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

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# Iron Ores - Determination of Loss on Ignition - Gravimetric Method Corrected by Hygroscopic Water

铁矿石 灼烧减量的测定 吸湿水校正重量法

(ISO 11536:2015 Iron Ores - Determination of Loss on Ignition - Gravimetric Method, MOD)

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#### **Foreword**

This document was drafted in accordance with the rules provided in GB/T 1.1-2020 *Directives* for Standardization - Part 1: Rules for the Structure and Drafting of Standardizing Documents.

This is Part 83 of GB/T 6730. See Appendix A for the published parts of GB/T 6730.

This document modifies and adopts ISO 11536:2015 Iron Ores - Determination of Loss on Ignition - Gravimetric Method.

In comparison with ISO 11536:2015, this document makes the following structural adjustments:

- --- The chapter "Terms and Definitions" is added to this document;
- ---Appendix B corresponds to Appendix A in ISO 11536:2015.

The technical differences between this document and ISO 11536:2015, and the causes for these differences are as follows:

- --- The normative references "GB/T 6730.1" and "GB/T 8170" are added (see Chapter 7 and 9.2.5), so as to adapt to the technical conditions of China;
- ---The preparation of laboratory specimens is modified (see Chapter 7; Chapter 6 of ISO 11536:2015): the specific requirements are clarified, so as to facilitate the practical operation;
- ---The method for preparing air balance samples is added (see 8.1; 7.1 of ISO 11536:2015), because it is necessary to specify the specific requirements for preparing air balance samples;
- ---The determination of the test end point is modified (see 8.5; 7.5 of ISO 11536:2015), so as to enhance the accuracy of the determination and maintain consistency with the mode of determining the moisture determination end point in GB/T 10322.5;
- ---The expression of precision (see 9.2.1; 8.2.1 of ISO 11536:2015) and the calculation formula of C value in correctness check (see 9.2.4, 8.2.4 of ISO 11536:2015) are modified, so as to comply with the requirements for precision expression in Chinese national standards;
- ---The expression of numerical value rounding-off is modified (see 9.2.5; 8.2.5 of ISO 11536:2015), and GB/T 8170 is used to replace the text description of 8.2.5 in ISO 11536:2015.

This document makes the following editorial modifications:

--- The title of the Standard is modified;

## Iron Ores - Determination of Loss on Ignition - Gravimetric Method Corrected by Hygroscopic Water

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

## 1 Scope

This document specifies the method for the determination of loss on ignition in iron ores using gravimetric method corrected by hygroscopic water.

This document is applicable to natural iron ores, iron concentrations and artificial lumpy iron ores, with a determination range (mass fraction) of:  $1.0\% \sim 10.0\%$ .

This document does not apply to the following circumstances:

- a) Processed iron ores containing metallic iron (direct reduced iron);
- b) Natural or processed iron ores with sulfur content exceeding 0.2%;
- c) Natural or processed iron ores with ferrous oxide content exceeding 1.0% (mass fraction).

#### 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 6730.1 Iron Ores - Preparation of Predried Test Samples for Chemical Analysis (GB/T 6730.1-2016, ISO 7764:2006, MOD)

GB/T 6730.3 Iron Ores - Determination of Hygroscopic Moisture in Analytical Samples - Gravimetric, Karl Fischer and Mass-loss Methods (GB/T 6730.3-2017, ISO 2596:2006, MOD)

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 10322.1-2014, ISO 3082:2009, IDT)

### 3 Terms and Definitions

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

## 4 Principle

The specimen is burned at 1,000 °C to a constant weight and cooled in a desiccator. Determine the mass loss of the specimen and express it as a mass fraction relative to the original mass, that is, the loss on ignition.

## **5** Reagents

Unless otherwise stated, only approved analytically pure reagents are used during the analysis.

Silica gel desiccant, dry at 105 °C for 4 h.

### 6 Instruments

- **6.1** Crucible (porcelain crucible or platinum crucible): with a capacity of  $15 \text{ mL} \sim 25 \text{ mL}$ , with a lid. The crucible and its lid shall be pretreated in a muffle furnace at 1,000 °C for 60 min. Before use, the crucible and its lid shall be stored in a vacuum desiccator (see 6.4). If the platinum crucible used for ignition loss analysis is used for flux melting, the platinum vessels shall be thoroughly cleaned, so as to prevent cross-contamination.
- **6.2** Balance: with a division value of 0.1 mg.
- **6.3** Muffle furnace: capable of maintaining a temperature of 1,000 °C  $\pm$  25 °C and providing sufficient air circulation, so as to prevent water vapor retention.
- **6.4** Vacuum desiccator: with an inner diameter of 150 mm  $\sim$  250 mm, with vacuum cock, which can maintain air pressure balance. The desiccator base plate shall be made of metal, ceramic or other heat-resistant materials, which will not break when in contact with the crucible at 1,000 °C. The edges of the desiccator shall be coated with a small amount of silicone grease or Vaseline. 150 g  $\sim$  200 g of silica gel shall be replaced every day (see Chapter 5). If a platinum crucible is used, then, metal desiccator base plate shall not be used.

## 7 Preparation of Laboratory Specimens

In accordance with GB/T 10322.1, take and prepare the specimens. Generally, the particle size of the specimens shall be less than 100  $\mu$ m. If the content of combined water or readily oxidizable substances in the specimens is high, the particle size shall be less than 160  $\mu$ m.

The stipulations for high content of combined water and readily oxidizable substances shall

comply with GB/T 6730.1.

## **8 Analytical Procedures**

#### 8.1 Specimens

Thoroughly mix the laboratory specimens (see Chapter 7) and take multiple specimens to ensure that each specimen is representative of the laboratory sample. The specimens shall be air balanced.

Air balance is to lay the test sample flat in a tray, with the density of the sample layer not greater than 0.1 g/cm<sup>2</sup>, in a laboratory environment, perform air balance for 2 h, and quickly mix it before testing.

#### 8.2 Number of Determinations

In accordance with Appendix B, the same specimen shall be independently determined at least twice.

**NOTE:** "independent" means that the results of the second and any subsequent determinations are not affected by the previous determination. In this analytical method, this condition means repeated determinations independently carried out in the same laboratory, by the same operator using the same equipment and the same test method on the same test object in a short period of time, including appropriate re-calibration.

#### 8.3 Determination of Hygroscopic Water

Two parallel specimens shall be simultaneously determined, one for the determination of loss on ignition and the other for the determination of hygroscopic water. The determination of hygroscopic water shall be carried out in accordance with GB/T 6730.3.

#### 8.4 Mass of Specimen

Weigh-take about 2.0 g  $\pm$  0.2 g of specimen (see 8.1), accurate to 0.0001 g.

#### 8.5 Measurement

Place the specimen in the crucible with a lid (see 6.1), put it into the muffle furnace (see 6.3), and burn it at  $1,000\,^{\circ}\text{C} \pm 25\,^{\circ}\text{C}$  for  $60\,\text{min} \pm 10\,\text{min}$  (make sure that there is no loose material in the muffle cavity that may adhere to the crucible). Take the crucible out of the muffle furnace and place it in the desiccator (see 6.4). Immediately place the lid on the crucible and let it cool to room temperature. Slowly release the pressure, take it out and quickly complete the weighing. Repeat this operation, until the mass difference after two consecutive burnings does not exceed 0.05% of the initial mass of the specimen, and take the mass after the last burning as the basis for calculation.

The operating temperature and speed of specimen weighing and empty crucible weighing shall

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