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

YS/T 1116.7-2016

**Methods for chemical analysis of tin anode slime--Part 7:
Determination of antimony content--Cerium sulfate titration**

锡阳极泥化学分析方法 第7部分：锑量的测定 硫酸铈滴定法

(English Translation)

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Foreword

YS/T 1116-2016 "tin anode mud chemical analysis method" is divided into seven parts.

- Part 1. Determination of tin content Potassium iodate titration;
- Part 2. Determination of bismuth content Na₂EDTA titration;
- Part 3. Determination of copper content, lead content and bismuth - Flame atomic absorption spectrometric method;
- Part 4. Determination of arsenic iodine titration;
- Part 5. Determination of indium - Flame atomic absorption spectrometric method;
- Part 6. Determination of gold and silver;
- Part 7. Determination of antimony content - Cerium sulfate titration method.

This part is part 7 of YS/T 1116-2016.

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

This part of the National Nonferrous Metals Standardization Technical Committee (SAC/TC243) proposed and centralized.

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Method for chemical analysis of tin anode Part 7. determination of antimony content Cerium sulfate titration

1 Scope

This part of YS/T 1116 specifies the method for the determination of antimony content in tin anode mud.

This part is applicable to the determination of antimony content in tin anode mud. The measurement range is 3.00% ~ 20.00%.

2 Method summary

The samples were decomposed with sulfuric acid and reduced with sulfuric acid. In the sulfuric acid-hydrochloric acid medium, the samples were treated with methyl orange and methylene blue at 80 °C ~ 90 °C as the indicator, with sodium sulfate standard titration solution titration.

3 Reagent

Unless otherwise stated, only the reagents and secondary water confirmed to be analytical were used in the analysis.

3.1

Potassium sulfate.

3.2

Sulfate.

3.3

Sulfuric acid ($\rho=1.84\text{g/mL}$).

3.4

Phosphoric acid ($\rho=1.42\text{g/mL}$).

3.5

hydrochloric acid ($\rho=1.19\text{g/mL}$).

3.6

standard solution of cerium ammonium sulfate titration. $c[\text{Ce}(\text{SO}_4)_2 \cdot (\text{NH}_4)_2\text{SO}_4] \sim 0.020\text{mol/L}$.

3.6.1

Preparation. Weigh 50 g of cerium ammonium sulfate $[\text{Ce}(\text{SO}_4)_2 \cdot (\text{NH}_4)_2\text{SO}_4 \cdot 4\text{H}_2\text{O}]$ in a 1000 mL beaker, 500 mL of water, slowly adding 300 mL of sulfuric acid (3.3) along the wall of the cup, heating at low temperature, stirring until dissolved, cooled to room temperature, filtered with absorbent cotton in the reagent bottle, diluted with water to 5L,

mix.

3.6.2

Calibration. Weigh 0.050g metallic antimony ($w_{\text{Sb}} \geq 99.99\%$), accurate to 0.0001g, placed in 500mL Erlenmeyer flask, add 1g Potassium sulfate (3.1), 30mL sulfuric acid (3.3), blowing a small amount of water along the cup wall, heated to the concentration of sulfuric acid smoke, remove, cool, add 0.1g sulfuric acid (3.2), blowing a small amount of water along the cup wall, continue to heat to the fudge of sulfuric acid smoke to the mouth, remove, cool, add 5mL phosphoric acid (3.4), along the cup wall to join (3.5), 4 drops of methyl orange solution (3.7) and 4 drops of methylene blue solution (3.8) were added to a solution of 50 mL of water, with the standard titration solution of cerium ammonium sulfate titration to red disappears, blue appears as the end point, with the blank test.

The actual concentration of the standard titration solution of cerium ammonium sulfate is calculated according to formula (1) $c = \frac{m}{60.88(V_1 - V_2)} \times 10^{-3}$ (1) where.

c - the actual concentration of ceramate ammonium standard titration solution in moles per liter (mol/L);

m --- the quality of metallic antimony, in grams (g);

V1 - the volume of the standard titration solution of cerium ammonium sulfate consumed in calibration, in milliliters (mL);

V2 - the volume of the standard titration solution of cerium ammonium sulfate consumed by the titration of the blank solution in milliliters (mL);