

ICS 77.040.30  
CCS H 17

**YS**

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

**YS/T 37.1-2018**

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**Methods for chemical analysis of high pure germanium dioxide  
-- Determination of chlorine content by silver nitrate  
turbidimetric method**

高纯二氧化锗化学分析方法 硝酸银比浊法测定氯量  
**(English Translation)**

**Issued on: October 22, 2018**

**Implemented on: April 1, 2019**

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**Issued by: Ministry of Industry and Information Technology of the  
People's Republic of China**

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### 1 Scope

This Part specifies the determination of the chlorine content in high-purity germanium dioxide.

This Part applies to the determination of the chlorine content in high-purity germanium dioxide, with a determination range of 0.0050% to 0.1000%.

### 2 Method Summary

The test material was dissolved in sodium hydroxide and neutralized to a weakly acidic state with nitric acid. In dilute nitric acid medium, chloride ions reacted with silver ions to form silver chloride colloid. The silver chloride colloid remained suspended in the solution in the presence of the stabilizer glycerol. Turbidity was measured at 430 nm using a spectrophotometer to indirectly determine the chloride content.

### 3 Reagents and Materials

#### 3.1

The water used in this Part is deionized water with a resistivity of no less than 18 Mohm·cm.

#### 3.2

Nitric acid ( $\rho = 1.42 \text{ g/mL}$ ), guarantee reagent.

**3.3**

Glycerol, analytical reagent.

**3.4**

Nitric acid (1+1), prepared from nitric acid (3.2).

**3.5**

Glycerol (1+1), prepared from glycerol 3.3.

**3.6**

Silver nitrate solution (5 g/L). Take 2.5 g of silver nitrate (analytical reagent) into a 250 mL beaker; dissolve completely in deionized water. Transfer to a 500 mL volumetric flask; make constant volume; and store in a 500 mL amber bottle. 1 mL of this solution contains 5 mg of silver nitrate.

**3.7**

Sodium hydroxide solution (400 g/L). Take 100 g of sodium hydroxide (analytical reagent) into a 500 mL beaker; dissolve completely in deionized water; cool to room temperature.

Transfer to a 250 mL volumetric flask; make constant volume; mix well; and store in a plastic bottle. 1 mL of this solution contains 400 mg of sodium hydroxide.

**3.8**

Sodium chloride [ $w(\text{NaCl}) \geq 99.99\%$ ]. Ignite at 400 °C~450 °C for 4 h to constant weight.

**3.9**

Chlorine standard stock solution. Accurately take 1.6485 g of sodium chloride (3.8); place in a 250 mL beaker; dissolve in an appropriate amount of water. Transfer to a 1000 mL volumetric flask; dilute with water; make constant volume to the mark; and mix well. 1 mL of this solution contains 1 mg of chlorine.

**3.10**

Chlorine Standard Stock Solution A. Pipette 10.0 mL of chlorine standard stock solution (3.9) to a 100 mL volumetric flask; dilute with water; make constant volume to the mark; and mix well. 1 mL of this solution contains 100 µg of chlorine.

**3.11**

Chlorine Standard Solution B. Pipette 10.0 mL of Chlorine Standard Stock Solution A (3.10) to a 100 mL volumetric flask; dilute with water; make constant volume to the mark; and mix well. 1 mL of this solution contains 10 µg of chlorine.

## 4 Instruments

Spectrophotometer.

### 5.1 Test materials

Take specimen according to Table 1, accurate to 0.0001g.

### 5.2 Parallel test

Perform two parallel tests.

### 5.3 Blank test

Perform a blank test along with the test material.

### 5.4 Determination

#### 5.4.1

Place the test material (5.1) in a 50 mL PTFE beaker; moisten with 2 mL of water. Add 3 mL of sodium hydroxide solution (3.7), heat to dissolve. Add 15 mL of water; neutralize with nitric acid (3.4) until a precipitate appears; then add more nitric acid until the precipitate dissolves, with an excess of 0.5 mL. Dilute the solution to the total volume according to Table

1, and mix well.

#### 5.4.2

Pipette the solution to a 25 mL colorimetric tube according to Table 1; additionally add 2 mL of nitric acid (3.4), 2 mL of glycerol (3.5), and 2 mL of silver nitrate (3.6); gently mixing after each addition. Dilute to the mark with water and mix well.

#### 5.4.3

Pipette the test solution (5.4.2) into a 20 mm absorption dish. Using the standard zero point as a reference and the reagent blank as the sample blank solution; measure its absorbance at a wavelength of 430 nm using a spectrophotometer. Subtract the absorbance of the blank test solution and find the corresponding mass of chlorine from the working curve.

### 5.5 Plotting of the working curve

Pipette 0 mL, 1.00 mL, 2.00 mL, 4.00 mL, 6.00 mL, 8.00 mL, and 10.00 mL of Chlorine

Standard Solution B (3.11) into a set of 25 mL colorimetric tubes, respectively. Follow the procedures in 5.4.2 and 5.4.3. Plot the working curve with the mass of chlorine as the abscissa and the absorbance as the ordinate. The correlation coefficient of the linear

equation of the curve shall be no less than 0.99.

## 6 Test Data Processing

The chlorine content is expressed as its mass fraction  $w(\text{Cl})$  and calculated according to Formula (1).

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