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Test method for chloride content of silicon - Ion chromatography method

硅中氯离子含量的测定 离子色谱法

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Test method for chloride content of silicon - Ion chromatography method

1 Scope

This Standard specifies ion chromatography test method for chloride content of silicon.

This Standard is applicable to the determination of chloride content of rod silicon, block silicon, granular silicon, and flake silicon. The determination range is from 1.0μg/g to the maximum solid solubility of chloride in silicon.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

GB/T 6679, *General rules for sampling solid chemical products*

GB/T 6682-2008, *Water for analytical laboratory use - Specification and test methods*

3 Method principle

Place the sample in a high-temperature tube type furnace to heat. After the sample is melt, chlorine shall be released in the form of chloride. Use a bubble absorption bottle that is filled with water to absorb. Then use ion chromatograph to determine the chloride content in the absorption solution.

4 Interference factors

4.1 The material purity of the sample tube and the sample container of the high-temperature tube type furnace shall affect the test results. If the purity is not enough, the impurities in the sample tube and the sample container shall evaporate at a high temperature.

4.2 The sample tube and the sample container of the high-temperature tube

prepared absorption bottles connected in series (8.3) to the exhaust port of the high-temperature tube type furnace. Adjust the flow of argon to 200mL/min~300mL/min. Argon enters absorption bottle through the high-temperature tube type furnace.

8.5.2 Heat test portion from room temperature to 1500°C and remain at 1500°C for 20min. Then cool to room temperature. During this process, chlorine in the sample is released in the form of chloride and absorbed by water in the absorption bottle.

8.5.3 Use ion chromatograph to determine the chloride content of the solution in the absorption bottle. When the chloride content in the sample is below or exceeds the standard curve drawing range, it shall adjust the sample quantity according to Table 1 to re-analyze. Refer to Annex A for the working conditions of ion chromatograph.

9 Test data processing

The chloride content w in test portion is calculated according to formula (1):

$$w = \frac{(c_1 + c_2 - c_0) \times V}{m} \qquad \cdots\cdots(1)$$

Where,

w - Chloride content in test portion, in micrograms per gram (μg/g);

c_1 - Content of chloride in the first absorption bottle, in micrograms per milliliter (μg/mL);

c_2 - Content of chloride in the second absorption bottle, in micrograms per milliliter (μg/mL);

c_0 - Sum of blank chloride in two absorption bottles, in micrograms per milliliter (μg/mL);

V - Total volume of water in two absorption bottles, in milliliters (mL);

m - Mass of test portion, in grams (g).

The calculation result is expressed to one digit after the decimal point.

10 Precision

10.1 For the measured values of two independent test results obtained under

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