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SN

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

SN/T 5242-2020

**Import and export textiles Determination of germanium content
Inductively coupled plasma mass spectrometry**

进出口纺织品 锗含量的测定 电感耦合等离子体质谱法

(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. Shaoxing Customs of the People's Republic of China, the Patent Office of the State Intellectual Property Office, China Inspection and Control Technology Group Co., Ltd.

the company.

1 Scope

This standard specifies the inductively coupled plasma mass spectrometry (ICP-MS) method for the determination of germanium in textiles.

This standard applies to the determination of germanium content in textiles.

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 6682 Analytical laboratory water specifications and test methods GB/T 8170
Numerical rounding rules and the expression and determination of limit values

3 Principle

After the sample is digested by microwave, the measured element in the sample solution undergoes a plasma process and enters the mass spectrometer through the nickel sampling cone.

The response value of the mass spectrum is directly proportional to the content of the element to be measured. Compared with the standard working curve, indium is used as the internal standard, and the internal standard method is used for quantification.

Calculate the content of germanium in the sample.

4 Reagents and materials

Unless otherwise specified, the reagents used are all premium grade pure, and the experimental water is the first grade water that meets the requirements in GB/T 6682.

4.1

Nitric acid (MOS grade, 65%).

4.2

Hydrogen peroxide (30% aqueous solution).

4.3

Nitric acid solution (2 98). Take 20 mL of nitric acid (4.1) and slowly add 980 mL of ultrapure water.

4.4

Germanium (Ge) standard stock solution (1000 µg/mL). the element standard solution within the validity period of the certificate of standard material can be used, and the concentration it is 1000 µg/mL.

4.6

Germanium standard intermediate working solution (10 µg/mL). Pipette 1.0 mL of germanium standard stock solution (4.4), use nitric acid solution (4.3) to determine Volume

to 100 mL.

4.7

Germanium standard working solution (reference concentration range is 0.2 µg/L~40.0 µg/L). the germanium standard intermediate working solution (4.6) is gradually diluted the medium is nitric acid solution (4.3).

4.8

Indium (In) standard stock solution (1000 µg/mL). as an internal standard in ICP-MS detection, a certificate of standard substance can be used the element standard solution within the expiry date of, the concentration is 1000 µg/mL.

4.9

Indium standard intermediate solution (10 µg/mL). Pipette 1.0 mL of indium standard stock solution (4.8), and dilute to volume with nitric acid solution (4.3) 100 mL.

4.10

Indium standard working solution (1.0 µg/mL). diluted from indium standard intermediate solution (4.9) to 1.0 µg/L, the medium is nitric acid solution (4.3).

4.11

Argon. The purity is greater than or equal to 99.999%.

Note: The prepared standard solution should be promptly transferred to the special plastic reagent bottle for standard solution storage, and stored in the refrigerator at 0 °C ~ 5 °C. The storage period is generally 1 month.

5 Apparatus and equipment

5.1

Inductively coupled plasma mass spectrometer.

5.2

Analytical balance. Sensitivity 1.0 mg.

5.3

Microwave digestion system.

5.4

Volumetric flask. 50 mL.

5.5

Pipette. 0.5 mL, 1.0 mL, 2.0 mL, 5.0 mL.

5.6

Water phase filter membrane. 0.45 µm.

5.7

Electrothermal digestion device.

6 Sample preparation and storage

The sample should be representative as a whole, should contain various yarn and fiber components of the fabric, and the number of samples should be sufficient for testing. will Cut the 10 g sample into pieces not larger than 5 mm x 5 mm, and mix them evenly. The sample is put into a clean container, sealed, and marked with a mark. test the sample should be stored at room temperature.

7.1 Sample digestion

Weigh two 0.5 g samples (for parallel test) from the shredded mixed samples, accurate to 0.001 g, and place them in the microwave digestion inner tank.

Add 5.0 mL of nitric acid (4.1), and then add 3.0 mL of hydrogen peroxide (4.2) to fully infiltrate the sample, and preheat the sample in a 120 °C electrothermal digester.

After 30 minutes of digestion, place it in a microwave digestion system for digestion. Refer to Table A.1 in Appendix A for the digestion procedure. After digestion, take out the digestion tank Cool to room temperature.

7.2 Constant volume of sample solution

Transfer the solution to a 50.0 mL volumetric flask, rinse the tank wall with water 3 times or more, combine the lotions and dilute to the mark, and mix. take 5mL water phase filter membrane (5.6), the filtrate should be used for instrument analysis and testing as soon as possible.

7.3 Instrument working conditions

It should be adjusted to the best condition according to the performance of the respective instruments. For working conditions, see Table B.1 in Appendix B.

7.4 Sample determination

The internal standard working solution (4.10) was added online in the analysis, and the internal standard calibration quantitative analysis method in the ICP-MS analysis method was used for determination.