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National Standard of the People's Republic of China

GB/T 14849.3-2007
Replace GB/T 14849.3-1993

Methods for chemical analysis of silicon metal—Part 3: Determination of calcium content

工业硅化学分析方法 第 3 部分:钙含量的测定

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Foreword

GB/T14849 *Methods for Chemical Analysis of Silicon Metal* is divided into four parts:

——Part 1: Determination of iron content 1, 10-phenanthroline spectrophotometric method

——Part 2: Determination of aluminum content Chrome azurol-S spectrophotometric method

——Part 3: Determination of calcium content

——Part 4: Inductively coupled plasma-atomic emission spectrometry to determine element content

This Part is Part 3.

Method I and Method II of this Part are flame atomic absorption spectrometry and chlorophosphonazo-I spectrophotometric method respectively.

If the analysis results are controversial, take Method I as the arbitration analysis method.

Method I of this Part is an amendment of GB/T 14849.3-1993 *Methods for chemical analysis of silicon metal: Determination of calcium content*. To reflect the coordination of standards, and make the standards of analysis method and product match well, the determination range(of quality fraction) of these two methods in this Part is changed from 0.05%-1.20% to 0.02%-0.30%, meanwhile, the clauses of “repeatability” and “quality assurance and control” is added.

This Part is proposed by China Nonferrous Metals Industry Association.

This Part is under the jurisdiction of the National Technical Committee on Nonferrous Metals of Standardization.

This Part is drafted by Fushun Aluminum Co., Ltd.

Method I in this Part is drafted by Zhengzhou Research Institute of CHALCO.

Method II in this Part is drafted by Fushun Aluminum Co., Ltd.

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The release conditions of previous versions of the standards replaced by this Part are as follows:

——GB/T 14849.3-1993

Methods for chemical analysis of silicon metal—Part 3: Determination of calcium content

Method I Flame atomic absorption spectrometry

1 Scope

This Method specifies the method for determination of calcium content in silicon metal.

This Method applies to the determination of calcium content in silicon metal. Determination range (quality fraction): 0.020%～0.30%

2 Method summary

The sample is decomposed by hydrofluoric acid and nitric acid, perchloric acid smoking is used to remove silicone, fluorine, etc., and residue is dissolved by hydrochloric acid. Lanthanum salt is used to control the interference of aluminum. Determine the absorbance of calcium by air-acetylene flame, at flame atomic absorption spectrometry wavelength of 422.7nm.

3 Reagents

3.1 Perchloric acid (ρ1.67g/L).

3.2 Hydrogen Chloride (ρ1.14g/L).

3.3 Nitric acid (1+1).

3.4 Hydrochloric acid (1+1).

3.5 Lanthanum salt solution (10g/L): 5.00g lanthanum oxide is put into 250mL beaker, and 15mL hydrochloric acid (3.4) is added. Dissolve by slightly heating, and cool to the room temperature. Put it into 500mL volumetric flask, and dilute with water to the scale and mix it evenly.

3.6 Calcium standard storage solution (500μg/mL): 0.6243g standard calcium carbonate, which is pre-dried at 105℃ and then cooled to the room temperature in dryer, is put into 300mL beaker. About 20mL water is added. Hydrochloric acid (3.4) is added dropwise until completely dissolved, and an excessive 10mL is added. Heat it to boil to remove carbon dioxide. Cool it to the room temperature. Put into 500mL volumetric flask, and dilute with water to the scale and mix it evenly.1mL solution contains 500μg calcium.

3.7 Calcium standard solution (50μg/mL): Take 25.00mL calcium standard storage

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