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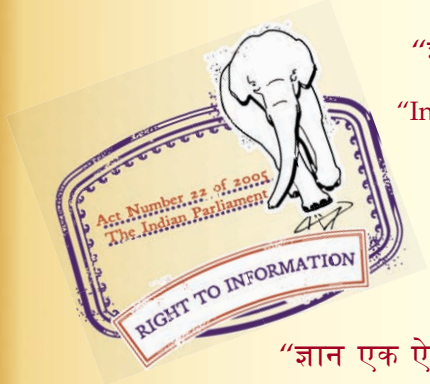

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METHOD FOR DETERMINATION OF SULPHATE CONTENT IN TEXTILE MATERIALS

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MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
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METHOD FOR DETERMINATION OF SULPHATE CONTENT IN TEXTILE MATERIALS

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METHOD FOR DETERMINATION OF SULPHATE CONTENT IN TEXTILE MATERIALS

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 31 July 1967, after the draft finalized by the Textile Chemistry Sectional Committee had been approved by the Textile Division Council.

0.2 In textile industry, textile materials undergo various treatments in course of which extraneous matter of various types, such as, sizing or finishing material, and water-soluble salts (chlorides and sulphates) is gathered by or added to the textile materials. Such water-soluble substances, if present, in more than certain quantities may have deleterious effect on the fibrous material or on other materials with which they are associated in use and may, therefore, it affect their performance in service. It is hoped that this standard will be useful for determining the sulphate content in aqueous extract of textile materials.

0.3 The gravimetric and volumetric methods for estimating the sulphate content in textile materials are prescribed in this standard.

0.4 In reporting the result of a test made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

1. SCOPE

1.1 This standard prescribes the methods for determination of water-soluble sulphate present in textile materials, and the procedure for extracting the textile materials with water.

2. PRINCIPLE

2.1 The aqueous extract of textile material is prepared, the sulphate content is determined, either gravimetrically or volumetrically and expressed as the percentage of the weight of the conditioned material.

3. SAMPLING

3.1 Sampling for Fibre and Yarn

3.1.1 *Lot (Fibre or Yarn)* — The quantity of fibre or yarn from the same source shall constitute a lot. If the lot contains more than 200 kg. of fibre or yarn, it shall be divided in sub-lots each weighing 200 kg or less.

*Rules for rounding off numerical values (*revised*).

3.1.2 From a sub-lot 15 increments each approximately weighing 10 g shall be taken from different parts so that a representative sample is obtained. All the increments thus collected shall be thoroughly mixed. This shall constitute the **test sample.**

3.2 Sampling for Fabrics

3.2.1 *Lot* (*Fabric*) — The quantity of fabric manufactured under relatively uniform conditions shall constitute a lot.

3.2.2 The number of pieces to be selected from a lot shall be as given below. The pieces thus selected shall constitute the **gross sample.**

Lot Size	*Sample Size*
Up to 100	3
101 ,, 300	4
301 ,, 500	5
501 and above	7

3.2.3 From each piece in the gross sample about 25 g of fabric shall be taken out from at least two different parts. The parts shall then be cut into further smaller pieces and thoroughly mixed. The pieces thus collected shall constitute the **test sample.**

4. TEST SPECIMENS

4.1 From the test sample draw at least two test specimens each weighing about 10 g. Cut the test specimens into small pieces. If the sample under analysis is loose fibre, take about 5 g of the test specimen.

5. CONDITIONING OF TEST SPECIMENS

5.1 Prior to test, the test specimens shall be conditioned for 24 hours to moisture equilibrium in a standard atmosphere at 65 $\pm$ 2 percent relative humidity and 27° $\pm$ 2°C (*see also* IS : 196-1966*).

6. APPARATUS

6.1 Flat-Bottom Flask — of a suitable capacity with a glass stopper.

6.2 Water-Cooled Condensers

6.3 Gooch Crucible — with asbestos pad.

*Atmospheric conditions for testing (*revised*).

7. QUALITY OF REAGENTS

7.1 Unless specified otherwise pure chemicals shall be employed in tests and distilled water (*see* IS : 1070-1960*) shall be used where the use of water as reagent is intended.

NOTE — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the test results.

8. PREPARATION OF AQUEOUS EXTRACT

8.1 Condition the test specimens to moisture equilibrium in the standard atmosphere and weigh accurately each test specimen.

8.2 Put a test specimen in the flask and add sufficient amount of water into it to make liquor to material ratio of 20 : 1 (*see* Note). Connect the flask to the condenser and bring rapidly to the boil and continue to boil the liquor gently for 60 minutes. Disconnect and remove the flask while the liquor is still boiling and close it immediately with the glass stopper fitted with the stopcock. Rapidly cool the flask to room temperature (27°C). Do not remove or open the tap until ready for filtration.

NOTE — If the test specimen is wool in any form, felt or loose fibre masses of any composition, the liquor to material ratio should be 50 : 1.

8.3 Similarly prepare separate extracts for each of the remaining test specimens.

9. GRAVIMETRIC METHOD

9.1 Reagents

9.1.1 *Barium Chloride Solution* — 2 percent (*w/v*).

9.1.2 *Hydrochloric Acid* — concentrated.

9.2 Procedure

9.2.1 Take a measured portion of extract. Filter through a suitable filter paper (*see* Note) and wash the filter paper with distilled water. Add concentrated hydrochloric acid drop by drop to the combined filtrate and washings until the solution is just acidic to litmus, then add 1 ml of acid per 100 ml of solution. Boil the solution for 5 minutes and leave it to cool overnight. Filter off any precipitate on a filter paper-pulp pad. Wash with water and heat the combined filtrate and washings to boiling. To the boiling solution add drop by drop 10 ml of hot barium chloride solution. Boil for 30 minutes and leave to cool overnight. Transfer the

*Specification for water, distilled quality (*revised*) (**Since revised**).

precipitate quantitatively to an ignited tared Gooch crucible with asbestos pad and wash with cold water until the washings are free from chloride. Ignite the crucible and its contents gently at first and finally at 800° to 900°C to constant weight.

NOTE — Whatman No. 41 paper is suitable.

9.2.2 Carry out the blank determination.

9.2.3 Calculate the percentage of water-soluble sulphate by either of the following formulae:

a) For all materials in yarn and fabric form other than wool (*see* Note 1):

$$P = \frac{823 \times (a - b)}{v}$$

b) For wool in any textile form and for felts and loose fibre masses of any composition (*see* Note 2):

$$P = \frac{2\,058 \times (a - b)}{v}$$

where

P = percentage, by weight, of water-soluble sulphates as sulphate ion;

a = weight, in g, of the precipitate obtained in the test (*see* **9.2.1**);

b = weight, in g, of the precipitate obtained as in blank (*see* **9.2.2**); and

v = volume, in ml, of extract taken for the test.

NOTE 1 — 100 ml of extract are equivalent to 5·0 g of conditioned test specimen.

NOTE 2 — 100 ml of extract are equivalent to 2·0 g of conditioned test specimen.

9.2.4 Repeat the test with the extracts of the remaining test specimens and calculate the percentage of water-soluble sulphate in each test specimen.

9.2.5 Calculate the average of the values obtained as in **9.2.3** and **9.2.4.**

10. VOLUMETRIC METHOD

10.1 Reagents

10.1.1 *Benzidine Hydrochloride Solution* — prepared as follows:

Dissolve 5 g of benzidine hydrochloride in 40 ml of 1 N hydrochloric acid and dilute the solution to 250 ml with 50 percent aqueous ethanol · (*v*/*v*). Heat the solution to boil, cool, filter if necessary, and store in a dark glass stoppered bottle.

10.1.2 *Alcohol* — 95 percent (*v/v*).

10.1.3 *Standard Sodium Hydroxide Solution* — 0·02 N.

10.1.4 *Standard Sulphuric Acid* — 0·02 N.

10.1.5 *Phenol Red Indicator* — 0·25 percent (*w/v*) prepared in 25 percent ethanol (*v/v*).

10.2 Procedure

10.2.1 Take a measured portion of extract (*see* **8.2**) and concentrate it to 20 ml. Add to this 20 ml of alcohol followed by 20 ml of benzidine hydrochloride solution. Allow the solution to stand for 30 minutes. Filter the solution under low suction through a suitable filter paper (*see* Note 1). Wash the precipitate with 5 ml of alcohol and repeat the washing 4 times more. After the test washing transfer the precipitate and filter paper to 250-ml conical flask and add 25 ml of distilled water. Add few drops of phenol red indicator. Heat the solution to boiling and cool. Add a known volume of standard sodium hydroxide solution to the contents of the flask, shake thoroughly to dissolve all the precipitate, add more phenol red indicator as required and back titrate the excess of sodium hydroxide with standard sulphuric acid.

NOTE 1 — Whatman No. 42 paper is suitable.

NOTE 2 — The accuracy of the above method may be checked by determining the sulphur content in 2 ml of sodium sulphate solution of known strength. 2 ml of sodium sulphate solution is taken in a beaker and 8 ml of alcohol is added. To this 4 ml of benzidine hydrochloride is added. The solution is allowed to stand for 30 minutes. The precipitate is filtered and titrated against 0·02 N sodium hydroxide as above.

10.2.2 Calculate the percentage of water-soluble sulphates by either of the following formulae:

a) For all materials in yarn and fabric form, other than wool (*see* Note 1):

$$P = \frac{A \times B \times 4{\cdot}8}{V} \times 20$$

b) For wool in any textile form, and for felts and loose fibre masses of any composition (*see* Note 2):

$$P = \frac{A \times B \times 4{\cdot}8}{V} \times 50$$

where

P = percentage, by weight, of water-soluble sulphate as sulphate ion;

A = volume, in ml, of standard sodium hydroxide solution;

B = normality of sodium hydroxide solution; and

V = volume, in ml, of the extract.

NOTE 1 — 100 ml of extract are equivalent to 5·0 g of conditioned test specimen.

NOTE 2 — 100 ml of the extract are equivalent to 2·0 g of conditioned test specimen.

10.2.3 Repeat the test with the remaining test specimens and calculate the percentage of water-soluble sulphate in each test specimen.

10.2.4 Calculate the average of the values obtained as in **10.2.2** and **10.2.3**.

11. REPORT

11.1 Report the values obtained as in **9.2.5** or **10.2.4** as the percentage of sulphate content as sulphate ions in textile materials.

NOTE — If the percentage of water-soluble sulphate is to be expressed as sodium sulphate, multiply P by 1·48.

11.2 Report also the method used (whether gravimetric or volumetric).

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