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Replacing GB/T 223.69-1997

Iron, steel and alloy - Determination of carbon contents - Gas-volumetric method after combustion in the pipe furnace

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Foreword

This Part of GB/T 223 replaces GB/T 223.69-1997 “Methods for chemical analysis of iron, steel and alloy - The gas-volumetric method after combustion in the pipe furnace for the determination of carbon content”.

Compared with GB/T 223.69-1997, main modifications of this Part are as follows:

- MODIFY the name to “Iron, steel and alloy - Determination of carbon contents - Gas-volumetric method after combustion in the pipe furnace”.
- MODIFY the equations for calculation of results and the units of the amount in the equations;
- NORMALIZE the description of the precision function.

Annexes A and B of this Part are informative annexes.

This Part is proposed by China Iron and Steel Association.

This Part is under the jurisdiction of National Technical Committee on Iron & Steel of Standardization Administration of China.

Drafting organization of this Part: China Iron & Steel Research Institute Group.

Main drafters of this Part: Cui Qiuhong, Wang Yuxing.

The historical editions of the standard replaced by this Part are as follows:

GB 223.69-1989, GB/T 223.69-1997.

Iron, steel and alloy - Determination of carbon contents - Gas-volumetric method after combustion in the pipe furnace

Warning: Personnel using this Part shall have practical experience in formal laboratory work. This Part does not indicate all possible security issues. It is the user's responsibility to take appropriate safety and health measures and to ensure compliance with the requirements of the relevant state laws and regulations.

1 Scope

This Part of GB/T 223 specifies a gas-volumetric method after combustion in the pipe furnace for the determination of the carbon content.

This Part applies to the determination of the carbon content with the mass fraction of 0.10 % ~ 2.00 % in steel, iron, high temperature alloys and precision alloys.

2 Normative references

The following standards contain provisions which, through reference in this Part of GB/T 223, constitute provisions of this Part. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this Part are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. For undated references, the latest edition of the normative document referred to applies.

GB/T 6379.1 Accuracy (trueness and precision) of measurement methods and results - Part 1: General principles and definitions

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 20066 Steel and iron - Sampling and preparation of samples for the determination of chemical composition

1 - oxygen bottle;
2 - partial pressure gauge (with flow meter and buffer valve);
3 - buffer bottle;
4 - gas washing bottle I;
5 - gas washing bottle II;
6 - drying tower;
7 - oxygen supply piston;
8 - glass ground stopper;
9 - pipe furnace;
10 - temperature controller (or pressure regulator):
11 - spherical drying pipe;
12 - desulfurizing pipe;
13 - capacity carbon detector (including serpentine pipe a, gas burette b, levelling bottle c, absorber d, small piston e, three-way piston f):
14 - porcelain pipe;
15 - porcelain boat.

Figure 1 -- Diagram of instrument and equipment

5.1 Oxygen purification device

5.1.1 Buffer bottle (see Figure 1)

5.1.2 Gas washing bottle I (see Figure 1), containing potassium permanganate - potassium hydroxide solution (4.4), and the amount of the solution loading is about a third of the volume of the gas washing bottle I.

5.1.3 Gas washing bottle II (see Figure 1), containing sulfuric acid (ρ about 1.84 g/mL). and the amount of sulfuric acid loading is about one third of the volume of the gas washing bottle II.

5.1.4 Drying tower (see Figure 1), containing asbestos (or soda lime) on the upper layer, and anhydrous calcium chloride on the lower layer, separated with glass wool (4.8) in the middle; the bottom and the top are also covered with glass wool.

5.2 Pipe furnace (see Figure 1)

With thermocouple and temperature controller. High-temperature heating equipment can also be high-frequency heating device.

5.3 Porcelain pipe (see Figure 1)

The porcelain pipe is of 600 mm length and 23 mm diameter (porcelain pipes of similar specifications can also be used). The thick end of the porcelain pipe is connected to the glass ground stopper, and the conical end is connected to the spherical drying pipe by a rubber pipe. Check whether it leaks before use, and then burn. The rubber stopper

7 Analysis procedure

WARNING: For combustion analysis, the danger comes mainly from burns during pre-calcining of the porcelain boat and melting. In analysis, use tweezers whenever possible to hold the porcelain boat and use suitable containers for the placement of the porcelain boat. There must be formal preventive measures when operating the oxygen bottle. Due to the risk of fire in the presence of high concentrations of oxygen in confined spaces, oxygen from the combustion process must be efficiently removed from the apparatus.

7.1 MOUNT the porcelain pipe, TURN on the power, HEAT up. For iron, carbon steel and low alloy steel samples, heat up to 1 200 °C ~ 1 250 °C; for refractory samples such as high alloy steel, high temperature alloy, heat up to 1 350 °C.

NOTE: Some high temperature alloys, such as cobalt-based alloys and titanium-based alloys, are difficult to melt with a pipe furnace, so an infrared absorption method after combustion in an induction furnace may be used for determination.

7.2 INTRODUCE oxygen, CHECK whether the piping and pistons of the entire apparatus leak. ADJUST the apparatus and maintain in normal working condition. After replacing the blocking solution in the levelling bottle (4.5 or 4.6), glass wool (4.8), desulfurization agent (4.3) and potassium permanganate - potassium hydroxide solution (4.4), high-carbon sample shall be burned several times to make carbon dioxide saturate before starting the analysis.

7.3 Blank test

The temperature of the solution in the absorption bottle and the leveling bottle and the temperature of the mixed gas to be tested shall be basically the same, otherwise, there will be positive and negative blank values. The sample shall be subjected to repeated blank test according to 7.6.1 (without the sample) and 7.6.2 before analysis, until a stable blank test value is obtained. Due to changes in room temperature and changes in water temperature in the condenser tube caused in analysis, the blank test shall be carried out often in the process of measuring the sample.

7.4 SELECT appropriate standard sample to measure according to the analysis procedures of 7.5 ~ 7.6.3, to check the apparatus; START the sample analysis only after the apparatus meet the requirements.

7.5 Sample amount

WASH the oil or dirt on the sample surface with suitable solvent (4.2). REMOVE residual washings by heating and evaporation.

WEIGH the sample amount according to table 1.