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Replacing GB/T 223.71-1991

Methods for Chemical Analysis of Iron, Steel and Alloy
The Gravimetric Method after Combustion in the Pipe
Furnace for the Determination of Carbon Content

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Foreword

This Standard was drafted by revising GB 223.71-91, *Methods for Chemical Analysis of Iron Steel and Alloy – the Combustion Gravimetric Method for the Determination of Carbon Content*, in accordance with the national standard of the People's Republic of China, GB/T 1.1-1993, *Directives for the Work of Standardization – Unit 1: Drafting and Presentation of Standards – Part 1: General Rules for Drafting Standards*, and GB 1.4-88, *Directives for the Work of Standardization – Rules for Drafting Chemical Analysis Methods*.

In accordance with 4.2.3 and 4.3.3 of Article 4 of GB/T 1.1-1993 and 6.10 of Article 6 of GB 1.4-88, "Foreword", "Normative References" of Article 2, "Sampling and Sample Preparation" of Article 6 and "Test Report" of Article 10 are added in this revision.

Annex A and Annex B of this Standard are informative.

As from the effective date of this Standard, this Standard replaces GB 223.71-91.

This Standard was proposed by the Ministry of Metallurgical Industry of the People's Republic of China.

This Standard shall be under the jurisdiction of the National Standardization Technical Committee on Steels.

This Standard was drafted by Shanghai Metallurgical Equipment Factory and Ministry of Metallurgical Industry Central Iron and Steel Research Institute.

The main drafters of this Standard: Jin Shenghui, Wang Yuxing, Guo Xiang, Cui Qiuhong.

This Standard was issued for the first time in the name of GB 223.1 (1)-81 in 1981; and it was revised in the name of GB 223.71-91 in 1991, in which the precision of method was decided.

Methods for Chemical Analysis of Iron, Steel and Alloy The Gravimetric Method after Combustion in the Pipe Furnace for the Determination of Sulfur Content

1 Application Scope

This Standard specifies the gravimetric method after combustion in the pipe furnace for the determination of sulfur content.

This Standard applies to the determination of carbon content of 0.10% $(m/m) \sim 5.00\%$ (m/m) in iron, steel, high temperature alloy and precision alloy.

2 Normative References

The provisions in following documents become the provisions of this Standard through reference in this Standard. All editions shown are effective when this Standard is issued. All standards will be revised, however, parties who reach an agreement based on this Standard are encouraged to study if the latest versions of the following standards are applicable.

GB 222-84, Method of Sampling Steel for Determination of Chemical Composition and Permissible Variations for Product Analysis

GB 6379-86, Precision of Test Methods – Determination of Repeatability and Reproducibility for a Standard Test Method by Interlaboratory Tests

3 Method Summary

Test sample and fluxing agent burn in a pipe furnace at high temperature (1 250° C \sim 1 350° C) by pumping in oxygen; carbon is fully oxidized into carbon dioxide. After removing sulphur dioxide, use the soda asbestos absorption flask of a known mass to absorb the carbon dioxide in the mixed gas. Weigh the increment of the absorption flask and calculated the carbon content of test sample based on the increment.

4 Reagents and Materials

4.1 Oxygen of purity not lower than 99.5% (m/m).

For the samples of iron, carbon steel and low alloy steel, raise the temperature to 1 200°C ~ 1 250°C; and for medium- and high-alloy steel, high-temperature alloy and precision alloy, raise the temperature to over 1 350°C.

NOTE For some high-temperature alloys, such as cobalt-base alloys and titanium-base alloys, it is hard to melt in a furnace pipe, so the they may be determined using the high-frequency infrared absorption method after combustion in induction furnace.

- **7.1.2** Pump in oxygen, check whether the pipeline and valve of the whole equipment leaks, place the porcelain boat loaded with test sample in the porcelain tube, use the silicone rubber stopper to seal the porcelain tube, observe the chromic acid saturated sulphuric acid solution in the quadruple ball and if there is any backflow, it indicates the equipment does not leak.
- **7.1.3** Connect the water absorption flask and carbon dioxide absorption flask, unwind the valves in succession, pump in oxygen (at the flow rate of $500 \text{ mL/min} \sim 800 \text{ mL/min}$), conduct gas washing for $3 \text{ min} \sim 5 \text{ min}$, and close the valves of oxygen and two absorption flasks. Use the long hook to pull back the porcelain boat. After weighing, repeat the operation of pumping in oxygen until the difference between two weighed values is less than 0.3 mg; and analysis may be started after putting the carbon dioxide absorption flask back.

NOTE

- 1 Pump in oxygen for the newly-loaded carbon dioxide absorption flasks for $20 \text{ min} \sim 30 \text{ min}$, analyze the test samples of high secondary carbon content and weigh. Pump in oxygen once again for $3 \text{ min} \sim 5 \text{ min}$, weigh and repeat $1 \sim 2 \text{ times}$, and the difference between two weighed values shall be less than 0.3 mg (when the weight of loss is much more than the value, it indicates that there is water loss, so the value is unusable).
- 2 When the height of soda asbestos in the carbon dioxide absorption flask becoming white is close to one third of the height of all soda asbestos, the soda asbestos must be replaced. During the mounting of the carbon dioxide absorption flask, operate quickly; or else, the soda asbestos will absorb the carbon dioxide and water in the air soon. The mounting of the carbon dioxide absorption flask can affect the analytical results. If the glass wool is spread uniformly on the bottom, carbon dioxide will rise uniformly during absorption and the absorption is satisfactory, and otherwise, the absorption is unsatisfactory.

7.2 Blank test

Spread fluxing agent (see Table 1) uniformly in the porcelain boat, carry out determination as specified in 7.3.3 and 7.3.4 and determine carefully the increment of the carbon dioxide absorption flask (m_1). The blank value is normally not greater than 0.3 mg; and if the blank value is abnormal, find the cause before continuing operation and carry out blank test once again.

Close the valves of oxygen and the two absorption flasks, take down the two carbon dioxide absorption flasks, use the rubber stopper to seal the gas inlet of the carbon dioxide absorption flask before placing on the balance, and use a long hook to pull back the porcelain. Determine the increment (m_2) of the absorption flasks after waiting for 5 min ~ 10 min.

7.3.5 Check the porcelain boat and if the slag is uneven and the section of slag has pores, it indicates the combustion is not complete, so test samples shall be weighed and determined once again.

NOTE

- 1 The time for preheating high-alloy test samples shall be increased; and oxygen may be pumped in by observing the rise of the liquid level of chromic acid saturated sulphuric acid in the quadruple ball.
- 2 During the continuous analysis of test samples, the content may be calculated as the difference between the weight measured this time minus the weight measured last time. If the analysis is interrupted for more than 20 min, oxygen shall be pumped in once again for gas washing for 3 min \sim 5 min and the weight of the carbon dioxide absorption flask shall be measured once again.

8 Analytical Result and Its Expression

The content content expressed in mass percentage is calculated in accordance with Formula (1).

$$C[\%(m/m)] = \frac{(m_2 - m_1) \times 0.272 \text{ 9}}{m} \times 100$$
(1)

where,

 m_1 —the mass of carbon dioxide measured in blank test, g;

 m_2 —the mass of carbon dioxide measured in the analysis of test sample, g;

m—the mass of test sample, g;

0.272 9—the coefficient for conversion of carbon dioxide into carbon.

9 Precision

The precision of this Standard is the 10 carbon levels selected from 8 laboratories in 1988; and each sulphur level is determined by each laboratory through three joints tests as specified in GB 6379. See Annex A for the test samples. See Annex B for the raw data (measured values) given by all laboratories.

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