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Replacing GB/T 11170-1989

Stainless steel - Determination of multi-element contents - Spark discharge atomic emission spectrometric method (Routine method)

不锈钢 多元素含量的测定 火花放电原子发射光谱法（常规法）

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Stainless steel - Determination of multi-element contents - Spark discharge atomic emission spectrometric method (Routine method)

1 Scope

This Standard specifies the analysis for the determination of carbon, silicon, manganese, phosphorus, sulfur, chromium, nickel, molybdenum, aluminum, copper, tungsten, titanium, niobium, vanadium, cobalt, boron, arsenic, tin and lead contents by spark discharge atomic emission spectrometric method.

This Standard applies to the determination of carbon, silicon, manganese, phosphorus, sulfur, chromium, nickel, molybdenum, aluminum, copper, tungsten, titanium, niobium, vanadium, cobalt, boron, arsenic, tin and lead contents in stainless steel. The determination range of each element is shown in Table 1.

2 Normative references

The terms in the following documents become the terms of this Standard by reference to this Standard. For dated references, all subsequent amendments (not including errata content) or revisions do not apply to this Standard. However, parties to agreements that are based on this Standard are encouraged to study whether the latest versions of these standards can be used. For undated references, the latest edition applies 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 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

Use the prepared block sample as an electrode; use a light source generator to excite and emit light between the sample and the counter electrode; introduce the beam into the spectrometer. After the light beam is dispersed by the dispersion element, measure the intensity of the selected internal standard line and analytical line. According to the calibration curve of standard samples, obtain the content of the to-be-measured element in the analyzed sample.

4 Instruments

The spark discharge atomic emission spectrometer – vacuum type or gas-filled type – is mainly composed of the following units.

4.1 Excitation light source

The excitation light source shall be a stable spark discharge light source.

4.2 Spark chamber

The spark chamber shall be specially designed for the use of argon gas. The spark chamber shall be mounted directly on the spectrometer, and shall have an argon-flushed spark stand, to accommodate the flat sample and the rod-shaped counter electrode. The argon gas circuit of the spark chamber shall be able to replace the air in the optical path between the analytical gap and the condenser, and provide an argon atmosphere for the analytical gap.

4.3 Argon gas system

The argon gas system mainly includes an argon gas container, a two-stage pressure regulator, a gas flow meter and a timing control part that can automatically change the argon gas flow rate according to the analysis conditions.

Since the purity and flow rate of argon gas have a great influence on the analysis and measurement value, it shall be ensured that the purity of argon gas is not less than 99.996%; otherwise, an argon gas purification device must be used, and the pressure and flow rate of argon gas in the spark chamber must be kept constant.

4.4 Counter electrode

Different counter electrodes shall be used for equipment of different models. Generally, a conical tungsten rod with a diameter of 4 mm ~ 7 mm and a top processing of 30° ~ 120° shall be used; also, a flat tungsten electrode with a diameter of 1 mm can be used. Each laboratory determines the time to replace the counter electrode according to the specific situation.

4.5 Spectrometer

In general, the reciprocal of the first-order spectral line dispersion of a spectrometer shall be less than 0.6 nm/mm, the focal length shall be 0.5 m ~ 1.0 m, and the wavelength range shall be 120.0 nm ~ 623.0 nm. The vacuum degree of the spectrometer shall work below 3 Pa, or be filled with high-purity nitrogen or argon gas (the gas does not absorb spectral lines with wavelengths less than 200 nm, and the purity is not lower than 99.999%).

4.6 Photometric system

The photometric system shall include a photomultiplier tube (or other photoelectric conversion device) for receiving signals, an integrating capacitor capable of storing each output electrical signal, a measuring unit for directly or indirectly recording the voltage on the capacitor, and a necessary switching circuitry provided for the required timing.

5 Sampling and sample preparation equipment

5.1 Sampling and sample preparation

Sampling and sample preparation shall be carried out in accordance with the requirements of GB/T 20066. The surface of the sample taken shall ensure that two points can be excited without overlapping, the recommended thickness is not less than 3 mm, and there is no physical defect.

5.2 Sample preparation equipment

The grinding equipment can be a grinding machine, a sandpaper disc or an abrasive band grinding machine, or can be a milling machine, a lathe, etc.

6 Standard samples, standardized samples and control samples

6.1 Standard samples

Standard samples are certified reference materials required for routine analysis to draw calibration curves. The content of each analytical element in the standard samples shall have an appropriate gradient, and the chemical value shall be accurate and free from physical defects. The selected standard samples shall be as close as possible to the type of samples to be tested.

6.2 Standardized samples

Due to changes in the state of the instrument, the measurement results deviate. In order to directly use the original calibration curve to obtain accurate results, use (1~2) samples to standardize the instrument. These samples are called standardized samples. Standardized samples, which shall be very uniform and shall have appropriate contents, can be selected from standard samples or specially smelted. When two-point standardization is used, the content is taken as the content near the upper limit and lower limit of the calibration curve of each element respectively.

6.3 Control samples

Control samples, which have similar metallurgical process and chemical composition to the analytical samples, are used to correct the measurement results of the analytical samples.

7 Preparation of instruments

7.1 Basic requirements

The spectrometer shall be placed in a shockproof and clean laboratory with an indoor temperature of 16 °C ~ 30 °C and a relative humidity of 40% ~ 70%. In the same calibration period, the maximum allowable indoor temperature difference is 5 °C/h.

7.2 Power supply

In order to ensure the stability of the instrument, the power supply voltage variation shall be within ±10%, and the frequency variation shall not exceed ±2%. It shall be

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