भारतीय मानक

Indian Standard

IS 13949 : 2024

वस्त्र प्रसंस्करण सहायक सामग्री — डाइमेथाइलोल डाइहाइड्रॉक्सी एथिलीन यूरिया (डीएमडीएचईयू) अभिकारक — विशिष्टि

(पहला पुनरीक्षण)

Textile Auxiliaries — Dimethylol Dihydroxy Ethylene Urea (DMDHEU) Reactant — Specification

(First Revision)

ICS 59.040

भारतीय मानक ब्यूरो
BUREAU OF INDIAN STANDARDS
मानक भवन, 9 बहादुर शाह ज़फर मार्ग, नई दिल्ली - 110002
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI - 110002
www.bis.gov.in www.standardsbis.in

January 2024 **Price Group 6**

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.

Various advantages and disadvantages associated with the use of dimethylol dihydroxy ethylene urea in textile industry are given in Annex A.

This standard was first published in 1994. The standard has been revised in the light of experience gained since its publication and to incorporate the following major changes:

a) Requirement for gel time has been specified;
b) Grade and purity of chemicals used have been specified;
c) BIS certification marking clause has been modified; and
d) References to Indian Standard have been updated.

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

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 2022 ‘Rules for rounding off numerical values (*second revision*)’. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

TEXTILE AUXILIARIES — DIMETHYLOL DIHYDROXY ETHYLENE UREA (DMDHEU) REACTANT — SPECIFICATION

(First Revision)

1 SCOPE

This standard specifies the requirements and methods of test for dimethylol dihydroxy ethylene urea (DMDHEU) reactant solution used in textile industry. It also specifies details of measurement of crease recovery angle to check its effectiveness as cross-linking agent.

2 REFERENCES

The standards given below contain provisions which through reference in this text, constitute provision of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent edition of these standards:

IS No.	*Title*
IS 188 : 2004	Textiles — Cotton poplin — Specification (*third revision*)
IS 1070 : 2023	Reagent grade water — Specification (*fourth revision*)
IS 4681: 1981	Method for determination of recovery from creasing of textile fabrics by measuring the angle of recovery (*first revision*)
IS 13360 (Part 10/ Sec 4) : 2001	Plastics — Methods of testing: Part 10 Thermosetting properties, Section 4 Determination of gel time and peak exothermic temperature of reacting thermosetting resins
IS 1390 : 2022/ ISO 3071 : 2020	Textiles — Determination of *p*H of aqueous extract (*third revision*)

3 REQUIREMENTS

3.1 General Requirements

The reactant shall be clear transparent solution free from turbidity and shall be of white to pale yellow colour.

3.2 Specific Requirements

Dimethylol dihydroxy ethylene urea (DMDHEU) shall meet the requirements given in Table 1.

4 CREASE RECOVERY ANGLE

The crease recovery angle of bleached, mercerized white cotton poplin conforming to IS 188 treated with the reactant solution by the method prescribed in Annex G shall be minimum 240° when tested by the method given in IS 4681.

5 PACKING

The material shall be suitably packed in waterproof packages as detailed in the contract or order.

6 MARKING

6.1 Each package shall be legibly and indelibly marked with the following:

a) Net mass of the resin solution;
b) Name of the material namely dimethylol dihydroxy ethylene urea
c) Solid content of the material;
d) Indication of source of the manufacture;
e) Month and year of manufacture; and
f) Any other information/instruction provided by the manufacture/required under law.

6.2 BIS Certification Marking

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act*, 2016 and the Rules and Regulations framed thereunder, and the product(s) may be marked with the Standard Mark.

7 SAMPLING

7.1 Lot

The quantity of dimethylol dihydroxy ethylene urea solution of one definite composition delivered to a buyer against one dispatch note shall constitute a lot.

7.2 Unless otherwise agreed to between the buyer and the seller, the number of packages to be selected

from each lot at random shall be as given below:

Sl No.	*Lot Size*	*Sample Size*
(1)	(2)	(3)
i)	up to 15	3
ii)	16 to 25	4
iii)	26 to 50	5
iv)	51 to 100	7
v)	101 to 150	10

7.3 Draw small quantities of the resin solution by suitable sampling instrument from at least three different parts of each container and mix them thoroughly so as to get a composite sample of desired mass.

7.4 The reactant solution shall be declared, as conforming to the requirements of this standard if all the composite specimens prepared in **7.3** meet the relevant requirements as specified in **3** and **4**.

Table 1 Dimethylol Dihydroxy Ethylene Urea — Specific Requirements

(*Clause* 3.2)

Sl No.	**Characteristic**	**Requirement**	**Method of Test, Ref to**
(1)	(2)	(3)	(4)
i)	Total solid content, percent, *Min* (*m*/*m*)	45	Annex B
ii)	*p*H of solution	7 to 8 without catalyst and 4 to 5 with catalyst	IS 1390
iii)	Free formaldehyde content, percent, *Max*	1.0	Annex C
iv)	Total formaldehyde content, percent	33.7 ± 2	Annex D
v)	Nitrogen content, percent	15.7 ± 2	Annex E
vi)	Nitrogen to formaldehyde molar ratio	1.0 to 1.3	Annex F
vii)	Active matter content, *Min*	30 percent with catalyst and 40 percent without catalyst	
viii)	Gel time, s	25 to 30 at 100 ºC	IS 13360 (Part 10/Sec 4)

ANNEX A

(*Foreword*)

USES, ADVANTAGES AND DISADVANTAGES OF DIMETHYLOL DIHYDROXY ETHYLENE UREA REACTANT

A-1 Dimethylol dihydroxy ethylene urea (DMDHEU) is extensively used in textile industry for wash and wear and for durable press finishing. The finish obtained with DMDHEU exhibits excellent crease recovery and is fast to repeated home launderings. However, due to high chlorine damage to finished fabric (mainly due to impurities present in DMDHEU), this product is not recommended for finishing of white goods. DMDHEU does not affect the light fastness of dyes in general and those of reactive dyes in particular. It is an excellent reactant for the deferred cure process. It gives low formaldehyde odour and thus it is convenient to stitch the garments from the sensitised cloth.

ANNEX B

(*Clause* 3.2.1 *and Table* 1)

METHOD FOR DETERMINATION OF TOTAL SOLID CONTENT OF THE RESIN SOLUTION

B-1 APPARATUS

B-1.1 A centrifuge with centrifuge bottles/tubes capable of rotating at 2 500 rev/min

B-1.2 A Weighing Balance — correct up to 1 mg

B-2 PROCEDURE

B-2.1 Weigh about 5 g of resin solution exactly in a tared centrifuge bottle/tube. Add to it about 50 ml (or more depending upon the capacity of the centrifuge bottle) of acetone and centrifuge at 2 500 rev/min for about 10 minutes when the solid matter separates out as a viscous oil.

B-2.2 Decant off the upper layer and add about 50 ml of fresh acetone to the oily residue in the centrifuge bottle and centrifuge at 2 500 rev/min for about 10 minutes. Repeat the process (3 times to 5 times) until the resin, free of water, separates out as solid or sticky mass.

B-2.3 After removal of acetone, weigh the centrifuge bottle containing the solid resin.

B-2.4 Calculate the total percent solid content (*S*) of the resin by mass of the resin solution by the formula:

$$S, \text{ percent} = \frac{\text{Mass of solid resin}}{\text{Mass of resin solution taken}} \times 100$$

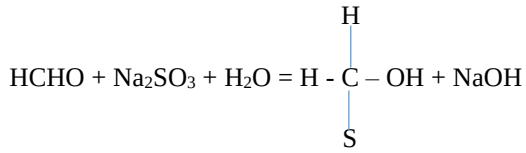
ANNEX C

(*Clause* 3.2 *and Table* 1)

METHOD FOR DETERMINATION OF FREE FORMALDEHYDE CONTENT OF THE RESIN SOLUTION

C-1 PRINCIPLE

A sample of the resin solution is reacted with sodium sulphite when free formaldehyde present in the solution forms a formaldehyde bisulphite adduct and sodium hydroxide is liberated. The mixture is then titrated with standard hydrochloric acid solution.



Formaldehyde adduct of Na_2SO_3

C-2 APPARATUS

C-2.1 A Conical Flask — 500 ml capacity

C-2.2 A Weighing Balance — with an accuracy up to 1 mg

C-2.3 A Pipette — 25 ml capacity

C-2.4 A burette

C-3 REAGENTS

C-3.1 Quality of Reagents

Unless otherwise specified analytical reagent grade chemicals with 99.0 percent purity shall be employed in tests and distilled water (*see* IS 1070) shall be used where the use of water as reagent is intended.

C-3.1.1 *Sodium Sulphite Solution* — 1 M prepared by dissolving 12.6 g of anhydrous sodium sulphite m 100 ml of water.

C-3.1.2 *Hydrochloric Acid Solution* — 1 N

C-3.1.3 *Thymolphthalein Indicator Solution* — Prepared by dissolving 0. 1 g indicator in 100 ml of 80 percent rectified spirit.

C-4 PROCEDURE

C-4.1 Take 20 ml of 1 M sodium sulphite solution in a conical flask previously placed in an ice bath. Add to it 3 drops of thymolphthalein indicator.

C-4.2 Neutralize the solution obtained in **C-4.1** carefully with 1 N hydrochloric acid until the colour disappears.

C-4.3 Weigh accurately 10 g of resin solution in a beaker and dilute to 100 ml after neutralizing with thymolphthalein.

C-4.4 Add the solution obtained in **C-4.3** to the solution obtained in **C-4.2**.

C-4.5 Titrate the solution obtained in **C-4.4** with 1 N hydrochloric acid to complete discolouration.

C-4.6 Calculate the free formaldehyde content (F) percent by mass of the resin solution by the formula:

$$F = 3.003 \times N \times V$$

where

N = normality of hydrochloric acid used for titration; and

V = volume in ml of I N hydrochloric acid required for titration.

ANNEX D

(*Clause* 3.2 *and Table* 1)

METHOD FOR DETERMINATION OF TOTAL FORMALDEHYDE CONTENT OF THE RESIN SOLUTION

D-1 REAGENTS

D-1.1 Sodium Hydroxide Solution — 2 N

D-1.2 Iodine Solution — 0.1 N

D-1.3 Starch solution

D-1.4 Sulphuric Acid Solution — 2 N

D-1.5 Sodium Thiosulphate Solution — 0.1 N

D-2 PROCEDURE

D-2.1 Weigh about 0.2 g of resin solution into a 250 ml Erlenmeyer flask and add to it 10 ml of 2 N sodium hydroxide solution and 50 ml of 0.1 N iodine solution. Let the mixture stand for 1 hour to $1\frac{1}{2}$ hours at 20 °C ± 2 °C in the closed flask.

D-2.2 Add to this solution 20 ml of 2 N Sulphuric acid and titrate with 0.1 N sodium thiosulphate solution, with stirring to the colourless end point.

NOTE — The addition of small amount solution makes the end point easier to detect,

D-2.3 Calculate the total formaldehyde (X) of the resin solution by the formula:

$$X = \frac{0.15 \times (V_1 - V_2)}{m}$$

where

V_1 = volume in ml of 0.1 N iodine taken;

V_2 = volume in ml of 0.1 N thiosulphate required; and

m = mass of the resin solution taken in g.

ANNEX E

(*Clause* 3.2 *and Table* 1)

METHOD FOR DETERMINATION OF NITROGEN CONTENT

E-1 APPARATUS

E-1.1 A weighing bottle

E-1.2 Kjeldahl Flask — with a loosely fitting pear shaped hollow glass stopper

E-1.3 A Water Bath — capable of working at a temperature of 100 °C ± 2 °C

E-1.4 A Volumetric Flask — 100 ml capacity

E-1.5 A Beaker — 250 ml capacity

E-1.6 Distillation assembly

E-2 REAGENTS

E-2.1 Concentrated sulphuric acid

E-2.2 Sucrose

E-2.3 Selenium catalyst

E-2.4 Potassium sulphate

E-2.5 Copper sulphate

E-2.6 A few pieces of pumice stone

E-2.7 Sulphuric Acid Solution — 0.05 N

E-2.8 Mixed Indicator Solution — obtained by dissolving 0.2 g methyl red and 0.1 g methylene blue in 100 ml of rectified spirit

E-2.9 Sodium Hydroxide Solution — 0.05 N

E-3 PROCEDURE

E-3.1 Weigh accurately about 0.5 g of the sample in a weighing bottle and transfer it to Kjeldahl flask. Add to it 25 ml of concentrated sulphuric acid and 0.5 g of sucrose.

E-3.2 Keep the Kjeldahl flask in a boiling water bath for one hour and then add 0.1 g selenium, 18 g potassium sulphate and 0.3 g of copper sulphate. Keep the flask in an inclined position and close with a loosely fitting pear shaped hollow glass stopper to prevent loss of sulphuric acid or entry of dust. Heat the mixture gently in a fume cupboard until the initial frothing has ceased. Heat the liquid to boiling point and continue boiling until the solution becomes clear. Boil the liquid for a further period of 2 hours and then cool the contents of the flask.

E-3.3 Transfer the contents of Kjeldahl flask to a 100 ml volumetric flask and make the solution up to the mark. Take 10 ml of this solution in a volumetric flask and add to it a few pieces of pumice stone.

E-3.4 Take 50 ml of sulphuric acid solution (0.05 N) and 3 drops of mixed indicator [*see* **E-2** (h)] in a beaker. Set up a distillation assembly. Add excess of sodium hydroxide solution (0.05 N) through the separating funnel and mix the contents of the flask by mild shaking. Distill about one-third of the total volume of the solution in the flask. Wash the dip tube with water, 1 collecting the washing in the beaker.

E-3.5 Titrate the excess of sulphuric acid in the beaker with sodium hydroxide solution (0.05 N).

E-3.6 Carry out a blank titration without the sample as described from **E-3.1** to **E-3.4**.

E-3.7 Calculate the nitrogen, percent by mass (N) by the following formula:

$$N = \frac{1.4\ (V_2 - V_1) \times 0.05}{m} \times \frac{100}{10}$$

where

V_1 = volume in ml of sodium hydroxide solution (0.05 N) used to neutralize excess of acid in the determination with the sample;

V_2 = volume of sodium hydroxide solution used in blank titration; and

m = mass in g of the sample taken for test.

ANNEX F

(*Clause* 3.2 *and Table* 1)

DETERMINATION OF NITROGEN TO FORMALDEHYDE MOLAR RATIO

F-1 Calculate the nitrogen to formaldehyde molar ratio (*N : F*) of the resin solution by the following formula:

$$\text{Molar Ratio} = \frac{N/14}{X/30}$$

where

N = percent nitrogen content of the resin solution and 14 represents molecular weight of nitrogen; and

X = percent total formaldehyde content of the resin solution and 30 represents molecular weight of formaldehyde.

ANNEX G

(*Clause* 4)

METHOD FOR TREATMENT FOR COTTON FABRIC WITH UREA FORMALDEHYDE

G-1 APPLICATION OF RESIN

G-1.1 The resin is applied by pad-dry-cure method and following recipe is used:

a) Resin = 100 g/l;

b) Catalyst (magnesium chloride) = 15 g/l;

c) Wet pick up = 80 percent with 2 dips and 2 nips; and

d) Fabric is then dried at 80 °C to 100 °C for 5 minutes and cured at 150 °C for 5 minutes.

ANNEX H

(*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
Central Coir Research Institute, Alappuzha	DR SHANMUGASUNDARAM O. L. DR S. RADHAKRISHNAN (*Alternate*)
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*)
Northern India Textile Research Association, Ghaziabad	DR M. S. PARMAR DR NIDHI SISODIA (*Alternate*)
Office of the Textile Commissioner, Mumbai	SHRI GAURAV GUPTA SHRI SANJAY CHARAK (*Alternate)*
SGS India Pvt Ltd, Mumbai	SHRI KARTHIKEYAN K. SHRI GAURAV SARASWAT (*Alternate*)
Shree Pushkar Chemicals & Fertilizers Ltd, Mumbai	DR N. N. MAHAPATRA
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

BIS is a statutory institution established under the *Bureau of Indian Standards Act*, 2016 to promote harmonious development of the activities of standardization, marking and quality certification of goods and attending to connected matters in the country.

Copyright

Review of Indian Standards

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the website-www.bis.gov.in or www.standardsbis.in.

This Indian Standard has been developed from Doc No.: TXD 07 (21071).

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110002
Telephones: 2323 0131, 2323 3375, 2323 9402 *Website*: www.bis.gov.in

Regional Offices:		*Telephones*
Central	: 601/A, Konnectus Tower -1, 6th Floor, DMRC Building, Bhavbhuti Marg, New Delhi 110002	2323 7617
Eastern	: 8th Floor, Plot No 7/7 & 7/8, CP Block, Sector V, Salt Lake, Kolkata, West Bengal 700091	2367 0012 2320 9474
Northern	: Plot No. 4-A, Sector 27-B, Madhya Marg, Chandigarh 160019	265 9930
Southern	: C.I.T. Campus, IV Cross Road, Taramani, Chennai 600113	2254 1442 2254 1216
Western	: Plot No. E-9, Road No.-8, MIDC, Andheri (East), Mumbai 400093	2821 8093

Branches : AHMEDABAD. BENGALURU. BHOPAL. BHUBANESHWAR. CHANDIGARH. CHENNAI. COIMBATORE. DEHRADUN. DELHI. FARIDABAD. GHAZIABAD. GUWAHATI. HIMACHAL PRADESH. HUBLI. HYDERABAD. JAIPUR. JAMMU & KASHMIR. JAMSHEDPUR. KOCHI. KOLKATA. LUCKNOW. MADURAI. MUMBAI. NAGPUR. NOIDA. PANIPAT. PATNA. PUNE. RAIPUR. RAJKOT. SURAT. VISAKHAPATNAM.

Published by BIS, New Delhi