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

SN/T 5249-2020

**Determination of iron, manganese, copper, aluminum, titanium, lead, chromium,
calcium, magnesium, zinc, potassium, sodium content in precipitated hydrated
silica by inductively coupled plasma atomic emission spectrometry**

沉淀水合二氧化硅中铁、锰、铜、铝、钛、铅、铬、钙、镁、锌、钾、钠含量的
测定 电感耦合等离子体原子发射光谱法

(English Translation)

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Foreword

This standard was drafted in accordance with the rules given in GB/T 1.1-2009.

This standard was proposed and managed by the General Administration of Customs of the People's Republic of China.

Drafting organizations of this standard. Xiamen Customs of the People's Republic of China, Chongqing Customs of the People's Republic of China, Fujian Zhengsheng Inorganic Materials Stock Co., Ltd.

1 Scope

This standard specifies the inductively coupled plasma atomic emission spectrometry (ICP-AES) for the determination of precipitated hydrated silica (commonly known as white carbon) Black) method of iron, manganese, copper, aluminum, titanium, lead, chromium, calcium, magnesium, zinc, potassium and sodium content.

This standard applies to the content of iron, manganese, copper, aluminum, titanium, lead, chromium, calcium, magnesium, zinc, potassium and sodium in precipitated hydrated silica.

Determination. The elements and measurement ranges are shown in Table 1.

2 Normative references

The following documents are indispensable for the application of this document. For dated reference documents, only the dated version applies to this article. For undated reference documents, the latest version (including all amendments) is applicable to this document.

GB/T 602 Preparation of standard solutions for impurity determination of chemical reagents
GB/T 6682 Analytical laboratory water specifications and test methods

3 Method summary

The test sample was digested with hydrofluoric acid and perchloric acid, in which the silicon was volatilized in the form of silicon tetrafluoride. Dissolve the residue in a hydrochloric acid solution, the test solution is measured with an inductively coupled plasma atomic emission spectrometer, and the content of each element is determined according to the calibration curve.

4 Reagents and materials

4.1

Unless otherwise specified, only superior grade pure reagents are used, and the experimental water should meet the requirements of secondary water specified in GB/T 6682.

4.2

Hydrofluoric acid ($\rho=1.13$ g/mL).

4.3

Perchloric acid ($\rho=1.76$ g/mL).

4.4

Hydrochloric acid ($\rho=1.19$ g/mL).

4.5

Hydrochloric acid solution (5 95).

4.6

Single element standard solution (1 000 $\mu\text{g/mL}$). iron, manganese, copper, aluminum, titanium, lead, chromium, calcium, magnesium, zinc, potassium and sodium standard solution the solution is prepared according to GB/T 602 method, or directly purchase a certified standard solution.

4.7

Mixed standard solution series. prepared by diluting each element standard solution (4.6) step by step, the medium is hydrochloric acid solution (4.5), the concentration of each element in the mixed solution of the calibration curve series is shown in Table 2.

5 Apparatus and equipment

5.1

Inductively coupled plasma atomic emission spectrometer, equipped with hydrofluoric acid resistant sampling system. Refer to Appendix A for the working conditions of the instrument.

5.2

Analytical balance. the accuracy is 0.1 mg.

5.3

Oven. The temperature can be maintained at $105\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$.

5.4

Platinum crucible, with a volume of about 75 mL.

5.5

Electric heating plate.

6.1 Sample preparation

6.1.1

Dry the sample at $105\text{ }^{\circ}\text{C}$ for 2 h before analysis, and cool it to room temperature in a desiccator.

6.1.2

Weigh 2.0 g of the sample to the nearest 0.1 mg. Make parallel determinations of two samples.

6.2 Blank test

Except that the sample to be tested is not added, the blank test is carried out according to step 6.3.

6.3 Sample decomposition and test solution preparation

6.3.1

Place the sample (6.1.2) in a platinum crucible (5.4), add an appropriate amount of water to fully wet the sample, and add 10 mL of hydrofluoric acid (4.2) and 0.5 mL perchloric acid

(4.3). Place the platinum crucible (5.4) on the electric heating plate (5.5), heat it until the perchloric acid smoke is exhausted, take it out, and cool it down.

6.3.2

Add 5.0 mL of hydrochloric acid (4.4), rinse the inner wall of the platinum crucible with a small amount of water, and heat to dissolve the residue. Cool to room temperature, move in a 50 mL plastic volumetric flask, dilute to the mark with water and shake well.

6.4 Drawing of calibration curve

Under certain working conditions of the instrument, a series of calibration working solutions are determined in sequence from low to high concentration. Take the spectral intensity as the ordinate, the element concentration is the abscissa to draw the calibration curve, and the linear correlation coefficient of each element calibration curve should not be less than 0.999.

6.5 Determination

Measure the spectral intensity of each measured element in the blank solution and the sample solution respectively, and find out the content of the measured element from the calibration curve.

7 Result calculation

The content of each element is calculated by mass fraction w , and the value is expressed in mg/kg, calculated according to formula (1).

Where.

c_i --the concentration of the measured element in the sample, in micrograms per milliliter ($\mu\text{g/mL}$);

c_0 --Concentration of sample blank, in micrograms per milliliter ($\mu\text{g/mL}$);

V --The volume of the sample solution, the unit is milliliters (mL);

m --the mass of the sample, the unit is grams (g).

Note: The calculation result is the average of two parallel determination results, and two decimal places are retained.

8 Precision

The precision was determined by 8 laboratories on 6 levels of samples, and the results were determined after statistical data. The results are shown in Table 3.

Note: m is the average of two determination results.