YS

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Determination of chemical content and physical properties of aluminum fluoride - Part 15: Free alumina content

氟化铝化学分析方法和物理性能测定方法 第 15 部分：游历氧化铝含量的测定

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Determination of chemical content and physical properties of aluminum fluoride - Part 15: Free alumina content

1 Scope

This document specifies a method for the determination of free alumina in aluminum fluoride. Gravimetric method is used as arbitration inspection method.

This document is applicable to the determination of free alumina content in aluminum fluoride. The gravimetric method is applicable to aluminum fluoride products, which are produced by wet processes. The chemical composition balance calculation method is applicable to aluminum fluoride products, which are produced by all processes. The measurement range is 0.1% ~ 15%.

2 Normative references

The contents of the following documents constitute essential provisions of this document through normative references in the text. Among them, for dated reference documents, only the version corresponding to the date applies to this document; for undated reference documents, the latest version (including all amendments) applies to this document.

GB/T 8170 Rules of rounding off for numerical values & expression and judgement of limiting values

YS/T 581.3 Determination of chemical content and physical properties of aluminum fluoride - Part 3: Determination of fluoride content

YS/T 581.4 Determination of chemical contents and physical properties of aluminum fluoride - Part 4: Determination of aluminum by the EDTA volumetric method

YS/T 581.5 Determination of chemical contents and physical properties of aluminum fluoride - Part 5: Determination of sodium by flame atomic absorption spectrometric method

YS/T 581.6 Determination of chemical contents and physical properties of aluminum fluoride - Part 6: Determination of silica content by the molybdenum blue photometric

3 Terms and definitions

There are no terms or definitions to be defined in this document.

4 Weight method

4.1 Principle

The fluoride salt in the specimen is dissolved in the hot boric acid-sodium hydroxide solution, whilst the free Al_2O_3 is not dissolved. After filtration, burn it and weigh the mass of the precipitate. Calculate the free alumina content.

4.2 Reagents

Unless otherwise stated, only reagents confirmed to be of analytical grade and distilled or deionized water or water of relative purity, are used in the analysis.

4.2.1 Boric acid-sodium hydroxide solution: Dissolve 12.5 g of boric acid and 30 g of sodium hydroxide in 500 mL of water. Mix well.

4.2.2 Phenolphthalein ethanol solution: 10 g/L.

4.3 Instruments and equipment

4.3.1 Platinum crucible: The top diameter is about 30 mm, the bottom diameter is about 20 mm, the height is about 35 mm. It is equipped with a lead lid.

4.3.2 High temperature furnace: It can control the temperature at 1000 °C ± 20 °C.

4.4 Specimen

Grind and mix the sample, to make it pass through a 74 μm standard sieve. Bake it in an oven at 110 °C ± 5 °C for 2 h. Cool it to room temperature, in a desiccator.

4.5 Test procedures

4.5.1 Sample

Weigh 1.0 g of specimen (4.4), accurate to 0.0001 g.

4.5.2 Parallel test

Conduct two tests in parallel. Take the average value.

4.5.3 Determination

4.5.3.1 Place the platinum crucible and lid (4.3.1) into the high temperature furnace

(4.3.2). Burn it, at 1000 °C ± 20 °C for 30 minutes. Take out the platinum crucible and lid. Put them into a desiccator. Cool it to room temperature. Weigh the platinum crucible and lid (m_0), accurate to 0.0002 g.

4.5.3.2 Place the specimen into a 400 mL beaker. Add 150 mL of water. Use a glass rod to stir it, to evenly distribute the specimen at the bottom of the beaker. Cover with a watch glass. Heat and boil for 15 min ~ 20 min (continuously stir and maintain the original volume). Then add 50 mL of boric acid-sodium hydroxide solution (4.2.1). Continue to heat and boil for 15 minutes (continuously stir and maintain the original volume). Remove it. Use slow filter paper to filter it. Transfer the precipitate to the filter paper. Use hot water to rinse it, until it is neutral (use phenolphthalein ethanol solution (4.2.2) for inspection).

4.5.3.3 Place the precipitate together with the filter paper into a constant weight platinum crucible (4.5.3.1). Ash it on the electric furnace. Then move it to a high temperature furnace (4.3.2), which has a temperature of 1000 °C ± 20 °C. Burn for 30 minutes. Take out. Put in a desiccator. Cool to room temperature. Weigh it (m_1), accurate to 0.0001 g.

4.6 Processing of test data

The content of free alumina is calculated as the mass fraction of free alumina $w_{Al_2O_3}$, which is calculated according to formula (1):

$$w_{Al_2O_3} = \frac{m_1 - m_0}{m} \times 100\% \quad \cdots\cdots (1)$$

Where:

m_1 - The mass of the platinum crucible, lid, sediment, in grams (g);

m_0 - The mass of the platinum crucible and lid, in grams (g);

m - The mass of sample, in grams (g).

Calculation results are expressed to two decimal places. Numerical rounding off is performed, in accordance with the provisions of GB/T 8170.

4.7 Precision

4.7.1 Repeatability

For the measured value of two independent test results, which are obtained under repeatability conditions, within the average range given below, the absolute difference between the two test results does not exceed the repeatability limit (r); those exceeding the repeatability limit (r) does not exceed 5%. The repeatability limit (r) is obtained, by linear interpolation or extrapolation method, according to the data in Table 1.

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