ICS 77.080.01
CCS H11

NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB/T 223.26-2008

Replacing GB/T 223.27-1994

Iron, steel and alloy - Determination of molybdenum content - The thiocyanate spectrophotometric method

钢铁及合金 钼含量的测定 硫氰酸盐分光光度法

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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1 Scope

GB/T 223.26-2008 is the Chinese national standard on iron, steel and alloy - determination of molybdenum content - the thiocyanate spectrophotometric method, 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 the determination of molybdenum by thiocyanate direct spectrophotometry and thiocyanate-butyl acetate extraction spectrophotometry. The method-1 of this part is applicable to the determination of molybdenum content of 0.10%~2.00% (mass fraction) in medium-and-low alloy steel, high- temperature alloy steel, precision alloy. The method-2 of this part is applicable to the determination of molybdenum content of 0.0025% ~ 0.20% (mass fraction) in pig iron, carbon steel, alloy steel.

2 Normative references

The provisions in following documents become the provisions of this standard through reference in this part of

GB/T 223. For the dated references, the subsequent amendments (excluding corrections) or revisions do not apply to this part; however, parties who reach an agreement based on this standard are encouraged to study if the latest versions of these documents are applicable. For undated references, the latest edition of the referenced document applies.

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

GB/T 6379.2 Measurement methods and results - Accuracy (trueness and precision) - Part 2: Determine the standard methods of measurement repeatability and reproducibility of the basic method

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

3 Method-1: Thiocyanate direct spectrophotometry

3.1 Principle In the sulfuric acid-perchloric acid medium, use stannous chloride to reduce iron and molybdenum. Molybdenum reacts with sodium thiocyanate to produce an orange-red complex. Measure the absorbance. In the color-developing solution, there is no impact if the amount of copper is less than

0.2 mg, the amount of vanadium is less than

0.05 mg, the amount of cobalt is less than

0.8 mg, the amount of antimony is less than

0.8 mg, the amount of chromium is less than

3.2 Reagents and materials Unless otherwise stated, only analytically pure reagents and distilled water or water of comparable purity are used in the analysis.

3.2.1 Hydrochloric acid, rho is about

3.2.2 Nitric acid, rho is about

3.2.3 Sulfuric acid, rho is about

1.84 g/mL, which is diluted to 1 + 1.

3.2.4 Sulfuric acid, rho is about

1.84 g/mL, which is diluted to 5 + 95.

3.2.5 Mixed acid of sulfuric acid-phosphoric acid, in 700 mL of water, slowly add 150 mL of sulfuric acid (rho is about

1.84 g/mL). After cooling it slightly, add 150 mL of phosphoric acid (rho is about

3.2.9 Iron solution, 20 g/L. Weigh

2.0 g of pure iron (the molybdenum content, in mass fraction, must be less than 0.001%). Place it in a 250 mL beaker. Add 40 mL of mixed acid of sulfuric acid-phosphoric acid (3.2.5). Heat to dissolve it. Add nitric acid (3.2.2) dropwise to oxidize it. Heat it until sulfuric acid smoke is produced. Take it off and cool it slightly. Add 40 mL of water. Heat to dissolve the salt. Cool it to room temperature. Transfer it to a 100 mL volumetric flask. Use water to dilute it to the mark. Mix it uniformly. 1 mL of this solution contains 20 mg of iron. min ~ 3 min. Remove to slightly cool it. Add 20 mL of water. Heat to dissolve the salt. Cool it down. Transfer it into a 100 mL volumetric flask. Use water to dilute it to the mark. Mix it uniformly.

3.5.3.3 Pipette two sets of

10.00 mL of test solution into two 50 mL volumetric flasks, respectively [where the iron content is less than 30 mg, add iron solution (3.2.9) to make up].

3.5.3.4 Add 4 mL of sulfuric acid (3.2.3) and 10 mL of perchloric acid (3.2.6) to one set of solution. Mix it uniformly. Add 10 mL of sodium thiocyanate solution (3.2.8). Mix it uniformly. Add 10 mL of stannous chloride solution (3.2.7) whilst shaking it. Use sulfuric acid (3.2.4) to dilute it to the mark. Mix it uniformly. This is the color-developing solution. In the other test solution, except for adding the sodium thiocyanate solution (3.2.8), the rest operations are same as that of the color-developing solution. This is the reference solution.

3.5.3.5 Place it at room temperature for 10 min ~ 15 min. Transfer part of the solution into a 1 cm ~ 2 cm absorption dish. Use the reference solution as a reference. At the wavelength of 470 nm of the spectrophotometer, measure the absorbance. Subtract the absorbance of the blank solution as made along with the sample. From the calibration curve, check the corresponding mass of molybdenum (μg) in the color-developing solution.

3.5.4 Drawing of calibration curve Weigh 9 parts of

0.30 g of pure iron (molybdenum's mass fraction is less than 0.001%). Place them in a 250 mL conical flask. Respectively pipette 0,

0.50 mL,

4.00 mL of molybdenum standard solution (3.2.10). Add 40 mL of the mixed acid of sulfuric acid-phosphoric acid (3.2.5). Heat to dissolve it. Follow the procedures of 3.5.3.2 ~ 3.5.3.5, until measuring the absorbance. Subtract the absorbance of the compensation solution. Use the mass of molybdenum (μg) as the abscissa and the absorbance as the ordinate, to draw the calibration curve.

3.6 Calculation of results The molybdenum content is expressed in mass fraction wMo, the value is expressed in % and calculated according to formula (1): Where: V1 - The volume of the divided test solution, in milliliters (mL); When the tungsten in the specimen is less than 5 mg, use phosphoric acid to mask it. When the tungsten in the specimen is more than 5 mg ~ 20 mg, add 2 ~ 3 g of tartaric acid to mask it. When the molybdenum is less than 0.010%, the ratio of molybdenum to tungsten is not more than 1:80. In the pipetted solution, when copper is more than 5 mg, add thiourea to mask it. When antimony is more than

0.15 mg, it interferes with this method.

4.2 Reagents and materials Unless otherwise stated, only analytically pure reagents and distilled water or water of comparable purity are used in the analysis.

4.2.1 Tartaric acid.

4.2.2 Pure iron (molybdenum's mass fraction shall be less than 0.0003%).

4.2.3 Butyl acetate.

4.2.4 Hydrochloric acid, rho is about

4.2.5 Nitric acid, rho is about

4.2.6 Phosphoric acid, rho is about

4.2.7 Perchloric acid, rho is about

4.2.8 Sulfuric acid, rho is about

4.2.14 Molybdenum standard solution

4.2.14.1 Molybdenum stock solution, 500 μg/mL. Weigh 0.2500 g of pure molybdenum (mass fraction is more than 99.9%). Place it in a 250 mL beaker. Add 10 mL of nitric acid (1 + 3). Heat to dissolve it. Take it off to cool it slightly. Transfer it into a 500 mL volumetric flask. Use water to dilute it to the mark. Mix it uniformly. 1 mL of this solution contains 500 μg of molybdenum. 20 mg of tungsten, produce perchloric acid smoke until the volume is 2 mL ~ 3 mL].

4.5.3.2 Take off the conical flask. Cool it slightly. Add 20 mL of water to dissolve the salt [for the specimen which contains 5 mg ~ 20 mg of tungsten, add another 2 g ~ 3 g tartaric acid (4.2.1); stir to dissolve it; add sodium hydroxide solution (4.2.10) dropwise to dissolve the tungsten; then use sulfuric acid (4.2.8) to neutralize it to acidic]. Cool it to room temperature. Transfer it into a 100 mL volumetric flask. Use water to dilute it to the mark. Mix it uniformly.

4.5.3.3 Pipette

20.00 mL of the test solution. Place it in a 125 mL separatory funnel which contains 10 mL