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**FZ**

TEXTILE INDUSTRY STANDARD OF  
THE PEOPLE'S REPUBLIC OF CHINA

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**Method for the determination of residual  
sulphur in viscose fibres - Direct iodimetry**

纤维素化学纤维残硫量测定方法

直接碘量法

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# **Method for the determination of residual sulphur in viscose fibres - Direct iodimetry**

## **1 Scope**

This Standard specifies the method for the determination of residual sulphur by redox-direct iodometry.

This Standard is applicable to fibres made by viscose process.

## **2 Normative references**

The following documents contain the provisions which, through reference in this Standard, become the provisions of this Standard. For dated references, their subsequent amendments (excluding corrigendum) or revisions do not apply to this Standard. However, the parties who enter into agreement based on this Standard are encouraged to investigate whether the latest versions of these documents are applicable. For undated reference documents, the latest versions apply to this Standard.

GB/T 601 Chemical reagent - Preparations of standard volumetric solutions

GB/T 4146 Textile terms and terminology (parts of chemical fibres)

GB/T 8170 Rules for rounding off of numerical values

GB/T 14334-2006 Sampling method for man-made staple fibres

## **3 Terms and definitions**

For the purpose of this Standard, the terms and definitions specified in GB/T 4146 apply.

## **4 Principle**

The sulphur attached to the viscose fibre reacts with sodium sulfite to produce sodium thiosulfate, which is titrated with iodine standard solution. The end point of the titration is judged according to the color change of the indicator. The residual sulphur content is calculated according to the volume consumed.

**7.3** Measuring cylinder: the capacity is 200 mL.

**7.4** Measuring cup: the capacity is 20 mL.

**7.5** Volumetric flask: the capacity is 100 mL.

**7.6** Pipette: the capacity is 50 mL.

**7.7** Iodine flask: the capacity is 500 mL.

**7.8** Ground spherical condenser tube.

**7.9** Vacuum filtration device, including vacuum pump, suction filter flask, buffer flask, etc.

**7.10** Buchner funnel.

**7.11** Cooling device.

**7.12** Heating device, ensuring constant temperature (100 °C) requirements.

**7.13** Container, which can store samples, and the quality is known.

**7.14** Thermometer, 0 ~ 100 °C, accuracy of 1 °C.

## **8 Sampling**

- Unpackaged laboratory samples are taken out as needed;
- Laboratory samples in batch samples are taken according to the provision of GB/T 14334-2006.

Do not take out rolls that have been accidentally damp, scratched or of which the packaging has been opened during transportation.

## **9 Test environment**

The test can be carried out in an environment with a temperature of 20 °C ± 5 °C, avoid sunlight indoors.

## **10 Analysis steps**

**10.1** Randomly TAKE about 20 g of sample, MIX them evenly and DIVIDE them into four parts, each of about 5 g, accurate to 0.001 g. Two of them are used for parallel test of residual sulphur content; the other two are used for moisture

$m_2$  - the mass of the sample and container after baking, in grams (g);

$m_w$  - the mass of the sample before baking, in grams (g).

It is calculated to three decimal places, and rounded off to two decimal places according to GB/T 8170.

## 11.2 Residual sulphur content of the sample

It is calculated according to formula (2):

$$S = \frac{(V_1 - V_0) \times c \times M}{m_s (1 - W)} \times 100 \quad \dots\dots\dots (2)$$

where

$S$  - the residual sulphur content of the sample, in milligrams per 100 grams (mg/100 g);

$V_1$  - the volume of iodine standard titration solution consumed by the sample, in milliliters (mL);

$V_0$  - the volume of iodine standard titration solution in the blank test, in milliliters (mL);

$c$  - the concentration of iodine standard titration solution, in moles per liter (mol/L);

$M$  - the molar mass of sulphur, in grams per mole (g/mol) ( $M = 32.06$ );

$m_s$  - the mass of the sample, in grams (g);

$W$  - the moisture content of the sample, %.

The result is calculated and expressed to one decimal place.

## 11.3 Allowable error

The moisture content test and the residual sulphur test shall have parallel tests, and the arithmetic mean of the two parallel test results shall be taken as the determination result. The relative error of the moisture content test shall not exceed 0.5 %; the relative error of the residual sulphur test shall not exceed 10 %.

## 12 Test report

a) sample name, source, batch number, etc.;

## Annex A

(informative)

### Preparation of iodine standard titration solution

#### A.1 Preparation of iodine standard titration solution [ $c(\frac{1}{2}I_2) = 0.05 \text{ mol/L}$ ]

WEIGH 6.5 g of iodine and 17.5 g of potassium iodide; DISSOLVE in 100 mL of water; DILUTE to 1000 mL; SHAKE well; STORE in a brown bottle.

#### A.2 Calibration of iodine standard titration solution [ $c(\frac{1}{2}I_2) = 0.05 \text{ mol/L}$ ]

MEASURE 35.00 mL ~ 40.00 mL of the prepared iodine solution; PLACE in an iodine flask; ADD 150 mL of water (15 °C ~ 20 °C); TITRATE with sodium thiosulfate standard titration solution [ $c(\text{Na}_2\text{S}_2\text{O}_3) = 0.05 \text{ mol/L}$ ]; ADD 2 mL of starch indicator (10 g/L) near the end point; continue to TITRATE until the blue color of the solution disappears.

At the same time, carry out a blank test of iodine consumed by water: TAKE 250 mL of water (15 °C ~ 20 °C); ADD 0.05 mL ~ 0.20 mL of the prepared iodine solution and 2 mL of starch indicator (10 g/L); TITRATE with sodium thiosulfate standard titration solution [ $c(\text{Na}_2\text{S}_2\text{O}_3) = 0.05 \text{ mol/L}$ ] until the blue color of the solution disappears.

The concentration of iodine standard titration solution [ $c(\frac{1}{2}I_2)$ ] is calculated according to formula (A.1):

$$c\left(\frac{1}{2}I_2\right) = \frac{(V_1 - V_2)c_1}{V_3 - V_4} \dots\dots\dots (A.1)$$

where

$c(\frac{1}{2}I_2)$  - the concentration of iodine standard titration solution, in moles per liter (mol/L);

$V_1$  - the volume of sodium thiosulfate standard titration solution, in milliliters (mL);

$V_2$  - the volume of sodium thiosulfate standard titration solution for the blank test, in milliliters (mL);

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