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NONFERROUS METAL INDUSTRY STANDARD
OF THE PEOPLE'S REPUBLIC OF CHINA

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**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

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Table of Contents

Foreword	3
1 Scope	4
2 Normative references	4
3 Method summary	5
4 Reagents and materials	5
5 Instruments	6
6 Samples	6
7 Analysis steps	6
8 Calculation of measurement results	7
9 Precision	8
10 Quality control and assurance	10

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

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

Impurity element	Measurement range (mass fraction)/%	Impurity element	Measurement range (mass fraction)/%
Silicon dioxide	0.005~0.30	Chromium trioxide	0.000 3~0.10
Ferric oxide	0.003~0.30	Vanadium pentoxide	0.000 2~0.10
Sodium oxide	0.001~1.30	Zinc oxide	0.000 3~0.10
Potassium oxide	0.002~0.30	Titanium dioxide	0.000 3~0.10
Copper oxide	0.000 3~0.10	Manganese oxide	0.000 3~0.10
Magnesium oxide	0.000 3~0.10	Gallium trioxide	0.000 3~0.10
Calcium oxide	0.005~0.30	Lithium oxide	0.000 2~0.20
Boron trioxide	0.002~0.30	Beryllium oxide	0.000 2~0.20

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)

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 µg/mL each of iron and calcium, 200 µg/mL of sodium, and 20 µg/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 Analysis steps

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.

7.4 Preparation of analytical solution

7.4.1 Place the specimen (7.1) in a polytetrafluoroethylene digestion vessel, add 12.0 mL of sulfuric acid (4.2), and digest in a microwave digester (5.3) until the sample is completely dissolved. Remove the digestion vessel, cool to room temperature, transfer the solution to a 100 mL volumetric flask, dilute to the mark with water, and mix thoroughly.

7.4.2 Place the specimen (7.1) in a polytetrafluoroethylene reaction cup, add 12.0 mL of sulfuric acid (4.2), and cover tightly. Place the reaction cup in a sample dissolver and cover tightly. Heat the dissolver to $240\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$ in an oven, maintain for 6 hours, remove the dissolver, and allow it to cool naturally to room temperature. Remove the reaction cup, transfer the solution to a 100 mL volumetric flask, dilute to the mark with water, and mix thoroughly.

7.5 Preparation of standard series solutions

7.5.1 Pipette 0 mL, 1.00 mL, 3.00 mL, 5.00 mL, 8.00 mL, and 10.00 mL of the silicon standard solution (4.7) into a set of 100 mL volumetric flasks. Add 10.00 mL of the aluminum matrix solution (4.6) to each flask. Dilute to the mark with water, mix thoroughly, and store in polyethylene bottles.

7.5.2 Pipette 0 mL, 1.00 mL, 3.00 mL, 5.00 mL, 8.00 mL, and 10.00 mL of potassium standard solution (4.7) into a set of 100 mL volumetric flasks. Add 10.00 mL of aluminum matrix solution (4.6) to each flask. Dilute to the mark with water, mix well, and store in polyethylene bottles.

7.6 Determination

Under the selected instrument's optimal working conditions, measure the standard series solutions (7.5) and the analytical solution (7.4).

8 Calculation of measurement results

The contents of the standard series solutions are directly input into the corresponding computer software system. Based on the strength values of the standard series solutions and the analytical solution, the computer calculates, corrects, and outputs the concentration ($\mu\text{g/mL}$) of the element to be measured in the analytical solution. The mass fraction of each element to be measured is calculated according to formula (1):

$$w_{(\text{MeO})} = \frac{c_{\text{Me}} \cdot V \cdot n \times 10^{-4}}{m} \times 100 \quad \dots\dots\dots (1)$$

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