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**NATIONAL STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA**

GB/T 3260.4-2013

Replacing GB/T 3260.4-2000

**Methods for chemical analysis of tin -- Part 4: Determination of
lead content -- Flame atomic absorption spectrometric method**

锡化学分析方法 第4部分：铅量的测定 火焰原子吸收光谱法

(English Translation)

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Foreword

GB/T 3260 "Methods for chemical analysis of tin" is divided into 14 parts.

- Part 1. Determination of copper content - Flame atomic absorption spectrometry;
- Part 2. Determination of iron content - 1,10-phenanthroline spectrophotometry;
- Part 3. Determination of bismuth content of potassium iodide spectrophotometric method and the flame atomic absorption spectrometry;
- Part 4. Determination of lead content by flame atomic absorption spectrometry;
- Part 5. Determination of antimony content - Malachite green spectrophotometry;
- Part 6. Determination of arsenic content - Malachite Green - arsenic heteropolyoxomolybdate spectrophotometry;
- Part 7. Determination of aluminum content electrothermal atomic absorption spectrometry;
- Part 8. Determination of zinc content - Flame atomic absorption spectrometry;
- Part 9. Determination of sulfur content of high frequency induction furnace combustion infrared absorption method;
- Part 10. Determination of cadmium content by flame atomic absorption spectrometry;
- Part 11. Determination of the amount of silver by flame atomic absorption spectrometry;
- Part 12. Determination of nickel content by flame atomic absorption spectrometry;
- Part 13. Determination of cobalt content by flame atomic absorption spectrometry;
- Part 14. Inductively coupled plasma atomic copper, iron, bismuth, lead, antimony, arsenic, aluminum, zinc, cadmium, nickel, cobalt and the amount of emitted light Spectroscopy.

This section GB/T 3260 Part 4.

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

This part is GB/T 3260.4-2000 "chemical analysis of tin Determination of lead content," the amendment. This standard GB/T 3260.4-2000 compared to the main changes are as follows.

--- By flame atomic absorption spectrometry replaced POLAROGRAPHY.

--- Detection limit reduced from 0.0010 to 0.00080%;

--- Text format has been modified;

--- Increasing the repeatability and reproducibility content.

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

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1 Scope

GB/T 3260 provisions of this part of the tin lead by flame atomic absorption spectrometric method for the amount.

This section applies to the determination of tin in lead content. Determination of the range of 0.00080% to 0.0500%.

2 Method summary

Samples with hydrochloric acid, nitric acid is completely dissolved in sulfuric acid medium, hydrochloric acid - hydrobromide exclude a large number of tin. Hydrochloric acid - nitric acid mixed media Using air - acetylene flame atomic absorption spectrometer wavelength 283.3nm measured absorbance lead.

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

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

3.2

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

3.3

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

3.4

hydrobromide (excellent pure analytical grade or purified by distillation).

3.5

Hydrogen peroxide (30%).

3.6

sulfate (11).

3.7

nitric acid (21).

3.8

hydrochloric acid - Mixed hydrobromic acid. hydrochloric acid hydrobromide (11).

3.9

hydrochloric acid - mixed nitric acid. hydrochloric acid, nitric acid (31), using now.

3.10

lead standard stock solution. Weigh 1.0000g metallic lead ($w_{\text{Pb}} \geq 99.99\%$) in 300mL beaker, add 60mL of nitric acid (3.7), Low dissolved completely, boiled to remove oxides of nitrogen, cooling water moved into 1000mL volumetric flask, dilute to volume, and mix. This solution 1mL containing 1mg lead.

3.11

Lead standard solution. Pipette 20.00mL lead standard stock solution (3.10) in 1000mL flask, add 60mL of nitric acid (3.7), Dilute with water to volume, and mix. This solution 1mL containing 20 μg lead.

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 0.11 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