

Simple Preparation of High Purity Potassium Nitrate

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Abstract: A new idea and method are discussed in purification of potassium nitrate by adding mixture of potassium hydroxide or potassium carbonate. The results indicate increasing temperature can improve Ca²⁺ and Mg²⁺ to precipitate completely.

Keywords: Potassium nitrate; Purification; Mixed base; Temperature condition

1. Introduction

The energy storage materials and optical fiber communication systems requires a great diversity of high quality inorganic substances. Extra pure potassium nitrate has a significant place among molten salt storage materials. Its quality must satisfy rather stringent requirements on the content of 3d transition metals and Cl⁻[1]. In this connection, a search for simple, highly effective processes for ultrapurification of potassium nitrate is of key importance. Potassium nitrate manufactured in China is of industry grade (93 wt %) and the energy storage materials need high purity potassium nitrate. Analysis of the reported processes for the preparation of extrapure inorganic substances, with application to potassium nitrate, indicates that crystallization from aqueous solutions is of very practical interest. The purity of the product depends on the conditions of the crystallization process and subsequent auxiliary steps (filtration and washing), as well as on impurity content [2-4]. One impediment to the use of crystallization for the ultrapurification of substances is isomorphism (isodimorphism), which favors the formation of host-impurity solid solutions. The formation of a solid solution of micro and macrocomponents depends on a variety of physicochemical factors, the most important of which are ionic radii and charges, the type of chemical bond, and lattice parameters [5]. But it is difficult to control the process. Here we report a simple method to produce high purity potassium nitrate.

2. Experimental Procedure

Industry grade potassium nitrate is gained from Qinghai Salt Lakes (wt 92%) and the other are analytical reagents. The Ca²⁺ and Mg²⁺ were determined by the coordination titration. The chlorine content was by chemical mercurimetry. The pH value used pH meter (Shanghai Fine Instrument Co. Ltd). The Fe³⁺ was determined by atomic absorption spectrophotometry (Shimadzu AA-7000).

An aqueous solution of industry grade potassium nitrate saturated at 25 °C and containing 4 wt % potassium carbonate and 1 wt % potassium hydroxide was boiled down at the boiling point of the solution (113 °C) with stirring for 10 mins, and then, adding solution of potassium hydrate to pH above 12. The aqueous solution was filtered at 113 °C

and adjusted the pH value to 7 with HNO₃ (2 mol/L). Finally, the filtrate was cooled to 25 °C with constant stirring. The precipitated KNO₃ crystals were isolated and thoroughly squeezed between filter papers to remove as much of the mother liquor as possible. Then followed, we took samples for subsequent analysis. The remaining KNO₃ crystals were washed with an aqueous potassium nitrate solution saturated at 25 °C (25 mL of the wash solution for 100 g of the crystals, as selected experimentally) and thoroughly squeezed as described above to remove the wash liquid. The crystals for analysis were dried at 100 °C to constant weight both before and after washing. Finally, we determined the degree of potassium nitrate purification relative to the initial impurity concentration.

3. Results and Discussion

The original contents respectively are 3.88 wt % of Ca²⁺ and Mg²⁺, 0.75 wt % of Fe³⁺ and 2.41 wt % by analysis. We use a single potassium hydroxide or potassium carbonate remove impurity Ca²⁺ and Mg²⁺ (Table 1). Analysis of the results obtained indicates that a single potassium hydroxide or potassium carbonate can't remove impurity completely even if the excess alkali used. The Ca²⁺ and Mg²⁺ impurity concentrations are in the range 3.83 to 1.28 wt %. The reason of the result is the different solubility product (K_{sp}) of produced alkali or salt (K_{sp}(Mg(OH)₂)=5.1x10⁻¹², K_{sp}(Ca(OH)₂)=4.7x10⁻⁶, 25 °C). So it is difficult to remove Ca²⁺ completely because of microsolubility of Ca(OH)₂. The Fe³⁺ and Cl⁻ are easy to remove because additional washing of the crystals improves the removal of the Cl⁻.

Table 1: Impurity contents under different pH values

pH\Impurity (%)	Ca ²⁺ and Mg ²⁺	Fe ³⁺	Cl ⁻
original contents	3.88	0.75	2.41
9(KOH)	2.17	0.15	0.06
9(K ₂ CO ₃)	1.83	0.11	0.06
10(KOH)	2.32	0.11	0.05
10(K ₂ CO ₃)	2.02	0.11	0.05
12(KOH)	1.28	---	0.04
12(K ₂ CO ₃)	1.39	---	0.04
14(KOH)	1.31	---	0.05
14(K ₂ CO ₃)	1.35	---	0.05

In order to remove Ca^{2+} and Mg^{2+} , we tried to use the mixture of potassium hydroxide and potassium carbonate. Interesting, it seemed the solution didn't appear precipitation when added the mixture of potassium hydroxide and potassium carbonate under room temperature. It must heat to precipitate Ca^{2+} and Mg^{2+} . The results obtained saw figure 1. It showed the impurity content gradually decreased with the increase of temperature. When boiled for 10 mins the impurities could be removed completely. This method can not only remove impurities but also easy to filter. We conclude that calcium carbonate and magnesium carbonate are formed at first when adding mixture of potassium hydroxide and potassium carbonate. Because magnesium carbonate is slightly soluble partly magnesium salt still causes impurities. When pH increases the magnesium ions change into insoluble magnesium hydroxide. So the impurities can be removed completely.

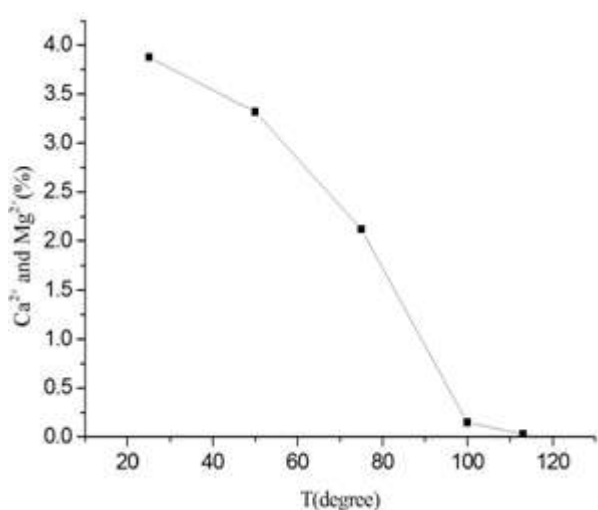


Figure 1: Impurities contents with temperature

4. Conclusion

High purity potassium nitrate can be obtained from a raw material of industry grade by mixture of potassium hydroxide and potassium carbonate to remove impurities. An increase in temperature at constant pressure will help Ca^{2+} and Mg^{2+} to precipitate. A new idea and method are provided for producing high purity potassium nitrate in industry.

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References

- [1] N Dabra, JS Hundal, KC Sekhar, A Nautiyal, Preparation and Characterization of the Ferroelectric Potassium Nitrate: Poly(vinyl alcohol) Composite Films[J], IEEE Transactions on Ultrasonics Ferroelectrics & Frequency Control, 2009, 56, 1627-1633.
- [2] OM Polishchuk, AA Fakeev, VZ Krasil'Shchik, AG Vendilo, Preparation of extrapure potassium nitrate[J], Inorganic Materials, 2012, 48, 836-840.

- [3] Korotkevich, I. B., Sorokina, R.I., and Bomshtein, V.E., Systems Sodium Nitrate-Iron(III) Nitrate-Water and Potassium Nitrate-Iron(III) Nitrate-Water at 25°C[J], Zh. Neorg. Khim., 1982, 27: 2676-2678.
- [4] Polishchuk, O.M., Fakeev, A.A., Potapova, N.V., and Vendilo, A.G., Behavior of Aluminum in Potassium Nitrate Solutions at 25°C, Russ. J. Inorg. Chem. 2008, 12, 521-525.
- [5] VP Danilov, VT Orlova, SK Myasnikov, EA Frolova, DF Kondakov, Effect of reaction conditions on the conversion of calcium nitrate into potassium nitrate[J], Theoretical Foundations of Chemical Engineering, 2008, 42 (5) :579-582.