

Translated English of Chinese Standard: GB/T223.26-2008

www.ChineseStandard.net → Buy True-PDF → Auto-delivery.

Sales@ChineseStandard.net

GB

NATIONAL STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA

ICS 77.080.01

H 11

GB/T 223.26-2008

Replacing GB/T 223.26-1989, 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 01, 2008

**Issued by: General Administration of Quality Supervision, Inspection and
Quarantine of PRC;**

Standardization Administration of PRC.

Table of Contents

Foreword	3
1 Scope	4
2 Normative references	4
3 Method-1: Thiocyanate direct spectrophotometry	5
4 Method-2: Thiocyanate-butyl acetate extraction spectrophotometry.....	8
5 Test report.....	13
Appendix A (Informative) Raw data of common precision test.....	14

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

Warning: Personnel who use this part shall have hands-on experience with formal laboratory work. This part does not address all possible safety issues. It is the responsibility of the user to take appropriate safety and health measures and to ensure compliance with the conditions set by the relevant national regulations.

1 Scope

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 2.4 mg.

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, ρ is about 1.19 g/mL.

3.2.2 Nitric acid, ρ is about 1.42 g/mL.

3.2.3 Sulfuric acid, ρ is about 1.84 g/mL, which is diluted to 1 + 1.

3.2.4 Sulfuric acid, ρ 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 (ρ is about 1.84 g/mL). After cooling it slightly, add 150 mL of phosphoric acid (ρ is about 1.70 g/mL). Mix it uniformly.

3.2.6 Perchloric acid, ρ is about 1.67 g/mL, which is diluted to 1 + 5.

3.2.7 Stannous chloride solution, 100 g/L. Weigh 10 g of stannous chloride ($\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$). Place it in a 250 mL beaker. Add 10 mL of hydrochloric acid (3.2.1). Heat to dissolve it and boil it. Cool it. Use water to dilute it to 100 mL. Mix it uniformly. Prepare it before use.

3.2.8 Sodium thiocyanate solution, 100 g/L.

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, 1.00 mL, 1.50 mL, 2.00 mL, 2.50 mL, 3.00 mL, 3.50 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 w_{Mo} , the value is expressed in % and calculated according to formula (1):

$$w_{Mo} = \frac{m_1 \times V}{m \times V_1 \times 10^6} \times 100 \quad \text{.....(1)}$$

Where:

V_1 - 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, ρ is about 1.19 g/mL.

4.2.5 Nitric acid, ρ is about 1.42 g/mL.

4.2.6 Phosphoric acid, ρ is about 1.70 g/mL.

4.2.7 Perchloric acid, ρ is about 1.67 g/mL.

4.2.8 Sulfuric acid, ρ is about 1.84 g/mL, which is diluted to 1 + 1.

4.2.9 Sulfuric acid, ρ is about 1.84 g/mL, which is diluted to 1 + 3.

4.2.10 Sodium hydroxide solution, 200 g/L.

4.2.11 Thiourea solution, 100 g/L.

4.2.12 Ammonium thiocyanate solution, 200 g/L.

4.2.13 Stannous chloride solution, 100 g/L. Weigh 10 g of stannous chloride ($\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$) into a 250 mL beaker. Add 10 mL of hydrochloric acid (4.2.4). Heat to dissolve it and boil it. Cool it down. Use water to dilute it to 100 mL. Mix it uniformly. Prepare it before use.

4.2.14 Molybdenum standard solution

4.2.14.1 Molybdenum stock solution, 500 $\mu\text{g/mL}$. 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 of sulfuric acid (4.2.9) in advance. Mix it uniformly. Add 5 mL of ammonium thiocyanate solution (4.2.12). Mix it uniformly. Add 10 mL of stannous chloride solution (4.2.13) [when the copper content is more than 5 mg, add 10 mL of thiourea (4.2.11)]. Mix it uniformly. When the iron thiocyanate fades to light red, immediately add 20.0 mL of butyl acetate (4.2.3). Shake it for 1 min. Let it stand for stratification. Discard the aqueous phase. Then along the mouth of bottle, add 5 mL of sulfuric acid (4.2.9). Mix it uniformly. Add 5 mL of stannous chloride (4.2.13). Shake it for 30 s. Let it stand for stratification. Discard the aqueous phase.

4.5.3.4 Use absorbent cotton to dry-filter the organic phase (discard the initial filtrate) in a 1 cm or 2 cm absorption dish (consistent with the calibration curve). Use butyl acetate (4.2.3) as a reference. Measure the absorbance at a wavelength of 470 nm of the spectrophotometer (when the specimen contains tungsten, the wavelength uses 500 nm). Subtract the absorbance of the blank solution along with the sample. From the calibration curve, check the corresponding mass of molybdenum (μg) in the color-developing solution.

4.5.4 Drawing of calibration curve

4.5.4.1 Molybdenum's mass fraction of 0.0020% ~ 0.010%

4.5.4.1.1 Weigh 6 parts of 1.000 g of pure iron (4.2.2). Respectively place them in six 125 mL conical flasks. Add 0, 1.00 mL, 2.00 mL, 3.00 mL, 4.00 mL, 5.00 mL of molybdenum standard solution (4.2.14.3). The rest operation is carried out based on the tungsten and copper contents of sample according to 4.5.3.1 ~ 4.5.3.3.

4.5.4.1.2 Use absorbent cotton to dry-filter the organic phase (discard the initial filtrate) in a 2 cm absorption dish. Use butyl acetate (4.2.3) as a reference. At the wavelength of 470 nm of the spectrophotometer (when the specimen contains tungsten, use the wavelength of 500 nm), measure the absorbance. Subtract the absorbance of the reagent blank. Use the mass of molybdenum as the abscissa and the absorbance as the ordinate, to draw the calibration curve.

m - The mass of the sample, in grams (g).

4.7 Precision

The precision test in this part was carried out in 9 laboratories in 1990 for the determination of six levels of molybdenum content; each laboratory made 3 determination of the molybdenum content of each level under the repeatability conditions as specified in GB/T 6379.1.

The raw data (measurement results) as reported by each laboratory are as shown in Appendix A (informative).

According to GB/T 6379.2, carry out statistical analysis of the obtained measurement results. The precision is as shown in Table 4.

Table 4 -- Precision results

Mass fraction of molybdenum / %	Repeatability limit r	Reproducibility limit R
0.0025 ~ 0.200	$r = 0.0001290 + 0.1078m$	$\lg R = -1.1646 + 0.6538 \lg m$

Where: m is the average of two measured values, in % (mass fraction).

The repeatability limit r and reproducibility limit R are obtained according to the equation as given in Table 2.

Under repeatability conditions, the absolute difference between the two independent test results obtained is not more than the repeatability limit (r). If it is more than the repeatability limit (r), it shall not exceed 5%;

Under reproducibility conditions, the absolute difference between the two independent test results obtained is not greater than the reproducibility limit (R). If it is more than the reproducibility limit (R), it shall not exceed 5%.

5 Test report

The test report shall include the following:

- Data such as sample identification, laboratory, analysis date;
- The extent of compliance with the provisions of this part;
- Analysis results and their representation;
- Anomalies as observed in the measurement;
- Operations that may have an impact on the results of the analysis but are not included in this part or optional operations.

This is an excerpt of the PDF (Some pages are marked off intentionally)

Full-copy PDF can be purchased from 1 of 2 websites:

1. <https://www.ChineseStandard.us>

- SEARCH the standard ID, such as GB 4943.1-2022.
- Select your country (currency), for example: USA (USD); Germany (Euro).
- Full-copy of PDF (text-editable, true-PDF) can be downloaded in 9 seconds.
- Tax invoice can be downloaded in 9 seconds.
- Receiving emails in 9 seconds (with download links).

2. <https://www.ChineseStandard.net>

- SEARCH the standard ID, such as GB 4943.1-2022.
- Add to cart. Only accept USD (other currencies - <https://www.ChineseStandard.us>).
- Full-copy of PDF (text-editable, true-PDF) can be downloaded in 9 seconds.
- Receiving emails in 9 seconds (with PDFs attached, invoice and download links).

Translated by: Field Test Asia Pte. Ltd. (Incorporated & taxed in Singapore. Tax ID: 201302277C)

About Us (Goodwill, Policies, Fair Trading...): <https://www.chinesestandard.net/AboutUs.aspx>

Contact: Wayne Zheng, Sales@ChineseStandard.net

Linkin: <https://www.linkedin.com/in/waynezhengwenrui/>

----- The End -----