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**NONFERROUS METALS INDUSTRY STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA**

YS/T 630-2016

**Chemical analysis method of alumina - Determination of
impurities in alumina - ICP-AES analytical method**

氧化铝化学分析方法杂质元素含量的测定电感耦合等离子体原子发射光谱法
(English Translation)

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1 Scope

This standard specifies the method for determining the content of impurity elements such as silicon dioxide, ferric oxide, sodium oxide, potassium oxide, copper oxide, magnesium oxide, calcium oxide, boron trioxide, chromium trioxide, vanadium pentoxide, zinc oxide, titanium dioxide, manganese oxide, gallium trioxide, lithium oxide, and beryllium oxide in alumina.

This standard applies to the determination of the content of impurity elements such as silicon dioxide, ferric oxide, sodium oxide, potassium oxide, copper oxide, magnesium oxide, calcium oxide, boron trioxide, chromium trioxide, vanadium pentoxide, zinc oxide, titanium dioxide, manganese oxide, gallium trioxide, lithium oxide, and beryllium oxide in alumina. The measurement range is shown in Table 1.

Table 1 -- Measurement range

2 Normative references

The following documents are essential for the application of this document. For any dated referenced document, only the dated version applies to this document. For any undated referenced document, the latest version (including all amendments) applies to this document.

GB/T 602 Chemical reagent - Preparations of standard solutions for impurity (ISO 6353-1:1982, NEQ)

Impurity element Measurement range (mass fraction)/%

Vanadium pentoxide

Impurity element Measurement range (mass fraction)/%

Silicon dioxide

Ferric oxide

Sodium oxide

Potassium oxide

Copper oxide

Magnesium oxide

Calcium oxide

Boron trioxide

Chromium trioxide

Zinc oxide

Titanium dioxide

Manganese oxide

Gallium trioxide

Lithium oxide

Beryllium oxide

3 Method summary

Under the action of a microwave digestion system, the test specimen is digested with sulfuric acid at high temperature and high pressure, and the test solution is introduced into argon plasma. Under the selected optimal operating conditions, the impurity element content is determined by an inductively coupled plasma spectrometer, and the matrix matching method is used to correct the influence of the matrix on the determination.

4 Reagents and materials

4.1

Argon (with a purity of $\geq 99.99\%$).

4.2

Sulfuric acid (1+2). Guaranteed or UP grade.

4.3

Sulfuric acid (1+1). Guaranteed or UP grade.

4.4

High-purity aluminum ($\geq 99.999\%$).

4.5

Deionized water or ultrapure water.

4.6 Aluminum matrix solution (20 mg/mL). Weigh 5.2873 g of high-purity aluminum

(4.4) into a 1000 mL beaker; add 400 mL of sulfuric acid (4.3), place the beaker on an electric stove, and slowly heat until completely dissolved. Cool, transfer the solution to a 500 mL volumetric flask, dilute to the mark with water, mix thoroughly, and store in a polyethylene bottle. 1 mL of this solution contains 20 mg of aluminum oxide.

4.7 Standard storage solution

4.7.1

Prepare standard solutions of silicon, iron, sodium, potassium, copper, magnesium, calcium, chromium, vanadium, zinc, titanium, manganese, gallium, lithium, beryllium, and boron according to GB/T 602, with the concentrations of silicon, potassium, vanadium, and titanium at 100 $\mu\text{g/mL}$ and those of iron, sodium, copper, magnesium, calcium, chromium, zinc, manganese, gallium, lithium, beryllium, and boron at 1000 $\mu\text{g/mL}$.

4.8 Mixed standard solution. Pipette 10.00 mL each of the iron and calcium standard solutions (4.7), 50.00 mL of the sodium standard solution (4.7), 5.00 mL each of the copper, magnesium, chromium, zinc, manganese, gallium, lithium, beryllium, and boron standard solutions (4.7), and 50.00 mL each of the vanadium and titanium standard solutions (4.7) into a 250 mL volumetric flask, dilute to the mark with water, mix thoroughly, and transfer to a polyethylene bottle for storage. This solution contains

40 $\mu\text{g/mL}$ each of iron and calcium, 200 $\mu\text{g/mL}$ of sodium, and 20 $\mu\text{g/mL}$ each of copper, magnesium, chromium, vanadium, zinc, titanium, manganese, gallium, lithium, beryllium and boron.

5 Instruments

5.1

Inductively Coupled Plasma-Atomic Emission Spectrometer (ICP-AES).

5.2

Dryer. New activated alumina is used as the desiccant.

5.3

Microwave digestion apparatus. with a rated temperature of 300 °C, and a digestion vessel volume of 100 mL.

5.4

Polytetrafluoroethylene sealed sample dissolver (autoclave).

5.5

Electronic balance. The sensitivity is 0.0001 g.

5.6

Oven. 300 °C±10 °C.

6 Samples

6.1

The sample shall pass through a sieve with an aperture of 0.125 mm.

6.2

The sample shall be dried in an oven (5.6) at 300 °C±10 °C for 2 hours and placed in a desiccator (5.2) to cool to room temperature.

7.1 Test specimen

Weigh 0.2 g of the sample (Chapter 6) to the nearest 0.0001 g.

7.2 Number of measurements

Two measurements shall be performed independently, and the average value shall be taken.

7.3 Blank test

A blank test shall be performed along with the specimen test.