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Steel and Iron - Determination of Hydrogen Content - Thermal Conductivity/Infrared Method after Fusion under Inert Gas

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Table of Contents

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Steel and Iron - Determination of Hydrogen Content - Thermal Conductivity/Infrared Method after Fusion under Inert Gas

1 Scope

This Part in GB/T 223 specifies methods of determining hydrogen content through thermal conductivity or infrared method after fusion under inert gas.

This Part is applicable to the determination of hydrogen content in steel and iron (mass fraction: 0.6 μg/g ~ 30.0 μg/g).

2 Normative References

The following documents are indispensable to the application of this Standard. In terms of references with a specified date, only versions with a specified date are applicable to this Standard. The latest version (including all the modifications) of references without a specified date is also applicable 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

Place reagent in a degassed graphite crucible; heat it up and fuse it under inert gas. In reagent, hydrogen is released in the form of molecular, and enters carrier gas flow. After isolating it from other gases with chromatographic column, conduct the detection in thermal conductivity cell. Hydrogen molecule passes through hot copper oxide with carrier gas flow, then, converts into water; conduct the detection in specific infrared pool. In accordance with changes in thermal conductivity or infrared absorption intensity signal, calculate the hydrogen content.

hydrogen determination sample, because of hydrogen transfer during the processing.

6.6 Other Samples

Materials in abnormal shapes shall be sampled in accordance with the materials. The operation of sampling can adopt certain modes of processing, such as cutting, sawing and wire-electrode cutting. Avoid overheat and contamination. Wire materials can be directly sampled.

7 Analytical Procedures

7.1 Instrument Preparation

7.1.1 Initiate the instrument in accordance with requirements in the instruction for use provided by instrument manufacturer; confirm that flow meter and pressure gauge on the instrument are indicating the specified position. Examine whether the impurity removal device, and filter and reagent of the auxiliary purification system are available; if they are unavailable, rinse them or replace them.

7.1.2 If necessary, conduct air-leak check of the instrument while the power is connected, and the water-cooling system is initiated.

7.1.3 Configurate parameters of the instrument; pre-heat and stabilize the instrument. The following analytical conditions are recommended:

a) Degassing power: 3,000 W or current 850 A;

b) Analytical power: 2,500 W or current 700 A;

c) Analytical time: 90 s.

7.1.4 If the instrument is powered off for a long time, start pre-heating for over 1 h after initiating the instrument. Confirm the stability of the instrument and that the various indexes reach the previously set value through blank test. After replacing the filter and purifying the reagent, the instrument is in a non-operating state; stabilize the instrument through 2 ~ 3 times of dry burning.

7.2 Blank Test

Conduct blank test before the procedure of analysis and determination. Conduct at least 3 blank value determinations; replace a new crucible in each determination. The deduction of blank value can be automatic or manual. When the function of automatic blank deduction is operated, the blank value displayed on the instrument shall be stable. When the difference among the 3 successive blank values is < 20%, input the average value of the last 3 blank values; start automatic deduction. In terms of manual deduction, sample with hydrogen content < 1 μg/g shall be the main object. Deduct one by one, namely, conduct a blank test first, then, prepare an individual sample.

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7.3.2.5 In accordance with the procedure described in the instruction for use of the instrument, store this calibration in the form of method; name this method.

7.3.3 Calibration frequency requirements

Instrument shall be calibrated for at least 1 time every year. When any of the abnormal circumstances emerge, instrument shall be re-calibrated to guarantee the reliability and validity of measurement:

a) Instrument manifests symptoms of being contaminated and long tail in any sample;

b) There are unexpected failures that might influence the analytical result;

c) Main parameters are modified.

Before and after each analysis, hydrogen standard substance/standard sample (4.8) in steel (iron) shall be adopted to confirm calibration (when the above-mentioned standard substance/standard sample does not exist, control sample can also be adopted as a replacement). When the result of measurement exceeds the uncertainty range of the standard value, find out reasons and re-calibrate.

7.4 Sample Analysis

7.4.1 Select a suitable analytical method (7.3.1.5 or 7.3.2.5). The adopted analytical conditions shall maintain consistency with the analytical conditions adopted for calibration. If the sample to be determined is powdered, corresponding standard substance/standard sample shall be adopted for calibration.

7.4.2 Input sample information in accordance with the procedure required in the instruction for use of the instrument.

7.4.3 Weigh-take 0.5 g ~ 1.0 g of sample (accurate to 0.001 g) (if there are any special requirements, the maximum weight can reach 5.0 g). Most instruments can automatically load the weight through the connection of balance and instruments. When this function is not equipped, manually input the weight in accordance with the requirements in the instruction for use of the instrument.

7.4.4 Place the sample (7.4.3) into a sample injector; analyze under selected analytical conditions. After analysis is completed, the instrument shall automatically display the analytical result.

8 Result Calculation

Hydrogen content shall be calculated by mass fraction (μg/g).

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