GB

NATIONAL STANDARD OF THE

PEOPLE'S REPUBLIC OF CHINA

ICS 77.080.20

H 11

GB/T 20125-2006

Low-alloy steel - Determination of multi-element contents - Inductively coupled plasma atomic emission spectrometric method

低合金钢 多元素含量的测定 电感耦合等离子体原子发射光谱法

Issued on: March 02, 2006 **Implemented on: September 01, 2006**

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

Standardization Administration of PRC.

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1 Scope

This standard specifies the method for the determination of silicon, manganese, phosphorus, nickel, chromium, molybdenum, copper, vanadium, cobalt, titanium, aluminum, by inductively coupled plasma emission spectrometry.

This method is suitable for the determination of silicon, manganese, phosphorus, nickel, chromium, molybdenum, copper, vanadium, cobalt, titanium, aluminum in carbon steel and low alloy steel, which has an iron mass fraction greater than 92%. For the determination range of multi-elements, refer to Table 1.

The silicon, titanium, aluminum, which is measured by this method, are acid-soluble silicon, acid-soluble titanium, acid-soluble aluminum.

When even only one of the components in the steel exceeds the upper limit of the content range in Table 1, this standard does not apply.

In steel, when the mass fraction of carbon and sulfur is greater than 1.0%, AND the mass fraction of tungsten and niobium is greater than 0.10%, this standard is also not applicable.

This standard specifies two alternative methods, to determine the content of elements in test solutions, one using an internal standard AND the other not using an internal standard.

2 Normative references

The provisions in following documents become the provisions of this Standard through reference in this Standard. For the dated references, the subsequent amendments (excluding corrections) or revisions do not apply to this Standard; however, parties who reach an agreement based on this Standard are encouraged to study if the latest versions of these documents are applicable. For undated references, the latest edition of the referenced document applies.

GB/T 20066-2006 Steel and iron - Sampling and preparation of samples for the determination of chemical composition

GB/T 6379 (all parts) Accuracy (trueness and precision) of measurement methods and results

3 Principles

The sample is dissolved by a mixed acid of hydrochloric acid and nitric acid AND diluted to a certain volume. If necessary, add yttrium as an internal standard. The atomized solution is introduced into an inductively coupled plasma emission spectrometer, to measure the emission light intensity of each elemental analysis line, OR simultaneously measure the emission light intensity of yttrium at 371.03 nm. Then calculate the emission light intensity ratio of each element.

4 Reagents and materials

Unless otherwise stated, only approved reagents of analytical grade and double distilled water or water of equivalent purity are used in the analysis.

4.1 High-purity iron: the mass fraction is greater than 99.98%; the content of the element to be tested is known.

4.2 Hydrochloric acid: ρ is about 1.19 g/mL.

4.3 Nitric acid: ρ is about 1.42 g/mL.

4.4 Perchloric acid: ρ is about 1.67 g/mL.

4.5 Sulfuric acid: ρ is about 1.84 g/mL.

4.6 Hydrogen peroxide: ρ is about 1.10 g/mL.

Weigh 1.0000 g of electrolytic manganese [mass fraction greater than 99.9%, use nitric acid (1 + 3) to wash the surface oxide film in advance; then wash it in absolute ethanol (4 ~ 5) times; take it out and store it in a drier for more than 12 h]. Put it in a 500 mL beaker. Add 50 mL of nitric acid (1 + 3). Heat to dissolve it. Boil to drive off nitrogen oxides. Remove and cool to room temperature. Transfer it into a 1000 mL volumetric flask. Use water to dilute it to the mark. Mix well.

1 mL of this solution contains 1000.0 μg of manganese.

4.9.2 Manganese standard solution, 100.0 μg/mL

Transfer 10.00 mL of manganese stock solution (4.9.1) into a 100 mL volumetric flask. Use water to dilute it to the mark. Mix well.

1 mL of this solution contains 100.0 μg of manganese.

4.10 Phosphorus standard solution

4.10.1 Phosphorus stock solution, 1000.0 μg/mL

Weigh 4.3936 g of standard potassium dihydrogen phosphate (KH_2PO_4) (pre-dried at 105 °C for 1 h. Place it in a desiccator. Cool it to room temperature). Place it in a 500 mL beaker. Use an appropriate amount of water to dissolve it. Boil it. Cool it. Transfer it into a 1000 mL volumetric flask. Use water to dilute it to the mark. Mix well.

1 mL of this solution contains 1000.0 μg of phosphorus.

4.10.2 Phosphorus standard solution A, 100.0 μg/mL

Transfer 10.00 mL of phosphorus stock solution (4.10.1) into a 100 mL volumetric flask. Use water to dilute it to the mark. Mix well.

1 mL of this solution contains 100.0 μg of phosphorus.

4.10.3 Phosphorus standard solution B, 10.00 μg/mL

Transfer 10.00 mL of phosphorus standard solution A (4.10.2) into a 100 mL volumetric flask. Use water to dilute it to the mark. Mix well.

1 mL of this solution contains 10.00 μg of phosphorus.

4.11 Nickel standard solution

4.11.1 Nickel stock solution, 1000.0 μg/mL

Weigh 1.0000 g of pure nickel (mass fraction greater than 99.9%). Put it in a 500 mL beaker. Add 50 mL of nitric acid (1 + 1). Heat to dissolve. Cool to room temperature. Transfer it into a 1000 mL volumetric flask. Use water to dilute it to the mark. Mix well.

1 mL of this solution contains 1000.0 μg of nickel.

4.11.2 Nickel standard solution, 100 μg/mL

Transfer 10.00 mL of nickel stock solution (4.11.1) into a 100 mL volumetric flask. Use water to dilute it to the mark. Mix well.

1 mL of this solution contains 100 μg of nickel.

4.12 Chromium standard solution

4.12.1 Chromium stock solution, 1000.0 μg/mL

Weigh 1.0000 g of pure chromium (mass fraction greater than 99.9%). Put it in a 500 mL beaker. Add 50 mL of hydrochloric acid (4.2). Heat to dissolve. Cool to room temperature. Transfer it to a 1000 mL volumetric flask. Use water to dilute it to the mark. Mix well.

1 mL of this solution contains 1000.0 μg of chromium.

4.12.2 Chromium standard solution, 100 μg/mL

Transfer 10.00 mL of chromium stock solution (4.12.1) into a 100 mL volumetric flask. Use water to dilute it to the mark. Mix well.

1 mL of this solution contains 100 μg of chromium.

4.13 Molybdenum standard solution

4.13.1 Molybdenum stock solution, 1000.0 μg/mL

Weigh 1.0000 g of metal molybdenum (mass fraction greater than 99.9%). Put it in a 500 mL beaker. Add 30 mL of nitric acid (1 + 1). Heat to dissolve. Cool it. Add 30 mL of sulfuric acid (4.5). Heat it, until white smoke of sulfuric acid is emitted. Cool to room temperature. Transfer it into a 1000 mL volumetric flask. Use water to dilute it to the mark. Mix well.

1 mL of this solution contains 1000.0 μg of molybdenum.

4.13.2 Molybdenum standard solution, 100.0 μg/mL

Transfer 10.00 mL of molybdenum stock solution (4.13.1) into a 100 mL volumetric flask. Use water to dilute it to the mark. Mix well.

1 mL of this solution contains 100.0 μg of molybdenum.

4.14 Copper standard solution

4.14.1 Copper stock solution, 500.0 μg/mL

1 mL of this solution contains 100.0 μg of cobalt.

4.16.3 Cobalt standard solution B, 10.00 μg/mL

Transfer 10.00 mL of cobalt standard solution A (4.16.2) into a 100 mL volumetric flask. Use water to dilute it to the mark. Mix well.

1 mL of this solution contains 10.00 μg of cobalt.

4.17 Titanium standard solution

4.17.1 Titanium stock solution, 250.0 μg/mL

Weigh 0.2500 g of titanium metal (mass fraction greater than 99.9%). Put it in a 400 mL polytetrafluoroethylene beaker. Add 5 mL of hydrofluoric acid. Immediately add 2 mL of nitric acid (4.3) dropwise. Heat to dissolve it. Cool it. Add 20 mL of sulfuric acid (4.5). Evaporate it at low temperature, until sulfuric acid smoke. Cool to room temperature. Use sulfuric acid (5 + 95), to transfer it into a 1000 mL volumetric flask. Use water to dilute it to the mark. Mix well.

1 mL of this solution contains 250.0 μg of titanium.

4.17.2 Titanium standard solution, 10.00 μg/mL

Transfer 10.00 mL of titanium stock solution (4.17.1) into a 250 mL volumetric flask. Use sulfuric acid (5 + 95) to dilute it to the mark. Mix well.

1 mL of this solution contains 10.00 μg of titanium.

4.18 Aluminum standard solution

4.18.1 Aluminum stock solution, 1000.0 μg/mL

Weigh 1.0000 g of pure aluminum (mass fraction greater than 99.9%). Put it in a 500 mL beaker. Add 100 mL of hydrochloric acid (1 + 1). Dissolve it on a water bath at 85 °C (1 ~ 3 days). After dissolving, cool it to room temperature. Transfer it into a 1000 mL volumetric flask. Use water to dilute it to the mark. Mix well.

1 mL of this solution contains 1000.0 μg of aluminum.

4.18.2 Aluminum standard solution A, 100.0 μg/mL

Transfer 10.00 mL of aluminum stock solution (4.18.1) into a 100 mL volumetric flask. Add 10 mL of hydrochloric acid (1 + 1). Use water to dilute it to the mark. Mix well.

1 mL of this solution contains 100.0 μg of aluminum.

4.18.3 Aluminum standard solution B, 10.00 μg/mL

7 Analytical procedures

7.1 Sample amount

Weigh 0.50 g of sample, accurate to 0.1 mg.

7.2 Blank test (equivalent to No.0)

Weigh 0.500 g of high-purity iron (4.1). Make a blank test with the sample.

7.3 Determination

7.3.1 Preparation of specimen solution

Place the sample (7.1) in a 200 mL beaker. Add 10 mL of water, 5 mL of nitric acid (4.3). Cover a watch glass, Heat slowly, until the bubbling stops. Add 5 mL of hydrochloric acid (4.2). Continue heating, until complete decomposition. If there is insoluble carbide, add 5 mL of perchloric acid (4.4); heat until perchloric acid smoke for (3 ~ 5) min; remove it; cool it; add 10 mL of water and 5 mL of nitric acid (4.3); shake well; add 5 mL of hydrochloric acid (4.2); heat to dissolve the salts. This solution cannot be used for measurement of Si. Cool it to room temperature. Transfer the solution quantitatively to a 100 mL volumetric flask. If the internal standard method is used, use pipetting to add 10 mL of yttrium internal standard solution (4.7.2). Use water to dilute it to the mark. Mix well.

7.3.2 Preparation of calibration curve solution

Weigh 7 parts of 0.500 g high-purity iron (4.1) into a 200 mL beaker. Dissolve it according to step 7.3.1. Cool it to room temperature. Transfer the solution to a 100 mL volumetric flask. Add the standard solution of the analyte, according to Table 4 and Table 5. If the calibration curve is found to be non-linear, it may add the calibration series (e.g., see Table 5). If the internal standard method is used, use pipette to add 10 mL of yttrium internal standard solution (4.7.2). Use water to dilute it to the mark. Mix well. In the standard solution, if there is a coexisting element other than the measured element (sodium, etc.), that affects the luminescence intensity of the measured element, the amount of this coexisting element shall be the same in the calibration curve series solution. It shall also add this coexisting element, which has equivalent amount as in the calibration curve series solution, into the sample solution.

If using an internal standard, prepare software, that uses Y (371.03 nm) as the internal standard and calculates the intensity ratio of each element to yttrium. The internal standard intensity shall be measured simultaneously with the analyte intensity.

Check the performance requirements of each instrument, which is given in 5.1.2 ~ 5.1.5.

7.4.2 Measurement of emission intensity

If measuring absolute strength, it shall be ensured that the temperature difference of all measuring solutions is within 1 °C. Use medium speed filter paper, to filter all solution. Discard the initial 2 mL ~ 3 mL of solution.

Use the lowest concentration calibration solution (number zero is equivalent to the blank test), to begin measuring the absolute intensity or intensity ratio.

Then measure 2 or 3 unknown test solutions. Then measure the calibration solution next to the lowest concentration. Then measure 2 or more unknown test solutions, and so on. For the measured element in each solution, integrate 5 times AND check the short-term stability as specified in 5.1.3. Then calculate the average intensity or average intensity ratio.

The average absolute intensity or average intensity ratio (I_i) of the tested element in each solution is subtracted from the average absolute intensity or average intensity ratio (I_0) in No.0, to obtain the net absolute intensity or net intensity ratio (I_N), as shown in formula (1).

$$I_N = I_i - I_0 \quad \cdots\cdots (1)$$

7.5 Correction of interference lines in analysis lines

First check the spectral interference of each coexisting element on the analytical line of the analyte. In the case of spectral interference, obtain the spectral interference correction factor, that is, the mass fraction of the analyte, when the mass fraction of the coexisting element is 1%.

7.6 Plotting of calibration curve

Take the net intensity or net intensity ratio as the Y axis; take the concentration of the analyte (μg/mL) as the X axis, for linear regression. The calculation of the correlation factor shall meet the requirements of 5.1.6.

8 Result calculation

According to the calibration curve (7.6), convert the net intensity or net intensity ratio of the test solution to the concentration of the corresponding analyte, expressed in μg/mL.

------ The End ------