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**YS**

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

**YS/T 226.4-2009**

Replacing YS/T 226.5-1994

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**Methods for chemical analysis of selenium. Part 4:  
Determination of mercury content. Dithizone-carbon  
tetrachloride titrated colorimetric-method**

硒化学分析方法 第4部分：汞量的测定双硫腙-四氯化碳滴定比色法  
**(English Translation)**

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## Table of Contents

|                               |
|-------------------------------|
| Foreword                      |
| 1 Scope                       |
| 2 Method summary              |
| 3 Reagents                    |
| 3.9 Mercury standard solution |

### 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 content of electricity Inductively coupled plasma mass spectrometry this section YS/T Part 4 226.

The partial replacement of YS/T 226.5-1994 "Determination of the amount of mercury and

selenium dithizone - CTC titration colorimetry." And YS/T 226.5-Compared to 1994, this section has the following main changes.

- Text format has been modified;
- 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.

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## 1 Scope

This section YS/T 226 specifies the method for determination of selenium in the amount of mercury.

This section applies to the determination of the amount of mercury and selenium.  
Measurement range. 0.0005% to 0.04%.

## 2 Method summary

The sample is dissolved in nitric acid, mercury with dithizone in pH4 ~ pH5 acid medium - carbon tetrachloride generated orange-red complex, take into Titrated colorimetric assay.

There edetate sodium in the presence of selenium impurity element other than silver outer not interfere with the determination. Interference silver, plus ammonium thiocyanate masking.

## 3 Reagents

The standard water is deionized water, conductivity is not more than  $6.67 \times 10^{-2} \mu\text{S} \cdot \text{cm}^{-1}$ .

Nitrate 3.1 (rho1.42g/mL).

### 3.2

nitric acid (1 + 1).

### 3.3

Disodium ethylenediaminetetraacetate solution (50g/L).

### 3.4

ammonium acetate solution (500g/L).

Ammonia 3.5 (1 + 1).

### 3.6

ammonium thiocyanate solution (500g/L).

### 3.7

pairs Diphenylthiocarbazone. Take 1g of finely dithizone dissolved in 200mL of carbon tetrachloride was added 200mL ammonium hydroxide (1 + 100) in the dispensing Funnel thoroughly shaken. After standing stratification, carbon tetrachloride layer was charged to another separating funnel, plus ammonium hydroxide (1 + 100) (the volume can be adapted when reduction), repeat the operation until the last ammonium hydroxide was colorless. All ammonium hydroxide solutions were combined, washed with hydrochloric acid (1 + 1) Dropwise to precipitate all dithizone solution colorless. Precipitated dithizone filtered with a glass funnel, washed with water 2 to 3 times, left at 40 °C Right drying.

### 3.8

pairs Diphenylthiocarbazone - carbon tetrachloride solution (0.01g/L).

## 3.9 Mercury standard solution

### 3.9.1

Mercury standard solution A. Weigh 0.1354g mercuric chloride dissolved in water and transferred to 1000mL volumetric flask, dilute to the mark with water, shake uniform. This solution 1mL containing 100µg mercury.

### 3.9.2

Mercury standard solution B. Pipette 50.00mL solution A, placed in 500mL flask, diluted with water to the mark. This solution 1mL containing 10µg mercury.

3.10 pairs Diphenylthiocarbazone - CTC calibration solution. Pipette 10.00mL mercury standard solution (3.9.2) (Vp), placed in 60mL separatory funnel, Diluted with water to 20mL, plus 5mL disodium edetate solution (3.3), shake the solution was adjusted with ammonia (3.5) and ammonium acetate (3.4) pH4 ~ pH5, add 1mL of ammonium thiocyanate solution (3.6), 10mL ammonium acetate solution (3.4), shake, dropping dithizone - carbon tetrachloride solution Extraction, start adding 5mL, 3mL, 2mL, 1mL, close to the end dropwise 0.50mL, 0.20mL. Each oscillation 1min, standing After separation, discard the orange-red organic phase was extracted into the organic phase was dark green date (such as last dithizone - carbon tetrachloride and excess solution was dark green when mixing colors can be generated dithizone - CTC volume of 1/2,1/3 is calculated). Write down the consumption of dithizone - carbon tetrachloride solution