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YS

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

YS/T 239.1-2010

Replacing YS/T 239.1-1994

**Methods for chemical analysis of antimony trisulfide. Part 1:
Determination of arsenic content. Cerium sulfate titration
method**

三硫化二锑化学分析方法 第1部分：锑量的测定硫酸铈滴定法
(English Translation)

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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 1.

The partial replacement of YS/T 239.1-1994 "Determination of antimony trisulfide chemical analysis of antimony potassium bromate volumetric method." versus YS/T 239.1-1994 compared to this part of the following changes.

- Potassium bromate volumetric method to cerium sulfate titration;
- Text format has been modified;
- Complements the precision and quality assurance and control provisions.

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

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1 Scope

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

This section applies to the determination of the amount of antimony trisulfide antimony. Measurement range. antimony mass fraction of 68.00% ~ 73.00%.

2 Method summary

Sample decomposition with sulfuric acid, hydrazine sulfate reducing pentavalent antimony, trivalent antimony with methyl orange as indicator, at 80 °C ~ 90 °C with cerium sulfate standard Quasi titration.

3 Reagents

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

3.1

potassium sulfate.

3.2

hydrazine sulfate.

3.3

sulfate (ρ 1.84g/mL).

3.4

hydrochloride (ρ 1.19g/mL).

3.5

sulfate (12).

3.6

hydrochloride (11).

3.7

methyl orange indicator (1g/L).

3.8

cerium sulfate standard titration solution [$c(\text{Ce}(\text{SO}_4)_2)$ about 0.1mol/L].

3.8.1

preparation. Weigh 40.50g cerium sulfate [$\text{Ce}(\text{SO}_4)_2 \cdot 4\text{H}_2\text{O}$], placed in 1000mL beaker, add 50mL sulfuric acid (3.3), stir, gradually warming on a hot plate heated solvent into a paste, and take the white smoke of sulfuric acid for about 20min, remove the cooled, sulfur 150mL Acid (3.5), and then slowly added 400mL of water, and stirred to dissolve clear, cooled and transferred to 1000mL volumetric flask, dilute to the mark with water, Mix well.

3.8.2

Calibration. Weigh 0.2500 three antimony [$w_{\text{Sb}} \geq 99.99\%$], were placed in 300mL Erlenmeyer flask with a small amount of water wet, add 12mL sulfuric acid (3.3), heated and dissolved to clear, remove the cooling. The following operations shall be conducted, according to 5.4.3,5.4.4.

Along with the calibration blank test.

Press (1) is calculated cerium sulfate standard titration solution (3.8) of the actual concentration.

$c = m \times 2000 (V_1 - V_0) \times 121.76$ (1) where.

c --- cerium sulfate standard titration solution (3.8) of the actual concentration in units of moles per liter (mol/L);

m --- quality pure antimony weighed in grams (g);

Antimony consumption volume V_1 --- titration with cerium sulfate standard titration solution (3.8) in milliliters (mL);

V_0 --- calibration blank solution consumed cerium sulfate standard titration solution (3.8) the volume in milliliters (mL);

121.76 --- antimony molar mass in grams per mole (g/mol).