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NATIONAL STANDARD OF THE

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ICS 73.060.10
D 31

GB/T 6730.63-2006

Iron ores - Determination of aluminum, calcium, magnesium, manganese, phosphorus, silicon and titanium content - Inductively coupled plasma atomic emission spectrometric method

铁矿石 铝、钙、镁、锰、磷、硅和钛含量的测定

电感耦合等离子体发射光谱法

(ISO 11535:1998, MOD)

Issued on: August 16, 2006 **Implemented on: January 01, 2007**

Issued by: General Administration of Quality Supervision, Inspection and Quarantine of the PRC;

Standardization Administration of the PRC.

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Foreword

This Standard modifies and adopts ISO 11535:1998 "Iron ores - Determination of various elements - Inductively coupled plasma atomic emission spectrometric method" (English edition).

This Standard, compared with ISO 11535:1998, has mainly been modified as follows:

- In 4.5, the method of preparing hydrochloric acid (1+1) is different: In ISO, use constant-boiling hydrochloric acid to prepare; in this Standard, use hydrochloric acid (ρ1.19 g/mL) to directly prepare;

- In 4.6, the preparation methods of Si, Al, Mg, Ti standard solutions have been changed: For the preparation of silicon standard solution, add 20 mL hydrochloric acid to acidify; this Standard does not add hydrochloric acid to acidify. The aluminum standard solution is prepared by acid dissolution; in this Standard, after adding 25 mL of sodium hydroxide solution (200 g/L) to dissolve the metal aluminum completely, use hydrochloric acid to acidify. The preparation of magnesium standard solution uses high-purity metal magnesium; this Standard uses high-purity magnesium oxide. The preparation of titanium standard solution uses hydrochloric acid to dissolve high-purity metal titanium; this Standard uses sulfuric acid to dissolve high-purity metal titanium and uses sulfuric acid (1+9) to dilute it;

- In 5.3, the volume of the platinum or platinum-alloy crucible for analysis has changed: The minimum volume is 40 mL; the minimum volume in this Standard is 30 mL;

- In 5.8, the suggested analytical lines for elements have been increased: ADD analytical lines of 315.89 nm for calcium, 285.21 nm for magnesium, 293.93 nm for manganese, 185.89 nm and 213.62 nm for phosphorus, and 323.45 nm for titanium;

- In 7.4.3, the iron base and the concentration and number of species of elements of calibration solutions have changed: USE 10-point 0.50g of Fe_2O_3 iron-based primers to prepare calibration solutions containing different concentrations of elements. This Standard uses 8-point 0.43g of Fe_2O_3 iron-based primers to prepare calibration solutions containing different concentrations of elements (see Annex C);

- In 8.4.5, the rounding of the significant figures is incorrect. In 8.4.5 a), b), and c), the fifth place is changed to the fourth place; the fourth place is changed to the third place; the sixth place is changed to the fifth place.

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Iron ores - Determination of aluminum, calcium, magnesium, manganese, phosphorus, silicon and titanium content - Inductively coupled plasma atomic emission spectrometric method

WARNING - This Standard may involve hazardous materials, operations and equipment. This Standard does not purport to address all of the safety problems associated with its use. Therefore, it is the responsibility of the user of this Standard to establish appropriate health and safety practices and ensure compliance with the provisions of relevant national regulations.

1 Scope

This Standard specifies a method for the determination of aluminum, calcium, magnesium, manganese, phosphorus, silicon and titanium by inductively coupled plasma atomic emission spectrometry (ICP-AES).

This Standard is applicable to the determination of the following elements in natural iron ores, iron ore concentrates and agglomerates, including sinter products. The measurement range of each element is shown in Table 1.

Table 1 -- Element and measurement range

Analysis element	Measurement range (mass fraction)/%
Al	0.020~5.00
Ca	0.010~8.00
Mg	0.010~3.00
Mn	0.010~3.00
P	0.013~2.00
Si	0.10~8.00
Ti	0.010~0.20

2 Normative references

The following documents contain provisions which, through reference in this Standard, constitute provisions of this Standard. For the dated references, their subsequent amendments (excluding corrections) or revisions do not apply to this Standard. However, the parties who enter into agreement based on this Standard are encouraged to investigate whether the latest editions of these

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documents are applicable. For undated reference documents, the latest editions apply to this Standard.

GB/T 6682 Water for laboratory use - Specifications (GB/T 6682-1992, neq ISO 3697:1987)

GB/T 6730.1 Methods for chemical analysis of iron ores - Preparation of predried test samples for chemical analysis (GB/T 6730.1-1986, eqv ISO 7764:1985)

GB/T 10322.1 Iron ores - Sampling and sample preparation procedures (GB/T 10322.1-2000, idt ISO 3082:1998)

GB/T 12806 Laboratory glassware - One-mark volumetric flasks (GB/T 12806-1991, neq ISO 1042:1983)

GB/T 12808 Laboratory glassware - One-mark pipettes (GB/T 12808-1991, neq ISO 648:1977)

3 Principle

USE a sodium carbonate-sodium tetraborate mixed flux to melt the test portion. USE hydrochloric acid to dissolve and leach the cooled melt. Decompose it by heating at low temperature and dilute to a predetermined volume. USE ICP spectrometer to measure the intensity of the element to be measured in the solution. According to the calibration graph prepared by the standard solution, calculate the final content of the element.

4 Reagents

During the analysis, use only reagents of recognized analytical grade and only water for laboratory use that conforms to GB/T 6682.

4.1 Iron oxide powder (Fe_2O_3>99.99%).

4.2 Anhydrous sodium carbonate, high quality grade.

4.3 Anhydrous sodium tetraborate, high quality grade.

4.4 Hydrochloric acid, ρ about 1.19 g/mL, high quality grade.

4.5 Hydrochloric acid, 1+1.

4.6 Standard stock solutions

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4.6.1 Silicon standard stock solution, 1000 μg/mL.

WEIGH 2.1393 g of silicon dioxide (>99.9%) which is previously burned at 1000°C for 45 min and cooled to room temperature in a desiccator; PLACE in a platinum crucible containing 5 g of anhydrous sodium carbonate and mix well. MELT in a muffle furnace at 1000°C for 15 min. USE 100 mL of warm water to heat and dissolve the melt in a polytetrafluoroethylene beaker; COOL to room temperature; TRANSFER to a 1000 mL volumetric flask; USE water to dilute to the mark and mix well; STORE in a polyethylene flask.

4.6.2 Aluminum standard stock solution, 1000 μg/mL.

WEIGH 1.0000 g of metal aluminum (99.99%) into a 250 mL polytetrafluoroethylene beaker; ADD 25 mL of sodium hydroxide solution (200 g/L). After heating and dissolving it completely, use hydrochloric acid (4.5) to neutralize, with an excess of 20 mL. BOIL until the solution is clear; COOL to room temperature; TRANSFER to a 1000 mL volumetric flask; USE water to dilute to the mark and mix well.

4.6.3 Calcium standard stock solution, 1000 μg/mL.

WEIGH 2.4972 g of calcium carbonate (>99.99%) dried to a constant mass at 110°C into a 250 mL beaker; COVER with a watch glass; slowly add 20 mL of hydrochloric acid (4.5); HEAT until completely dissolved; BOIL to drive off carbon dioxide; COOL to room temperature; TRANSFER into a 1000 mL volumetric flask; USE water to dilute to the mark and mix well.

4.6.4 Magnesium standard stock solution, 1000 μg/mL.

WEIGH 1.6582 g of magnesium oxide (>99.9%) which is heated at 850°C for 30 min and cooled to room temperature in a desiccator; PLACE it in a 250 mL beaker; ADD 20 mL of hydrochloric acid (4.5); HEAT to dissolve completely; COOL to room temperature; TRANSFER to a 1000 mL volumetric flask; USE water to dilute to the mark and mix well.

4.6.5 Manganese standard stock solution, 1000 μg/mL.

WEIGH 1.0000 g of metal manganese (>99.9%) from which surface oxides have been removed into a 250 mL beaker; ADD 20 mL of hydrochloric acid (4.5); HEAT to dissolve completely; COOL to room temperature; TRANSFER to a 1000 mL volumetric flask; USE water to dilute to the mark and mix well.

4.6.6 Phosphorus standard stock solution, 1000 μg/mL.

WEIGH 4.3936 g of reference potassium dihydrogen phosphate dried to a constant mass at 110°C into a 400 mL beaker; ADD 200 mL of water; completely

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