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NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB/T 14849.2-2007

Replacing GB/T 14849.2-1993

Methods for chemical analysis of silicon metal - Part 2: Determination of aluminum content - Chrome azurol S spectrophotometric method

工业硅化学分析方法 第2部分：铝含量的测定 铬天青-S分光光度法

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Foreword

GB/T 14849 Methods for Chemical Analysis of Silicon Metal is divided into 4 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 - flame atomic absorption spectrometry, chlorophosphonazo I spectrophotometric method; - Part

4.Inductively coupled plasma-atomic emission spectrometry to determine element content. This is Part 2 of GB/T 14849. This Part will replace GB/T 14849.2-1993 Silicon metal - Determination of aluminium content--Chrome azurol S spectrophotometric method. Compared with GB/T 14849.2-1993, the main changes are as follows: - "Repeatability" and "quality assurance and control" is added. This Part was proposed by China Nonferrous Metals Industry Association. This Part shall be under the jurisdiction of the National Technical Committee on Nonferrous Metals of Standardization. This Part was responsibly drafted by Fushun Aluminium Co., Ltd. Main drafters of this Part. Xu Tieling, Ji Chunlei, Yang Yuhong, and Xu Tieling. The previous version of the standard replaced by this Part is as follows: - GB/T 14849.1-1993. Methods for chemical analysis of silicon metal - Part 2. Determination of aluminum content - Chrome azurol S spectrophotometric method

1 Scope

GB/T 14849.2-2007 is the Chinese national standard on methods for chemical analysis of

silicon metal - part 2: determination of aluminum content - chrome azurol s spectrophotometric method, in the field of metallurgy. The /T suffix marks it as a recommended standard: it is not compulsory by itself, but it becomes binding as soon as a contract, a tender or a customer specification calls it up - which in practice is how most foreign buyers meet it. It was issued on 25 October 2007 by the General Administration of Quality Supervision, Inspection and Quarantine of the People's Republic of China; Standardization Administration of China, and has been in force since 1 April 2008. Classification: ICS 77.120.10, CCS H12. This page is published from the official record of the standard held by the Chinese standards administration: the identification, the dates, the classification and the issuing body are taken from there. The clause text, the tables and the numeric limits are in the document itself, which is delivered complete in English translation.

This Part specifies the method for determination of aluminum content in silicon metal. This Part applies to the determination of aluminum content in silicon metal. Determination range (mass fraction). 0.02%~0.30%.

2 Method summary

The specimen is decomposed with hydrofluoric acid and nitric acid. Sulfuric acid's smoking removes silicon, fluorine, etc. The residue is dissolved with hydrochloric acid. Ascorbic acid is used to mask the interference of ferrum. A purplish-red complex compound is formed by the aluminum and chrome azurol-S, in hexamethylenetetramine medium of pH5.5-6.1.At wavelength of 510nm of the spectrophotometer, MEASURE the absorbance.

3 Reagents

3.1 Hydrofluoric acid (rho1.14g/mL).

3.2 Nitric acid (1+1).

3.3 Sulfuric acid (1+1).

3.4 Hydrochloric acid (1+1).

3.5 Ascorbic acid solution (10g/L). Prepare it when use.

3.6 Hexamethylenetetramine solution (300g/L).

3.7 Chrome azurol S acetic acid solution (0.3g/L). WEIGH 0.30g of chrome azurol-S and PUT into a beaker. ADD 25mL of water and 25mL of anhydrous alcohol. After dissolved, ADD 475mL of water. USE anhydrous alcohol to dilute to 1000mL. MIX evenly.

3.9 Aluminum standard solution. PITETTE 10.00mL of aluminum standard storage solution (3.8) into a 500mL volumetric flask.

4 Instruments

Spectrophotometer.

5 Sample

The test sample shall all pass through 0.149mm standard sieve. And USE magnet to remove iron powder.

6 Analytical steps

6.1 Specimen WEIGH 1g of the test sample (5), accurate to 0.0001g.

6.2 Determination number of times PERFORM twice independent determinations and TAKE the average values.

6.3 Blank test MAKE a blank test along with the specimen (6.1).

6.4 Determination

6.4.1 The specimen (6.1) is put in 100mL platinum dish. ADD 0.5mL of sulphuric acid (3.3) and 20mL-25mL of hydrofluoric acid (3.1).

6.5 Drawing of working curve

6.5.1 PIPETTE blank test solution (6.3) that is equal to the volume of the test solution B. according to Table

1.Respectively PLACE into a set of 100mL volumetric flasks. Then respectively ADD 0mL, 1.00mL, 2.00mL, 3.00mL, 4.00mL, and 5.00mL of standard aluminum solution (3.9).

6.5.2 TAKE the mass of aluminum as the abscissa; absorbance as ordinate; DRAW the working curve.

7 Calculation of the analysis results

CALCULATE the mass fraction of aluminum (w) by Formula (1), values expressed in %.

8 Precision

8.1 Repeatability Under the repeatability condition, within the range of average value given as follows, for the determination values obtained from 2 independent test results, the absolute values of these 2 test results shall not exceed repeatability limit (r).

8.2 Allowable difference The difference value of analysis results among the laboratories shall not exceed the allowable difference listed in Table 3.