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YS

**NONFERROUS METALS INDUSTRY STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA**

YS/T 226.6-2009

Replacing YS/T 226.10-1994

**Methods for chemical analysis of selenium. Part 6:
Determination of sulfur content. Diphenylcarbazide
spectrophotometry**

硒化学分析方法 第6部分：硫量的测定对称二苯氨基脲分光光度法
(English Translation)

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Foreword

YS/T 226 "selenium chemical analysis method" is divided into 13 parts.

- Part 1. Determination of bismuth content - Hydride Generation - Atomic Fluorescence Spectrometry
- Part 2. Determination of antimony content Hydride Generation - Atomic Fluorescence Spectrometry
- Part 3. Determination of aluminum content Chromazurol S- cetylpyridinium bromide spectrophotometric method
- Part 4. Determination of mercury content of dithizone - CTC colorimetric titration
- Part 5. Determination of the amount of silicon molybdenum blue spectrophotometry
- Part 6. Determination of sulfur content symmetrical diphenyl semicarbazide spectrophotometry
- Part 7. Determination of magnesium content - Flame atomic absorption spectrometry
- Part 8. Determination of copper content - Flame atomic absorption spectrometry
- Part 9. Determination of iron content by flame atomic absorption spectrometry
- Part 10. Determination of nickel content by flame atomic absorption spectrometry
- Part 11. Determination of lead content by flame atomic absorption spectrometry
- Part 12. Determination of the amount of selenium sodium thiosulfate volumetric method
- Part 13. silver, aluminum, arsenic, boron, mercury, bismuth, copper, cadmium, iron, gallium, indium, magnesium, nickel, lead, silicon, antimony, tin, tellurium, titanium, zinc contents Inductively Coupled Plasma Mass Spectrometry this section YS/T Part 6 226.

The partial replacement of YS/T 226.10-1994 "Determination of selenium in the reduction of sulfur content of distillation absorptiometry." And YS/T 226.10-Compared to 1994, this section has the following main changes.

- Measured by the scope of 0.0005 percent to 0.03 percent to 0.0005% to 0.01%;
- By the turbidimetric absorptiometry to symmetrical Diphenyl urea spectrophotometry;
- Supplementary terms of quality assurance and control;
- Increase the repeatability limit, will be allowed to poor reproducibility limit.

The non-ferrous metal part by the National Standardization Technical Committee and centralized.

This section is responsible for drafting unit. Jinchuan Group Limited.

Participated in the drafting of this section. Beijing Nonferrous Metal Research Institute, Guangzhou Nonferrous Metal Research Institute.

1 Scope

This section YS/T 226 provides for the determination of selenium in sulfur content.

This section applies to the determination of selenium in sulfur content. Measurement range. 0.0005% to 0.01%.

2 Method summary

Sample with nitric acid, hydrobromic acid decomposition and separation of volatile selenium, sulfur is oxidized to SO_4^{2-} . In ethanol containing ammonia medium, SO_4^{2-} in the past the presence of quantitative amounts of BaCrO_4 following reaction.

$\text{BaCrO}_4 + \text{SO}_4^{2-} = \text{BaSO}_4 + \text{CrO}_4^{2-}$ Released CrO_4^{2-} in hydrochloric acid medium with symmetrical diphenyl semicarbazide form soluble purple complex wavelength spectrophotometer at 542nm measured absorbance, the standard curve method to calculate the amount of sulfur.

3 Reagents

Unless otherwise indicated, the second part of the water are distilled water, reagents were pure class distinctions.

3.1

barium chromate (BaCrO_4 content of not less than 98%).

3.2

hydrobromide ($\rho 1.38\text{g/mL}$).

3.3

Glacial acetic acid ($\rho 1.67\text{g/mL}$).

3.4

hydrochloride (rho1.19g/mL) of high purity.

3.5

HCl (1 + 1).

3.6

hydrochloric acid (1 + 11).

Nitrate 3.7 (rho1.42g/mL) of high purity.

3.8

Ammonia (rho0.90g/mL).

3.9

ethanol.

3.10

pairs nitrophenol (1g/L).

3.11

sodium chloride solution (25g/L).

3.12

Symmetrical Diphenyl semicarbazide solution. Weigh 0.8g diphenylmethyl semicarbazide was dissolved in symmetrical 100mL of absolute ethanol, was filtered in a brown bottle, Added 1mL hydrochloric acid (3.6) as a stabilizer.

Calcium 3.13 ammonia solution. to 200mL ammonia (3.8) was added 130mL of water, 1.2g calcium chloride, after shaking stored in polyethylene bottles.

3.14

mixed acid solution. 0.42mL of hydrochloric acid (3.4) and 14.7mL glacial acetic acid (3.3) diluted mixed with water to 200mL.

3.15

barium chromate suspension. Weigh 0.5g barium chromate (3.1) was dissolved in 200mL mixed acid solution (3.14), and stored in polyethylene bottles. use Before fully shaken.

3.16

sulfur standard stock solution. Weigh accurately 1.3865g pre-bake at 105 °C 2h potassium sulphate reference reagent, placed in 200mL beaker in, with the amount of water dissolved and transferred to 1000mL volumetric flask, constant volume of water. This solution 1mL containing 0.25mg sulfur.

3.17

sulfur standard solution. Pipette 20.00mL sulfur standard stock solution (3.16) in 500mL flask, constant volume of water. This solution 1mL containing 10 μ g sulfur.