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

YS/T 775.6-2011

**Methods for chemical analysis of lead anode slime. Part 6:
Determination of copper content. Iodine titration method**

铅阳极泥化学分析方法 第6部分：铜量的测定碘量法

(English Translation)

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Foreword

YS/T 775-2011 "lead anode mud chemical analysis method" is divided into seven parts.

- Part 1. Determination of lead content - Na₂EDTA titration;

Part 2. Determination of Bismuth Flame Atomic Absorption Spectrometry and Na₂EDTA Titration;

- Part 3. Determination of arsenic content - Potassium bromate titration;

- Part 4. Determination of antimony content - Flame atomic absorption spectrometry and cerium sulphate titration;

- Part 5. Determination of gold and silver;

- Part 6. Determination of copper content - Iodometric method;

Part 7. Determination of arsenic, copper and selenium - Inductively coupled plasma atomic emission spectrometric method.

This section is part 6.

The arbitration analysis of this part and the overlapping range of Part 7 is carried out using the method of this part.

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

This part is the national nonferrous Metals Standardization Technical Committee (SAC/TC243).

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1 Scope

This part of YS/T 775 specifies the determination of copper content in lead anodic mud.

This part is applicable to the determination of copper content in lead anode mud, and the determination range is 2.00% ~ 12.00%.

2 Method summary

Samples with hydrochloric acid, nitric acid, sulfuric acid dissolved, with hydrogen bromide to remove arsenic, antimony, tin and other interference elements. Control solution pH value of 3 to 4, with fluoride Hydrogenated ammonium masking iron, adding potassium iodide and divalent copper, the precipitation of iodine as a indicator, with sodium thiosulfate standard titration solution titration to the solution becomes colorless for the end.

3 Reagent

Unless otherwise stated, only the reagents identified as analytical reagents and distilled or deionized water or comparable purity were used in the analysis.

3.1

Potassium iodide.

3.2

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

3.3

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

3.4

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

3.5

hydrobromic acid ($\rho=1.49\text{g/mL}$).

3.6

glacial acetic acid ($\rho=1.05\text{g/mL}$).

3.7

ammonia ($\rho=0.90\text{g/mL}$).

3.8

Starch solution (5 g/L).

3.9

Ammonium fluoride in saturated solution.

3.10

potassium thiocyanate solution (100g/L). Weigh 10g potassium thiocyanate in a 400mL beaker, add water and dilute to 100mL. plus Into the 2g potassium iodide (3.1) dissolved, add 2mL starch solution (3.8), while the side of the drop of iodine solution (about 0.04mol/L) to just blue Color, then titration with sodium thiosulfate titration solution to blue just disappeared.

3.11

copper standard solution. Weigh 1.0000g metal copper ($w_{\text{Cu}} \geq 99.99\%$) placed in 500mL conical beaker, add 20mL nitric acid (1 1), cover the surface of the dish, low temperature heating dissolved completely, add 5mL sulfuric acid (3.4), steamed to smoke white smoke, remove the cooling, water washing table and the cup wall, slightly so that the salt dissolves, remove the cooling, into the 500mL volumetric flask, diluted with water to the mark, mix. This solution 1mL Containing 2 mg of copper.

3.12

sodium thiosulfate standard titration solution [$c(\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}) \sim 0.04\text{mol/L}$].

3.12.1

Preparation. Weigh 100g sodium thiosulfate ($\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$) placed in a 1000mL beaker, add 2g anhydrous sodium carbonate, Add 500mL of water dissolved, into the 10L brown reagent bottle. Diluted with boiling and cooled water to about 10 L, and 1 mL of trichloromethane was added Alkane, mix well for one week.

3.12.2

Calibration. Remove four 25.00 mL copper standard solution (3.11) In a 500 mL conical beaker, wash the cup wall with water. According to points