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CCS H12



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

GB/T 14849.4-2014

Replacing GB/T 14849.4-2008

**Methods for chemical analysis of silicon metal-Part 4:
Determination of impurity contents-Inductively coupled plasma
atomic emission spectrometric method**

工业硅化学分析方法 第4部分：杂质元素含量的测定 电感耦合等离子体原子发射
光谱法

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Foreword

GB/T 14849 "Industrial Silicon Chemical Analysis Method" is divided into 9 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 spectrometric method,
chlorophosphonazo I spectrophotometric method; - Part

4.Determination of impurity contents - Inductively coupled plasma atomic emission
spectrometric method; - Part

1 Scope

GB/T 14849.4-2014 is the Chinese national standard on methods for chemical analysis of silicon metal-part 4: determination of impurity contents-inductively coupled plasma atomic emission spectrometric 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 5 December 2014 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 August 2015.

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 of GB/T 14849 specifies the determination methods of iron, aluminum, calcium, manganese, titanium, nickel, copper, chromium, vanadium, magnesium, cobalt, phosphorus, potassium, sodium, lead, zinc, boron in industrial silicon. This part applies to the determination of iron, aluminum, calcium, manganese, titanium, nickel, copper, chromium, vanadium, magnesium, cobalt, phosphorus, potassium, sodium, lead, zinc, boron in industrial silicon. The measurement range of each element is as shown in Table 1.

Table 1 -- Measurement range Element Mass fraction / % Element Mass fraction / % Iron
0.020 ~

1.00 Phosphorus 0.0010 ~

0.10 Manganese 0.0050 ~

0.50 Potassium 0.0010 ~

0.50 Titanium 0.0050 ~

0.50 Sodium 0.0010 ~

0.50 Nickel 0.0050 ~

0.50 Lead 0.0030 ~

0.10 Copper 0.0010 ~

0.50 Zinc 0.0010 ~

0.50 Chromium 0.0010 ~

0.50 Boron 0.0005 ~

0.20 Vanadium 0.0005 ~ 0.20 - -

2 Normative references

The following documents are essential to the application of this document. For the dated documents, only the versions with the dates indicated are applicable to this document; for the undated documents, only the latest version (including all the amendments) are applicable to this standard.

GB/T 6682 Water for analytical laboratory use - Specification and test methods

GB/T 8170 Rules of rounding off for numerical values & expression and judgement of limiting values

3 Summary of method

The sample is dissolved by the use of hydrofluoric acid or nitric acid. The perchloric acid is smoked to remove silicon, fluorine, etc.

4 Reagents

The reagents and water used in this standard refer to, unless otherwise specified, the analytical reagents and the grade water as specified in GB/T 6682.

4.1 Perchloric acid ($\rho =$

1.67 g/mL), excellent grade pure.

4.9 Sodium hydroxide solution (400 g/L).

4.10 Metal iron ($\text{Fe} \geq 99.99\%$).

4.11 Metal aluminum ($\text{Al} \geq 99.99\%$).

4.12 Calcium carbonate (base reagent or excellent grade pure).

4.13 Metal manganese ($\text{Mn} \geq 99.99\%$).

4.14 Metal titanium ($\text{Ti} \geq 99.99\%$).

4.15 Metal nickel ($\text{Ni} \geq 99.99\%$).

4.16 Metal copper ($\text{Cu} \geq 99.99\%$).

4.17 Potassium dichromate (base reagent or excellent grade pure).

4.22 Potassium chloride (spectral pure).

4.23 Sodium chloride (spectral pure).

4.24 Metal lead ($\text{Pb} \geq 99.99\%$).

4.25 Metal zinc ($\text{Zn} \geq 99.99\%$).

5 Instruments

Inductively coupled plasma optical emission spectrometer. Light source. Plasma source, using power of 750 W ~ 1750 W.

6 Specimen

The specimen shall pass through a

0.149 mm standard sieve.

7 Analytical procedures

7.1 Samples According to the preparation method (7.4) of the analytical test solution, analyze the weighed sample amount and the constant volume of different elements, which shall comply with the requirements of Table

2. The weighed sample amount is accurate to 0.0001 g.

7.2 Number of measurements Perform two measurements independently and take the average.

7.3 Blank test Carry out the blank test along with the sample.

7.5 Preparation of series standard solution

7.5.1 Add a suitable amount of standard solution B (4.29) to a set of 100 mL volumetric flask. The medium and acidity are the same as the sample solution. Use water to dilute it to the mark. Shake it uniformly. Use the specimen in which no standard solution is added as the blank solution. The content of the element to be determined shall be within the range of the plotted working curve. The amount of the series standard solution is determined by the accuracy requirements. Take at least 6 points.

7.6 Determination

7.6.1 The recommended analytical lines for each element to be tested are as shown in Table 3.

8 Calculation of analytical results

Calculate the mass fraction of the element to be tested according to formula (1). The value is expressed in %. The value is expressed and judged according to the GB/T 8170 numerical rounding off rule and limit value.

9 Precision

9.1 Repeatability limit The measured values of two independent tests are obtained under repetitive conditions.

9.2 Reproducibility limits The absolute difference between two independent test results obtained under Mass fraction / % reproducibility conditions is not greater than the