Annals of R.S.C.B., ISSN:1583-6258, Vol. 25, Issue 2, 2021, Pages. 3164 - 3170
Received 20 January 2021; Accepted 08 February 2021.

# Obtaining Potassium Nitrate by the Conversion Method

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**Abstract.** The results of a study of the physicochemical parameters of potassium nitrate production by the method of conversion of magnesium nitrate with potassium chloride are presented. The optimal technological parameters for the production of potassium nitrate. On the optimal conditions for obtaining potassium nitrate, as well as the ratio of reacting substances brucite in nitric acid with the formation of magnesium nitrate, followed by the conversion of magnesium nitrate with a liquid of potassium chloride are presented.

***Keywords:****brucite, magnesium hydroxide, nitric acid,potassium chloride, magnesium nitrate, potassium nitrate,bischofite liquid.*

## INTRODUCTION

In the potash industry, an urgent task is to significantly increase the chlorine-free potash fertilizers production.Potassium nitrate is widely used in agriculture and contains two essential nutrients, potassium and nitrogen.At present, in our country, the need for potash fertilizers is satisfied by potassium chloride, which contains the undesirable element chlorine, and effective potassium fertilizers such as potassium sulfate and potassium nitrate are produced in small quantities, as a result of which the country's need for potash fertilizers is satisfied by foreign manufacturers.In the Republic of Uzbekistan, the need for potash fertilizers is satisfied by potassium chloride, which contains the undesirable element chlorine, as well as effective potassium fertilizers, like potassium sulfate and nitrate, are produced in small quantities, as a result of which they have to be imported.

Currently, there are no large producers of these fertilizers in the cities of Uzbekistan, which is due to the lack of a developed raw material base for obtaining chlorine-free potash fertilizers.Basically, to obtain potassium nitrate, conversion methods are used [1], based on the interaction of throw with potassium chloride according to the reaction:

$$Mg(NO_3)_2 + 2KCl \leftrightarrow 2KNO_3 + MgCl_2 \quad +Q \qquad (1)$$

Out of these potassium salts as raw materials, the greatest interest is potassium chloride - KCl, which is the most common mineral from the nitrate group, as well as a large tonnage of potassium chloride in the production of potash fertilizers.

The basis of the above process is the salts solubility in the $K^+$, $Mg^{2+}$//$CI^-$, $NO_3^-$ system. The components of this system have been studied quite well in a wide temperature range [1].

Potassium chloride is an extremely functional mineral fertilizer involved in the basic physiological processes of crop growth.The potassium consumption per unit of the formed crop is significantly higher than those other elements of mineral nutrition. This is especially true for crops that form a large amount of sugar, starch, fat; their potassium content reaches 6-8%.In vegetable and fruit crops with the use of potassium chloride, the fruits setting and formation sharply increases, their taste, aroma and keeping quality improve.In potatoes, the amount of starch in the tubers increases.The amount of sucrose in the sugar beet roots increases. In fruits and berries, the content of vitamin C, as well as pectin substances, increases.Cotton, wheat, barley, rye, rice, potatoes, sugar beets, sunflowers, buckwheat, millet, some types of vegetables and other crops are the most demanding for the consumption of potassium and giving the highest effect from its use.

At this time, mastering the potassium nitrate technology production is one of the urgent tasks in the mineral fertilizer industry of Uzbekistan.The introduction of this technology into production makes it possible to obtain potassium nitrate at low prices in comparison with foreign

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analogues.In addition, potash nitrate localization is carried out, which largely plays a role in the development of agriculture, hence the economy of Uzbekistan [2].

Another important requirement for complex fertilizers for greenhouses is the absence in their harmful impurities composition, in particular chlorine, since most vegetable crops are chlorophobic.At the same time, potassium chloride, which contains up to 47% chlorine, is used as a potassium source in the most industrial complex fertilizers. For this reason, such complex fertilizers cannot be used in greenhouses [3].

Potassium nitrate is industrially obtained in various ways from potassium-containing substances [4,5]. In many methods, the potassium nitrate yield does not reach 90%, and the secondary products utilization formed in the process is one of the main production problems.The proposed method is considered waste-free competitive production. The magnesium chloride solution released as a by-product is used for the production of magnesium chlorate defoliant, as well as in industrial cooling systems.

Magnesium nitrate in the first stage is obtained in a reactor equipped with a stirrer by the magnesium hydroxide (brucite) interaction and weak nitric acid 321 kg (57%). The reaction between the above substances proceeds with the heat release with constant stirring, the solution boiling is observed.

$$Mg(OH)_2 + HNO_3 \rightarrow Mg(NO_3)_2 + H_2O$$

In the process, brucite is taken in excess of 85 kg (10% by weight) so that the solution has a neutral or slightly alkaline medium in order to avoid the release of elemental chlorine when mixed with potassium chloride.In an acidic environment, potassium chloride forms a very corrosive substance that accelerates the corrosion of metal structures, especially at high temperatures.A hot (with 80-90 °C temperature) magnesium nitrate solution with admixtures of an excessive amount of brucite and acid-insoluble particles is sent to the conversion stage with potassium chloride.

Potassium chloride 245 kg before conversion with magnesium nitrate is dissolved (at 80-90 °C) in an amount of 0.630 $m^3$ of water to obtain a 28% solution of this salt.After complete dissolution of potassium chloride, it is mixed with a magnesium nitrate solution, as a result of which potassium nitrate and magnesium chloride solutions are formed in the system.

$$Mg(NO_3)_2 + 2KCl \leftrightarrow 2KNO_3 + MgCl_2 \quad +Q$$

The solution is filtered on a suction filter to remove excess brucite and other impurities, which are returned back to the neutralization stage.

## EXPERIMENTAL

**Experimental reagents and instruments.** For research, brucite was used in terms of dry matter of the following composition (wt%):$Mg(OH)_2$ – 87.0; CaO – 3.0;SiO – 3.0; $Fe_2O_3$ – 0.5humidity – 1.0. To establish the physicochemical parameters of the obtaining potassium nitrate process, magnesium chloride solution, scientific research was carried out in laboratory conditions.A glass reactor equipped with a mechanical stirrer and a jacket for heating and cooling was used for the experiments.

In the studies, brucite ($Mg(OH)_2$) of Russian production, nitric acid (with 56-58% concentration) of “Fargonaazot” JSC and potassium chloride from Dekhkanabad potassium chloride were used as starting materials.

Brucite was dissolved in non-concentrated nitric acid to prepare a magnesium nitrate solution. The process proceeds with the heat release, which initiates the solution boiling and the magnesium nitrate formation with 43-45% concentration:

$$Mg(OH)_2 + HNO_3 \rightarrow Mg(NO_3)_2 + H_2O \quad (2)$$

To avoid water foaming and boiling, brucite was neutralized with constant stirring and dispensing brucite in portions.

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**Experimentalmethod.** In the next step, potassium chloride was dissolved by heating water at 80-90°C to obtain a saturated solution (28%) of this salt. At the same time, the mass ratio of potassium chloride to magnesium nitrate was 1.2:1. The excess of potassium chloride makes it possible to shift the conversion equilibrium to the right, thereby reducing the number of nitrates in the mother liquor.

Further, the prepared potassium chloride solution was added to the magnesium nitrate solution at 80-90°C temperature. The reaction between these substances is exothermic, accompanied by heat release:

$$Mg(NO_3)_2 + 2KCl \leftrightarrow 2KNO_3 + MgCl_2 + Q \quad (3)$$

After some time, equilibrium will come to this system. The starting materials and the salts solubility formed are not very different from each other, and they all dissolve well in water.

After mixing magnesium nitrate and potassium chloride, after a certain time, the salt ions are in equilibrium. In order to shift the equilibrium towards the target products, one of the formed salts must be removed from the system. Magnesium chloride crystallizes poorly; upon saturation it forms very small crystals, which later turn into a monolithic mass.Potassium nitrate can be recovered from the system in the large crystals form (crystallization degree 90-95%).

*Table 1.***Temperature dependence of salt solubility**

| **Solubility,indegree** | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| Name | 10°C | 20°C | 30°C | 40°C | 50°C | 60°C | 70°C | 80°C | 90°C |
| $KNO_3$ | 17,3 | 24 | 31,4 | 39 | 44 | 52 | 58 | 62,8 | 66,9 |
| $MgCl_2$ | 36 | 36,8 | 37,6 | 38 | 38,6 | 39,2 | 39,6 | 40 | 42 |
| $Mg(NO_3)_2$ | 41 | 42,3 | 43,6 | 44,8 | - | - | - | - | - |
| KCl | 23,7 | 25,4 | 27,1 | 28,6 | 29,9 | 31,3 | 32,6 | 33,8 | 35,1 |

After mixing magnesium nitrate and potassium chloride, the solution is filtered from insoluble substances. The solution after mixing magnesium nitrate and potassium chloride contains acid-insoluble particles, an excess of brucite, as well as impurities coming with potassium chloride. Unwanted impurities remain on the surface of the filter material and are returned to the brucite neutralization stage with weak nitric acid.

The purified solution after filtration was evaporated until a moisture content of 20-22% of water was reached at 110-112°C until the solution was saturated. Then the solution was cooled to 20°C for complete precipitation of potassium nitrate crystals. After cooling, the crystals were separated from potassium nitrate on a vacuum filter, then the product was dried in an oven at a temperature of 55-60°C.

## RESUTSANDDISCUSSION

At the preparation stage of a magnesium nitrate solution, the solution must have a neutral or slightly alkaline medium. In the acidic solution case, the remaining nitric acid in the conversion stage of potassium chloride with magnesium nitrate did not react, releasing nitrosyl chloride according to the following scheme:

$$KCl + HNO_3 \rightarrow KNO_3 + NOCl + Cl_2 + H_2O \quad (3)$$

Nitrosyl chloride is a very toxic and corrosive substance, highly corrosive to equipment, as a result of which they quickly fail [6]. And also in order to maintain the necessary pH of the medium, carbonates or hydroxides of alkali metals can be added.

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*Table 2.***Composition of crystals and mother liquors, one stripped off, at different temperatures after cooling**

| Experience№ | Temperature, °C | Motherliquor, at % | | $KNO_3$crystals, at % | |
|---|---|---|---|---|---|
| | | $Mg^{2+}$ | $Cl^-$ | $Mg^{2+}$ | $Cl^-$ |
| 1 | 105 | 4.35 | 13.61 | 2.51 | 7.11 |
| 2 | 106 | 4.52 | 13.75 | 2.50 | 7.33 |
| 2 | 107 | 4.61 | 13.80 | 2.49 | 7.42 |
| 3 | 108 | 4.67 | 13.86 | 2.48 | 7.59 |
| 4 | 109 | 4.75 | 14.12 | 1.16 | 6.45 |
| 5 | 110 | 5.77 | 16.13 | 1.51 | 6.43 |
| 6 | 111 | 5.19 | 14.50 | 1.48 | 6.30 |
| 7 | 112 | 5.94 | 15.48 | 1.56 | 8.13 |
| 8 | 113 | 5.83 | 16.38 | 1.67 | 7.01 |
| 9 | 114 | 5.81 | 16.40 | 1.68 | 7.05 |
| 10 | 115 | 5.80 | 16.41 | 1.69 | 7.09 |

It turns out that the optimal evaporation temperature for this system is in the range of 110-112°C. In this case, the concentration of potassium nitrate reaches 31% of the mass. Below 110°C, the solution is not completely saturated, and above 112°C, other complex salts, for example, carnallite, begin to precipitate [7, 8].Then the solution was cooled to 20°C for complete precipitation of crystals of potassium nitrate. Further cooling of the solution does not significantly affect the crystallization process, it simply increases the energy consumption for cooling, and therefore, cooling below 20°C is considered inappropriate.

*Table 3.***Results of potassium nitrate and mother liquor analyzes**

| Thenameofindicators | $KNO_3$content | | $MgCl_2$ (solution) |
|---|---|---|---|
| | $KNO_3$ (crystals obtained) | $KNO_3$ (washedcrystals) | |
| Number of $Mg^+$ions, at % | 0.72 | 0.09 | 5.78 |
| Number of Cl ions $^-$, at % | 2.33 | 0.3 | 14.9 |
| Basicsubstance, at % | 90.7 | 99.3 | 22 |
| Humidity, at % | 6.1 | 0.26 | - |

It can be seen from the table that the mother liquor of the third sample corresponds to $MgCl_2$ with a concentration of 20-25%, potassium nitrate crystals contain chlorine ions, which are higher than the permissible value. Chlorine ions after cold washing are reduced to the required amount.

After washing, the content of potassium nitrate increases significantly, the chlorine content decreases to the required value, which fully complies with SS 19790-74.

## CONCLUSIONS

In this work, based on the analysis of the, $Mg^{2+}$//$CI^-$,$NO_3^-$solubility diagram, the following interval of the main technological parameters variation is selected; $Mg(NO_3)_2$:KCI=1.2:1; the conversion duration is 5-10 minutes, the crystallization temperature is 5-20°C, the crystallization duration is 15-20 minutes.

The $Mg(NO_3)_2$:KCI ratio effect, the conversion temperature and duration, as well as the kinetics of crystallization at temperatures of 5, 10, and 20°C (tab) was studied.

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*Table 4.***Influence of technological parameters on the potassium nitrate obtaining process by the conversion method**

| № | Conversion ratio KCI/$Mg(NO_3)_2$ | Duration of conversion, min | Crystallizationtemperature, °C | Crystallization duration, min | Filtration rate, кg/$m^2$·h bysolidphase | $K_2O$ yield, % |
|---|---|---|---|---|---|---|
| 1 | 1:1 | 5 | 5 | 10 | 1238.72 | 87.5 |
| 2 | | 10 | | | 1231.66 | 88.2 |
| 3 | | 5 | 10 | 20 | 1221.33 | 88.6 |
| 4 | | 10 | | | 1269.87 | 89.3 |
| 5 | | 5 | 20 | 30 | 1355.71 | 82.1 |
| 6 | | 10 | | | 1377.24 | 81.0 |
| 7 | 1.2:1 | 5 | 5 | 10 | 1362.11 | 93.6 |
| 8 | | 10 | | | 1298.10 | 95.5 |
| 9 | | 5 | 10 | 20 | 1251.06 | 97.2 |
| 10 | | 10 | | | 1288.23 | 98.1 |
| 11 | | 5 | 20 | 30 | 1391.12 | 98.4 |
| 12 | | 10 | | | 1356.42 | 98.7 |
| 13 | 1.5:1 | 5 | 5 | 10 | 1254.23 | 80.1 |
| 14 | | 10 | | | 1212.31 | 81.2 |
| 15 | | 5 | 10 | 20 | 1268.66 | 82.6 |
| 16 | | 10 | | | 1233.52 | 83.9 |
| 17 | | 5 | 20 | 30 | 1301.11 | 85.3 |
| 18 | | 10 | | | 1286.35 | 86.1 |

As the obtained data show, in the studied intervals after the conversion process, regardless of the conversion conditions at 120°C, a solid phase does not form in the system. After a given duration of the conversion process, the system was cooled to a certain crystallization temperature with stirring at a stirrer speed of 60-120 rpm and cooling at 3-5°C/min.The resulting solid product and the liquid phase were analyzed for the content of $K^+$, $Mg^{2+}$, $Cl^-$and nitrogen in the form of nitrate according to the procedures [9, 10].

As a result of a decrease in the potassium nitrate solubility in the system, the latter crystals are formed from a suspension with KCI:$Mg(NO_3)_2$=1.2:1. The clarification degree within 20 minutes reaches more than 93.6-98.7%, depending on the experimental conditions.

In order to obtain industrial samples of potassium nitrate and a magnesium chloride solution, the process was carried out at the section of consumer goods in the Soda workshop.

The technological process for the potassium nitrate production on an industrial scale is developed according to the following sequence:

- dissolving brucite in nitric acid by forming a magnesium nitrate solution;
- the reaction of potassium chloride with magnesium nitrate;
- filtration of the resulting solution;

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- solution evaporation;
- cooling the solution to 20°C to isolate crystals of potassium nitrate;
- separation of potassium nitrate crystals.

After mixing salt solutions, this system is in equilibrium. To obtain the target products, the reaction must be shifted to the right by removing one of the substances of potassium nitrate or magnesium chloride in the form of crystals from the system. In this case, magnesium chloride is impossible and impractical to crystallize in solution.

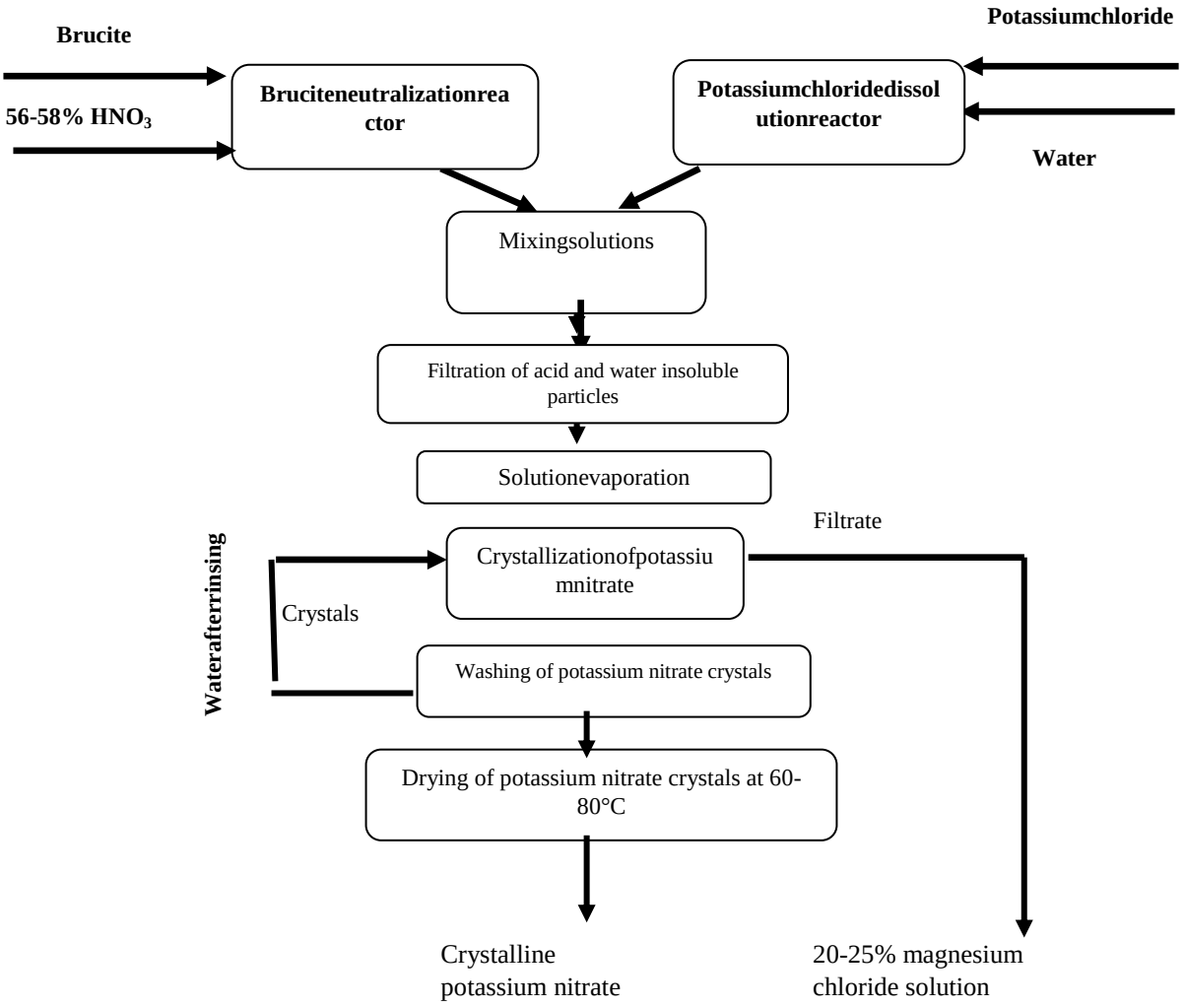


**Figure-1.**Potassium nitrate production processes

In order to crystallize potassium nitrate, the solution is evaporated to saturation with respect to potassium nitrate at a temperature of 110-112°Catatmospheric pressure. Above 112°C, crystals of carnallite begin to precipitate, probably the joint salts of the ions present in the solution.Below 110°C, potassium nitrate is not completely saturated, the yield of potassium nitrate decreases, therefore, the content of magnesium chloride in the mother liquor decreases, and becomes clogged with potassium nitrate.

It has been found that the optimum temperature for evaporation at atmospheric pressure is 111°C. At this temperature, the potassium nitrate crystals yield is maximum, the solution of magnesium chloride after the separation of crystals of potassium nitrate contains few impurities of other salts.

The saturated solution is cooled to 20°C. At this temperature, the yield of crystals (90-93 wt%) of potassium nitrate was obtained (190 kg) using a suction filter. In the case of a suction filter, the product contains 10-15% moisture, if using a centrifuge for filtration, the crystals are drier, and therefore, it contains less impurity of other salts dissolved in moisture. Further cooling leads to

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unnecessary energy consumption, and has little effect on the crystallization process. The sequence of technological processes for obtaining potassium nitrate is shown below in Fig. 1.

Experiments carried out on an industrial scale also show that magnesium chloride with a concentration of 20-25% remains in the mother liquor, with traces of other salts ($KNO_3$, KCl). The amount of the obtained mother liquor was 520 kg.

Upon cooling, the crystals were finely dispersed, which is why the moisture content of the crystals obtained on the Nutsch filter was 35-40%. In addition, humidity was influenced by the effectiveness of the Nutsch filter. According to the calculations based on the experiment carried out at the section of the consumer goods shop of the Soda shop, the losses amounted to 23%.

After washing, the content of potassium nitrate increases significantly, the chlorine content decreases to the required value, which fully complies with SS 19790-74.

## FUNDING

As a result, studies show that the obtained potassium nitrate can be used as a chlorine-free potassium fertilizer. The $KNO_3$content is 98-99%, and the degree of use of the $K^+$ ion is 97-98%. In the proposed method for producing potassium nitrate, as a by-product, a solution of magnesium chloride is used to obtain the defoliant of magnesium chlorate, as well as in industrial cooling systems. This method allows you to get potassium nitrate at a lower cost compared to other methods and is considered a competitive production.

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