

ICS 77.120.01
CCS H 13

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

YS/T 775.4-2011

**Methods for chemical analysis of lead anode slime. Part 4:
Determination of antimony content. Flame atomic absorption
spectrophotometry and cerium sulfate titrimetric method**

铅阳极泥化学分析方法 第4部分：锑量的测定火焰原子吸收光谱法和硫酸铈滴定
法

(English Translation)

Issued on: December 20, 2011

Implemented on: July 1, 2012

Issued by: Ministry of Industry and Information Technology

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Foreword

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

- Part 1. Determination of lead content Na₂EDTA titration;
- Part 2. Determination of bismuth content - Flame atomic absorption spectrometric method and Na₂EDTA titration;
- Part 3. Determination of arsenic content potassium bromate titration;
- Part 4. Determination of antimony content - Flame atomic absorption spectrometry and cerium sulfate titration;
- Part 5. Gold and silver contents - Fire assay gravimetric method;
- Part 6. Determination of copper content iodometry;
- Part 7. arsenic, copper and selenium content - Inductively coupled plasma atomic emission spectrometry.

This is Part 4.

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

The content ranges overlap arbitration analysis using method two.

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

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Methods for chemical analysis of lead anode slime Part 4. Determination of antimony content A method of flame atomic absorption spectrometry

1 Scope

This method specifies the method for the determination of antimony content of lead anode mud.

This method is applicable to the determination of lead anode mud antimony content was measured in the range of from 5.00% to 13.00%.

2 Method summary

Sample with a mixed acid of hydrochloric acid and nitric acid decomposition, plus antimony tartrate complex. In hydrochloric acid medium, atomic absorption spectrometer wavelength 217.6nm at using air - acetylene flame measured absorbance of antimony.

3 Reagents

Unless otherwise indicated in the analysis using only recognized as analytical grade reagents and distilled or deionized water or equivalent purity.

3.1

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

Nitrate 3.2 ($\rho=1.42\text{g/mL}$).

3.3

hydrochloric acid (119).

3.4

mixed acid. hydrochloric acid, nitric acid (31).

3.5

tartaric acid solution (200g/L).

3.6

Antimony standard stock solution. Weigh 0.5000g metal antimony ($w_{Sb} \geq 99.99\%$), placed in 250mL beaker, add tartaric acid and 5g 30mL of nitric acid (3.2), dissolved by heating on a hot plate, remove, cooled to room temperature, transferred to 500mL volumetric flask, dilute to the mark with water and mix.

This solution 1mL 1mg containing antimony.

3.7

Antimony standard solution. Pipette 20.00mL antimony standard stock solution (3.6) in 200mL volumetric flask, was added 40mL of hydrochloric acid (3.1), 10mL tartaric acid solution (3.5), dilute to the mark and mix. This solution 1mL containing 100 μ g antimony.

4 Instrument

Atomic absorption spectrometer, with antimony hollow cathode lamp.

Under optimum working conditions, who can achieve the following index value can be used.

Sensitivity. antimony characteristic concentration should not exceed 0.30 μ g/mL.

Precision. measure 10 times the absorbance of the highest concentration of the standard solution, the standard deviation should not exceed the average absorbance of 1.0%, with the most Low concentrations of the standard solution (not a "zero" concentration of the solution) absorbance measured 10 times, and the standard deviation is not more than the mean absorbance of the highest concentration of the standard 0.5% luminosity.

Linear curve. The curve is divided into five sections according to concentration, than the difference between the absorbance of the absorbance values of the highest and the lowest segment section shall not Less than 0.7.