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

YS/T 775.3-2011

**Methods for chemical analysis of lead anode slime. Part 3:
Determination of arsenic content. Potassium bromate titrimetric
method**

铅阳极泥化学分析方法 第3部分：砷量的测定溴酸钾滴定法
(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 3.

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

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

This part is responsible for drafting units. Shaanxi Dongling Smelting Co., Ltd.

This part of the drafting unit. Beijing Nonferrous Metals Research Institute.

This part participates in the drafting unit. Tongling Nonferrous Metals Group Holdings Co., Ltd., Hechi City South Nonferrous Smelting Co., Ltd., Tin Mine Mountain flash Star antimony industry limited liability company.

1 Scope

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

This part is applicable to the determination of arsenic in lead anode mud, the determination range is 1.00% ~ 10.00%.

2 Method summary

Samples were dissolved in sulfuric acid, in hydrochloric acid medium, at 40 °C ~ 60 °C, with methyl blue - methyl orange as an indicator, first with cerium sulfate drop Determination of antimony, and then titration of potassium bromate standard titration of arsenic.

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

urea.

3.2

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

3.3

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

3.4

Sulfuric acid (1:1).

3.5

methyl orange solution (2g/L).

3.6

times methyl blue solution (1g/L).

3.7

arsenic standard solution. Weigh 1.3203g arsenic trioxide ($w_{\text{As}_2\text{O}_3} \geq 99.99\%$, pre-drying at 100 °C ~ 105 °C 1h, placed in Dryer, cooled to room temperature), placed in 250mL beaker, add 20mL sodium hydroxide solution (200g/L) dissolved to clear, add 100mL water, 10mL sulfuric acid (3.4), cooled to room temperature, into the 1000mL volumetric flask, diluted with water to the mark, mix. This solution 1mL containing 1mg arsenic.

3.8

Cerium Sulfate Solution. $c[\text{Ce}(\text{SO}_4)_2 \cdot 4\text{H}_2\text{O}] \sim 0.05\text{mol/L}$. Weigh 20 g of cerium sulfate $[\text{Ce}(\text{SO}_4)_2 \cdot 4\text{H}_2\text{O}]$ 1000mL beaker, add 300mL water, 100mL sulfuric acid (3.4) dissolved completely, cooling, into the 1000mL volumetric flask, with water dilute Release to the scale, mix.

3.9

standard solution of potassium bromate ($c\text{KBrO}_3 \sim 0.007\text{mol/L}$).

3.9.1

preparation. Weigh 1.2g potassium bromate in a 500mL beaker, add.200mL water dissolved completely, cooling, into the 2500mL capacity Bottle, diluted with water to the mark, mix.

3.9.2

Calibration. Remove four 10.00 mL of arsenic standard solution (3.7) in 250 mL Erlenmeyer flask, dilute with water to 100 mL, add 20 mL Hydrochloric acid (3.2), heated to $40^\circ\text{C} \sim 60^\circ\text{C}$, plus 2 drops of methyl orange solution (3.5) and 4 drops of methyl blue solution (3.6), with potassium bromate standard drops Set the solution titrated to blue and green, that is the end point. The volume difference of potassium bromate standard titration solution (3.9) consumed by parallel calibration does not exceed 0.10 mL. Follow the calibration to do blank test.

Calculate the concentration of potassium bromate standard titration solution according to formula (1) Concentration.

$c\text{KBrO}_3 =$

$(V_1 - V_0) \times 74.923$