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Textiles - Determination of total content of boron

纺织品 总硼含量的测定

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Textiles - Determination of total content of boron

WARNING -- Personnel using this document shall have practical experience in formal laboratory work. This document does not address all possible security issues. It is the user's responsibility to take appropriate safety and health measures and ensure compliance with the conditions stipulated in the relevant national laws and regulations.

1 Scope

This document describes a method for the determination of the total content of boron in textiles using an inductively coupled plasma optical emission spectrometer (ICP-OES).

This document applies to various textile materials and their products.

2 Normative references

The following documents contain the provisions which, through normative reference in this document, constitute the essential provisions of this document. For the dated referenced documents, only the versions with the indicated dates are applicable to this document; for the undated referenced documents, only the latest version (including all the amendments) is applicable to this document.

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

3 Terms and definitions

There are no terms and definitions that need to be defined in this document.

4 Principle

After the sample is digested by concentrated nitric acid-hydrogen peroxide microwave, the digestion solution is diluted to the metered volume, and the emission intensities of boron and internal standard yttrium are measured by inductively coupled plasma optical emission spectrometer (ICP-OES). The internal standard method is used for quantification, the concentration of boron is determined according to the standard working curve, and the total content of boron in the sample is calculated.

5 Reagents and materials

5.1 Water: Water shall meet the requirements of GB/T 6682, grade 2 or above.

5.2 Concentrated nitric acid: The concentration is 65% (mass fraction); a guaranteed reagent.

5.3 Nitric acid solution: The concentration is 5% (volume fraction); measure 50 mL of concentrated nitric acid (5.2), and dilute it to 1000 mL with water (5.1).

5.4 Hydrogen peroxide: The concentration is 30% (mass fraction), a guaranteed reagent.

5.5 Boron (B) standard substance: CAS. No7440-42-8, and the mass concentration is 1000 mg/L.

5.6 Yttrium (Y) standard substance: CAS. No7440-65-5, and the mass concentration is 1000 mg/L, internal standard substance.

5.7 Boron standard stock solution: Accurately measure an appropriate amount of boron standard substance (5.5), and use nitric acid solution (5.3) to prepare a boron standard stock solution with a mass concentration of 10 mg/L.

5.8 Internal standard yttrium standard stock solution: Accurately measure an appropriate amount of yttrium standard substance (5.6), and use nitric acid solution (5.3) to prepare a yttrium standard stock solution with a mass concentration of 10 mg/L.

NOTE: The validity period of the standard stock solution is 6 months when stored in the dark at 0 °C~4 °C. When turbidity, precipitation, or color change occurs, prepare it again.

5.9 Standard working solution: Dilute boron standard stock solution (5.7) with nitric acid solution (5.3) as needed to form a series of standard working solutions of different concentrations (all contain 0.10 mg/L yttrium standard solution).

NOTE: The standard working solution is valid for 3 months when stored in the dark at 0 °C~4 °C. When turbidity, precipitation, or color change occurs, prepare it again.

6 Instruments and equipment

6.1 Inductively coupled plasma optical emission spectrometer (ICP-OES).

6.2 Microwave digestion instrument: temperature sensing type.

6.3 PTFE digestion vessel.

6.4 Analytical balance: The division value is 0.0001 g.

- c) Auxiliary gas flow rate: 0.2 L/min;
- d) Atomizing gas flow rate: 0.65 L/min;
- e) Vibration pump flow rate: 1.3 mL/min;
- f) Wavelength of boron analytical spectral line: 249.772 nm;
- g) Wavelength of yttrium analytical spectral line: 371.029 nm.

7.2.2 Determination

7.2.2.1 Determination of the working curve

According to the test requirements and instrument conditions, set the analysis conditions of the instrument, and ignite the plasma torch; after the torch is stable, measure the spectral intensities of the boron element and the internal standard yttrium in the series of standard working solutions in the order of concentration from low to high at the corresponding wavelength. Draw the standard working curve with the boron standard working solution concentration as the abscissa and the spectral intensity ratio of boron to yttrium as the ordinate.

7.2.2.2 Determination of sample liquid

According to the instrument conditions set in 7.2.2.1, measure the spectral intensities of the boron element and the internal standard yttrium in the sample liquid (7.1), and calculate the concentration of the boron element from the working curve.

NOTE 1: The test solution volume needed is about 3 mL.

See Appendix A for the ICP-OES spectrum of boron under the above analysis conditions.

7.2.3 Blank test

Except that no sample is added, the operation is carried out according to the above-mentioned determination steps.

8 Calculation and presentation of the result

8.1 Calculation of the result

The content of boron in the sample is calculated according to formula (1).

$$X = \frac{(C - C_0) \times V}{m} \dots\dots\dots (1)$$

where:

X -- the total content of boron in the sample, in milligrams per kilogram (mg/kg);

C -- the mass concentration of boron in the sample solution, in milligrams per liter (mg/L);

C_0 -- the mass concentration of boron in the blank solution, in milligrams per liter (mg/L);

V -- the metered volume of the sample solution, in milliliters (mL);

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

8.2 Result presentation

The mean of two parallel determination results is taken as the test result, and the result is rounded to one decimal place. When it is lower than the limit of quantification, the test result is "not detected".

9 Limit of quantitation, recovery, and precision

9.1 Limit of quantitation

The limit of quantitation is 5.0 mg/kg.

9.2 Recovery

The standard addition method is used, and the measured recovery shall be greater than 80%.

9.3 Precision

In the same laboratory, the same operator uses the same equipment, according to the same test method, and independently tests the same measured object twice in a short period of time; the relative standard deviation of the two test results obtained is not more than 10%, the probability of greater than 10% is not more than 5% is the premise.

10 Test report

The test report shall at least include the following information:

- a) the number of this document;
- b) sample description;

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