

ICS 77.120.01
CCS H 13

YS

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

YS/T 239.3-2010

Replacing YS/T 239.3-1994

**Methods for chemical analysis of antimony trisulfide. Part 3:
Determination of free sulfur content. Burning and sodium
hydroxide titration method**

三硫化二锑化学分析方法 第3部分：游离硫量的测定燃烧中和滴定法
(English Translation)

Issued on: November 22, 2010

Implemented on: March 1, 2011

Issued by: Ministry of Industry and Information Technology

Table of Contents

Foreword

1 Scope

2 Method summary

3 Reagents

3.7 Sodium hydroxide standard titration solution [$c(\text{NaOH})$ about 0.02mol/L]

Foreword

YS/T 239 "Methods for chemical analysis of antimony trisulfide" There are seven sections.

--- Part 1. Determination of antimony content cerium sulfate titration;

--- Part 2. Determination of the amount of sulfur compounds and combustion titration method;

--- Part 3. Determination of free sulfur content and combustion titration method;

--- Part 4. aqua regia insolubles - Gravimetric method;

--- Part 5. Determination of arsenic content of arsenic molybdenum blue spectrophotometric method;

--- Part 6. Determination of iron content of phenanthroline spectrophotometry;

--- Part 7. Determination of lead content by flame atomic absorption spectrometry.

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

This is Part 3.

The partial replacement of YS/T 239.3-1994 "antimony trisulfide chemical analysis of combustion iodometric method for the determination of free sulfur." versus YS/T 239.3-1994 compared to this part of the following changes.

--- Text format has been modified;

--- Complements the precision and quality assurance and control provisions.

This standard by the national non-ferrous metals Standardization Technical Committee (SAC/TC243) centralized.

1 Scope

This section YS/T 239 specifies the method for the determination of free sulfur content of antimony trisulfide.

This section applies to the determination of free sulfur content of antimony trisulfide.

Measurement range. free sulfur mass fraction of 0.0050% to 0.20%.

2 Method summary

With carbon tetrachloride and leaching of free sulfur compound isolated by filtration substrate, evaporating the solvent and drying, high temperature combustion in a stream of oxygen, sulfur conversion Sulfur dioxide, hydrogen peroxide absorbed and converted into sulfuric acid, methyl red - methylene blue solution as indicator, titrated with standard sodium hydroxide solution Titrated.

3 Reagents

Unless otherwise indicated, the reagents used in this section refer to water and recognized analytical reagent and tertiary water.

3.1

potassium hydroxide.

3.2

anhydrous calcium chloride.

3.3

CTC.

3.4

Absorption of hydrogen peroxide solution (298).

3.5

phenolphthalein indicator (10g/L ethanol).

3.6

methyl red - methylene blue mixed indicator. 20 unit volume of methyl red ethanol solution (0.3g/L) and 3 per unit volume of methylene blue solution (1g/L) and mixed.

3.7 Sodium hydroxide standard titration solution [c (NaOH) about 0.02mol/L]

3.7.1

preparation. Weigh 0.8g of sodium hydroxide, into 250mL beaker, dissolved in water freshly

boiled cooled and transferred to 1000mL capacity Volumetric flask, and dilute to the mark with the new boiling cooling water, and mix. Stored in plastic bottles.

3.7.2

Calibration. Weigh 5.0000g advance by potassium hydrogen phthalate (reference reagent) 100 °C ~ 105 °C drying 2h, resting 500mL beaker, add freshly boiled water after cooling to dissolve clear, transferred to 1000mL volumetric flask, dilute with freshly boiled water after cooling to the mark, and mix. 1mL solution containing 0.0050g of potassium hydrogen phthalate.

Pipette accurately 20.00mL potassium hydrogen phthalate standard solution was placed in 300mL conical flask, add 40mL freshly boiled cooled Water, 2 drops of phenolphthalein indicator (3.5) dropwise with sodium hydroxide standard titration solution to light red is the end.

Along with the calibration blank test.

According to equation (1) calculate the standard volumetric sodium hydroxide solution (3.7) of the actual concentration.

$$c = V \times 0.0050 \times 1000 (V_1 - V_0) \times 204.2 \quad (1)$$