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YS

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

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**Methods for Chemical Analysis of Bauxite - Part 4:
Determination of Iron Oxide Contents
(English Translation)**

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1 Scope

This Document specifies the 1,10-phenanthroline spectrophotometric method and the potassium dichromate titration method for the determination of ferric oxide content in bauxite.

This Document is applicable to the determination of ferric oxide content in bauxite. The determination range (mass fraction) is 0.20% ~ 15.00% for the 1,10-phenanthroline spectrophotometric method; and >15.00% ~ 40.00% for the potassium dichromate titration method.

2 Normative References

The provisions in following documents become the essential provisions of this Document through reference in this Document. For the dated documents, only the versions with the dates indicated are applicable to this Document; for the undated documents, only the latest version (including all the amendments) is applicable to this Document.

GB/T 6682 Water for analytical laboratory use - Specification and test methods

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

3 Terms and Definitions

For the purposes of this Document, there are no terms and definitions required to be defined.

4 1,10-Phenanthroline Spectrophotometric Method

4.1 Principle

The test material is fused by alkali and leached with hydrochloric acid; under appropriate acidity conditions, trivalent iron is reduced to divalent iron using hydroxylamine hydrochloride.

In an acetate buffer medium, the divalent iron forms an orange-red complex with 1,10-phenanthroline; and its absorbance is measured using a spectrophotometer at a wavelength of

510 nm.

4.2 Reagents or materials

Unless otherwise specified, only reagents confirmed to be of analytically pure and water conforming to Grade 2 as specified in GB/T 6682 shall be used for the analysis.

4.2.1

Sodium hydroxide (guaranteed reagent).

4.2.2

Hydrochloric acid (1+1).

4.2.3

Hydrochloric acid (1+3).

4.2.4 Hydroxylamine hydrochloride solution (50 g/L). Weigh 25 g of hydroxylamine

hydrochloride; dissolve in 200 mL of water; dilute to 500 mL with water; and mix well.

4.2.5 1,10-phenanthroline solution (5 g/L). Weigh 2.5 g of 1,10-phenanthroline into a beaker;

add 100 mL of anhydrous ethanol to dissolve it; dilute to 500 mL with water; and mix well.

4.2.6 Acetic acid-sodium acetate buffer solution (with pH of 4.9). Weigh 272 g of sodium acetate trihydrate ($\text{CH}_3\text{COONa} \cdot 3\text{H}_2\text{O}$); dissolve in 500 mL of water; add 240 mL of glacial acetic acid ($\rho = 1.05 \text{ g/mL}$); dilute to 1000 mL with water; and mix well.

4.2.7 Ferric oxide standard stock solution. Weigh 0.5000 g of ferric oxide ($w_{\text{Fe}_2\text{O}_3} \geq 99.99\%$) previously ignited at 600°C for 2 h into a 250 mL beaker; moisten with a small amount of water;

add 40 mL of hydrochloric acid (4.2.2) along the beaker wall; cover with a watch glass. Heat until completely dissolved; and cool. Transfer to a 1000 mL volumetric flask; dilute to the mark with water; and mix well. 1 mL of this solution contains 0.5 mg of ferric oxide. A

certified reference material may also be used.

4.2.8 Ferric oxide standard solution. Pipette 20.00 mL of the ferric oxide standard stock solution (4.2.7) into a 100 mL volumetric flask; dilute to the mark with water; and mix well. 1mL of this solution contains 0.1 mg of ferric oxide. Prepare immediately before use.

4.3 Apparatus

4.3.1

Silver crucible. 30mL.

4.3.2

High-temperature furnace. Capable of controlling a temperature at $750^{\circ}\text{C} \pm 20^{\circ}\text{C}$.

4.3.3

Spectrophotometer.

4.3.4

Drying oven. Capable of controlling a temperature at $110^{\circ}\text{C} \pm 5^{\circ}\text{C}$.

4.4 Sample

The sample particle size shall be less than 75 μm . Pre-dry the sample in the drying oven (4.3.4) at $110^{\circ}\text{C} \pm 5^{\circ}\text{C}$ for 2 h; then place it in a desiccator and allow it to cool to room temperature before use.

4.5.1 Test material

Weigh 0.25 g of the sample (4.4), accurate to 0.0001 g.

4.5.2 Parallel test

Perform two parallel tests and take the average value.

4.5.3 Blank test

Perform a blank test alongside the test material (4.5.1).

4.5.4 Determination

4.5.4.1

Place the test material (4.5.1) into the silver crucible (4.3.1); and cover it with 3 g of sodium hydroxide (4.2.1). Place the crucible in the high-temperature furnace (4.3.2); gradually raise the temperature from room temperature to 750°C ; and fuse for 20 min (blank fusion for 5 min). Remove the crucible; rotate it to ensure the melt adheres evenly to the inner walls; and allow it to cool.

4.5.4.2

Rinse the outer wall and bottom of the crucible with water. Place the crucible in a short-stemmed funnel with diameter of 10 cm; insert the funnel into a 250 mL volumetric flask containing 40 mL of hydrochloric acid (4.2.2). Using hot water, wash the melt from the crucible into a volumetric flask and swirl the flask. Once the test material has completely dissolved, wash the crucible in portions using hot water and 3 mL of hydrochloric acid (4.2.3); then rinse both the crucible and the funnel thoroughly with hot water. Allow the solution to cool to room temperature; dilute to the mark with water; and mix well.

4.5.4.3

According to Table 1, transfer an aliquot of the test solution into a 100 mL volumetric flask; and dilute to approximately 50 mL with water. Sequentially add 5 mL of hydroxylamine hydrochloride solution (4.2.4), 5 mL of 1,10-phenanthroline solution (4.2.5), and 20 mL of acetic acid-sodium acetate buffer solution (4.2.6). Dilute to the mark with water; mix well; and let stand for 20 min. If the room temperature is low, the color development time may be extended appropriately.

Table 1 -- Volume of an aliquot of the test solution

4.5.4.4

Transfer the solution (4.5.4.3) into a 1 cm absorption cell, using water as the reference.

Measure the absorbance at a wavelength of 510 nm of spectrophotometer. Subtract the absorbance of the reagent blank, prepared alongside the test material, from the measured absorbance; then determine the corresponding mass of ferric oxide from the working curve.

4.5.5 Plotting of the working curve

4.5.5.1

Pipette 0 mL, 0.50 mL, 1.00 mL, 2.00 mL, 4.00 mL, 6.00 mL, and 8.00 mL of the ferric oxide standard solution (4.2.8) into a series of 100 mL volumetric flasks, respectively; then proceed according to procedure 4.5.4.3.

4.5.5.2

Transfer a portion of the solution (4.5.5.1) into a 1 cm absorption cell, using water as a reference. Measure the absorbance at a wavelength of 510 nm of spectrophotometer. After subtracting the absorbance of the zero-concentration solution, plot the working curve with the mass of ferric oxide on the x-axis and the absorbance on the y-axis.

4.6 Processing of test data

The ferric oxide content is expressed as the mass fraction of ferric oxide $w_{\text{Fe}_2\text{O}_3}$ and calculated according to Formula (1).