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Iron, Steel and Alloy - Determination of Calcium and Magnesium - Inductively Coupled Plasma Mass Spectrometric Method

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Iron, Steel and Alloy - Determination of Calcium and Magnesium - Inductively Coupled Plasma Mass Spectrometric Method

1 Scope

This Part in GB/T 223 specifies methods of determining calcium and magnesium content through inductively coupled plasma mass spectrometric method.

This Part is applicable to the determination of calcium (mass fraction: 0.0002% ~ 0.0035%) and magnesium (mass fraction: 0.0003% ~ 0.05%) in iron, steel and alloy.

2 Normative References

The following documents are indispensable to the application of this Standard. In terms of references with a specified date, only versions with a specified date are applicable to this Standard. The latest version (including all the modifications) of references without a specified date is also applicable to this Standard.

GB/T 6379.1 Accuracy (Trueness and Precision) of Measurement Methods and Results - Part 1: General Principles and Definitions (GB/T 6379.1-2004, ISO 5725-1: 1994, IDT)

GB/T 6379.2 Accuracy (Trueness and Precision) of Measurement Methods and Results - Part 2: Basic Method for the Determination of Repeatability and Reproducibility of a Standard Measurement Method (GB/T 6379.2-2004, ISO 5725-2: 1994, IDT)

GB/T 6682 Water for Analytical Laboratory Use - Specification and Test Methods (GB/T 6682-2008, ISO 3696: 1987, MOD)

GB/T 12806 Laboratory Glassware - One-mark Volumetric Flasks (GB/T 12806-2011, ISO 1042: 1998, NEQ)

GB/T 12808 Laboratory Glassware - One-mark Pipettes

GB/T 20066 Steel and Iron - Sampling and Preparation of Samples for the Determination of Chemical Composition (GB/T 20066-2006, ISO 14284: 1996, IDT)

3 Principle

Dissolve reagent in the mixed acid of hydrochloric acid, nitric acid and hydrofluoric acid.

If necessary, adopt perchloric acid to generate fumes. After dilution, import the sample solution into inductively coupled plasma mass spectrometer with a peristaltic pump. During the operation of the inductively coupled plasma mass spectrometer, measure the mass spectrometry signal intensity of the element.

Conduct matrix matching of calibration blank, calibration solution, and main elements in the sample to be tested, and acid adopted for reagent decomposition. Adopt internal standard to compensate the instrument's drifting effect and matrix effect.

4 Reagents

Unless it is otherwise indicated, high-purity reagent (mass fraction of calcium and magnesium < 0.0001%) or reagent with equivalent purity, and first-grade water stipulated in GB/T 6682 shall be adopted in analysis. If the requirements cannot be satisfied, use an acid purification unit (5.3) to purify the reagents.

4.1 Hydrochloric acid, $\rho = 1.19$ g/mL.

4.2 Nitric acid, $\rho = 1.42$ g/mL.

4.3 Hydrofluoric acid, $\rho = 1.19$ g/mL.

4.4 Perchloric acid, $\rho = 1.67$ g/mL.

4.5 Calcium standard stock solution, 1,000 $\mu\text{g/mL}$.

Weigh-take 2.4972 g of calcium carbonate (mass fraction $\geq 99.99\%$) that is already dried at 110 °C for 1 h and cooled down to room temperature in a dryer. Place it in 400 mL beaker; add 100 mL of hydrochloric acid (1+1) to dissolve it. After cooling it down, transfer it into 1,000 mL volumetric flask. Use water to dilute to the constant volume; thoroughly mix it.

1 mL of this solution contains 1,000 μg of calcium.

4.6 Magnesium standard stock solution, 1,000 $\mu\text{g/mL}$.

Weigh-take 1.6582 g of magnesium oxide (mass fraction $\geq 99.99\%$) that is already burnt at 850 °C for 30 min and cooled down to room temperature in a dryer. Place it in 400 mL beaker; add 100 mL of hydrochloric acid (1+1). After heating it up and dissolving it, cool it down to room temperature; transfer it into 1,000 mL volumetric flask. Use water to dilute to the constant volume; thoroughly mix it.

1 mL of this solution contains 1,000 μg of magnesium.

4.7 Mixed standard solution of calcium and magnesium

4.7.1 Mixed standard solution of calcium and magnesium, 100 $\mu\text{g/mL}$.

7.2 Blank Test

Conduct blank test in accordance with the sample.

7.3 Determination

7.3.1 Preparation of sample solution

In terms of carbon steel and low-alloy steel sample: place the sample (7.1) in 100 mL quartz beaker. Add 10 mL of water and 5 mL of nitric acid (4.2) [add 0.5 mL of hydrofluoric acid (4.3) to high-silicon sample], heat it up till it boils. After eliminating nitrogen oxide, take it down; cool it down, then, transfer it into 100 mL plastic volumetric flask; add 1.0 mL of internal standard solution of scandium (4.8.3). Use water to dilute to the constant volume, then, thoroughly mix it for later determination.

In terms of high-carbon steel and alloy steel sample: place the sample (7.1) in 100 mL quartz beaker. Add 3 mL of hydrochloric acid (4.1) and 1 mL of HNO_3 (4.2); use heating plate to heat it up till it boils. Add 2 mL of perchloric acid (4.4), heat it up till it begins to fume and stable perchloric acid fume reflux is formed around the bottle mouth or on the wall. Continue to heat it up, till white perchloric acid fume vanishes. Cool it down, then, add 3 mL of hydrochloric acid (4.1) and 1 mL of HNO_3 (4.2); conduct low-temperature heating till salt is dissolved. If there are hydrolysable elements in alloy steel, add 0.5 mL of hydrofluoric acid (4.3) for complexation. Cool it down, then, transfer to 100 mL plastic volumetric flask; add 1.0 mL of internal standard solution of scandium (4.8.3). Use water to dilute to the constant volume, then, thoroughly mix it for later determination.

NOTE: microwave digestion system can also be adopted for the preparation of sample solution, but the laboratory shall determine an optimized experimental plan in accordance with conditional experiments.

7.3.2 Preparation of calibration solution

Respectively transfer 0, 0.20 mL, 0.50 mL, 1.00 mL, 2.00 mL and 5.00 mL of mixed standard solution of calcium and magnesium (4.7.3), and 1.00 mL, 2.00 mL and 5.00 mL of mixed standard solution of calcium and magnesium (4.7.2) in 9 plastic volumetric flasks (100 mL). Respectively add 10 mL of Fe matrix solution (4.11) and 1.00 mL of scandium internal standard solution (4.8.3). Add acid that is consistent with sample dissolution; use water to dilute to the constant volume, then, thoroughly mix it. Solution that does not contain the mixed standard solution of calcium and magnesium is calibration blank solution.

7.3.3 Preparation of instrument

Before analysis, ignite plasma; pre-heat it for 30 min ~ 60 min in accordance with the requirements in the instruction for use to stabilize the instrument. During this process, pump in water or mass spectrometer cleaning solution (4.10) to rinse nebulizer and

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Contact: Wayne Zheng, Sales@ChineseStandard.net

Linkin: <https://www.linkedin.com/in/waynezhengwenrui/>

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