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**NATIONAL STANDARD OF THE  
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**GB/T 22661.3-2008**

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**Chemical analysis methods of potassium fluoborate - Part 3:  
Determination of potassium fluoborate content - Distillation-sodium  
hydrogen oxidizes titration volumetric method**

氟硼酸钾化学分析方法 第3部分：氟硼酸钾含量的测定 氢氧化钠容量法

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### Foreword

GB/T 22661 "potassium fluoroborate chemical analysis method" is divided into 10 parts.

- Part 1. Preparation and storage of test samples;
- Part 2. Determination of the weight of water in the wet method;
- Part 3. Determination of potassium fluoride boric acid sodium hydroxide volumetric method;
- Part 4. Determination of magnesium by flame atomic absorption spectrometry;
- Part 5. Determination of calcium content by flame atomic absorption spectrometry;
- Part 6. Determination of silicon content - Molybdenum blue spectrophotometric method;
- Part 7. Determination of sodium content by flame atomic absorption spectrometry;
- Part 8. Determination of the free boronic acid sodium volumetric method;
- Part 9. Determination of the chlorine content of mercuric nitrate volumetric method;
- Part 10. Determination of phosphorus pentoxide content - Molybdenum blue spectrophotometric method. This section GB/T 22661 Part 3. This part is proposed by the China Nonferrous Metals Industry Association. The non-ferrous metal part by the National Standardization Technical Committee. This section is drafted. Hunan Nonferrous fluorine chemical limited liability company. Participated in the drafting of this section. Fluoride Chemicals Co., Ltd., Zhengzhou Research Institute, Aluminum Corporation of China, Hengyang City State Friends Chemical Technology Co., Ltd. The main drafters of this section. Li Zhijian, Zhu Liang, Liao Zhihui, Xue Xu Jin, Chen Chun, Wang Jianping, Feng Jingdong, Liu Zhihong Huang Ju in particular, Liu Min. Chemical analysis methods of potassium fluoroborate Part 3. Determination of the content of potassium fluoroborate Sodium hydroxide volumetric method

### 1 Scope

GB/T 22661.3-2008 is the Chinese national standard on chemical analysis methods of potassium fluoborate - part 3: determination of potassium fluoborate content - distillation-sodium hydrogen oxidizes titration volumetric method, in the field of metallurgy. The /T suffix marks it as a recommended standard: it is not compulsory by itself, but it becomes binding as soon as a contract, a tender or a customer specification calls it up - which in practice is how most foreign buyers meet it. It was issued on 29 December 2008 by the General Administration of Quality Supervision, Inspection and Quarantine of the People's Republic of China; Standardization Administration of China, and has been in force since 1 November 2009. Classification: ICS 77.120.10, CCS H61. This page is published from the official record of the standard held by the Chinese standards administration: the identification, the dates, the classification and the issuing body are taken from there. The clause text, the tables and the numeric limits are in the document itself, which is delivered complete in English translation.

This section GB/T 22661 specifies the method for determination of potassium fluoride boric acid, potassium fluoride boric acid content. This section applies to the determination of fluorine in potassium fluoborate Potassium Content. Measurement range.  $\geq 95\%$ .

## 2 Normative references

The following documents contain provisions which, through reference

GB/T 22661 in this section constitute provisions of this section. For dated reference documents Member, all subsequent amendments (not including errata content) or revisions do not apply to this section, however, encouraged to reach under this section Parties to research agreement to use the latest versions of these documents. For undated reference documents, the latest versions apply to this section.

GB/T 22661.1-2008 Chemical analysis methods of potassium fluoborate Part 1. Preparation and storage of test samples

## 3 Method summary

Sample in the calcium chloride solution, thermal hydrolysis, hydrochloric acid hydrolyzate occur with sodium hydroxide standard solution titration, calculated according to the consumption of fluorine Boric acid and potassium content.

## 4 Reagents

4.1 methyl orange indicator. 1g/L.

4.2 Neutral Calcium chloride solution. 300g/L. Preparation. 300g of anhydrous calcium chloride solution according to 1000mL ratio of preparation, such as no clear solution to be filtered with filter paper, add methyl orange indicator (4.1) was raised to orange.

4.3 sodium hydroxide standard titration solution.  $c(\text{NaOH}) = 0.2500\text{mol/L}$ .

4.3.1 preparation of stock solution of sodium hydroxide Weigh 2020g sodium hydroxide dissolved in 10L carbon dioxide-free water, the reservoir for organic glass bottle. Depending sodium carbonate Low sodium content, add two barium chloride (estimate each containing 1g of sodium carbonate plus 5g two barium chloride) was allowed to stand for several hours, so that barium carbonate precipitation was complete. Pipette the supernatant, add appropriate amount of sodium sulfate, after mixing overnight. 1L solution of sodium hydroxide containing about 5mol, whichever supernatant using or filter.

4.3.2 preparation of sodium hydroxide standard titration solution Pipette 500mL of sodium hydroxide stock solution (4.3.1) in plexiglass flask, add no carbon dioxide to pure water will dilute 10L, mix, put Calibration set the next day.

4.3.3 Calibration sodium hydroxide standard titration solution Weigh 1.2763g of potassium hydrogen phthalate reference reagent (at  $110\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$  drying 2h ~ 3h, cooled to room temperature in a desiccator) to 250mL flask, add carbon dioxide-free water 80mL, heated to dissolve, add 1 drop of methyl orange indicator (4.1), with the desire calibration hydroxide Sodium Standard Solution (4.3.2) titration until the solution is yellow as the end point mutation.

4.3.4 the actual concentration of sodium hydroxide standard titration solution (Ba) according to formula (1).