monopotassium phosphate;
- separation of monopotassium phosphate crystals;
- washing and drying of monopotassium phosphate.

The technology was tested on a model plant simulating production conditions and an experimental batch of monopotassium phosphate was produced.

Technological process. Monosodium phosphate solution is fed to the stage of conversion of monosodium phosphate with potassium chloride. The process of conversion and evaporation of the solution occurs at a temperature of 100°C until the sodium chloride present in the solution is precipitated. Next, the pulp is fed to the filtration. The solid phase - sodium chloride is sent for washing and drying to obtain a commercial product - sodium chloride, and the liquid phase is sent to the

crystallizer for cooling to a temperature of 20-30 ° C and crystallization of monopotassium phosphate. The precipitated crystals of monopotassium phosphate are separated from the mother liquor, washed and dried. At that, the mother liquors are returned to the stage of conversion of monosodium phosphate with potassium chloride.

As a result of testing the technology, an experimental batch of monopotassium phosphate was produced in the amount of 50 kg, which was transferred for testing. Data on the analysis of the chemical composition and commercial properties of the product are shown in Table 8.

Table 8. Chemical and physical-chemical parameters and comparative characteristics of the obtained monopotassium phosphate by the conversion method

№	Name of indicators	Indicators	
		according to TU	Actually
1	Appearance	white powder	white powder
2	Mass fraction of total phosphates, (P ₂ O ₅), %, not less than	50.0	50.0
3	Mass fraction of potassium, (K ₂ O), %, not less than	33.0	33.0
4	Mass fraction of moisture, %, no more	0.5	0.35
5	Hydrogen index of 1% solution, units pH	4.0-4.5	4.3

6	Mass fraction of water-insoluble residue, %, max	0.1	0.01
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The monopotassium phosphate obtained during the tests fully met the requirements. TU 2186-021-32496445-00 (RF). Thus, the tests performed showed the possibility of obtaining monopotassium phosphate from solutions of monosodium phosphate based on purified EPA

from phosphorites by conversion with potassium chloride. The tests carried out on the pilot plant show adequate reproducibility of the results of laboratory studies regarding the technological parameters of the process and the level of quality of the products obtained.

4. Conclusions

For the physicochemical substantiation of the possibility of obtaining monopotassium phosphate, theoretical and experimental studies were carried out, which made it possible to develop the technology of chlorine-free phosphorus-potassium fertilizers by converting monosodium phosphate with potassium chloride.

The field of crystallization of KH_2PO_4 on the solubility diagram of the system K^+ , Na^+ // Cl^- , H_2PO_4^- - H_2O at 25°C occupies a greater part of the square area than at 100°C . When considering the isothermal solubility diagram, it can be seen that as the temperature grows, the crystallization areas of KH_2PO_4 sharply decrease; therefore, their solubility grows significantly. The saturation region of KH_2PO_4 at a temperature of 100°C is several times lower than at a temperature of 25°C .

The crystallization field of NaCl at a temperature of 100°C significantly increases compared to the crystallization field at 25°C due to a decrease in the KH_2PO_4 field. Consequently, the solubility of KH_2PO_4 increases several times, and such a solution, when cooled, is able to precipitate this salt.

The KH_2PO_4 - NaH_2PO_4 - NaCl - KCl - H_2O system was used to analyze the phase composition of industrial processes.

Based on the analysis of phase equilibria using the solubility diagram of the four-component water-salt system K^+ , Na^+ // Cl^- , H_2PO_4^- - H_2O at 25°C and 100°C , data were obtained on the possibility of isolating potassium dihydrogen phosphate and sodium chloride by crystallizing them from saturated solutions.

The influence of technological parameters such as solution concentration, temperature and solution cooling rate on the

process of crystallization of monopotassium phosphate from a conversion solution obtained by the conversion method from solutions of monosodium phosphate obtained on the basis of EPA flotation potassium chloride was studied.

The slow cooling rate contributes to the precipitation of fewer impurities in the sediment. With a decrease in the cooling rate, the amount of Na_2O and Cl^- impurities in the product decreases by about a factor of two. Increasing the concentration of the solution over 30% leads to an increase in the content of impurities in the finished product.

The optimal conditions for obtaining potassium dihydrogen phosphate by converting a solution of monosodium phosphate with flotation potassium chloride and preliminary isolation of sodium chloride are temperature - no more than 25.0°C , solution concentration - at least 24-27%, solution cooling rate - 10.0°C / hour. The chemical composition of the resulting product meets the requirements of GOST.

The rheological properties of conversion solutions were studied at temperatures of 20 - 80°C depending on pH. As pH rises from 3.8 to 4.5, the densities of the initial and stripped off solutions rise as well and amount to 1.240-1.246 and 1.389-1.396 g/cm^3 at 20°C and 1.228-1.234 and 1.375-1.382 at 80°C , respectively.

As pH rises, the viscosities of the solutions rise as well and amount to 3.41-3.96 and 3.82-4.39 mPa s at 20°C and decrease to 1.10-1.29 and 1.23-1.45 mPa s at 80°C , respectively. The initial and stripped off solutions of monosodium phosphate have good rheological properties.

Based on the results obtained, a basic technological scheme for the production of monopotassium phosphate and sodium chloride is proposed. The optimal parameters of the

technological process were established and the material balance and norms of the technological regime compiled.

The tests carried out on the pilot plant

show adequate reproducibility of the results of laboratory studies over the technological parameters of the processes and the level of quality of the products obtained.

References

- Vakal S., Vakal V., Artyukhov A. et al. Granulated organo-mineral fertilizers: the process of formation and investigation of porous phosphate-diatomite shell. *Appl Nanosci.* 2023, vol. 13, pp. 5157–5164 <https://doi.org/10.1007/s13204-022-02718-w>
- FAOSTAT Statistics division of the Food and Agricultural Organization of the United Nations. 2019.
- Dwivedi SL, Scheben A, Edwards D et al. Assessing and exploiting functional diversity in germplasm pools to enhance abiotic stress adaptation and yield in cereals and food legumes. *Front Plant Sci.* 2017, vol. 8, p. 30. <https://doi.org/10.3389/fpls.2017.01461>
- Hu Y., Cheng H., Tao S. Environ mental and human health challenges of industrial livestock and poultry farming in China and their mitigation. *Environ Int.* 2017, vol. 107, pp. 111–130. <https://doi.org/10.1016/j.envint.2017.07.003>
- Ait-El-Mokhtar M, Anli M, Laouane BR et al. Food security and climate change. In: Kahime K, El Hidan M, El Hiba O, Sereno D, Bounoua L (eds) Handbook of research on global environmental changes and human health. *IGI Global, Pennsylvania,* 2019, pp. 53–73. <https://doi.org/10.4018/978-1-5225-7775-1.ch004>
- Nicholson CF, Stephens EC, Jones AD et al. Food security outcomes in agricultural systems models: current status and recommended improvements. *Agric Syst.* 2021, vol.188, p. 103028. <https://doi.org/10.1016/j.agsy.2020.103028>
- Grote U, Fasse A, Nguyen TT, Erenstein O. Food security and the dynamics of wheat and maize value chains in Africa and Asia. *Front Sustain Food Syst.* 2021, vol.4, pp. 617009. <https://doi.org/10.3389/fsufs.2020.617009>
- EPA National Pollutant Discharge Elimination System Permit Regulation and Effluent Limitations Guidelines and Standards for Concentrated Animal Feeding Operations. (ed Agency, E. P), 2003. pp.7176–7227.
- Ait-El-Mokhtar M., Labbilta T., Anli M. et al. Phosphate Glass-Based Controlled-Release Fertilizers Improve Wheat Growth, Yield and Grain Nutritional Quality Under Field Conditions. *Gesunde Pflanzen.* 2022, vol. 74. pp. 715–727. <https://doi.org/10.1007/s10343-022-00647-w>
- Meshalkin V.P., Pochitalkina I.A., Bessolova Y.A. et al. Methodology of Organization of Closed Water Circulation of Enterprises for the Production of Extraction of Phosphoric Acid and Mineral Fertilizers. *Russ J Gen Chem.* 2022, vol. 92, pp. 2993–2997. <https://doi.org/10.1134/S1070363222120581>
- Gorbovskiy K.G., Kazakov A.I., Norov A.M. et al. Effect of Impurities on Thermal Decomposition Kinetics of Mineral Fertilizers Based on $(\text{NH}_4)_2\text{HPO}_4$ in Self-Generated Atmosphere. *Russian Journal of Applied Chemistry.* 2018, vol. 91, pp. 1057–1067. <https://doi.org/10.1134/S1070427218070017>
- Alimov U.K., Reimov A.M., Namazov S.S. et al. The insoluble part of phosphorus fertilizers, obtained by processing of phosphorites of central kyzylkum with partially ammoniated extraction phosphoric acid. *Russian Journal of Applied Chemistry.* 2010, vol. 83, pp. 545–552. <https://doi.org/10.1134/S107042721030328>
- Gorbovskiy K.G., Norov A.M., Malyavin A.S. et al. Physicochemical properties of carbamide-containing nitrogen-phosphorus-

- potassium fertilizers conditioned by magnesium salts. *Theor Found Chem Eng* . 2015, vol. 49, pp. 467–470. <https://doi.org/10.1134/S0040579515040089>
14. Aguilar K.K. Preparation of potassium dihydrogen phosphate by the conversion method. Diss. tech. Sciences. St. Petersburg, 2004, 187 p.
 15. Dormeshkin O.B., Vorobyov N.I. Production of non-chlorine water-soluble complex fertilizers. Minsk, 2006, 248 p. ISBN 985-434-623-4.
 16. Shatilov V.I. Waste-free technology for obtaining water-soluble chlorine-free NPK fertilizers based on potassium phosphate. Diss. ... cand. tech. Sciences. Minsk, 2004, 265p.
 17. Dormeshkin O.B., Vorobyov N.I., Shatalo V.I. Waste-free technological process for obtaining a chlorine-free water-soluble complex fertilizer based on potassium phosphate. *Chemical technology*, 2014, vol.15, no. 6, pp. 24-32. Minsk, Belarus.
 18. Mazunin S.A., Chechulin V.L., Frolova S.I., Kistanova N.S. Technology of Obtaining of Potassium Dihydro-phosphate in the System with Salting-Out. *Russian Journal of Applied Chemistry*. 2010, vol. 82, no.3, pp. 553–561.
 19. Mohichehra Shaymardanova et al. Technology for obtaining monopotassiumphosphate from a solution of monosodiumphosphate and flotation potassium chloride by the conversion method. *News of the National University OF Uzbekistan*, 2021, pp. 125-130. ISSN 2181-7324.

MONO-NATRIUM FOSFAT VƏ KALIUM XLORİD ƏSASINDA MONO-KALIUM FOSFATIN ALINMA PROSESİNİN TƏDQIQI

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Xülasə: Mərkəzi Qızılqum fosforitlərindən təmizlənmiş ekstraksiyalı fosfor turşusu əsasında alınmış natrium dihidrofosfat məhlulları ilə Tübeqatan yatağından kalium xloridin kalium dihidrogenfosfata konversiya prosesinin tədqiqatlarının nəticələri təqdim olunur. 25°C və 100°C temperaturda kalium dihidrofosfatın əmələ gəlməsinə B:M komponentlərinin nisbətinin təsiri öyrənilmişdir. Kalium dihidrofosfatın ayrılması üçün optimal texnoloji parametrlər müəyyən edilmişdir. Kalium dihidrofosfatın istehsalı zamanı əmələ gələn məhlulların reoloji xassələri müəyyən edilmişdir. Alınan nəticələr rentgen faza və İQ-spektroskopik analiz üsulları ilə təsdiqlənmişdir. Kalium dihidrofosfatın element tərkibi müəyyən edilmişdir.

Açar sözlər: kalium monofosfat, kalium xlorid, həll olma diaqramı, çevrilmə, natrium və kalium dihidrofosfatları.

**ИССЛЕДОВАНИЕ ПРОЦЕССА ПОЛУЧЕНИЯ МОНОКАЛИЕВОГО ФОСФАТА
НА ОСНОВЕ МОНОНАТРИЙФОСФАТА И ХЛОРИДА КАЛИЯ**

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Аннотация: Представлены результаты исследований процесса конверсии флотационного хлорида калия Тюбегатанского месторождения растворами дигидрофосфата натрия, полученного на основе очищенной экстракционной фосфорной кислоты из фосфоритов Центральных Кызылкумов, в дигидрофосфат калия. Исследовано влияние соотношения компонентов Т:Ж на образование дигидрофосфата калия при температурах 25°C и 100°C. Определены оптимальные технологические параметры выделения дигидрофосфата калия. Определены реологические свойства растворов, образующихся в процессе получения дигидрофосфата калия. Полученные результаты подтверждены рентгенофазовым и ИК-спектроскопическим методами анализа. Элементный состав дигидрофосфата калия установлен методом электронной микроскопии.

Ключевые слова: монофосфат калия, флотация хлорида калия, диаграмма растворимости, конверсия, дигидрофосфаты натрия и калия.