

ICS 77.120.01

CCS H 13

YS

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

YS/T 239.7-2010

**Methods for chemical analysis of antimony trisulfide. Part 7:
Determination of lead content. Flame atomic absorption
spectrometric method**

三硫化二锑化学分析方法 第7部分：铅量的测定火焰原子吸收光谱法
(English Translation)

Issued on: November 22, 2010

Implemented on: March 1, 2011

Issued by: Ministry of Industry and Information Technology

Table of Contents

Foreword
1 Scope
2 Method summary
3 Reagents
3.6 Lead standard stock solution
3.7 Lead standard solution
4 Instrument

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 is Part 7.

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

Division into new parts.

This standard by the national non-ferrous metals Standardization Technical Committee (SAC/TC243) centralized.

1 Scope

This section YS/T 239 provides for antimony trisulfide determination of lead content.

This section applies to the determination of the amount of lead antimony trisulfide.

Measurement range. 0.0020% mass fraction of Pb to 0.050%.

2 Method summary

Sample dissolution with hydrochloric acid and nitric acid evaporated, the repetitive addition hydrobromide volatile antimony. In dilute hydrochloric acid medium, using air - acetylene flame at Atomic absorption spectrometer wavelength 283.3nm measured absorbance lead.

3 Reagents

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

3.1

hydrochloride (rho1.19g/mL).

Nitrate 3.2 (rho1.42g/mL).

3.3

hydrobromide (rho1.50g/mL).

3.4

hydrochloride (11).

3.5

nitric acid (11).

3.6 Lead standard stock solution

Weigh 1.0000g lead ($w_{Pb} \geq 99.99\%$) in 200mL beaker, add 20mL of nitric acid (3.5), heat gently to dissolve completely, boil the oxides of nitrogen purge, cooled to room temperature. Moved into 1000mL volumetric flask, supplemented with 20mL of nitric acid (3.5), diluted with water to the mark, mix uniform. This solution 1mL lead 1mg.

3.7 Lead standard solution

Pipette 10.00mL lead standard stock solution (3.6) in 100mL flask, add 5mL hydrochloric acid (3.4), diluted with water to the mark Degree, and mix. This solution 1mL lead 100 μ g.

4 Instrument

Atomic absorption spectrometer, with a lead hollow cathode lamp.

Under optimum working conditions for those who can reach the following indicators can be used.

--- Characteristic concentration. a solution in the measurement matrix consistent solution, characterized in lead concentration should not exceed 0.20 μ g/mL.

--- Precision. standard solution with the highest concentration of 10 times the absorbance measurements, the standard deviation should not exceed the average absorbance of 1.0%;

With the lowest concentration of standard solution (not "zero" concentration of the standard) measuring the absorbance of 10 times, the standard deviation should not exceed the maximum 0.5% concentration of the standard average absorbance.

--- Linear curve. The curve is divided into five sections according to concentration, absorbance difference between the absorbance difference between the highest and the lowest segment of the segment