भारतीय मानक

Indian Standard

IS 13627 : 2023

वस्त्र रंजक सामग्री — पानी में घुलनशील रंजकों की विलेयता का निर्धारण

(पहला पुनरीक्षण)

Textile Dyestuffs — Determination of Solubility of Water-Soluble Dyes

(First Revision)

ICS 59.040; 71.040.50

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December 2023 **Price Group 4**

Textile Speciality Chemicals and Dyestuffs Sectional Committee, TXD 07

FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Textile Speciality Chemicals and Dyestuffs Sectional Committee had been approved by the Textiles Division Council.

The term solubility is used in this standard in connection with the ability of a solution or dispersion of water-soluble dye to pass a given filter-medium without depositing a residue on it. The experimental set-up used serves as a model for practical dyeing conditions only and the method should not be used for determining solubility in the scientific, physico-chemical sense.

The trade of water-soluble dyes for the colouration of textiles, paper and leather has entered a truly international dimension. Where the product specification contains values for solubility, the present standard provides a method for its determination by the customer and the supplier.

This standard was first published in 1993. The standard has been revised in the light of experience gained since its publication and to incorporate the following major changes:

a) Title of the standard has been modified;

b) Grade and purity of chemicals used have been specified; and

c) Temperature for determination of solubility has been changed to, temperature at which dye is applied on substrate in place of 90 ºC.

The composition of the Committee responsible for the formulation of this standard is given in Annex A.

In reporting the results of a test or analysis 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 : 2022 ‘Rules for rounding off numerical values (*second revision*)’.

Indian Standard

TEXTILE DYESTUFFS — DETERMINATION OF SOLUBILITY OF WATER-SOLUBLE DYES

(First Revision)

1 SCOPE

This standard prescribes a method for the determination of the solubility of water-soluble dyes in the temperature range between 40 °C and 90 °C.

2 DEFINITIONS

2.1 Solubility

The term solubility and the values determined by this method are used as intrinsic values. No agreement is given to solubility in the physical sense.

2.2 Solubility Limit

Plotting filter residue and time needed to filter solutions/dispersions of a given dye at a given temperature as a function of the quantity per unit volume of dye solution normally exhibit quite a clear break. Its abscissa value is called the solubility limit.

3 SAFETY PRECAUTIONS

3.1 It is the user's responsibility to use safe and proper techniques in handling materials and equipment. Manufacturers shall be consulted for specific details such as safety data sheets and other recommendations.

3.2 Good laboratory practices have to be followed. Working with vacuum and chemicals (including dyes) requires safety measures such as wearing safety glasses, goggles or face shields, gloves, aprons and dust control.

4 APPARATUS, MATERIALS AND REAGENTS

4.1 Wide-mouthed Erlenmeyer flask, capacity 500 ml.

4.2 Thermostatically controlled heating bath with magnetic stirrer; stirrer rod size 400 mm long by 6 mm diameter, speed of stirrer 500 rev/min to 600 rev/min.

4.3 Thermostatic device with circulation pump to adjust temperature of Nutsche filter.

4.4 Heatable double-walled Nutsche filter of stainless steel or porcelain; inner diameter 70 mm, capacity at least 200 ml.

4.5 Suction bottle, capacity 1 liter to 2 liters.

4.6 Piston or membrane pump of sufficiently high suction capacity to create a full vacuum (at least 50 kPa).

4.7 Apparatus to adjust and maintain a given vacuum, preferably coupled with a manometer.

4.8 Stopwatch to measure flow-through time.

4.9 Circular paper filter, 70 mm in diameter (Whatman No. 2).

NOTES

1 Filter paper other than the size and number mentioned may be used if agreed to between the parties concerned.

2 Unless otherwise specified analytical reagent grade chemicals with 99.0 percent purity shall be employed in tests.

4.10 Solvent — Distilled or fully demineralized water; an amount of 200 ml is designated as normal. Additions to the solution are possible, but must be indicated together with the solubility values. Their influence on the volume must be taken into account with the water volume. However, no account is taken of changes in volume as a function of temperature, or those caused by the addition of solid dye.

5 PRINCIPLE

5.1 Several solutions of known concentrations of the dye to be tested, including the solubility limit, at temperature X °C, are prepared. The solutions are then filtered under suction at this temperature in a heatable Nutsche filter.

5.2 The solubility of dye is normally determined at at temperature at which it is applied on substrate. The temperature depends on class of dyestuff. In selecting the temperature, the manufacturers' recommendations should be followed. The temperature must be indicated (solubility at 90 °C, 60 °C, room temperature etc).

5.3 The solubility limit is determined by visual assessment of the filter residues and the measured flow-through time of the filtrate. Solubility is given in terms of the following limits:

a) 1 g/l to 10 g/l in intervals of 1 g;
b) 10 g/l to 50 g/l in intervals of 5 g;
c) 50 g/l to 100 g/l in intervals of 10 g; and
d) > 100 g/l in intervals of 20 g.

6 PREPARATION OF SOLUTIONS

6.1 For determining solubility at 90 °C, make paste of a known amount of the test dye and introduce into the wide-mouthed Erlenmeyer flask with a portion of the 200 ml distilled or fully demineralised water at about 60 °C, but not in excess of the dissolving temperature. When the dye is completely wetted out, fill the flask with the rest of the water,

6.1.1 Place the solution into the heating bath maintained at about 90 °C, switch on the magnetic stirrer and maintain heating for about 5 minutes at 90 °C ± 2 °C. Continue stirring at this temperature for a further 5 minutes (total stirring time of 10 minutes).

6.1.2 To determine the solubility of a dye at 90 °C, filter the solution immediately after stirring.

6.2 For determining solubility at temperatures below 90 °C, make paste of a known graduated amount of the test dye and introduce into a wide mouthed Erlenmeyer flask with a portion of the 200 ml distilled or fully demineralised water at the desired dissolving temperature until the dye is completely wetted out. Then fill the flask with the rest of the water.

6.2.1 Place the solution into the heating bath maintained at the desired dissolving temperature, stir the solution for 10 minutes and then filter. Adjust the temperature of the heating bath so that the desired dissolving temperature is attained.

7 FILTERING THE SOLUTIONS

7.1 Pre-heat the Nutsche filter to the test temperature by pumping through the heating medium from the thermostatic device, and maintain at this temperature throughout the entire filtration operation.

7.2 Immediately before filtering, wet out two filter papers in the nutsche filter in a double layer with at least 50 ml water at the test temperature.

7.3 Adjust the vacuum before filtration so that 200 ml water at the test temperature passes through the filters in a flow-through time of 5 seconds to 8 seconds (3 kPa to 4 kPa is approximately equivalent to a pressure of 300 mm H_2O to 400 mm H_2O).

7.4 Filter the dye solution at the desired temperature and measure the flow through time with a stop-watch.

7.5 If the solution does not filter within 2 minutes at a stabilized vacuum, filter it for an additional maximum 2 minutes under full vacuum.

7.6 After the solution has flowed through, hydro extract the filters uniformly under full vacuum for 1 minute.

7.7 Allow the filters to dry completely at room temperature.

8 EVALUATION

8.1 Compare the filters of the various dye solutions of known concentrations visually. The solubility limit is exceeded when filter residues are seen. Residues that are difficult to see may possibly be detected by rubbing on the filter.

8.2 The flow-through time serves as a further evaluation criterion. A sudden sharp increase in the flow-through time when moving up the range of solution concentrations indicates that the solubility limit is exceeded.

8.3 Residues in the flask after pouring the liquor out into the Nutsche filter are also a clear indication that the concentration has exceeded the solubility limit.

9 REPORT

9.1 The test report shall include the following:

a) particulars of the dyestuff under test;
b) solubility of the dyestuff (g/l) and dissolving temperature;
c) additions (where applicable); and
d) deviations from this specification (for example, other filter qualities or amounts of solvent other than 200 ml) (where applicable).

10 EXPLANATORY COMMENTS

10.1 The test method described above has given good results over several years. However, it should be pointed out that test conditions which deviate from those specified above may lead to quite different results. In some cases, this may affect entire groups of dyes and in others only individual members of a range.

For example, the results may be influenced when:

a) *Another filter is used* — The Whatman filter papers 2 and 4 used in the USA, for example, are relatively dense. Normally when these filters are used only the flow through time is affected. However, dyes that do not form true solutions may not be filterable through these denser filters. On the other hand, the cheese cloth filters formerly used are considerably more permeable and lead to higher solubility values. The filter selected for the test represents a compromise with respect to permeability and should do full justice to the practical conditions;

b) *Other dissolving temperatures are used* — Many dyes dissolve just as well at temperatures appreciably below 90 °C or the given test temperature. However, exceptions may occur. On the other hand, there are dyes whose solubility depends heavily on the dissolving temperature, that is, there are dyes which readily dissolve at 90 °C but are difficult to dissolve at 85 °C. However, in most cases these dyes are well known in commercial practice and must be tested with special care;

c) *Other stirring times are used* — Again; many dyes are impervious to modifications of this type, while others go into solution at a lower rate;

d) When water of different hardness or an electrolyte addition is used; and

e) *If results in the concentration series are contradictory* — Repeat with independently prepared solutions on both sides of the supposed concentration of the solubility limit.

ANNEX A

(*Foreword*)

COMMITTEE COMPOSITION

Textile Speciality Chemicals and Dyestuffs Sectional Committee, TXD 07

Organization	*Representative(s)*
Department for Jute and Fibre Technology Institute of Jute Technology University of Calcutta, Kolkata	PROF A. K. SAMANTA (***Chairperson***)
Ahmedabad Textile Industry's Research Association, Ahmedabad	SHRIMATI DEEPALI PLAWAT SHRIMATI FAHIMUNNISA KHATIB (*Alternate*)
Ama Herbals Laboratories Pvt Ltd, Lucknow	SHRI Y. A. SHAH
Archroma India Pvt Limited, Mumbai	SHRI RAJESH RAMAMURTHY SHRIMATI PRACHI NARVEKAR (*Alternate*)
Atul Limited (Colors Business), Valsad	SHRI RAJARAM JAMDADE SHRI ARINDAM CHAKRABORTY (*Alternate*)
Bio Dyes India Pvt Ltd, Goa	DR BOSCO HENRIQUES
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Central Institute for Research on Cotton Technology, Mumbai	DR SUJATA SAXENA DR A. S. M. RAJA (*Alternate*)
Department for Jute and Fibre Technology Institute of Jute Technology University of Calcutta, Kolkata	DR D. DAS
Global Organic Textile Standard, (GOTS), Thane	SHRI RAHUL BHAJEKAR MISS PRACHI GUPTA (*Alternate*)
Indian Jute Industries Research Association, Kolkata	DR S. K. CHAKRABARTI SHRI SANDIP BASU (*Alternate*)
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Office of the Textile Commissioner, Mumbai	SHRI GAURAV GUPTA SHRI SANJAY CHARAK (*Alternate)*
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Textiles Committee, Mumbai	SHRI KARTIKEYA DHANDA SHRIMATI SHILPI CHAUHAN (*Alternate*)
The Arvind Mills Limited, Ahmedabad	SHRI RAJARSHI GHOSH SHRI UMASANKAR MAHAPATRA (*Alternate*)
The Bombay Textile Research Association, Mumbai	DR PADMA S. VANKAR SHRI M. P. SATHIANARAYANAN (*Alternate*)

Organization	*Representative(s)*
The South India Textile Research Association, Coimbatore	DR PRAKASH VASUDEVAN SHRI S. SIVAKUMAR (*Alternate*)
The Synthetic and Art Silk Mills Research Association, Mumbai	SHRIMATI (DR) MANISHA MATHUR SHRIMATI ASHWINI SUDAM (*Alternate*)
U P Textile Technology Institute, Kanpur	DR ARUN KUMAR PATRA DR SHUBHANKAR MAITY (*Alternate*)
Wool Research Association, Thane	SHRIMATI (DR) MRINAL CHOUDHARI
BIS Directorate General	SHRI J. K. GUPTA, SCIENTIST 'E'/DIRECTOR AND HEAD (TEXTILES) [REPRESENTING DIRECTOR GENERAL (*Ex-officio*)]

Member Secretary
SHRI HIMANSHU SHUKLA
SCIENTIST 'B'/JOINT DIRECTOR
(TEXTILES), BIS

Bureau of Indian Standards

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Review of Indian Standards

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