

## SODIUM SULPHATE FROM DRY MIXED SALTS OF LAKE KARAUMBET

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### ANNOTATION

The results of the processing of dry mixed salts of lake Karaumbet and the isolation of mirabilite are presented. It was shown that the maximum solubility of dry mixed salts in water at 25°C is observed at T:W = 1:(2.5-3) and amounts to 91.08-93.02% of the total initial mass. The degree of dissolution of the main components of the salt at T:W = 1:3 after 5 minutes reaches 89.25%, and after 15 minutes 93.02% at a temperature of 25°C and 92.5% after 5 minutes and 91.61% after 10 minutes and 93.02% after 15 minutes at a temperature of 50°C.

When sedimentation (clarification) of suspension proceeds most intensively in the first 15 minutes and after 30 minutes the degree of clarification at 20°C reaches 94.59%. At temperatures of 40, 60 and 80°C, the degree of clarification is 95.51%.

At cooling of the clarified solution to temperature -5°C, the sodium sulfate content in the solution decreases from 15.31% to 2.23%. The maximum yield of sodium sulfate is 85.43% of the initial content of dry mixed salts in the solution. The filtration rate of the thickened sediment by mirabilite reaches 2743 kg/m<sup>2</sup>·h.

With an increase in frequency rate of return of the mother liquor, the sodium sulfate content decreases from 30.81% to 30.08%, the magnesium sulfate content increases from 0.68% to 1.33%, sodium chloride from 0.31% to 0.73% and sulfate calcium from 0.08% to 0.11%.

It was shown that by returning the mother liquor from the sodium sulfate filtration stage to the stage of mirabilite dissolution with juice pairs and the subsequent introduction of sodium hydroxide and sodium carbonate, the highest grade sodium sulfate yield reaches a maximum value with a main product content of at least 99.4%.

**Key words:** dry mixed salts, dissolution, cooling, mirabilite, filtration, sodium sulfate, lime-soda cleaning.

## INTRODUCTION

Sodium sulfate is widely used in many industries and its demand is increasing from year to year. Its main consumers are the pulp and paper, glass, chemical, metallurgical industries and the production of synthetic detergents. To a lesser extent, sodium sulfate is in demand in the textile, soap, leather industries, medicine and veterinary medicine [1-3]. In addition, sodium sulfate is used for other purposes [4]. The development of the extraction of natural sodium sulfate on a comparatively large scale dates back to the beginning of the second half of the XVIII century and is associated with its use in glass melting.

Industrial methods for obtaining sodium sulfate involve the use of both natural raw materials and associated or by-products of chemical industries [5-10].

The main amount of sodium sulfate is obtaining by dehydration of mirabilite [11-13]. Various dehydration methods provide sodium sulfate of various quality. So when dehydrated in a drum dryer, a clumping, caking product with a low sodium sulfate content is obtaining. Mirabilite is converted to anhydrous sodium sulfate by melting, melting-evaporating, dropping, dehydrating mirabilite in an autoclave by drying with air or flue gases, diluted with air, dissolving mirabilite with subsequent evaporation, dehydration using volatile solvents [2, 3, 9, 14-17 ].

Scientific and technological progress in industries using sodium sulfate, as well as the development of new industries, have increased the quality requirements for sodium sulfate. The highest requirements for the quality of sodium sulfate are made by the industries that produce synthetic detergents. This is due to the fact that the microcomponents introduced into them, which sharply worsen the quality of synthetic detergents, are very sensitive to the impurities of some divalent metals. The content of sodium sulfate should not be lower than 99.4%, with the practical absence of bivalent metals, especially iron [18].

The production of sodium sulfate by the conversion of sodium chloride with sulfuric acid is also gradually losing importance due to the fact that the resulting product is of poor quality, since it contains up to 3-4% NaCl, and the main substance is not more than 95% [19].

Sodium sulfate from sulfate-containing solutions or from mirabilite is often obtaining by salting out using inorganic salts, bases and organic solvents. For this purpose, sodium chloride is most often used, obtained as a waste from the basin or factory processing of natural brines and salts. Using this method, sodium sulfate is obtaining in Germany from wastes from the processing of Hartsalz to chloride and potassium sulfate, in the United States through the integrated processing of brine from lake Searles and brines of the Big Salt lake [20-22].

In the USA, dehydration of mirabilite is carried out in three stages: melting at 55°C in a rotary kiln, evaporation and drying of the mixture in a tubular steam dryer at 103°C, and final drying of the solid product in a drum dryer at 140°C [2].

Sodium sulfate from sulfate-containing solutions or mirabilite is also obtaining by salting out. Inorganic salts, bases, organic solvents are used as salting out substances [23-25].

To obtain sodium sulfate, mixed salts of  $\text{NaCl} + \text{MgSO}_4 \cdot 7\text{H}_2\text{O}$  are recommended as salting out agents [15]. However, an increase in the content of impurities of sodium chloride and magnesium leads to a deterioration in the quality of the finished product.

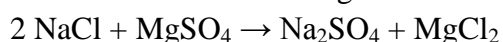
The use of sodium hydroxide as a salting out reagent makes it possible to isolate 90-95% sodium sulfate from the solution [26]. The residual amount of sodium sulfate in the solution

depends on the concentration of caustic soda. This method allows the processing of mirabilite containing organic impurities.

In India, anhydrous sodium sulfate is obtained from the salt deposits of one of the inland lakes. The solid phase of the composition (mass. %): NaCl - 49.5; Na<sub>2</sub>SO<sub>4</sub> - 35.2; Na<sub>2</sub>CO<sub>3</sub> - 4.5; but. - 4.5 is brought into contact with the brine composition (mass. %): NaCl - 16.0; Na<sub>2</sub>SO<sub>4</sub> - 7.0; Na<sub>2</sub>CO<sub>3</sub> - 1.0 at 50°C and a technical product containing 99.6% of the basic substance is obtained. The total yield of sodium sulfate is 87.5% [27].

Obtaining sodium sulfate using ethyl, methyl, propyl alcohols, acetone as a salting out component consists in mixing the liquid phase after melting mirabilite with an organic solvent and separating the solid phase [2]. This method allows you to get a better product than salting out with sodium chloride.

In another method, sodium sulfate is salted out from water-methanol solutions of magnesium chloride at a temperature of 30-60°C according to the reaction



During the conversion of magnesium sulfate, sodium chloride forms sodium sulfate, which precipitates [28].

Uzbekistan has large raw material resources of sodium sulfate found in the salt deposits of the Aral Sea: Akkaly, Kushkantau, Tumryuk deposits in the Republic of Karakalpakstan, glauberite deposits in the Ferghana Depression and the Tien Shan intermontane basins [1, 29, 30]. The Tumryuk mirabilite deposit is one of the most accessible raw material sources of sodium sulfate, characterized by a minimum content of impurity salts of halite, gypsum epsomite.

The brine of lakes of chloride-sulfate salts (Barsakelmes, Karaumbet, Dengiz-Kul deposits [31]) can also serve as cheap local raw material sources for producing sodium sulfate. The lack of development of deposits for processing local raw materials inhibits the organization of large-scale production of sodium sulfate.

Scientific and technological progress and the development of new industries have increased the quality requirements for sodium sulfate. This is due to the fact that the impurities contained, in particular iron, calcium, chlorine, sharply worsen the quality of synthetic detergents and special types of paper. Currently produced in a limited amount of sodium sulfate from the natural mirabilite of the Tumryuk deposit does not meet the increased requirements. At present, the natural mirabilite of the Tumryuk deposit is processed into sodium sulfate using a two-stage evaporation of the clarified solution [9, 29, 32]. In the first stage, the solution is evaporated to a content of 50-60% sodium sulfate, it is separated from the mother liquor and dried. In the second stage, the mother liquor is evaporated to a wet state and dried. The result is 39-64% sodium sulfate of the highest grade with a sodium sulfate content of at least 99.4% and 36-61% of grades 1 and 2.

Despite the great demand for premium grade sodium sulfate, and the presence of a large raw material base, the product in the republic is produced in a small amount. This is primarily due to the lack of acceptable technologies for processing the natural mirabilite of the Tumryuk field and the dry mixed salts of lake Karaumbet into high-quality sodium sulfate [33].

## RESEARCH METHODS

Industrial reserves of lake Karaumbet are represented by brine, halite and dry mixed salts. Dry mixed salts are represented by 754 thousand tons of sodium chloride, 2452 thousand tons of sodium sulfate, 613 thousand tons of magnesium chloride.

For research, samples of dry mixed salts of the composition (mass. %) were selected:  $\text{Na}_2\text{SO}_4$  - 60.69;  $\text{NaCl}$  - 18.81;  $\text{MgCl}_2$  - 15.30;  $\text{MgSO}_4$  - 0.42;  $\text{CaCl}_2$  - 0.31; n.o. - 3.98;  $\text{H}_2\text{O}$  - the rest.

Initial raw materials and the received products analyzed on the maintenance of following components: calcium, magnesium, sulphur, chlorine, carbonates, the insoluble rest in water, a moisture, known methods of the chemical analysis [14, 15].

## RESULTS AND ITS DISCUSSION

Researches of the influence of the duration of the process of dissolving dry mixed salts in water showed that after 5 minutes the degree of dissolution reaches 89.25%, and after 15 minutes - 93.02% at 25°C and 91.5% after 5 minutes at 50°C, which indicates 15 minutes are enough for dissolving dry mixed salts in water (table 1).

The maximum solubility of dry mixed salts is observed at T:W = 1:(3-4) and is 93.02%. Therefore, to obtain solutions with the maximum dissolution of dry mixed salts, it is necessary to maintain T:W = 1:3, at a temperature of 25°C and a process duration of 30 minutes.

Table 1

The influence of T:W on the solubility of dry mixed salts and the composition of the suspension at a temperature of 25°C and the duration of the process 30 minutes

№	T:W	The ionic composition, mass. %					The composition of the solutions, mass. %				The degree of dissolution of DMS,%
		$\text{Na}^+$	$\text{Mg}^{2+}$	$\text{Ca}^{2+}$	$\text{Cl}^-$	$\text{SO}_4^{2-}$	$\text{Na}_2\text{SO}_4$	$\text{MgCl}_2$	$\text{NaCl}$	$\text{CaSO}_4$	
1	1:2,0	9,41	1,40	0,024	3,97	14,35	21,15	5,48	6,55	0,08	73,96
2	1:2,5	7,83	1,16	0,024	3,30	11,93	17,57	4,55	5,44	0,08	91,08
3	1:3,0	6,82	1,01	0,024	2,88	10,40	15,31	3,97	4,74	0,08	93,02
4	1:4,0	5,45	0,81	0,024	2,30	8,32	12,23	3,17	3,79	0,08	93,02

Dry mixed salts contain more than 50% mirabilite. Saturated solutions were cooled to reduce the sodium sulfate content. The content of sodium sulfate when cooled to a temperature of +5 ÷ -5°C decreases from 15.48% to 2.23-2.82% (table. 2).

From the data obtained it follows that when cooled to a temperature of 0°C, the sodium sulfate content in the solution of dry mixed salts decreases from 15.31% to 2.51% with an increase in the yield of  $\text{Na}_2\text{SO}_4$  in the solid phase from 11.64 to 83.61%. In this case, the content of the remaining components of the solution increases.

When freezing mirabilite from solutions of dry mixed salts of Karaumbet, up to 200 kg of mirabilite is released from each ton of solution. The output of sodium sulfate is 81.58 - 85.43%

of the initial content in the solution. Mirabilite isolated from a solution of dry mixed salts is cleaner than the natural Tumryuk deposit.

In order to improve the quality and yield of sodium sulfate from mirabilite and to intensify the process of evaporation, the mother liquors after the first stage of evaporation were returned to the stage of dissolution of mirabilite. However, after 3-4 cycles of dissolution of mirabilite and evaporation of sodium sulfate solutions, the content of impurities in the mother liquor increases significantly, further contaminating sodium sulfate (table 3).

Table 2

The effect of temperature on the chemical and salt compositions of solutions of dry mixed salts and the yield of mirabilite

T, °C.	The chemical composition of the liquid phase, mass. %					The salt composition of the liquid phase, mass. %				Exit Na <sub>2</sub> SO <sub>4</sub> , %
	Na <sup>+</sup>	Mg <sup>2+</sup>	Ca <sup>2+</sup>	Cl <sup>-</sup>	SO <sub>4</sub> <sup>2-</sup>	Na <sub>2</sub> SO <sub>4</sub>	MgCl <sub>2</sub>	NaCl	CaSO <sub>4</sub>	
25	7,92	1,01	0,022	5,83	10,41	15,31	3,97	4,74	0,08	-
20	6,32	1,06	0,024	6,08	9,19	13,52	4,14	4,94	0,08	11,69
15	5,49	1,16	0,026	6,62	7,14	10,51	4,55	5,32	0,09	31,35
10	4,22	1,30	0,029	7,46	3,89	5,68	5,08	6,06	0,10	62,90
5	3,51	1,41	0,032	8,14	1,96	2,82	5,54	6,61	0,11	81,58
0	3,44	1,43	0,032	8,22	1,75	2,51	5,52	6,68	0,11	83,61
-5	3,37	1,44	0,032	8,29	1,56	2,23	5,64	6,74	0,11	85,43

Table 3

Changes in the chemical composition of the liquid phase, depending on the frequency of use of the mother liquor after evaporation

№	The frequency of use of the mother liquor	The chemical composition of the liquid phase, mass. %					The salt composition of the liquid phase, mass. %			
		Na <sup>+</sup>	Mg <sup>2+</sup>	Ca <sup>2+</sup>	Cl <sup>-</sup>	SO <sub>4</sub> <sup>2-</sup>	Na <sub>2</sub> SO <sub>4</sub>	MgCl <sub>2</sub>	NaCl	CaSO <sub>4</sub>
1	Source	10,10	0,14	0,023	0,19	20,89	30,81	0,68	0,31	0,08
2	Unitary	10,12	0,20	0,026	0,31	20,77	30,63	0,98	0,51	0,09
3	Twofold	10,10	0,23	0,030	0,37	20,66	30,45	1,13	0,61	0,10
4	Thrice-repeated	10,08	0,25	0,030	0,41	20,54	30,27	1,24	0,68	0,10
5	Fourfold	10,02	0,27	0,036	0,44	20,42	30,08	1,33	0,73	0,11

For research, we used a saturated solution of sodium sulfate obtained by dissolving mirabilite in water in the presence of a mother liquor from the stage of separation of sodium sulfate. The solution fed to the residue contained (mass. %): Na<sub>2</sub>SO<sub>4</sub> - 30.81%; MgSO<sub>4</sub> - 0.68; NaCl - 0.38; CaSO<sub>4</sub> - 0.08.

With an increase in the frequency of use of the mother liquor after separation of sodium sulfate, the content of sodium sulfate decreases slightly (from 30.81 to 30.08%), but the remaining components increase.

So, the content of magnesium sulfate increases from the initial 0.68% to 1.33%, sodium chloride from 0.31% to 0.73% and calcium sulfate from 0.08% to 0.11% after a four-fold return of the mother liquor to the stage dissolution of mirabilite. Therefore, clarified sodium sulfate solutions were purified from calcium and magnesium impurities by precipitation with carbonate and sodium hydroxide. Sodium carbonate was introduced in an amount providing a ratio of  $\text{CaO}:\text{CO}_2 = 1:(0.90-1.05)$ , and sodium hydroxide in an amount of 100% of the stoichiometry for the formation of magnesium hydroxide. Hydroxide and sodium carbonate were introduced simultaneously in the form of 20% solutions after the fourth cycle of the return of the mother liquor from the evaporation stage to the stage of dissolution of mirabilite. A saturated solution of sodium sulfate after the fourth cycle contains (mass. %):  $\text{Na}_2\text{SO}_4$  - 30.08;  $\text{MgCl}_2$  - 1.33;  $\text{NaCl}$  - 0.73;  $\text{CaSO}_4$  - 0.11.

Hydroxide and sodium carbonate were introduced simultaneously after the fourth cycle of the return of the mother liquor from the evaporation stage to the stage of dissolution of mirabilite.

The influence of the ratio of  $\text{Ca}:\text{CO}_3$  on the composition of the liquid phase at a rate of sodium hydroxide of 100% on the formation of magnesium hydroxide, after separation of precipitation, are shown in table 4.

Table 4

The effect of the molar ratio of  $\text{Ca}:\text{CO}_3$  on the chemical composition of the liquid phase at 100% normal sodium hydroxide

№	Ca:CO <sub>3</sub>	The chemical composition of the liquid phase, mass. %					The salt composition of the liquid phase, mass. %			
		Na <sup>+</sup>	Mg <sup>2+</sup>	Ca <sup>2+</sup>	Cl <sup>-</sup>	SO <sub>4</sub> <sup>2-</sup>	Na <sub>2</sub> SO <sub>4</sub>	MgSO <sub>4</sub>	NaCl	CaSO <sub>4</sub>
1	-	10,02	0,33	0,032	0,44	21,39	30,08	1,33	0,73	0,11
2	1:0,90	10,15	-	0,003	0,43	20,59	30,46	-	0,71	0,01
3	1:1,00	10,12	-	-	0,42	20,54	30,39	-	0,70	-
4	1:1,05	10,09	-	-	0,41	20,50	30,33	-	0,69	-

As a result of the precipitation of calcium carbonate and magnesium hydroxide, solutions are obtained that are practically free of calcium and magnesium salts. If, with a molar ratio of  $\text{Ca}:\text{CO}_3 = 1:0.90$ , a small amount of dissolved calcium sulfate is retained, then with a ratio of 1:1 and 1:1.05 it is absent.

The precipitation of calcium carbonate and magnesium hydroxide proceeds quickly and after 20-25 minutes the degree of clarification of the solution is reached 80-85%. The filtration rate of the condensed sediment in a vacuum installation with a discharge of 400 mm hg does not exceed 82 kg/m<sup>2</sup>·h. Separation of the precipitate should be carried out by centrifugation.

Studies on the evaporation of purified solutions of sodium sulfate were carried out at a temperature of 100°C, under vacuum, with a residual pressure of 82.16-86.26 kPa. Removing 35% of water from the evaporated solution occurs within 15 minutes with the formation of pulp with a ratio of W:T = 5:1.

Removing 50% water leads to the precipitation of 55% sodium sulfate and the formation of pulp with a ratio of W:T = 3:1. The evaporation can be carried out to a concentration of sodium sulfate in the pulp 60-65%. At higher concentrations, the pulp is poorly separated.

Researchers of the filtration process of pulps formed during evaporation and cooling of a sodium sulfate solution was carried out in a laboratory setup consisting of storage units, a thermostat, and filtration units. Belting was used as filter cloth.

Results of experiments on the filtration of pulps formed during the evaporation and cooling of sodium sulfate solutions at a residual pressure of 84 kPa and a filter area of  $63.59 \cdot 10^{-4} \text{ m}^2$  are presented in table. 5.

According to the resulted data filterability and speed of a filtration of the pulps containing  $\text{Na}_2\text{SO}_4$ , with increase of parity W:T enlarge.

The filtration rate for solid and liquid phases strongly depends on the thickness of the layer of solid residue formed on the filters. The smaller the thickness of the solid residue on the filter, the greater the filtration performance for pulp, solid and liquid phases.

After separation of the pulp with a precipitate of sodium sulfate by filtration and drying at  $200^\circ\text{C}$ , a product with a sodium sulfate content of at least 99.4% is obtaining.

Based on the results of laboratory experiments, a basic technological scheme was developed, the material balance of processing mirabilite extracted from dry mixed salts of Karaumbet into high-quality sodium sulfate. The technological scheme is shown in figure 1.

The essence of the proposed method for producing sodium sulfate consists in dissolving dry mixed salts in water, separating water-insoluble residues, cooling the clarified solution, filtering mirabilite, dissolving the separated mirabilite in water and mother liquor, purifying the solution, filtering sodium sulfate, drying it, and returning the mother liquor at the stage of dissolution of mirabilite [4].

**Table 5**  
**Filterability of pulp with  $\text{Na}_2\text{SO}_4$  sediment**

Ratio W:T	Number pulps, g	Time ( $\tau$ ), sec	Solid thickness draft (h), mm	Filterability ( $\Phi \cdot 10^{-3}$ ), $\text{m}^4/\text{N} \cdot \text{hour}$	Filtration rate, $\text{kg} / \text{m}^2 \cdot \text{s}$		
					on the pulp	solid phase	by filtrate
1:1	447,1	73	20,1	0,165	0,964	0,482	0,482
	300,0	45	13,5	0,120	1,048	0,524	0,524
	150,0	21	6,8	0,065	1,124	0,562	0,562
2:1	447,1	47	13,4	0,227	1,496	0,499	0,997
	300,0	28	9,0	0,142	1,685	0,562	1,123
	150,0	13	5,0	0,103	1,815	0,605	1,210
3:1	447,1	35	10,1	0,259	2,009	0,502	1,507
	300,0	20	6,8	0,205	2,359	0,590	1,769
	150,0	9	3,5	0,117	2,621	0,655	1,966
4:1	447,1	27	8,1	0,287	2,604	0,521	2,083
	300,0	15	5,5	0,235	3,145	0,629	2,516
	150,0	7	2,8	0,128	3,370	0,674	2,696
4,5:1	447,1	22	7,5	0,320	3,196	0,576	2,620
	300,0	12	5,1	0,268	3,932	0,709	3,223
	150,0	5	2,6	0,164	4,718	0,851	3,867

A scheme of material flows and a material balance for the production of premium grade sodium sulfate from the dry mixed salts of lake Karaumbet have been developed (fig. 1).

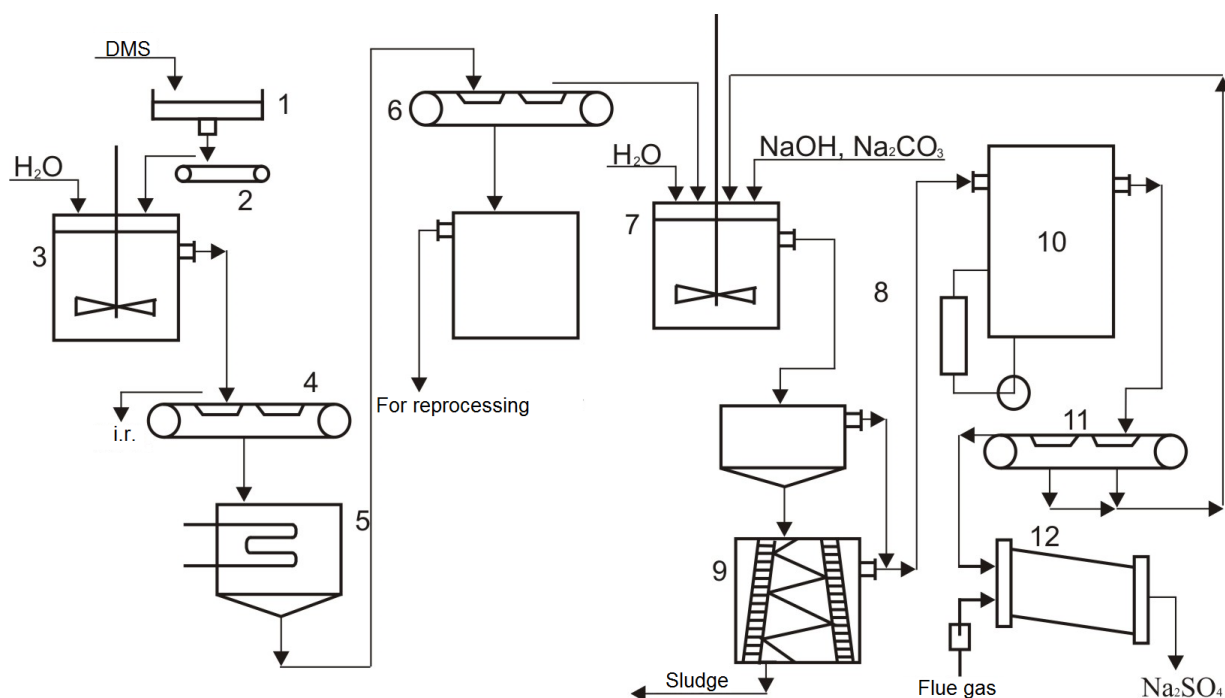


Fig. 1. Schematic diagram of the production of premium grade sodium sulfate from dry mixed salts of lake Karaumbet

According to the scheme, dry mixed salts from lake Karaumbet from the receiving hopper (pos. 1) are fed through a dispenser (pos. 2) to the dissolution reactor (pos. 3). Technical water is also supplied here to maintain T:W. From the reactor, the dissolution product enters the belt vacuum filter (pos. 4), where the separation into liquid and solid phases occurs. The solid phase is water-insoluble residues. The filtrate is a solution of sodium chloride, magnesium and sodium sulfate. Next, the filtrate is cooled in the mold (pos. 5) and fed to the belt vacuum filter (pos. 6). The liquid phase is directed to obtain compounds of magnesium and sodium chloride. The solid phase in the reactor (pos. 7) is dissolved in water and the mother liquor from the stage of evaporation of the sodium sulfate solution. Solutions of soda ash and sodium hydroxide are simultaneously fed into the reactor (pos. 7). From the reactor, the suspension is fed into the sump (pos. 8). The condensed part from the sump is fed to a centrifuge (pos. 9) to separate the sludge, and the liquid phase, together with the clarified part from the sump, is sent to the residue (pos. 10). From the residue, sodium sulfate pulp is fed to the filter (pos. 11). The mother liquor is sent to the stage of dissolution and purification of the mirabilite solution, the solid phase enters the dryer for drying in the drum dryer (pos. 12) and then to the cooling and finished products warehouse.

The technology for producing premium grade sodium sulfate with a maximum yield of a product suitable for the production of synthetic detergents has been tested and implemented at JV LLC "Sulfate Sodium".

## CONCLUSION

Thus, the studies showed the possibility of processing mirabilite extracted from the dry mixed salts of lake Karaumbet into sodium sulfate of the highest grade, suitable for the production of synthetic detergents and for other industries. For this, it is necessary to clear the

clarified mirabilite solutions from calcium and magnesium impurities with carbonate and sodium hydroxide, and the mother liquor is evaporated to a concentration of 60-65%, followed by filtration and drying of sodium sulfate, returning the mother liquor to the stage of dissolution of mirabilite and purification of sodium sulfate solution.

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