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**NATIONAL STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA**

GB/T 20125-2006

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

(English Translation)

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Table of Contents

Foreword...	3
1 Scope...	5
2 Normative references...	6
3 Principles...	6
4 Reagents and materials...	6
5 Instruments and equipment...	12
6 Sampling and preparation of specimen...	14
7 Analytical procedures...	15
8 Result calculation...	17
9 Precision...	18
10 Test report...	19
Appendix A (Normative) Standardized operation of measuring instrument...	20
Appendix B (Informative) Additional information on common precision tests...	23

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.

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.

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.

4.8.1 Silicon stock solution, 500.0 µg/mL

Weigh 0.5348 g of silica (mass fraction greater than 99.9%, pre-fired at 1000 °C for 1 h, placed in a desiccator and cooled to room temperature). Place it in a platinum crucible, which contains 3 g of anhydrous sodium carbonate. Stir it evenly. Cover (1 ~ 2) g of anhydrous sodium carbonate on the top.

4.9.1 Manganese stock solution, 1000.0 µg/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;

4.10.1 Phosphorus stock solution, 1000.0 µg/mL

Weigh 4.3936 g of standard potassium dihydrogen phosphate (KH₂PO₄) (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.

4.17.1 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.

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).

5 Instruments and equipment

Usual laboratory equipment and inductively coupled plasma atomic emission spectrometer (ICP-AES).

5.1 Inductively coupled plasma atomic emission spectrometer (ICP-AES)

If, after the spectrometer is optimized according to 7.4.1 ~ 7.4.5, it meets the performance index of 5.1.2 ~ 5.1.5, THEN, it meets the usage requirements.

Spectrometers can be either simultaneous or sequential. However, it must have the function of measuring the internal mark, at the same time. Otherwise, the internal standard method cannot be used.

5.1.4 Long-term stability

Make 3 measurements, of the average value of the absolute intensity or intensity ratio of the calibration solution, which has the highest concentration of each element.

Calculate the standard deviation of the seven average values.

5.1.5 Background equivalent concentration and detection limit (see Appendix A.2)

Elements Wavelength/nm Possible interfering elements

For the analysis line, which contains only the element to be detected in the solution, calculate the background equivalent concentration (BEC) and detection line (DL); the results must be lower than the values in Table 3.

5.1.6 Linearity of curve

The linearity of the calibration curve is checked by calculating the correlation coefficient, which must be greater than 0.999.

6 Sampling and preparation of specimen

Take samples AND prepare specimen, according to GB/T 20066 or appropriate national standards.

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.

7.3.1 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).

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.

7.4.1 Optimization of instrument

Turn on ICP-AES and run for at least 1 h, before taking measurements.

Measure the most concentrated calibration solution. Adjust the instrument parameters, according to the operating procedures and guidelines, which are provided by the instrument manufacturer. gas (outer, middle or center) flow rate, torch position, entrance slit, exit slit, photomultiplier tube voltage, analysis line wavelength in Table 2, prewash time, integration