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**Methods for chemical analysis of aluminium and
aluminium alloys - Part 29: Determination of
molybdenum content - Thiocyanate spectrophotometry**

铝及铝合金化学分析方法

第 29 部分：钼含量的测定 硫氰酸盐分光光度法

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Foreword

GB/T 20975, Methods for chemical analysis of aluminium and aluminium alloys, is divided into 31 parts:

- Part 1: Determination of mercury content;
- Part 2: Determination of arsenic content;
- Part 3: Determination of copper content;
- Part 4: Determination of iron content - Orthopenanthroline photometric method;
- Part 5: Determination of silicon content;
- Part 6: Determination of cadmium content - Flame atomic absorption spectrometric method;
- Part 7: Potassium periodate spectrophotometric method;
- Part 8: Determination of zinc content;
- Part 9: Determination of lithium content - Flame atomic absorption spectrometric method;
- Part 10: Determination of tin content;
- Part 11: Determination of tin content;
- Part 12: Determination of titanium content;
- Part 13: Determination of vanadium content - N-benzoyl-Nphenylhydroxylamine spectrophotometric method;
- Part 14: Determination of nickel content;
- Part 15: Determination of boron content;
- Part 16: Determination of magnesium;
- Part 17: Determination of strontium content - Flame atomic absorption spectrometric method;
- Part 18: Determination of chromium content;
- Part 19: Determination of zirconium content;

Methods for chemical analysis of aluminium and aluminium alloys - Part 29: Determination of molybdenum content - Thiocyanate spectrophotometry

Caution -- The personnel who uses this Part shall have hands-on experience in formal laboratory work. This Part does not address all possible security issues. It is the responsibility of the user to take appropriate safety and health measures and to ensure compliance with the conditions which are set by the relevant national regulations.

1 Scope

This Part of GB/T 20975 specifies the method for the determination of molybdenum content in aluminum and aluminum alloys by thiocyanate spectrophotometry.

This Part applies to the determination of molybdenum content in aluminum and aluminum alloys. Determination range: 0.005% ~ 12.0%.

2 Normative references

The following documents are indispensable for the application of this document. For dated references, only the dated version applies to this document. For undated references, the latest edition (including all amendments) applies to this document.

GB/T 6682, Water for analytical laboratory use. Specification and test methods

GB/T 8170-2008, Rules of rounding off for numerical values & expression and judgement of limiting values

3 Method summary

Dissolve the sample in acid; use sodium hydroxide sediment to separate most of the elements such as iron, copper, cobalt, nickel, and rhodium; use citric acid to cover tungsten. In the sulfuric acid medium, use iron sulfate as a catalyst; use ascorbic acid and thiourea to reduce molybdenum (VI) to molybdenum (V); make molybdenum and thiocyanate form a colored complex; measure the

absorbance at a wavelength of 460 nm of a spectrophotometer, so as to calculate the molybdenum content.

4 Reagents

Unless otherwise stated, in the analysis, only use analytical reagent and grade-2 water in accordance with GB/T 6682.

4.1 Hydrochloric acid ($\rho = 1.19$ g/mL).

4.2 Nitric acid ($\rho = 1.42$ g/mL).

4.3 Hydrofluoric acid ($\rho = 1.15$ g/mL).

4.4 Sodium hydroxide solution (400 g/L).

4.5 Sodium hydroxide solution (40 g/L).

4.6 Sulfuric acid (1+1).

4.7 Citric acid solution (500 g/L).

4.8 Potassium thiocyanate solution (500 g/L).

4.9 Ascorbic acid solution (100 g/L), prepare when necessary.

4.10 Thiourea solution (80 g/L).

4.11 Ferric sulfate solution: weigh 0.100 0 g of metallic iron; place it in a beaker; add 15 mL of sulfuric acid (4.6); heat to completely dissolve; cool; transfer to a 1 000 mL volumetric flask; use water to dilute to the mark; mix well. 1 mL of this solution contains 100 μ g of iron.

4.12 Molybdenum standard stock solution: weigh 1.839 8 g of ammonium molybdate in a beaker; place it in a 100 mL beaker; use a small amount of water to dissolve; transfer it to a 1 000 mL volumetric flask; use water to dilute to the mark; mix well. 1 mL of this solution contains 1 000 μ g of molybdenum.

4.13 Molybdenum standard solution A: pipette 10.00 mL of molybdenum standard stock solution (4.12) into a 200 mL volumetric flask; use water to dilute to the mark; mix well. 1 mL of this solution contains 50 μ g of molybdenum.

4.14 Molybdenum standard solution B: pipette 20.00 mL of molybdenum standard solution (4.13) into a 100 mL volumetric flask; use water to dilute to the mark; mix well. 1 mL of this solution contains 10 μ g of molybdenum.

immediately use water for cooling; then, add 0.5 mL of ferric sulfate solution (4.11), 2 mL of potassium thiocyanate solution (4.8), 2 mL of ascorbic acid solution (4.9), 1 mL of thiourea solution (4.10); immediately shake well when each reagent is added. Use water to fix volume; mix well; place for 30 min.

7.4.3 When the molybdenum content is $>1.0\% \sim 12.0\%$, dry-filter the test solution; transfer 10.00 mL of the filtrate and place in a 100 mL volumetric flask; use sodium hydroxide solution (4.5) to fix volume; mix well. Transfer 10.00 mL (V) of the diluted filtrate into a 25 mL volumetric flask; add 1 mL of citric acid solution (4.7); shake well; add 6 mL of sulfuric acid (4.6); immediately use water for cooling; then, add 0.5 mL of ferric sulfate solution (4.11), 2 mL of potassium thiocyanate solution (4.8), 2 mL of ascorbic acid solution (4.9), 1 mL of thiourea solution (4.10); immediately shake well when each reagent is added. Use water to fix volume; mix well; place for 30 min.

7.4.4 Transfer some of the solution into the corresponding absorption dish (consistent with the working curve); use water as the reference; measure the absorbance at 460 nm of the spectrophotometer; subtract the blank absorbance of accompanied sample; find the corresponding molybdenum mass (m_1) from the working curve.

7.5 Drawing of the working curve

7.5.1 Working curve 1: applicable to molybdenum content of $0.005\% \sim 0.10\%$

Pipette 0 mL, 0.20 mL, 0.50 mL, 1.00 mL, 2.00 mL, 3.00 mL, 5.00 mL of molybdenum standard solution (4.14); respectively place in the 25 mL volumetric flasks; add sodium hydroxide solution (4.5) to 10 mL; perform the following according to 7.4.2; pipette some of the solution in a 2 cm absorption dish; use water as the reference; measure the absorbance at 460 nm of the spectrophotometer. Take the molybdenum mass as the abscissa and the absorbance as the ordinate to draw the working curve.

7.5.2 Working curve 2: applicable to molybdenum content of $>0.10\% \sim 12.0\%$

Pipette 0 mL, 0.40 mL, 1.00 mL, 1.50 mL, 2.00 mL, 3.00 mL, 5.00 mL of molybdenum standard solution (4.13); respectively place in the 25 mL volumetric flasks; add sodium hydroxide solution (4.5) to 10 mL; perform the following according to 7.4.2; pipette some of the solution in a 0.5 cm absorption dish; use water as the reference; measure the absorbance at 460 nm of the spectrophotometer. Take the molybdenum mass as the abscissa and the absorbance as the ordinate to draw the working curve.

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