GB

NATIONAL STANDARD OF THE

PEOPLE'S REPUBLIC OF CHINA

ICS 73.060.10

CCS D 31

GB/T 6730.61-2022

Replacing GB/T 6730.61-2005

Iron Ores - Determination of Carbon and Sulfur Content - High Frequency Combustion with Infrared Absorption Method

铁矿石 碳和硫含量的测定 高频燃烧红外吸收法

Issued on: October 12, 2022 **Implemented on: February 1, 2023**

Issued by: State Administration for Market Regulation;

Standardization Administration of the People's Republic of China.

Table of Contents

Iron Ores - Determination of Carbon and Sulfur Content - High Frequency Combustion with Infrared Absorption Method

WARNING---the personnel adopting this document shall have formal laboratory work experience. This document does not point out all possible safety issues, and the user is responsible for taking appropriate safety and health measures and ensuring the compliance with the conditions specified in the relevant national laws and regulations.

1 Scope

This document specifies the determination of carbon and sulfur content in iron ore by high frequency combustion with infrared absorption method.

This document is applicable to the determination of carbon and sulfur content in natural iron ore, iron ore concentrate, sinter, pellets and their products. The determination range (mass fraction) is: carbon 0.01% ~ 2.5%, sulfur 0.001% ~ 2.0%.

2 Normative References

The contents of the following documents constitute indispensable clauses of this document through the normative references in the text. In terms of references with a specified date, only versions with a specified date are applicable to this document. In terms of references without a specified date, the latest version (including all the modifications) is applicable to this document.

GB/T 6682 Water for Analytical Laboratory Use - Specification and Test Methods

GB/T 6730.1 Iron Ores - Preparation of Predried Test Samples for Chemical Analysis

GB/T 6730.3 Iron Ores - Determination of Hygroscopic Moisture in Analytical Samples - Gravimetric, Karl Fischer and Mass-loss Methods

GB/T 8170 Rules of Rounding off for Numerical Values & Expression and Judgement of Limiting Values

GB/T 10322.1 Iron Ores - Sampling and Sample Preparation Procedures

GB/T 12806 Laboratory Glassware - One-mark Volumetric Flasks

GB/T 12807 Laboratory Glassware - Graduated Pipettes

GB/T 12808 Laboratory Glassware - One-mark Pipettes

3 Terms and Definitions

This document does not have terms or definitions that need to be defined.

4 Principle

The specimen is heat and burned in the oxygen flow of a high-frequency induction furnace, and the generated carbon dioxide (or carbon monoxide) and sulfur dioxide are carried by oxygen to the measurement chamber of the infrared analyzer. Carbon dioxide (or carbon monoxide) and sulfur dioxide absorb the infrared energy of a specific wavelength, and the absorption energy is proportional to its concentration. In accordance with the change of the energy received by the detector, determine the carbon and sulfur content.

5 Reagents and Materials

Unless it is otherwise specified in analysis, use only approved reagents of analytical purity and Grade-2 water specified in GB/T 6682 or water of equivalent purity.

5.1 Magnesium perchlorate: anhydrous, particle size: 0.7 mm ~ 1.2 mm.

5.2 Caustic soda asbestos: granular.

5.3 Compound flux: carbon content (mass fraction) less than 0.002%, sulfur content (mass fraction) less than 0.0005%; the mass ratio of tungsten, tin, iron and molybdenum is about 6 : 1 : 2 : 1.

5.4 Tungsten particles: carbon content (mass fraction) less than 0.002%, sulfur content (mass fraction) less than 0.0005%.

5.5 Tin particles: carbon content (mass fraction) less than 0.002%, sulfur content (mass fraction) less than 0.0005%. If necessary, use acetone (see 5.6) to clean the surface and dry at room temperature.

5.6 Acetone: the carbon content (mass fraction) in the evaporated residue is less than 0.005%.

5.7 Pure iron flux: carbon content (mass fraction) less than 0.002%, sulfur content (mass fraction) less than 0.0005%.

5.8 Barium carbonate: fine powder with a content (mass fraction) greater than 99.9%. At 105 °C, dry for 3 h, and cool in a desiccator.

5.9 Potassium sulfate: content (mass fraction) greater than 99.9%. At 105 °C, dry for 1 h, and cool in a desiccator.

an oxygen-enriched atmosphere of a high-temperature furnace, then, store in a desiccator.

6.4 Air source, including the following parts:

a) Carrier gas system: including oxygen container, two-stage pressure regulator and sequential control part to ensure the supply of appropriate pressure and rated flow;

b) Power gas source system: including power gas, two-stage pressure regulator and sequential control part that can supply appropriate pressure and rated flow.

6.5 High-frequency induction furnace, which shall satisfy the requirements for the melting temperature of specimen.

6.6 Control system, including the following parts:

a) Microprocessor system: including central processing unit, memory, keyboard input device, information center display screen and analysis result printer, etc.;

b) Control function: including loading and unloading of crucible and furnace table lifting, cleaning, analysis condition selection device, monitoring and alarm interruption of analysis process, collection, calculation, correction and processing of analysis data, etc.

6.7 Measuring system, which is mainly composed of a balance (with a sensitivity of less than 1.0 mg), infrared analyzer and electronic measuring component, etc.

7 Sampling and Sample Preparation

7.1 Laboratory Specimen

In accordance with GB/T 10322.1, conduct sampling and sample preparation. Generally, the particle size of the specimen shall be less than 100 μm. If the content of chemically combined water or easily oxidized substances in the specimen is high, the particle size shall be less than 160 μm. The stipulations of the high content of chemically combined water and easily oxidized substances shall comply with GB/T 6730.1.

7.2 Preparation of Pre-dried Specimen

Thoroughly mix the laboratory specimen and use the partial sample reduction method for the sampling. In accordance with GB/T 6730.1, at 105 °C ± 2 °C, pre-dry the specimen for 2h, then, cool it to room temperature in a desiccator, and reserve it for later use. If there is a large amount of chemically combined water or a high content of easily oxidized substances in the sample, then, adopt the air balance method in GB/T 6730.3 to pre-dry the specimen.

When preparing a series of reference materials, its content can be appropriately adjusted in accordance with the carbon and sulfur content of the specimen to be tested.

8.6.2 Use carbon and pyrite reference materials for multi-point calibration

In accordance with the carbon and sulfur content of the specimen to be tested, select three reference materials (the carbon and sulfur content of the specimen to be tested is within the range of the carbon and sulfur content of the three reference materials) of the same type of iron ore and successively conduct the measurement. If the measurement results are within the admissible error, confirm the linearity of the system, otherwise, the linearity of the system shall be re-adjusted. In accordance with the carbon and sulfur content, adjust the sampling size, so that the sampling size of the specimen is as consistent as possible with the sampling size of the reference materials.

8.6.3 Use carbon and pyrite reference materials for single-point calibration

In accordance with the carbon and sulfur content of the specimen to be tested, select a reference material (the carbon and sulfur content of the specimen to be tested is as close as possible to the carbon and sulfur content of the reference material) of the same type of iron ore and conduct the measurement. If the measurement results are within the admissible error, confirm the linearity of the system, otherwise, the linearity of the system shall be re-adjusted. In accordance with the carbon and sulfur content, adjust the sampling size, so that the sampling size of the specimen is as consistent as possible with the sampling size of the reference material.

8.7 Specimen Analysis

In accordance with 7.2, weigh-take the specimen; in accordance with the mode of 8.7 a) or 8.7 b), add flux. Use the same conditions and procedures as the determination of the reference materials to conduct the measurement.

a) Place the weighed sample in a burnt crucible (see 6.3) and add 2.0 g of compound flux (see 5.3).

b) Place the weighed sample in a burnt crucible (see 6.3) covered with 0.9 g of pure iron flux (5.7), add 0.2 g of tin particles (see 5.5) or a flattened and folded tin tube (see 5.13), then, cover it with 0.4 g of pure iron flux (see 5.7) and 1.9 g of tungsten particles (see 5.4).

9 Result Calculation and Presentation

9.1 Measurement of Carbon and Sulfur Content

In accordance with the relations between the absorption energy and the concentration of carbon and sulfur, obtain the carbon and sulfur content from the calibration curve.

9.2 General Processing of Analysis Results

This is an excerpt of the PDF (Some pages are marked off intentionally)

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