

ICS 77.100



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

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**Ferronickels - Determination of sulfur content - Infrared
absorption method after induction furnace combustion
(English Translation)**

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1 Scope

This document specifies the determination of sulfur content in ferronickel. It adopts the infrared absorption method in which an induction furnace is used.

This document applies to the determination of sulfur content in ferronickel. The measurement range (mass fraction) is 0.002%~0.26%.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any 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, Measurement methods and results -- Accuracy (trueness and precision)

-- Part 2.Determine the standard methods of measurement repeatability and reproducibility of the basic method

GB/T 6379.3, Accuracy (trueness and precision) of measurement methods and results -- Part 3.Intermediate measures of the precision of a standard measurement method

GB/T 8170, Rules of rounding off for numerical values and expression and judgement of

limiting values

GB/T 25050, Ferronickel ingots or pieces -- Sampling for analysis

GB/T 25051, Ferronickel shot -- Sampling for analysis

3 Terms and definitions

There are no terms or definitions that require definition in this document.

4 Principle

The test material is heated and burned in an oxygen stream in an induction furnace.

Under the action of flux and accelerators, sulfur is converted into sulfur dioxide, which is carried by the oxygen stream to an infrared absorption cell. Use an infrared detector to measure its absorption of infrared light of a specific wavelength. The sulfur content is determined by the change in infrared energy received by the detector.

5.1 Oxygen

The purity should be at least 99.5% (mass fraction). When organic contaminants are suspected in the oxygen, an oxidizing agent (copper (II) oxide or platinum tube) heated at 600°C should be used. Then, an appropriate carbon dioxide absorbing agent and a water absorbent should be used for absorption and purification.

5.2 Alkali asbestos

Use inert ceramic that is impregnated with sodium hydroxide. Particle size is 0.7 mm~1.2 mm. It is used to absorb carbon dioxide.

5.3 Magnesium perchlorate [Mg(ClO₄)₂]

Particle size is 0.7 mm~1.2 mm. It is used for moisture absorption.

5.5.1 General requirements

Adding a flux can make the small particles of the specimen stick together. It shall make the induction effect in the furnace more effective. It shall produce a melt with good fluidity.

Adding an accelerator can.

a) Create a good coupling induction medium for refractory samples (fine-grained samples, materials with complex compositions). It shall allow for induction heating and melting of refractory samples;

b) Increase the combustion temperature;

c) When the specimen volume is small, adding an accelerator can increase the total

crucible content. It shall create a good coupling medium.

The flux and accelerator used should have a low sulfur content. It should be used simultaneously during the calibration procedure. Various factors (oxygen, crucible, flux, and accelerator) contribute to the sulfur blank value. When the sulfur content of the sample to be measured is $\leq 0.05\%$ (mass fraction), the blank value should not exceed

0.0005% (mass fraction). When the sulfur content of the sample to be measured is $> 0.05\%$ (mass fraction), the blank value should not exceed 0.001% (mass fraction).

NOTE: Certain materials act as both fluxes and accelerators.

5.5.2 Flux

Common fluxes are tin, copper plus tin, copper or tungsten.

5.5.3 Accelerator

Common accelerators are copper, iron, tungsten or nickel.

5.6 Steel or ferronickel certified reference samples

The sulfur content (mass fraction) is 0.001%~0.27%.

6.1 Sulfur analyzer

6.1.1

High-frequency induction furnace infrared sulfur analyzers are available from a variety of manufacturers. The instrument should be operated according to the manufacturer's instructions. A pressure regulator is required to control the oxygen pressure entering the instrument's combustion furnace, as specified by the manufacturer.

6.1.2

Use a quartz tube filled with alkali asbestos (5.2) and magnesium perchlorate (5.3) to purify the oxygen. In standby mode, the flow rate should be maintained at approximately 0.5 L/min.

6.1.3 Install a glass wool (5.4) filter between the high-frequency induction furnace

chamber and the analyzer. Replace it when necessary. Clean the furnace chamber, furnace column, and filter screen regularly to remove oxide residues.

6.2 Ceramic crucible and crucible cover

A ceramic crucible is needed to hold the sample and other necessary additives for the subsequent melting process. The crucible should be precisely sized and fit the support so

that the sample in the crucible is directly inside the heating induction coil.

Typical dimensions of a crucible are.

The crucible and crucible cover should comply with the instrument manufacturer's specifications. They shall be able to withstand the combustion temperature in the induction furnace. They shall not produce sulfur-containing chemicals to meet the blank value requirements specified in the document.

To eliminate the effects of possible sulfur contamination in the crucible on the measurement, calcinate the crucible in a high-temperature furnace with air or oxygen for at least 1 h at 1100°C. After cooling, store in a desiccator or sealed container.

Alternatively, use a resistance furnace with a combustion tube and flow oxygen through the tube. Precondition the crucible in a similar manner.

6.3 Crucible clamp

Capable of gripping the crucible (6.2).

Height

Outer diameter

Inner diameter

Wall thickness

Bottom thickness

Crucible cover aperture more than 10 mm.

7 Sampling and sample preparation

7.1

Sampling shall be carried out in accordance with the provisions of GB/T 25050 or

GB/T 25051. Sample preparation shall be carried out according to the normal agreed procedures. In case of any dispute, the supplier and the purchaser may negotiate separately.

7.2

Laboratory samples are usually in the form of pellets, drill cuttings or milling cuttings. No further processing is required.

7.3

Laboratory samples that have been ground or drilled and contaminated with oil or grease should be cleaned with high-purity acetone and allowed to air dry.