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Ferronickel - Determination of carbon, sulfur, silicon, phosphorus, nickel, cobalt, chromium and copper contents - Spark atomic emission spectrometry

镍铁 碳、硫、硅、磷、镍、钴、铬和铜含量的测定 火花源原子发射光谱法

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Foreword

This document was drafted in accordance with the rules given in GB/T 1.1-2020 "Directives for standardization - Part 1: Rules for the structure and drafting of standardizing documents".

Attention is drawn to the possibility that some elements of this document may be the subject of patent rights. The issuing body shall not be held responsible for identifying patents.

This document was proposed by the China Iron and Steel Association and is under the jurisdiction of the National Technical Committee on Pig Iron and Ferroalloys of Standardization Administration of China (SAC/TC 318).

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Warning

Persons using this document need practical experience of working in a formal laboratory. This document does not set out all the possible safety problems; it is the responsibility of the user to adopt appropriate safety and health measures and to ensure compliance with the conditions laid down by the relevant national regulations.

1 Scope

This document describes the determination of the carbon, sulfur, silicon, phosphorus, nickel, cobalt, chromium and copper contents of ferronickel by spark atomic emission spectrometry.

It applies to the routine analysis of the chemical composition of ferronickel products. The determination range for each element is given in Table 1: carbon from 0.017 % to 2.5 %, sulfur from 0.018 % to 0.25 %, silicon from 0.06 % to 4.2 %, phosphorus from 0.005 % to 0.040 %, nickel from 14.5 % to 42.0 %, cobalt from 0.10 % to 2.2 %, chromium from 0.25 % to 2.3 % and copper from 0.003 % to 0.25 %, all as mass fractions.

2 Normative references

The contents of the following documents constitute indispensable provisions of this document through normative reference in the text. For dated references, only the version corresponding to that date applies; for undated references, the latest version - including all amendments - applies.

GB/T 6379.1 Accuracy (trueness and precision) of measurement methods and results - Part 1: General principles and definitions · 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 6379.6 Accuracy (trueness and precision) of measurement methods and results - Part 6: Use in practice of accuracy values · GB/T 25050 Ferronickel ingots or blocks - Sampling for chemical composition analysis · GB/T 25051 Ferronickel granules - Sampling for chemical composition analysis

3 Terms and definitions

No terms and definitions need to be defined for this document.

4 Principle

A discharge takes place between the prepared block sample of ferronickel and the counter-electrode under the action of the spark source. The spark melts the surface of the sample, then vaporises and excites it so that it emits light. When the atoms or ions of the

elements to be determined are excited, the electrons move between energy levels within the atom, and the transition from a higher to a lower level produces the characteristic spectral lines. The spectral intensities of the selected analytical lines and internal standard lines are measured, and the content of each element is calculated from that intensity - or intensity ratio - by means of the calibration curve.

5 Reagents and materials

5.1 Electrode brush - a steel wire brush, used to remove the black deposit left on the tip of the electrode after the sample has been excited.

5.2 Certified reference materials - used to plot the calibration curve. They shall be a series of certified spectrometric reference materials of ferronickel whose chemical composition covers the determination range of the elements to be analysed, with a suitable gradient. Choosing an unsuitable series will bias the results, so the choice deserves full attention. In plotting the calibration curve, several reference materials of differing content are normally used as one series, and their composition and structure should be as close as possible to those of the samples to be analysed.

5.3 Standardisation samples - where a change in the state of the instrument causes the measured values to deviate, one or two samples are used to correct the instrument against the original calibration curve; these are the standardisation samples. They shall be homogeneous and of suitable content, and may be selected from the reference materials used for the calibration curve or from other homogeneous, stable samples of suitable content. Where two-point standardisation is used, the contents are taken near the upper and lower ends of the curve of each element.

5.4 Control samples - shall have a chemical composition and structure close to those of the sample to be analysed. They are used to check further the results obtained for the sample from the calibration curve, and may be certified reference materials, reference standards or in-house control samples.

6 Apparatus

Any model of spark atomic emission spectrometer may be used. It shall comprise the excitation source, the spark chamber, the argon system, the counter-electrode, the dispersing system and the measuring system, and shall meet the following requirements: a) the excitation source shall be a stable spark discharge source; b) the purity of the argon in the argon system shall be not less than 99.995 %; c) the vacuum in the optical path of the dispersing system shall be below 3 Pa, or the path shall be filled with a high-purity inert gas that does not absorb wavelengths below 200 nm and whose purity is not less than 99.999 %.

7 Sampling and sample preparation

7.1 Sampling shall be carried out in accordance with GB/T 25050 or GB/T 25051. The sampling shall ensure that the sample is homogeneous and free from shrinkage cavities and cracks.

7.2 The measuring face of the analytical sample shall be large enough to cover the excitation aperture of the spark stand - normally a diameter greater than 16 mm and a thickness greater than 5 mm. The surface shall be flat and clean, and shall be ground before measurement using a grinding wheel, a cup wheel or a belt grinder, or machined on a milling machine. The abrasives used are aluminium oxide, zirconium oxide, silicon carbide and the like, with a grain size normally between 0.124 mm and 0.25 mm. The reference materials, the standardisation samples, the control samples and the analytical samples shall all be ground under the same conditions.

8 Instrument preparation and analytical conditions

8.1 Basic requirements. The spectrometer shall be placed in a vibration-proof, clean laboratory at a room temperature of 16 °C to 30 °C and a relative humidity below 70 %. The maximum permitted variation of the room temperature is 5 °C.

8.2 Analytical conditions and analytical lines. The choice of analytical conditions depends on the analytical software and on the model of spectrometer; different analytical lines may be used for the elements of ferronickel within the contents of Table 1. The recommended analytical conditions are given in Table 2: a gap between sample and counter-electrode of 3.0 mm to 6.0 mm, a flushing time of 2 s to 10 s, a pre-integration time of 3 s to 20 s, an integration time of 2 s to 20 s, and argon flows of 3 L/min to 25 L/min during flushing, 2.5 L/min to 10 L/min during pre-integration and 2.5 L/min to 7 L/min during integration. The analytical and internal standard lines are given in Table 3, together with the elements that may interfere. Note: an internal standard line is not normally used for phosphorus.

9 Analytical procedure

9.1.1 Plotting the calibration curve. Using the spark atomic emission spectrometer under the selected working conditions, excite a series of ferronickel spectrometric reference materials (see 5.2), each at least three times. Plot the calibration curve of emission intensity - or intensity ratio - against content or content ratio, normally choosing a first-order or second-order regression curve. Where a second-order curve shows marked curvature, the curve shall be calibrated in segments. Where necessary, correction shall be made for interfering elements. The ferronickel reference materials shall cover the content range of the elements to be analysed (see Table 1), using at least four levels and keeping a suitable gradient.