भारतीय मानक **IS 18340 : 2023**

Indian Standard

विनाइल सल्फ़ोन — विशिष्टि

Vinyl Sulphone — Specification

ICS 71.080.99

भारतीय मानक ब्यूरो
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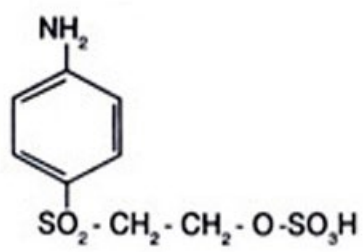
August 2023 **Price Group 6**

Dye Intermediates Sectional Committee, PCD 26

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Dye Intermediates Sectional Committee has been approved by the Petroleum, Coal and Related Products Division Council.

Vinyl Sulphone ($C_8H_{11}O_6NS_2$) is an important intermediate used for making azo dyes. It is described as 4-aminophenyl-beta-hydroxyethyl sulphone sulphate ester. It is represented by the following structural formula:



VINYL SULPHONE
Molecular Mass: 281.31
CAS Number: 2494-89-5

This standard stipulates the requirements and methods of test for vinyl sulphone (VS).

The bags in which the material is stored or transported may also be labelled with pictograms, signal word, hazard statement, and precautionary statement as given in Annex F, which are derived from GHS guidelines. At the time of publication, the latest edition of GHS guidelines was referred and are subject to revision and parties to agreement, are encouraged to investigate the possibility of applying the most recent labels as indicated.

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

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

VINYL SULPHONE — SPECIFICATION

1 SCOPE

This standard prescribes the requirements and the methods of sampling and tests for vinyl sulphone.

2 REFERENCES

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

IS No.	*Title*
IS 1070 : 2023	Reagent grade water — Specification (*fourth revision*)
IS 5299 : 2001	Methods of sampling and tests for dye intermediates (*first revision*)
IS 14887 : 2014	Textiles — High Density Polyethylene(HDPE)/Polypropylene (PP) woven sacks for packing 50 kg food grains (*first revision*)

3 REQUIREMENTS

3.1 Description

The material shall be in the form of off-white powder.

3.2 The material shall also comply with the requirements given in Table 1, when tested according to the methods prescribed in col (4) of Table 1.

4 PACKING AND MARKING

4.1 Packing

The material shall be packed in HDPE/PP woven sacks (*see* IS 14887). Each container shall be securely closed.

4.2 Marking

4.2.1 Each bag shall bear legibly and indelibly the following information:

a) Name of the material;
b) Name of the manufacturer/supplier, complete address and his recognized trade-mark, if any;
c) Gross, net and tare mass;
d) Lot or batch number;
e) Month and year of manufacturing;
f) Shelf life of the material; and
g) Any other statutory requirements.

4.2.2 All bags in which the material is stored or transported shall also be prominently and clearly marked with '**DANGER**' in red letters.

4.2.3 *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 products may be marked with the Standard Mark.

5 SAMPLING

5.1 The method of drawing representative samples of the material shall be as prescribed in **4** of IS 5299.

5.2 Number of Tests

5.2.1 Test for assay by nitrite value shall be conducted on the individual samples.

5.2.2 Tests for the determination of remaining characteristics, namely, assay by HPLC, hydrolysis value, matter insoluble in sodium carbonate solution and 4-chloroaniline content by HPLC shall be conducted on the composite sample.

5.3 Criteria for Conformity

5.3.1 *For Individual Samples*

The lot shall be declared as conforming to the requirement assay by nitrite value if each of the individual test results satisfies the relevant requirements given in Table 1.

5.3.2 *For Composite Sample*

For declaring the conformity of the lot to the requirements of all other characteristics tested on the composite sample (*see* **5.2.2**), the test results for

each of the characteristics shall satisfy the relevant requirements given in Table 1.

6 TESTS

6.1 PREPARED SAMPLE

Dry the material at 105 °C ± 1 °C to constant mass. Grind and mix well. Transfer the material to a wide-mouthed bottle and stopper it. Do not expose the sample to an atmosphere containing acidic or alkaline fumes. Use this prepared sample for tests.

6.2 Tests shall be carried out on the prepared sample (*see* **6.1**) according to the methods prescribed in col (4) of Table 1.

6.3 Quality of Reagents

Unless specified otherwise, 'pure chemicals' and distilled water (*see* IS 1070) shall be employed in tests.

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

Table 1 Requirements for Vinyl Sulphone

(*Clauses* 3.2, 5.3.1, 5.3.2 *and* 6.2)

Sl No.	Characteristic	Requirement	Method of Test, Ref to Annex
(1)	(2)	(3)	(4)
i)	Assay (by nitrite value), percent by mass (on dry basis), *Min*	96.0	A
	or		
	Assay (by HPLC[1)]), percent area (on dry basis), *Min*	95.0	B
ii)	Hydrolysis value, percent by mass (on 100 percent basis), *Min*	93.0	C
iii)	Matter insoluble in sodium carbonate solution, percent by mass, *Max*	0.6	D
iv)	4-Chloroaniline content, percent by mass (on 100 percent basis), *Max*	0.05	E

[1)]In case of disputes, determination of assay by HPLC shall be the referee method.

ANNEX A

[*Table* 1*, Sl No.* (i)]

DETERMINATION OF VINYL SULPHONE CONTENT (ASSAY) BY NITRITE VALUE

A-1 REAGENTS

A-1.1 Concentrated Hydrochloric Acid

A-1.2 Potassium Bromide

A-1.3 Standard Sodium Nitrite Solution, 0.1 N

A-1.4 Potassium Starch Iodide Indicator Papers

A-1.5 Ice

A-2 PROCEDURE

Weight 10 g to 14 g dry powder in 250 ml glass beaker. Add distilled water approximately 150 ml and stir with glass rod to make a smooth slurry. Add 20 percent soda ash solution (approximately 7 ml to 10 ml) to dissolve the powder to make clear solution. Transfer the solution to 500 ml volumetric flask along with little distilled water wash. Make volume exactly 500 ml by adding distilled water. Stir the contents well with magnetic stirrer. Take 50 ml of the solution by using pipette into 1 000 ml beaker. Add 200 ml to 250 ml distilled water. Add ice cubes to make the temperature around 10 °C. Weigh and add 1 g potassium bromide into the cold solution. Add Hydrochloric acid to make the *p*H acidic (*p*H around 2 to 2.5 on *p*H paper) approximately 25 ml is required. Take 0.1 N sodium nitrite in the burette. Titrate this solution against 0.1 N sodium nitrite solution with constant stirring by using magnetic stirrer. Check the end point to put the spot-on starch iodide paper, the end point shows faint blue ring on starch iodide paper. Check the sodium nitrite solution consumed by burette reading.

A-3 CALCULATION

Assay (by nitrite value), percent by mass =

$$\frac{V_1 \times N_1 \times 281.31}{M \times 10}$$

where

V_1 = volume, of standard sodium nitrite solution used in the titration in ml;

N_1 = Normality, of sodium nitrite solution, N; and

M = Mass, in g, of the material taken for the test.

ANNEX-B

[*Table* 1*, Sl No.* (i)]

DETERMINATION OF VINYL SULPHONE CONTENT (ASSAY) BY HIGH PERFORMANCE LIQUID CHROMATOGRAPHY

B-1 OUTLINE OF METHOD

High-performance liquid chromatography or high-pressure liquid chromatography (HPLC) is a chromatographic method that is used to separate a mixture of compounds in analytical chemistry and biochemistry so as to identify, quantify or purify the individual components of the mixture.

B-2 APPARATUS

B-2.1 HPLC — Binary gradient liquid chromatography system, with UV detector capable of being operated under conditions suitable for resolving the individual constituents into distinct peak may be used.

B-2.1.1 *Column* — C18 Column of 100 Å with length 250 mm, internal diameter 4.6 mm and particle size 5 µm or equivalent.

B-2.2 Analytical Balance

B-3 REAGENTS

B-3.1 Ammonium Acetate — HPLC grade

B-3.2 Methanol — HPLC grade

B-3.3 Water — HPLC grade

B-3.4 Vinyl Sulphone — of known purity

B-4 SAMPLE PREPARATION

Weigh accurately 0.100 g vinyl sulphone sample in 100 ml volumetric flask. Dissolve it in water and make it up to 100 ml with water. Take 2.5 ml of this diluted solution into 100 ml volumetric flask and adjust to 100 ml. Now this solution is around 25 ppm.

B-5 BUFFER PREPARATION

Take 0.610 0 g of ammonium acetate in to the 100 ml of volumetric flask. Add 30 ml of methanol to it.

B-6 FLOW RATE — 1.00 ml/min

B-7 MOBILE PHASE — buffer solution

B-8 COLUMN OVEN TEMPERATURE — 40 ºC

B-9 INJECTION VOLUME — 20 µl

B-10 RUN TIME — 25 min to 30 min

B-11 WAVELENGTH — 254 nm.

B-12 PEAK TIME

B-13 CALCULATION

Calculate the peak area of individual constituent pertaining to vinyl sulphone the chromatogram of the material. The concentration of the constituent may be obtained on the basis of peak area on chromatogram obtained with known amount of pure vinyl sulphone.

$$\text{Vinyl Sulphone, percent} = \frac{A}{Total\ Area} \times 100$$

where

A = area of vinyl sulphone peak in sample.

Vinyl Sulphone : 4.83 min

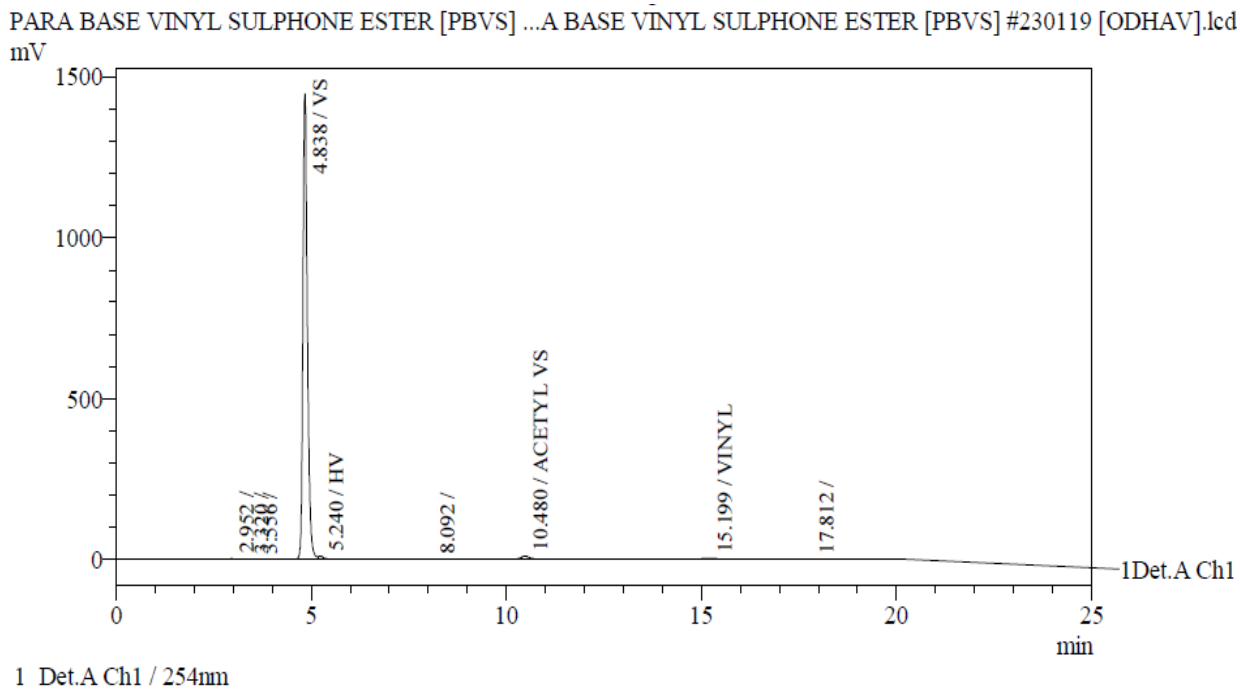


FIG. 1 TYPICAL CHROMATOGRAM

ANNEX C

[*Table* 1, *Sl No*. (ii)]

DETERMINATION OF HYDROLYSIS VALUE

C-1 REAGENTS

C-1.1 Standard Sodium Hydroxide Solution — 0.1 N

C-1.2 Phenolphthalein Indicator

C-1.3 Ice

C-2 PROCEDURE

Weigh and take 1 g sample in to 1 000 ml glass beaker. Add approximately 150 ml to 200 ml distilled water into the beaker and dissolve the sample by stirring. Add ice cubes into the beaker to get temperature around 10 °C. Add phenolphthalein indicator 10 drops to 12 drops. Take 0.1 N sodium hydroxide solution into burette. Titrate the sample against 0.1 N sodium hydroxide solution. End point will be pale pink colour. Note down the burette reading. Fill the burette with 0.1 N sodium hydroxide solution and adjust to zero reading. Keep the sample on hot plate and warm the solution to 60 °C with constant stirring till the pink colour disappears. Add 0.1 N sodium hydroxide solution from burette slowly under constant stirring, till a stable pink colour is obtained. If necessary, add few drops of phenolphthalein to ascertain the correct end point. Note down this hot burette reading.

C- 3 CALCULATION

Hydrolysis value, percent by mass =

$$\frac{V_1 \times N_1 \times 281.31}{M \times 10}$$

where

V_1 = Volume, in ml, of standard sodium hydroxide solution used in the titration (hot).

N_1 = Normality, of sodium hydroxide; and

M = Mass, in g, of the material taken for the test,

ANNEX D

[*Table* 1, *Sl No*. (iii)]

DETERMINATION OF MATTER INSOLUBLES IN SODIUM CARBONATE SOLUTION

D-1 REAGENTS

D-1.1 Sodium Carbonate Solution — 20 percent (*m/v*)

D-2 PROCEDURE

Weigh exactly 10 g of the dry powder sample and pour into 250 ml beaker. Add 100 ml demineralized water and stir with glass rod to make smooth slurry. Add 20 percent sodium carbonate (soda ash) solution to get *p*H 4.5 to 5.5 and stir well on magnetic stirrer to get clear solution. Add additional demineralized water to make up the volume to 200 ml (approximately). Take Whatman filter paper No. 42 of 110 diameter and soak in water and dry in oven till it is dry. Weigh the dried filter paper and note down the weight (initial weight). Place the dried Whatman paper in Buchner's funnel and add 50 ml water and start the vacuum pump to fix up properly. Pour the dissolved solution from the beaker on the filter paper and suck all the solution. Wash the filter paper by using fresh water from the wash bottle 4 to 5 times and suck properly. Take out the Whatman filter paper and keep in oven at 105 ºC for 20 min till it is completely dried. Keep the dried Whatman paper in desiccator and allow to cool to room temperature. Weigh the dried filter paper and note down the weight (final weight).

D-3 CALCULATION

Matter insoluble in sodium carbonate solution, percent by mass = $\frac{m \times 100}{M}$

where

m = mass, in g, of the residue; and

M = mass, in g, of the material taken for the test.

ANNEX E

[*Table* 1, *Sl No.* (iv)]

DETERMINATION OF 4-CHLOROANILINE CONTENT IN VINYL SULPHONE BY HIGH PERFORMANCE LIQUID CHROMATOGRAPHY

E-1 APPARATUS

E-1.1 HPLC — binary gradient liquid chromatography system, with UV detector capable of being operated under conditions suitable for resolving the individual constituents into distinct peak may be used.

E-1.1.1 *Column* — C18 Column of 100 Å with length 250 mm, internal diameter 4.6 mm and particle size 5 µm or equivalent.

E-1.2 Analytical Balance

E-2 REAGENTS

E-2.1 Methanol — HPLC grade

E-2.2 Water — HPLC grade

E-2.3 Acetonitrile — HPLC grade

E-2.4 Sodium Carbonate

E-2.5 Trichloromethane (Chloroform)

E-2.6 4-Chloroaniline — of known purity

E-3 STANDARD PREPARATION

Weigh accurately 0.100 g 4-chloroaniline into 100 ml volumetric flask. Make it up to 100 ml with methanol. Take 0.5 ml of the diluted solution in 100 ml volumetric flask and make it up to 100 ml with methanol. This is 5 ppm solution.

E-4 SAMPLE PREPARATION

Weigh accurately 1.000 0 g of sample in 100 ml beaker and dissolve in 60 ml water. Add 20 percent sodium carbonate solution to get the *p*H between 8 to 9. Take this solution into the separating funnel and add 10 ml of trichloromethane (chloroform) and shake well. Allow the content settle down for 15 min to 20 min. Take 1 ml from bottom layer in to the 10 ml of volumetric flask. Add methanol to make up the volume to 10 ml. This solution is ready for analysis.

E-5 BUFFER PREPARATION

Take 60 ml of acetonitrile in to 100 ml volumetric flask and make it up to 100 ml with water.

E-6 FLOW RATE — 1.00 ml/min

E-7 MOBILE PHASE — buffer solution

E-8 COLUMN OVEN TEMPERATURE — 40 ºC

E-9 INJECTION VOLUME — 20 µl

E-10 RUN TIME — 15 min

E-11 WAVELENGTH — 240 nm (photodiode array detector)

E-12 PEAK TIME

4-Chloroaniline : 5.67 min

E-13 CALCULATION

Calculate the peak area of 4-chloroaniline. The concentration of the constituent may be obtained on the basis peak area on chromatogram obtained with known amount of pure 4-chloroaniline.

4-Chloroaniline, percent by mass =

$$\frac{A_2 \times V_1 \times W_1 \times B_2}{A_1 \times V_2 \times W_2 \times B_1} \times 100$$

where

A_2 = area of 4-chloroaniline;
V_1 = injection volume of standard 4-chloroaniline;
W_1 = mass, in g of standard 4-chloroaniline;
B_2 = total volume of sample, in ml;
A_1 = area, of standard 4-chloroaniline;
V_2 = injection volume of sample, in ml;
W_2 = mass, in g of sample; and
B_1 = total volume, in ml of standard 4-chloroaniline.

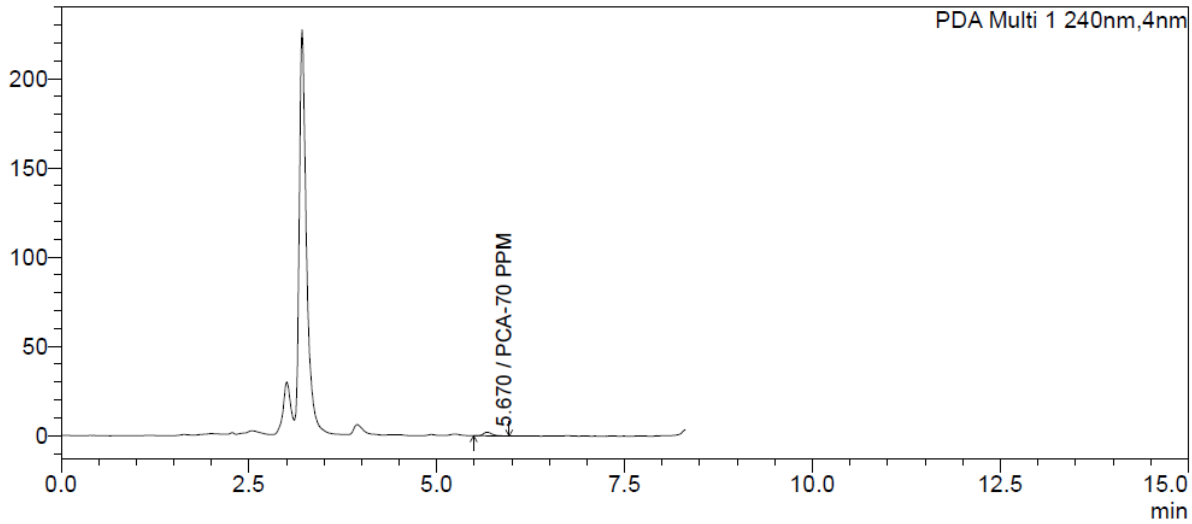


FIG. 2 TYPICAL CHROMATOGRAM

ANNEX F

(*Foreword*)

PICTOGRAMS, SIGNAL WORD, HAZARD STATEMENT AND PRECAUTIONARY STATEMENT

Pictogram(s)	:	
Signal Word	:	**DANGER**
Hazard Statement(s)	:	H318 : Causes serious eye damage
Precautionary Statement(s)	:	**Prevention:** P264 + P265 : Wash hands thoroughly after handling. Do not touch eyes. P280 : Wear protective gloves/protective clothing/eye protection/face protection. **Response:** P305 + P354 + P338 : If in eyes, immediately rinse with water for several minutes. Remove contact lenses, if present and easy to do. Continue rinsing. P317 : Get medical help.

ANNEX G

(*Foreword*)

COMMITTEE COMPOSITION

Dye Intermediates Sectional Committee, PCD 26

Organization	*Representative(s)*
Institute of Chemical Technology, Mumbai	PROF GANAPATI SUBRAY SHANKARLING **(*Chairperson*)**
Aarti Industries Limited, Mumbai	DR VAISHALI BHANDARY DR SANJEEV KUMAR DIXIT (*Alternate*)
Ankleshwar Research and Analytical Infrastructure Limited, Ankleshwar	SHRI MANSUKH H. VEKARIA
Alkyl Amines Chemicals Limited, Mumbai	SHRI S.V. NIKUMBHE SHRI KIRAT PATEL (*Alternate*)
Archroma India Private Limited, Thane	DR RAJESH RAMAMURTHY SHRI HEMANT MHADESHWAR (*Alternate*)
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Central Revenue Control Laboratory, New Delhi	SHRI V. SURESH SHRI SHIVRAJ SINGH (*Alternate* I) SHRI MRITUNJOY MAITY (*Alternate* II)
Colourtex Industries Limited, Mumbai	DR PANKAJ DESAI SHRI R. K. JAISWAL (*Alternate*)
Deepak Nitrite Limited, Vadodara	SHRI SAILASH RAVAL SHRI RAJENDRA SHINDE (*Alternate*)
Defence Research Development Organization, Ministry of Defence, New Delhi	DR PRADEEP K. GUPTA
Dystar, Mumbai	DR MONIKA SINGH
Ecological and Toxicological Association of Dyes, Vadodara	DR PARITI SIVA RAMA KUMAR
Gujarat Dyestuffs Manufacturers Association, Ahmedabad	SHRI YOGESH PARIKH SHRI ANIL M. JAIN (*Alternate* I) SHRI HARESH BHUTA (*Alternate* II)
Gujarat Narmada Valley Fertilizers Company Limited, Ahmedabad	SHRI R. M. PATEL SHRI C. S. PATEL (*Alternate*)
Gujarat Pollution Control Board, Gandhinagar, Ahmedabad	SHRI D.M. THAKER

Organization	*Representative(s)*
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Huntsman Textile Effects, Mumbai	MS PRACHI JOSHI
Indian Beauty and Hygiene Association, Mumbai	MS MALATHI NARAYANAN
Indian Chemical Council, Mumbai	SHRI P. S. SINGH
Jay Chemicals Industries Private Limited, Ahmedabad	SHRI VILPESH YADAV SHRIMATI MAITRI VYAS (*Alternate*)
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Meghmani Dyes and Intermediates Limited, Ahmedabad	SHRI MANOHAR MAHESHWARI SHRI RAMESH SHINGARE (*Alternate*)
Ministry of Environment Forest and Climate Change, New Delhi	SHRI N. SUBRAHMANYAM
Sudarshan Chemical Industries Limited, Pune	DR R. SRIDHARAN
The Bombay Textile Research Association, Mumbai	MS SHITAL PALASKAR
The Dyestuff Manufactures Association of India Office, Mumbai	SHRI V. R. KANETKAR
BIS Directorate General	SHRIMATI MEENAL PASSI, SCIENTIST 'F'/SENIOR DIRECTOR AND HEAD (PETROLEUM, COAL AND RELATED PRODUCTS) [REPRESENTING DIRECTOR GENERAL (*Ex-officio*)]

Member Secretary
SHRI ABHIJEET SINGH
SCIENTIST 'B'/ASSISTANT DIRECTOR
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Amend No.	Date of Issue	Text Affected

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