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GB

National Standard of the People's Republic of China

GB/T 4103.14-2009

Methods for chemical analysis of lead and lead alloys - Part 14: Determination of cadmium content

- Flame atomic absorption spectrophotometry

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Table of Contents

Pretace	3
1 Scope	
2 Methods and Principles	
3 Reagents	
4 Instruments	
5 Analytical Steps	7
6 Calculation of analytical results	8
7 Precision	9
8 Quality assurance and control	10
References and Original Chinese Documents	11

Preface

- GB/T 4103 " Methods for chemical analysis of lead and lead alloys" is divided into 16 parts:
- Part 1: Determination of tin content;
- Part 2: Determination of antimony content;
- Part 3: Determination of copper content;
- Part 4: Determination of iron content;
- Part 5: Determination of bismuth content;
- Part 6: Determination of arsenic content;
- Part 7: Determination of selenium content:
- Part 8: Determination of tellurium content;
- Part 9: Determination of calcium content;
- Part 10: Determination of silver content;
- Part 11: Determination of zinc content;
- Part 12: Determination of thallium content;
- Part 13: Determination of aluminum content;
- Part 14: Determination of cadmium content Flame atomic absorption spectrophotometry;
- Part 15: Determination of nickel content Flame atomic absorption spectrometry;
- Part 16: Determination of copper, silver, bismuth, arsenic, antimony, tin, zinc contents Photoelectric direct-reading emission spectroscopy.

This is Part 14.

This Part was proposed by the China Nonferrous Metals Industry Association.

This part is administered by the National Standardization Technical Committee for nonferrous metals.

The responsible drafting organizations of the Part: Zhuzhou Smelter Group Co., Ltd., and Henan Yuguang Gold & Lead Co., Ltd.

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Methods for chemical analysis of lead and lead alloys

Part 14: Determination of cadmium content

Flame atomic absorption spectrophotometry

1 Scope

This Part of GB/T 4103 specifies the determination of cadmium content in lead and lead-cadmium alloys.

This Part applies to the determination of cadmium content in lead and leadcadmium alloys.

Measuring range: 0.000 1% -0.01%; 0.3% – 2.0%.

2 Methods and Principles

Samples shall be dissolved in nitrate; USE lead sulfate to precipitate and separate lead; USE air-acetylene flame, at wavelength 228.8 nm of atomic absorption spectrometer, to measure absorbance of cadmium. USE standard curve method to calculate cadmium content.

3 Reagents

Unless otherwise stated, water used to prepare the solution and analysis in this test shall be Grade 1 Water; all test utensils used in the test shall be immersed with dilute nitrate (1 + 4), and shall be thoroughly washed with Grade 1 Water.

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3.1 Nitrate (ρ 1.42 g / mL), superior grade purity.
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- 3.2 Sulfate (p 1.84 g / mL); superior grade purity.
- 3.3 Nitrate (1 + 1).
- 3.4 Nitrate (1 + 3).
- 3.5 Sulfate (1 + 1).
- 3.6 Cadmium standard storage solution: WEIGH 0.500 0 g of metal cadmium (mass fraction of cadmium ≥ 99.99%) into 250 mL beaker; ADD 20 mL of nitrate

(3.1); COVER with watch glass; HEAT it until completely dissolved; BOIL to expel nitrogen oxides; COOL it; MOVE into 1 000 mL volumetric flask; USE water to dilute the volume to the mark; SHAKE well. 1 mL of this solution contains 0.5 mg of cadmium.

3.7 Cadmium standard solution: PIPETTE 10.00 mL of cadmium standard storage solution (3.6) into 500 mL volumetric flask; ADD 20 mL of nitrate (3.3); USE water to dilute the volume to the mark; MIX well; 1 mL of this solution contains 10 µg of cadmium.

4 Instruments

Atomic absorption spectrometer, attached with cadmium hollow cathode lamp. Under the optimum working conditions; those instruments which can achieve the following targets may be used.

- Characteristic concentration: In solution consistent with the matrix of measuring sample solution, the characteristic concentration of cadmium shall be less than 0.05 µg / mL;
- **Precision**: USE standard solution of the highest concentration to measure absorbance for 10 times, its standard deviation shall not exceed 1.0% of the average absorbance; USE standard solution of the lowest concentration (not the standard solution of "zero" concentration) to measure absorbance for 10 times, its standard deviation shall not exceed 0.5% of the average absorbance of standard solution of the highest concentration;
- Linearity of working curve: DIVIDE the working curve into five sections equally, based on concentration; the ratio between the absorbance difference of the highest section AND the absorbance difference of the lowest section shall not be less than 0.8:

Reference operating conditions of the instruments are shown in Table 1.

Table 4

Mass factor of	0.0002			0.50	1.00	1.50	
cadmium / %	0.0002	0.0002 0.002	0.0020	0.0050	0.50	1.00	1.50
R / %	0.0001	0.0005	0.0010	0.04	0.05	0.06	
Note: Reproducibility (R) is 2.8S _R ; S _R is the reproducibility standard deviation.							

8 Quality assurance and control

Standard samples of national-level or industrial-level shall be used (when these 2 samples are not available, controlled standard sample may be used for substitution); effectiveness of this analytical method shall be calibrated weekly or biweekly. When the process is out of control, root cause shall be found out; After the error is corrected, calibration shall be repeated.

References and Original Chinese Documents

[1] GB/T 4103.14-2009 Methods for chemical analysis of lead and lead alloys - Part 14: Determination of cadmium content - Flame atomic absorption spectrophotometry. http://www.chinesestandard.net/Default.aspx?PDF-English-lb=GB/T%204103.14-2009

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