ICS 77.080.01
CCS H11

NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB/T 223.69-2008

Replacing GB/T 223.69-1997

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

钢铁及合金 碳含量的测定 管式炉内燃烧后气体容量法

Issued on: May 13, 2008 **Implemented on: November 1, 2008**

Issued by: General Administration of Quality Supervision, Inspection and Quarantine of the People's Republic of China;
Standardization Administration of China

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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

GB/T 223.69-2008 is the Chinese national standard on iron, steel and alloy - determination of carbon contents - gas-volumetric method after combustion in the pipe furnace, in the field of metallurgy. The /T suffix marks it as a recommended standard: it is not compulsory by itself, but it becomes binding as soon as a contract, a tender or a customer specification calls it up - which in practice is how most foreign buyers meet it. It was issued on 13 May 2008 by the General Administration of Quality Supervision, Inspection and Quarantine of the People's Republic of China; Standardization Administration of China, and has been in force since 1 November 2008. Classification: ICS 77.080.01, CCS H11. This page is published from the official record of the standard held by the Chinese standards administration: the identification, the dates, the classification and the issuing body are taken from there. The clause text, the tables and the numeric limits are in the document itself, which is delivered complete in English translation.

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

3 Principle

The sample and flux are combusted in the high temperature (1 200 °C ~ 1 350 °C) pipe furnace with oxygen introduced, and the carbon is completely oxidized to carbon dioxide. After removing the sulfur dioxide, collect the gas mixture in the gas burette, and measure the volume. Use potassium hydroxide solution to absorb carbon dioxide, and then measure the volume of the remaining gas. The difference between the volume of gas before and after

absorption is the volume of carbon dioxide, which is used to calculate the carbon content.

4 Reagents and materials

4.1 Oxygen, with the purity of not less than 99.5 % (volume fraction). If oxygen is suspected of containing organic impurities, an oxidizing catalyst [copper oxide (II) or platinum] pipe heated to the temperature of above 45 °C shall be added before using the oxygen purification device.

4.2 Solvent, applicable to washing the oil or dirt on the surface of the sample, such as acetone.

4.3 Active manganese dioxide (or silver vanadate), granular. When there is not suitable chemically active grade manganese dioxide, it can be prepared as follows: To prepare about 50 g of active manganese dioxide, DISSOLVE 200 g of manganese tetra-sulfate manganese sulfate ($MnSO_4 \cdot 4H_2O$) in

2.5 L of water in a 4 L beaker, ADJUST it to alkaline with ammonia (rho is about

0.90 g/mL), ADD 1 L of freshly prepared ammonium persulfate solution (225 g/L), HEAT the solution to boil and keep boiling for another 10 min. During the heating and boiling, in order to keep the solution being ammoniac, ammonia shall be continuously added to settle the precipitate. If the supernatant does not clear or precipitate does not settle quickly, it may add 50 mL ~ 100 mL of ammonium persulfate solution (225 g/L), boil for 10 min and keep the solution always being ammoniac. ALLOW the solution to stand for some time so that the manganese dioxide is completely precipitated. SIPHON the supernatant carefully and WASH the precipitate with 3 L or 4 L of warm water at 500 mL to 600 mL each using decantation method. After each wash and before decantation, fully stir the manganese dioxide in water to make it settle. Finally, wash with dilute sulfuric acid solution [add 2 drops of sulfuric acid per 1 000 mL of solution (rho is

1.84 g/mL)] twice in the same manner. During this period, PREPARE a funnel with the diameter of 15 cm, TAKE a filter plate with the diameter of 5 cm and PLACE it on the funnel, and COVER the filter plate with a thin layer of purified asbestos pulp (it may also use a Brinell porcelain funnel instead of a filter plate). After the last wash, TRANSFER the manganese dioxide to the filter, RINSE it with warm water until it is free of sulfate ions, and PLACE it on a porcelain plate and DRY in an oven at 105 °C. Finely GROUND the manganese dioxide in a mortar so that it passes through a sieve with the diameter of

0.8 mm, and then sufficiently DRY at 105 °C.

4.4 Potassium permanganate - potassium hydroxide solution, WEIGH 30 g of potassium hydroxide and DISSOLVE in 70 mL of potassium permanganate saturated solution.

4.5 Sulfuric acid blocking solution, ADD 1 mL of sulfuric acid (rho is about

1.84 g/mL) to 1 000 mL of water, and ADD a few drops of methyl orange solution (1 g/L) until there is a stable light red.

4.6 Sodium chloride blocking solution, WEIGH 26 g of sodium chloride and DISSOLVE in 74 mL of water, ADD a few drops of methyl orange solution (1 g/L), ADD sulfuric acid (1 + 2) dropwise until there is a stable light red.

4.7 Flux, granular tin, copper, copper oxide, vanadium pentoxide, iron powder. The carbon content of each flux shall generally not exceed 0.005 0 % by mass. The blank test shall be carried out before use, and the blank value shall be deducted from the measured value of the sample.

5 Instruments and equipment

In analysis, except for the following provisions, only ordinary laboratory instruments and equipment are used. Instruments and equipment installation are shown in Figure 1. 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

5.1.1 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 (rho 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.