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**NATIONAL STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA**

GB/T 4324.5-2012

Replacing GB/T 4324.5-1984

**Methods for chemical analysis of tungsten -- Part 5:
Determination of arsenic content -- Hydrogenate atomic
absorption spectrometry**

钨化学分析方法 第5部分：砷量的测定 氢化物原子吸收光谱法
(English Translation)

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Foreword

GB/T 4324 "chemical analysis of tungsten" is divided into 28 parts.

- Part 1. Determination of lead content - flame atomic absorption spectrometry;
- Part 2. Determination of bismuth content hydride generation atomic absorption spectrometry;
- Part 3. Determination of tin content hydride generation atomic absorption spectrometry;
- Part 4. Determination of antimony content hydride generation atomic absorption spectrometry;
- Part 5. Determination of arsenic content by hydride generation atomic absorption spectrometry;
- Part 6. Determination of iron content of phenanthroline spectrophotometry;
- Part 7. Determination of cobalt content by inductively coupled plasma atomic emission spectrometry;
- Part 8. Determination of the amount of nickel by inductively coupled plasma atomic emission spectrometry and flame atomic absorption spectrometry Diacetyldioxime Gravimetric method;
- Part 9. Determination of cadmium content by inductively coupled plasma atomic emission spectrometry and flame atomic absorption spectrometry;
- Part 10. Determination of copper content - Flame atomic absorption spectrometry;
- Part 11. Determination of aluminum content by inductively coupled plasma atomic emission spectrometry;
- Part 12. Determination of the amount of silicon tetrachloride - molybdenum blue spectrophotometric method;
- Part 13. Inductively coupled plasma calcium atomic emission spectrometry;

- Part 14. Chlorinated Volatile residue amount after Gravimetric method;
- Part 15. Determination of magnesium content - Flame atomic absorption spectrometry and inductively coupled plasma atomic emission spectrometry;
- Part 16. Determination of loss on ignition - Gravimetric method;
- Part 17. Determination of sodium content by flame atomic absorption spectrometry;
- Part 18. Determination of potassium content by flame atomic absorption spectrometry;
- Part 19. Determination of titanium content of two methane antipyrine spectrophotometry;
- Part 20. vanadium content - Inductively coupled plasma atomic emission spectrometry;
- Part 21. Determination of chromium content by inductively coupled plasma atomic emission spectrometry;
- Part 22. manganese content - Inductively coupled plasma atomic emission spectrometry;
- Part 23. Determination of sulfur content of combustion conductance method and frequency combustion infrared absorption method;
- Part 24. Determination of phosphorus content - Molybdenum blue spectrophotometric method;
- Part 25. Determination of oxygen pulse heating inert gas fusion - infrared absorption method;
- Part 26. Determination of nitrogen pulse heating inert gas fusion - thermal conductivity method and Nessler reagent spectrophotometric method;
- Part 27. Determination of carbon content in the high-frequency combustion infrared absorption method;
- Part 28. Determination of molybdenum content - Thiocyanate spectrophotometric method.

This section GB/T 54324 Part of.

This section drafted in accordance with GB/T 1.1-2009 given rules.

This Part replaces GB/T 4324.5-1984 "chemical analysis of tungsten and molybdenum blue photometric method for determination of arsenic content." This portion of the GB/T 4324.5-Compared to 1984, the main technical changes are as follows.

- Determination by the "molybdenum blue photometric method" to "hydride generation atomic absorption spectrometry";
- Range of tungsten added blue, purple tungsten, carbide, ammonium paratungstate, measured by the scope of "0.00008% to 0.020%" with "0.00005% ~ 0.020% ";

--- In order to ensure the application of different sample dissolution methods in the "sample" in the fine, the average particle size of the tungsten carbide coarse particles were defined;

--- The "measurement frequency" in the "three" to "independently of the two measurements, the average value";

--- Tungsten powder, tungsten, oxide, tungstic acid, ammonium paratungstate were modified sample dissolution method, an increase of tungsten blue, purple tungsten, carbide, metatungstate Ammonium dissolving method;

--- Increasing the foreword, precision and test reports, and a standard format editing.

The non-ferrous metal part by the National Standardization Technical Committee (SAC/TC243) centralized.

This section is drafted. Chenzhou Diamond Tungsten Products Co., Ltd., Ganzhou Nonferrous Metallurgy Research Institute, Hunan Nonferrous Metals Co. Shizhuyuan Liability company.

1 Scope

GB/T 4324 provisions of this part of the tungsten powder, tungsten trioxide, tungsten, blue, purple tungsten, carbide, tungsten acid, ammonium metatungstate, ammonium paratungstate Determination of arsenic content.

This Part applies to tungsten powder, tungsten measure, tungsten trioxide, tungsten blue, purple tungsten, carbide, tungsten acid, ammonium metatungstate, ammonium paratungstate arsenic content set. Determination range of 0.00005% to 0.020%.

2 Method summary

Tungsten powder, tungsten, blue, fine particles of tungsten carbide and sodium hydroxide, hydrogen peroxide decomposition; tungsten trioxide, tungstic acid, ammonium paratungstate, metatungstate acid with ammonium hydroxide decomposition; purple tungsten, carbide coarse particles after burning into tungsten trioxide decomposition with sodium hydroxide. Phosphate tungsten complex in hydrochloric acid medium Stroma, thiourea, ascorbic acid and titanium trichloride restore the pentavalent arsenic to trivalent by flow injection - hydride generator and atomic absorption spectrometer MS determination of arsenic content.

3 Reagents

Unless otherwise indicated in the analysis using only recognized as analytical grade reagents and distilled or deionized water or equivalent purity.

3.1

Hydrogen peroxide ($\rho = 1.10\text{g/mL}$), pure class distinctions.

3.2

phosphoric acid ($\rho = 1.69\text{g/mL}$).

3.3

Sodium hydroxide solution (100g/L).

3.4

hydrochloride (14).

3.5

thiourea solution (100g/L).

3.6

ascorbic acid solution (50g/L).

3.7

titanium trichloride solution. Pipette 10mL conc titanium trichloride solution [15% ~ 20% (w/w)], hydrochloride (19) was diluted to 100mL.

The time is now equipped with.

3.8

solution of potassium borohydride (10g/L). Weigh 2g 0.6g potassium borohydride and sodium hydroxide dissolved in 200mL of water. The time is now equipped with.

3.9

tungsten matrix. the nature of the sample should be substantially similar to the arsenic content is not more than 0.00005%.

3.10

arsenic standard stock solution. Weigh 0.1320g advance by 110 °C drying of arsenic trioxide benchmark reagents, beaker, add 1mL sodium hydroxide solution (3.3) was dissolved with hydrochloric acid (19) and move to 1000mL volumetric flask, dilute to the mark with hydrochloric acid (19) Degree, shake. This solution 1mL containing 0.1mg arsenic.

3.11

Arsenic Standard Solution A. Pipette 10.00mL arsenic standard stock solution (3.10) in 200mL volumetric flask, with hydrochloric acid solution (19) diluted to the mark, shake, solution 1mL containing 5 μg arsenic.