International Journal of Aquatic Science
ISSN: 2008-8019
Vol 12, Issue 02, 2021

Study Of The Influence Of Technological Parameters On The Quality Of Potassium Nitrate

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Abstract: The state of the world production of potassium nitrate with substantiation of the actuality of solving problem has been analyzed in the article. The influence of technological parameters on the outdegree of potassium oxide and the quality of potassium nitrate obtained by the conversion method has been investigated. The quality of the product has been determined by modern methods of physical and chemical analysis. The optimal technological parameters for the production of potassium nitrate by the conversion method from potassium chloride and ammonium nitrate have also been determined.

Keywords: potassium chloride, conversion, potassium nitrate, ammonium chloride, ammonium nitrate, technology, temperature, concentration.

1. INTRODUCTION.

Chlorine, along with fluorine, belongs to the group of halogens. However, fluorine shows more non-metallic (oxidizing) properties and therefore is a more aggressive and harmful impurity. According to agrochemical studies [1], when the content of fluorine in fertilizers is up to 3%, there is no negative effect on plants. A similar value is adopted in most of the current standards for regulating the chlorine content in chlorine-free fertilizers.
At the same time, improving the quality is one of the priority directions in the development of the production of mineral fertilizers, and, according to Belarusian agrochemists, the chlorine content in fertilizers used in greenhouses should not exceed 1% [2-3].

The largest exporters of chlorine-free, water-soluble, complex fertilizers are Kemiro Agro (Finland), Norsk Hydro (Norway), Agrohanza (Poland), OJSC Buiskiy chemical plant (Russia), Nutri SI (Belgium), Haifa Chemicals and Israil Chemicals (Israel), Vicksburg

Chemicals Co. (USA), SQM SA, PCS Ymbes SCM (Chile), The Arab Potash Company Ltd (Jordan) [2].

According to published data on projects for the construction of NPK-fertilizers production [2], during 1997-2000, the planned commissioning of new capacities for the production of potassium nitrate in the world made up over 474 thousand tons / year. The growth rate of the global market for chlorine-free, water-soluble fertilizers is estimated at 4% per year. Meanwhile, to present time, a sufficiently large amount of researches has been accumulated in the development of technological processes for obtaining various types of chlorine-free, water-soluble, complex fertilizers. Of all the known methods, conversion methods for the production of chlorine-free, water-soluble fertilizers - potassium nitrate, sulfate, carbonate and phosphate - have a particular interest, since all the initial substances for the implementation of these processes are produced at the chemical enterprises of the republic. [3-11]

In work [12], in order to increase the degree of conversion and purity of the final product, contacting is carried out in a device consisting of two columns and two intermediate containers having a ratio of useful volumes equal to 1: (0.3-0.35), connected pipelines in sequence, the top of one column is an intermediate tank from another column, by processing in the first column of the K-form of the cation exchanger, supplied from above with a solution of ammonium nitrate, in the second column NH4 - the form of the cation exchanger of potassium chloride solutions with the outputs of the obtained solutions of potassium nitrate and ammonium chloride.

In work [11], the yield of the desired product has been increased by the flotation method. This is achieved in the process of interaction of sodium nitrate and potassium chloride at 80-90 ° C, cooling the reaction mixture for 3.5-4 hours and isolating the desired product by flotation. A mixture of octadecylamine in the amount of 50-75 g / t of salts is used as a flotation reagent. The method allows increasing the yield of potassium nitrate to 98% [10-11].

In [2], in order to simplify the process and reduce energy consumption for the product yield, potassium nitrate and ammonium chloride are crystallized separately. However, the conversion process, the crystallization of potassium nitrate and ammonium chloride takes a long time; 5; 5 and 2 hours at 100 ° C, 0 and 60 ° C, respectively.

2. METHODOLOGY

The conversion process was carried out for 1-5 minutes in an isothermal reactor at a maximum temperature of 90 °C.

The following physicochemical methods of analysis were used in the studies: electron-microscopic, thermoanalytical, and X-ray phase analysis.

The morphology and microstructure of the samples were measured using a SEM - EVO MA 10 scanning electron microscope (Carl Zeiss, Germany); the local elemental composition of the powders was determined using an EDX energy-dispersive elemental analyzer (Oxford Instrument). During sample preparation, the sample was dried and mounted on a microscope stage, over which aluminum foil with a double-sided adhesive was glued. Powder was glued onto this foil, then the object stage was installed in the working chamber of the microscope, from which air was evacuated to create a vacuum. For measurements, an accelerating voltage of 10 kV was applied to the filament. At the same time, the working distance was 8.5 mm. Local elemental analysis was obtained at a scale of 100 μm using the Aztec Energy Advanced software [17-18].

TG-DSC conditions: Thermoanalytical studies of the presented samples were carried out on a Netzsch Simultaneous Analyzer STA 409 PG (Germany) with a K-type thermocouple

(Low RG Silver) and aluminum crucibles. All measurements were carried out in an inert nitrogen atmosphere with a nitrogen flow rate of 50 ml / min. The temperature range of measurements was 25-370 °C; the heating rate was 5 k / min. The amount of sample per measurement is 5-10 mg. The measuring system was calibrated with a standard set of substances KNO_3, In, Bi, Sn, Zn [19-20].

Conditions for X-ray phase analysis: Identification of the samples was carried out on the basis of diffraction patterns that were recorded on a XRD-6100 apparatus (Shimadzu, Japan), computer-controlled. CuKα radiation (β filter, Ni, 1.54178, current mode and tube voltage of 30 mA, 30 kV) and a constant detector rotation speed of 4deg / min in increments of 0.02 deg were used. (ω / 2θ coupling), and the scanning angle varied from 4 to 800 [21-22].

3. RESEARCH RESULTS

In this work, to study the possibility of obtaining potassium nitrate by the conversion method, based on the analysis of the solubility diagram of the K +, NH^{4+}, // Cl-, NO_3 — H_2O system, the optimal range of variation of technological parameters has been selected.

The influence of the KCl: NH_4NO_3 ratio, the temperature and duration of conversion, as well as the kinetics of crystallization at temperatures of 5, 10 and 20 ° C have been studied.

After conversion, the system was cooled to a certain crystallization temperature with stirring at a mixer speed of 50-100 rpm and cooling at 2-14 ° C / min.

The resulting solid product and liquid phase were analyzed for the content of K +, Cl- and nitrogen in the form of ammonium nitrate according to a well-known method. [10-11].

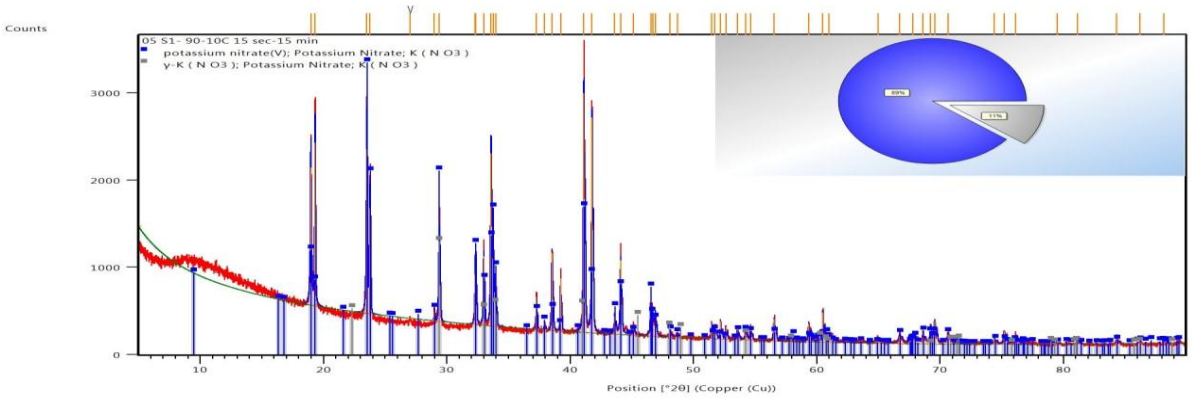
As shown by experimental studies, when the conversion solutions are cooled, potassium nitrate crystals precipitate in the system with the formation of a suspension, the W: T ratio in which fluctuates in the range 3.42-7.44, depending on the technological conditions of crystallization. Within 10 minutes, the degree of color of the suspension reached more than 70%.

Table 1
Influence of technological parameters on analytical indicators of potassium nitrate obtaining

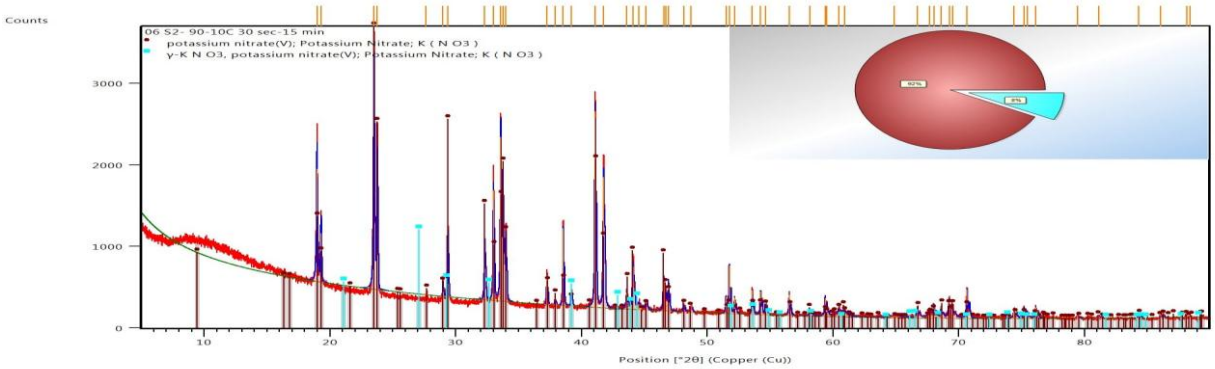
As the obtained data show (Table 1), the degree of K2O yield fluctuates in the range of 29.49-54.53%, the values of which are practically not affected by the conversion duration, since its increase from 15 to 2400 minutes increases the yield by only 2.53%.

	Conversion ratio KCl / NH_4NO_3	Continuator conversion rate, min	Temp chris thal lization, ^0C	Duration cryst allization, min	Filtration rate, kg / m^2 * h by solid phase	Moisture solid phase,%	L:S	K_2O yield, %
.		1	5	15	2 695,06	12,11	4,84:1	44,34
.		10			1 694,94	15,60	4,62:1	47,40
.		1		30	1 087,80	10,03	4,52:1	48,31
.		10			1 894,90	10,22	4,02:1	50,54
.		15сек	10	15	2 659,68	9,68	5,44:1	48,31
.		30сек			3 062,65	12,85	5,36:1	50,90
		45сек			3 103,78	12,86	5,22:1	51,76
	1:1	1			1 774,86	12,73	4,38:1	47,40
		10			2 213,85	12,87	4,14:1	47,45
0		1		30	1 884,86	14,21	4,70:1	48,84
1		10			2 173,56	14,28	4,31:1	49,97
2		1	20	15	1 576,43	16,85	5,26:1	38,15
3		10			1 801,17	19,89	5,60:1	38,49
4		1		30	1 380,75	10,57	5,49:1	38,93
5		10			1 585,39	11,67	5,15:1	40,75
6		5	5	15	1 264,15	9,14	4,63:1	46,51
7		10			1 284,13	9,76	3,90:1	49,85
8		20			1 561,21	9,86	3,75:1	46,44
9		40			1 380,06	9,18	3,52:1	52,86

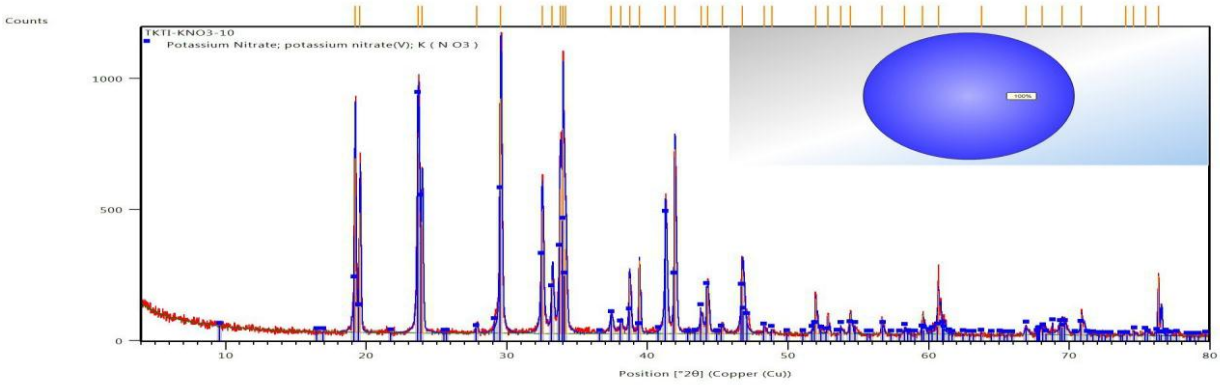
0	**1,09:1**	1	5	30	814,97	5,52	4,06:1	52,00
1		5			1128,13	6,35	3,98:1	52,96
2		10			1142,86	7,3	3,91:1	53,05
3		20			1066,31	7,52	3,93:1	53,24
4		40			1246,89	7,3	3,93:1	54,53
5		5	10	15	1546,12	14,0	4,87:1	44,40
6		10			1185,79	10,72	4,98:1	42,07
7		20			953,58	10,59	4,97:1	44,31
8		5	10	30	1404,97	8,37	4,5:1	46,33
9		10			952,27	9,5	5,3:1	43,64
0		20			1274,98	12	5,35:1	43,36
1	**1,2:1**	1	5	15	1224,67	13,29	4,8:1	44,66
2		10			1863,05	15,05	4,47:1	41,09
3		1	5	30	1009,33	9,12	5,52:1	43,25
4		10			2425,56	16,39	4,94:1	45,78
5		1	10	15	1553,66	12,83	5,83:1	40,64
6		10			1780,08	17,58	5,98:1	35,52
7		1	10	30	1251,42	12,26	5,63:1	39,18
8		10			1458,92	14,73	5,52:1	39,63
9		1	20	15	1682,04	18,16	7,39:1	29,52
0		10			1000,72	15,06	6,96:1	31,41
1		1	20	30	1131,56	12,4	7,44:1	30,32
2		10			1345,18	15,73	6,83:1	31,78


Counts
05 S1- 90-10C 15 sec-15 min
potassium nitrate(V); Potassium Nitrate; K (N O3)
y-K (N O3); Potassium Nitrate; K (N O3)
3000
2000
1000
0
10
20
30
40
50
60
70
80
Position [°2θ] (Copper (Cu))

а)


Counts
06 S2- 90-10C 30 sec-15 min
potassium nitrate(V); Potassium Nitrate; K (N O3)
y-K N O3, potassium nitrate(V); Potassium Nitrate; K (N O3)
3000
2000
1000
0
10
20
30
40
50
60
70
80
Position [°2θ] (Copper (Cu))

б)


Counts
TKTI-KNO3-10
Potassium Nitrate; potassium nitrate(V); K (N O3)
1000
500
0
10
20
30
40
50
60
70
80
Position [°2θ] (Copper (Cu))

в)

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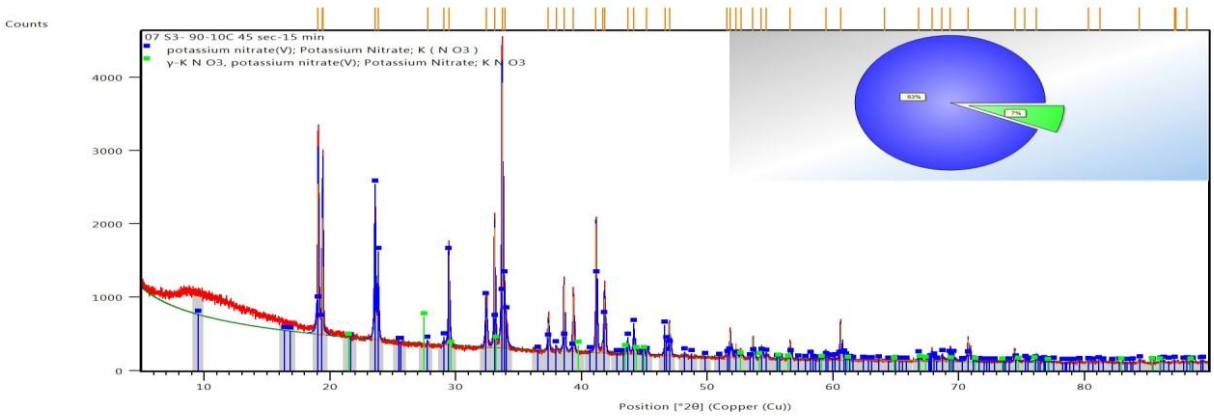

г)

Fig. 1. X-ray diffraction patterns of potassium nitrate obtained with the duration of the conversion process, (sec): a-15; b-30; в-30; g-60.

The duration of the conversion has a significant effect on the crystal structure of the resulting potassium nitrate X-ray studies (Fig. 1) showed that with an increase in the duration of conversion from 15 to 60 sec, the content of the γ-form of potassium nitrate crystals decreases from 12 to 0%. Consequently, at the beginning of the process, the γ-form crystallizes, which gradually turns into the α form.

At a temperature of 5 ° C, a maximum clarification degree is observed. However, an increase in the crystallization temperature of more than 10 ° C at a KCl: NH_4NO_3 ratio leads to a decrease in the K_2O yield from 5.23 to 33%. It should be noted that when the ratio KCl:NH_4NO_3 = 1.2: 1, the K_2O yield decreases by more than 6.5% compared to KCl: NH_4NO_3 = 1.09: 1.

It was found that the maximum yield of K2O is observed at a ratio of KCl: NH_4NO_3 = 1.09: 1 at a temperature of 5 ° C and fluctuates within 46.51-54.53%, and at 10 ° C this indicator decreases to 42.07-46.33 %.

The filtration rate of the samples under the same conditions is quite high (Table 1) and amounts to 1066.31-1561.21 kg / m2 h, therefore, the moisture content of raw potassium nitrate crystals does not exceed 9.90 and 14.30% at 5 and 10 ° C, respectively.

Studies of crystallization processes and filtration rate showed that with an increase in the number of nucleus from 5 to 20% relative to the mass of the solid phase. The filtration rate and the relative yield of K_2O increase by 15-20 kg / m2 h and 2-3%, and the W: S ratio decreases from 4.38; 4.14 to 3.30; 3.01, which duration of conversion is from 1 to 10 minutes, respectively.

The moisture content of the products after filtration is 8.87-12.87%, the chlorine content in the dry unwashed product ranges from 1.7-30%. These indicators primarily depend on the granulometric composition and structure of crystals.

Analytical sieving machines of the AS 200 series, controlled by an electromagnetic drive EP 0642844, have been used to determine the particle size distribution of the products. It was found that all samples contained particles with average diameters from 45 to 1000 μm. Prevailing is the presence of particles with a diameter of 250 μm in the product (Fig. 2), where the proportion of these particles at the KCl: NH4NO3 ratios of 1.09: 1 and 1: 1 is 40 and 50%, respectively.

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At a cooling temperature of 5 oC and a decrease in the cooling rate of the product from 7.1 to 2 oC / min (Fig. 2a), the content of particles with a diameter of 500 μm increases simultaneously with a decrease in the proportion of particles with a size of 125 μm from 32 to 22%.

At 10 oC, the same regularity is observed, i.e. the proportion of particles with a size of 500 μm increases to no more than 30%, and the proportion of particles with a size of 125 μm decreases to 21% by cooling to 20 ° C (Fig. 2b).

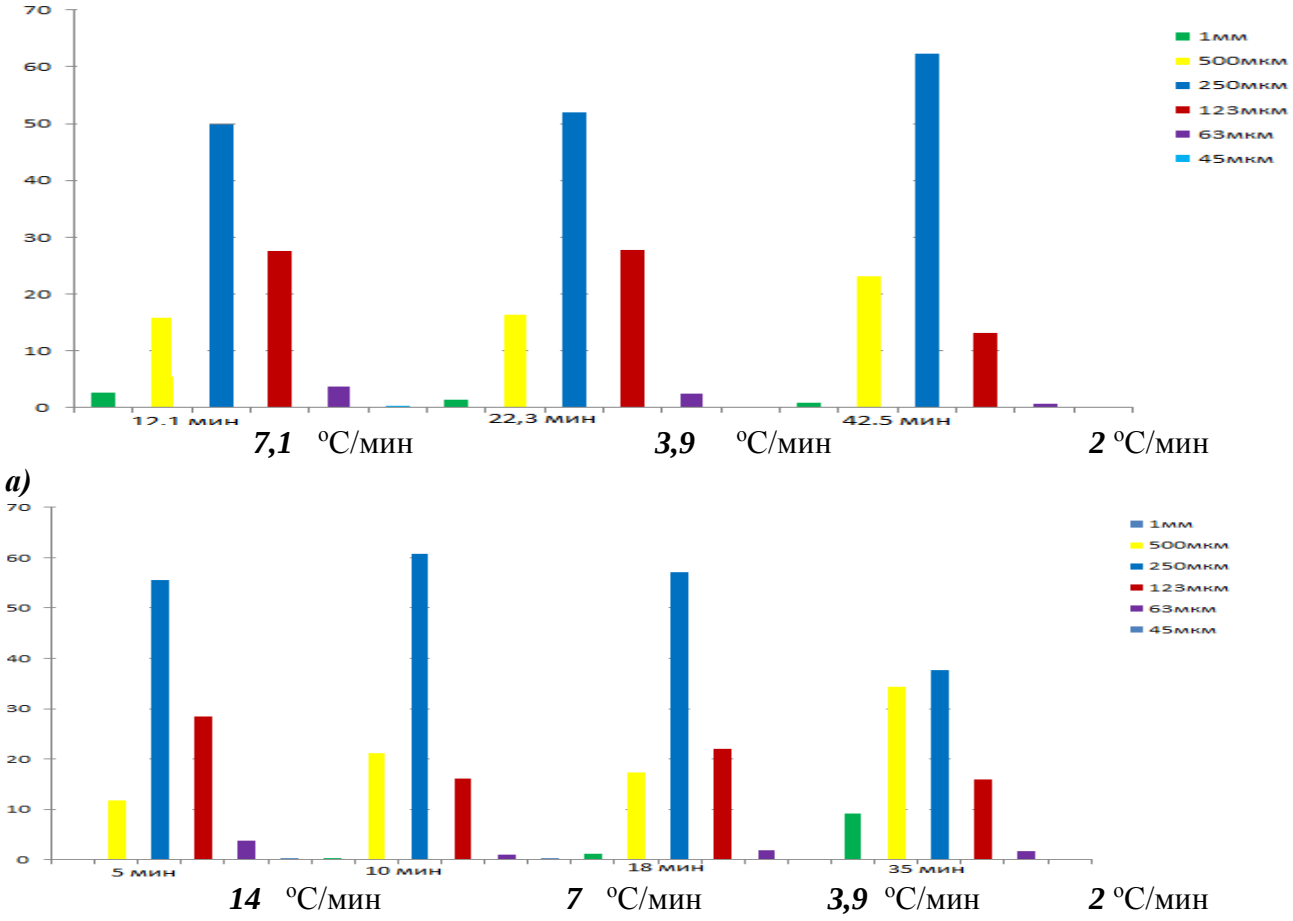

Fig2. Influence of technological parameters of the crystallization process on the granulometric composition of potassium nitrate crystals at KCl: NH_4NO_3 = 1: 1, and

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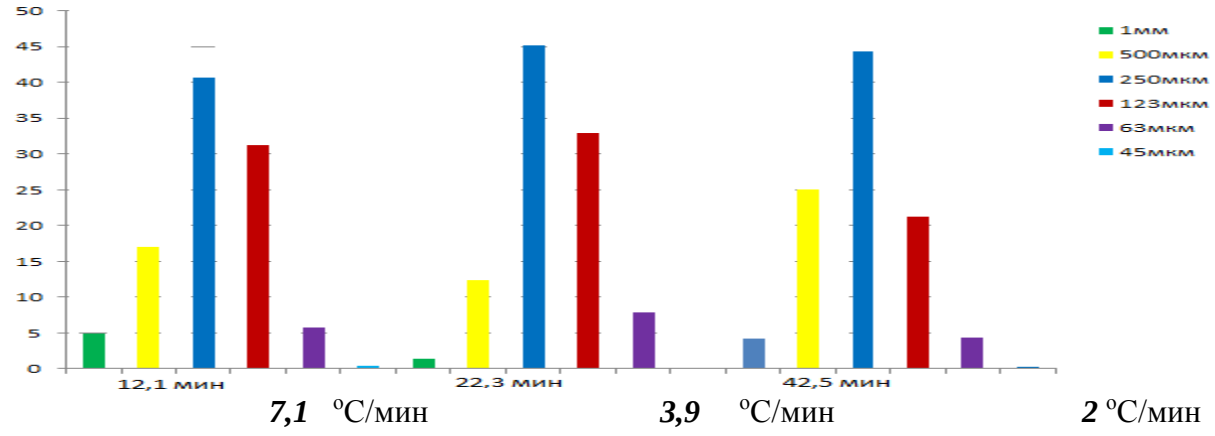
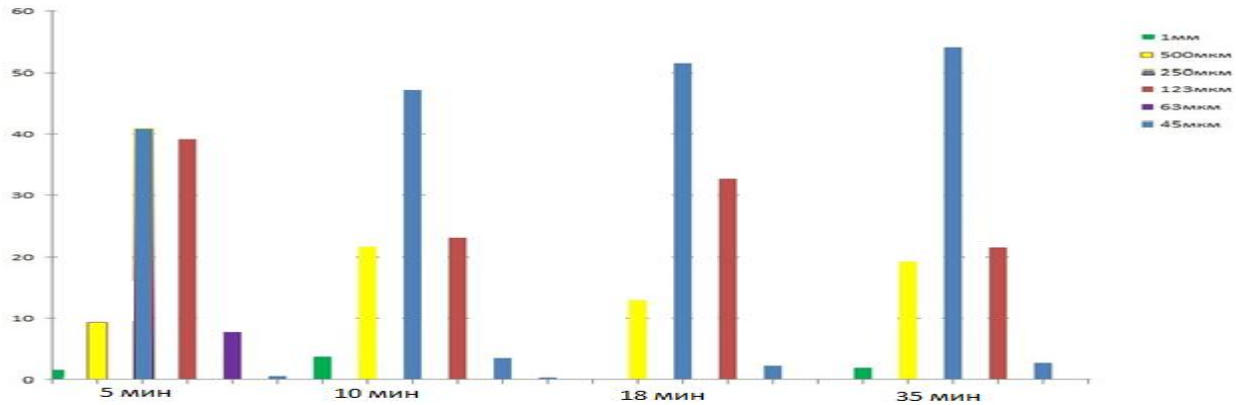
temperature, °C: a-5; b-20 °C.



7,1 °C/мин ***3,9*** °C/мин ***2*** °C/мин

а)



14 °C/мин ***7*** °C/мин ***3,9*** °C/мин ***2*** °C/мин ***б)***

Fig3. Influence of technological parameters of the crystallization process on the granulometric composition of potassium nitrate crystals at KCl: NH_4NO_3 = 1.09: 1 and temperature, °C: a-5; b-20 °C.

With an increase in the duration of the process, the fraction of particles with a size of 500 mcm practically does not change with a continuous increase in the content of a particle with an average diameter of 250 μm.

With a decrease in the cooling rate from 7.1 to 2 ° C / min, the proportion of particles with a size of 500 mcm is more than 20%, and 250 μm is more than 60%, due to a decrease in the proportion of other fractions. (fig. 3a).

When cooled to 20 ° C with cooling progress from 5 to 35 minutes, the content of particles with a size of 500 mcm in the product increases by 30% or more, while the proportion of other fractions in the product decreases.

To determine the morphology of crystals and their sizes, an optical and scanning electrode microscope SEM-EVOMA10 (Zeiss, Germany) has been used.

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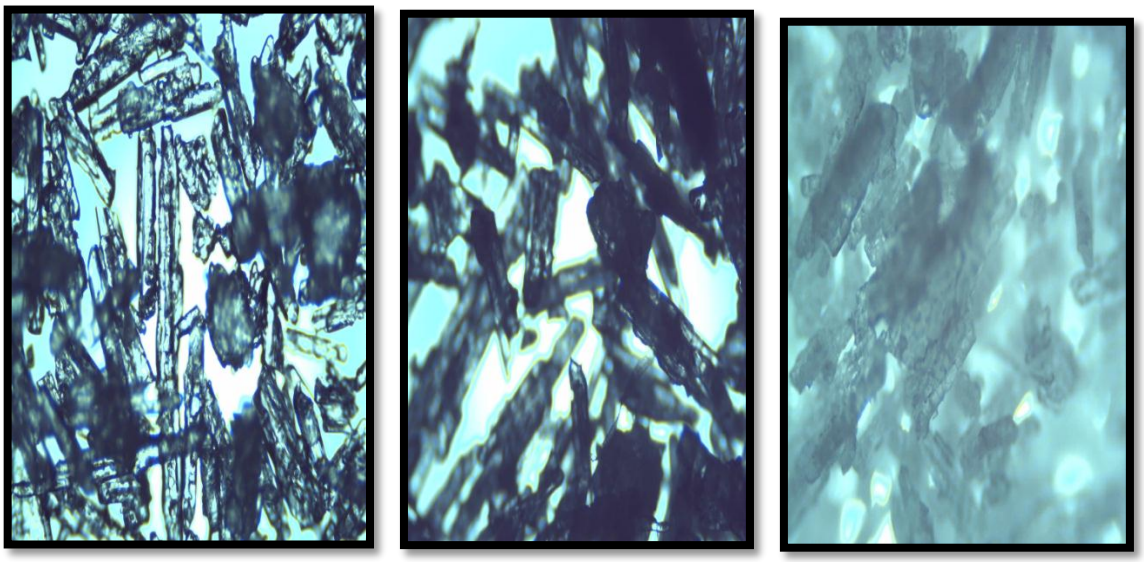
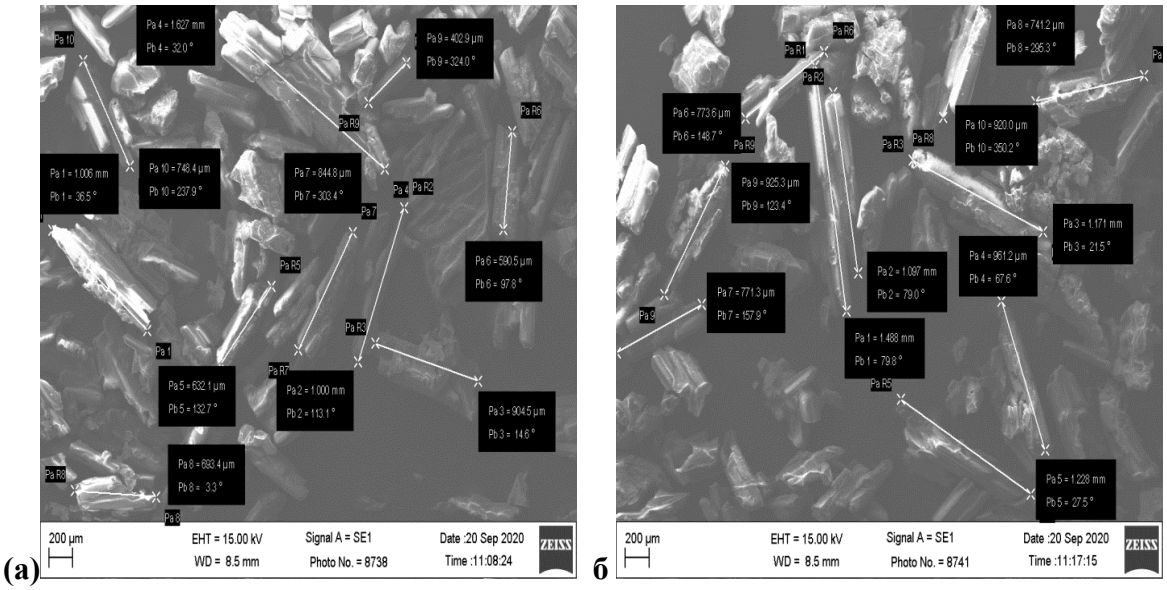


а) **(б)** **(с)**

Fig. 4. Micrograph of potassium nitrate crystals after drying at a temperature of 20 °C (a), 132.8 °C (b) and 338 °C (c) conversion duration - 1 min (optimal).



(а) **б**

Fig. 5. Morphological studies and particle sizes of potassium nitrate powder.

According to the obtained data (Fig. 4.5), the length of the particles reaches up to Pa4-1.627 mm in the studied intervals of variation of technological parameters, large prismatic crystals of potassium nitrate with the size Lxhxb- 0.125: 1.627 x 0.1-0.5 x 0.1-0 3 mm are formed, (fig. 4.5).

In all experiments, large crystals were formed and therefore the filtration rate was at least 814.97 kg / m2 * h, which provides a high degree of crystal washing from chlorine to its content of less than 0.3%

Due to the fact that potassium nitrate is a dangerously explosive substance, the drying process at various temperatures and their effect on the chemical and mineralogical composition

of products have been further studied. For this purpose, thermoanalytical studies of the obtained products have been carried out on a Netzsch Simultaneous Analyzer STA 409 PG device (Germany), with a K-type thermocouple (Low RG Silver) and aluminum crucibles.

All measurements have been carried out in an inert nitrogen atmosphere with a nitrogen flow rate of 50 ml / min. The temperature range of measurements was 25-370 ° C, the heating rate was 5 K / min. The amount of sample per measurement is 5-10 mg. The measuring system was calibrated with a standard set of substances KNO3, In, Bi, Sn, Zn. The analysis revealed that in the temperature range of 20-320 ° C one endothermic peak is observed without changing the sample mass, which it characterizes a phase transition (the first order - a transition from a metastable crystalline state to a stable state Tmax = 132.3oC and ΔQ = -53.27 J / g) (fig. 6).

The second peak (Tmax = 338.4oC and ΔQ = - 73.84 J / g) has been determined due to the melting and partial decomposition of the sample. The decomposition process begins after 330 ° C and proceeds with a weight loss of 0.96% up to 340 ° C.

Taking into account the above, the process of thermal treatment of potassium nitrates obtained by conversion has been carried out at 133 and 339 ° C in 30 min. Original potassium nitrate and heat treatment products have been analyzed to determine the chemical composition (Table 2).

Table 2
The chemical composition of heat treatment products

№	Heat treatment at t, °C	Content of components, wt%				
		K^+	NO_3^-	NO_2^-	Cl^-	NH_4^+
1	Original	39,09	59	-	0,39	0,7
2	132,8	31,82	62	0,002	0,64	0,7
3	338,2	34,54	62	0,01	0,14	0,1

Chemical analysis data show that with an increase in temperature, the content of nitrite nitrogen increases to 6% due to the thermal decomposition of potassium nitrate located on the surface of crystals (Fig. 4, Table 3).

To determine the mineralogical composition, the diffraction patterns of the heat treatment products have been identified (Fig. 6, Table 3), which were recorded on an XRD-6100 apparatus (Shimadzu, Japan) controlled by a computer.

CuKα radiation (β filter, Ni, 1.54178, current mode and tube voltage 30 mA, 30 kV) has been used and a constant detector rotation speed of 4 deg / min with a step of 0.02 deg. (ω / 2θ-adhesion), the scanning angle varied from 4 to 80 °.

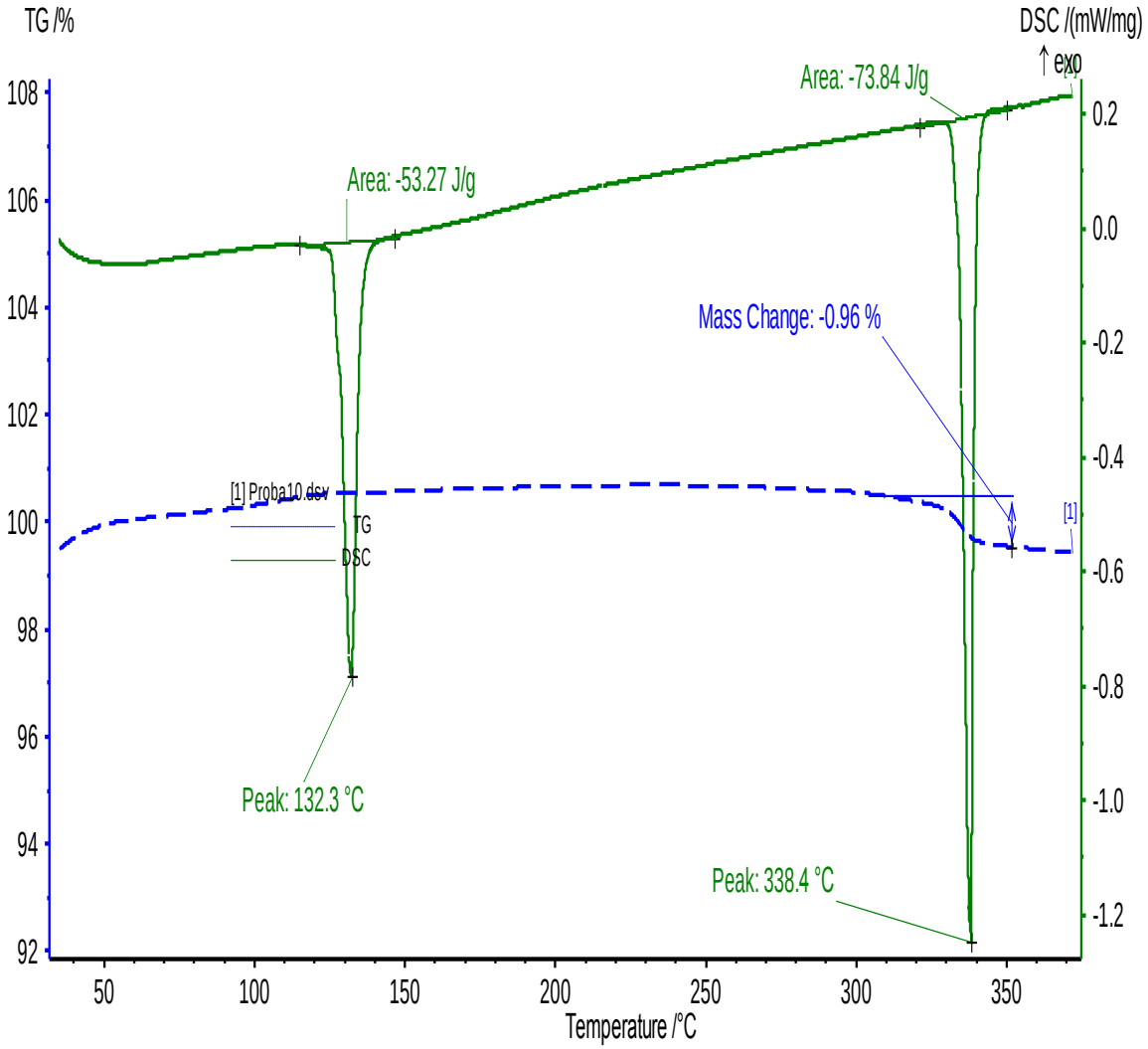
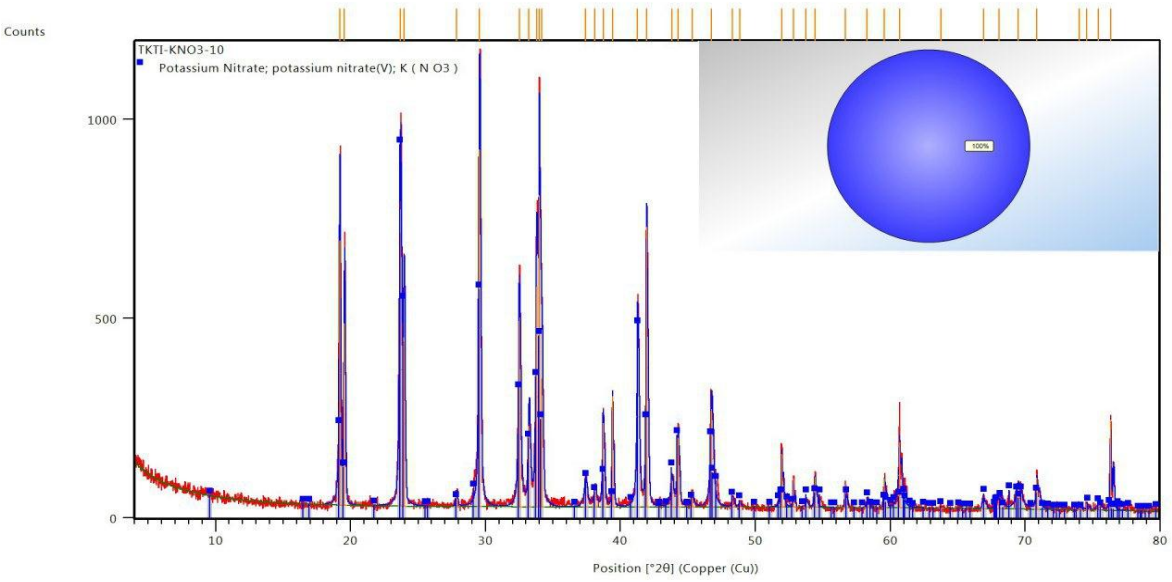
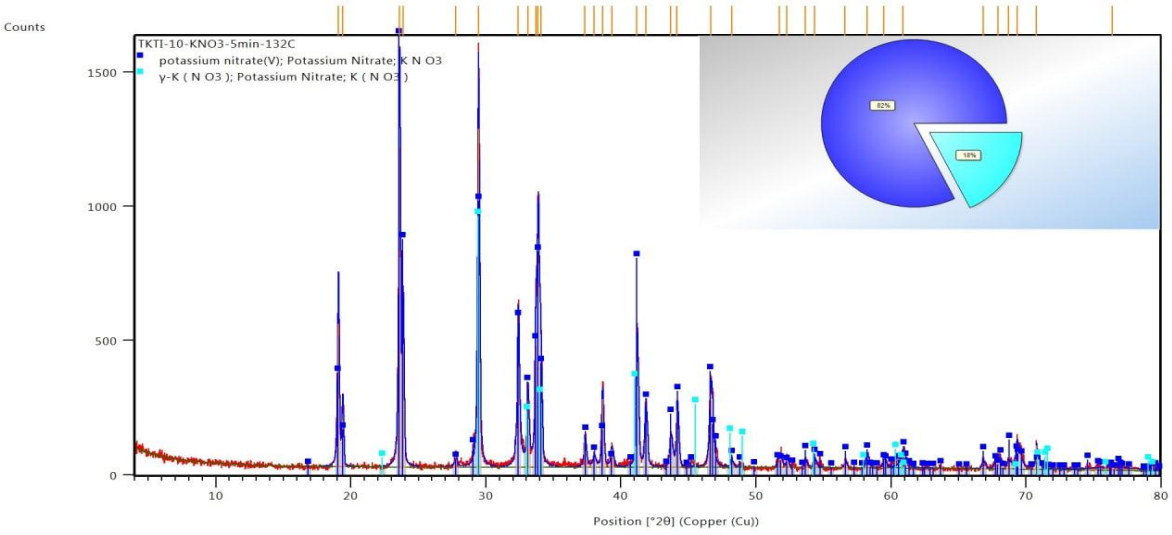


Fig. 6. TG-DSC curve of potassium nitrate obtained under optimal conditions.

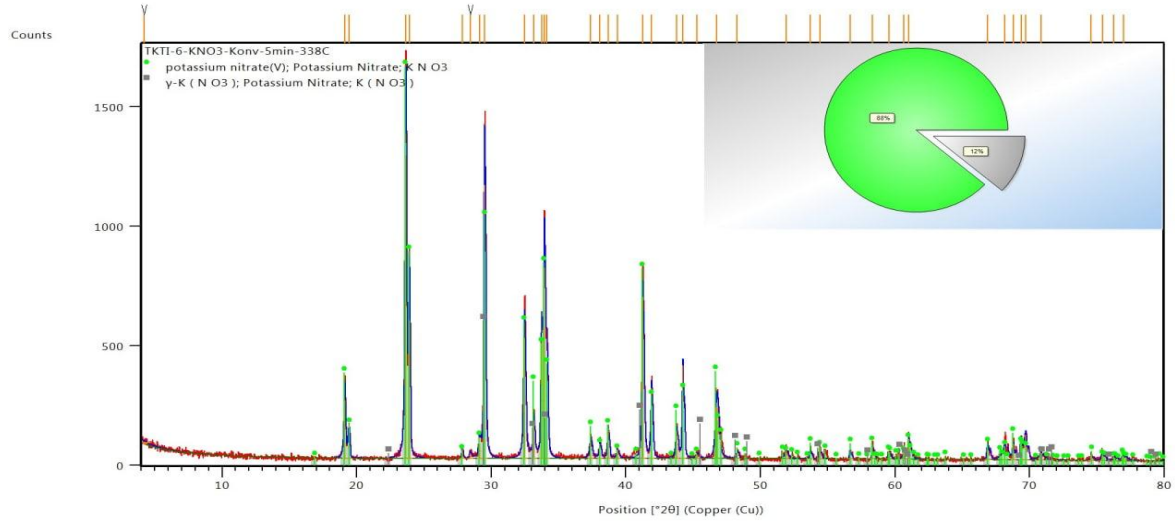

Counts
TKTI-KNO3-10
Potassium Nitrate; potassium nitrate(V); K (N O3)
100%
1000
500
0
10
20
30
40
50
60
70
80
Position [°2θ] (Copper (Cu))

а)


Counts
TKTI-10-KNO3-5min-132C
potassium nitrate(V); Potassium Nitrate; K N O3
γ-K (N O3); Potassium Nitrate; K (N O3)
82%
18%
1500
1000
500
0
10
20
30
40
50
60
70
80
Position [°2θ] (Copper (Cu))

б)

№	20 °C		132 °C		338 °C	
	D	J/J_o	D	J/J_o	D	J/J_o
1	4,617	70	4,650	43	4,640	19
2	4,535	53	4,576	16	3,760	100
3	3,751	82	3,767	97	3,720	50
4	3,711	51	3,728	40	3,024	88
5	3,019	100	3,031	100	2,755	41
6	2,752	50	2,761	39	2,670	11
7	2,694	23	2,704	19	2,654	39
8	2,649	64	2,657	43	2,639	64
9	2,635	82	2,643	62	2,624	31
10	2,618	33	2,627	25	2,186	53
11	2,323	22	2,328	20	2,154	23
12	2,284	26	2,189	35	2,046	30
13	2,185	49	2,157	18	1,943	21
14	2,152	74	2,049	18	1,937	11
15	2,044	20	1,946	23		
16	1,942	29	1,940	11		
17	1,759	15				
18	1,525	24				
19	1,246	24				



c)

Fig. 7. X-ray diffraction pattern of potassium nitrate crystals heated to a temperature of 20 ° C (a), and 132.8 (b) ° C 338, ° C (c).

Intensities of peaks between planar distances of potassium nitrate
Table 3

Analysis of diffractograms (Fig. 7) showed that with increasing temperature, new crystalline β and γ forms of potassium nitrate appear in the samples and their content increases

from 3, 18 to 13 and 15%, respectively, the form increases not only due to the γ form and due to the crystalline modification α form.

At room temperature, the α-form is stable, having rhombic symmetry with the unit cell dimensions a = 5.4142 A, ˚ b = 9.1654 A, ˚ c = 6.4309 A, ˚ Vc = 319.122 A˚ 3. When heated to 4030 K, the α-phase passes into the β-phase with rhombohedra symmetry. By cooling, the β-phase first transforms to 397 K into a new γ-phase with a ferroelectric trigonal structure and only then at 383 K into the α-phase [16].

4. CONCLUSION

The results of the conducted studies show that it turns out that the temperature in the range of 5-20 ° C practically does not affect the duration of the conversion of potassium chloride by ammonium nitrate. At a cooling rate of 2.0-7.1 ° C / min, to temperatures of 5-10 ° C, large prismatic crystals are formed within no more than 30 minutes, providing a high filtration rate of 1064.3-1561.2 kg / m2h with a product yield of more than 54 , 53% with a content of not more than 0.3% chlorine.

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