भारतीय मानक
Indian Standard

IS 8637 : 2020

एच एसिड — विशिष्टि

(दूसरा पुनरीक्षण)

H Acid — Specification

(Second Revision)

ICS 71.080.40

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

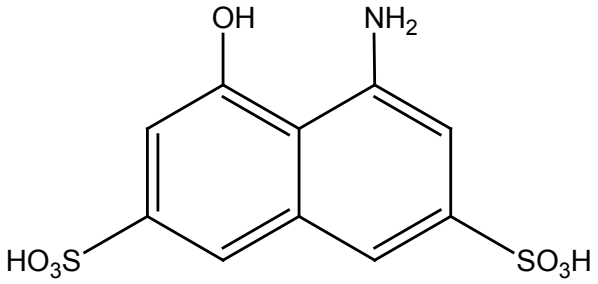
August 2020 **Price Group 4**

Dye Intermediates Sectional Committee, PCD 26

FOREWORD

This Indian Standard (Second Revision) was adopted by the Bureau of Indian Standards, After the draft finalized by the Dye Intermediates Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

H acid ($C_{10}H_9O_7NS_2$) is an important intermediate used for making azo dyes. It is described as 1-amino-8-naphthol-3, 6-disulphonic acid. It is represented by the following structural formula:



H ACID
(Molecular Mass 319)

This Standard was first published in 1977 and revised in 1986. The first revision was carried out to modify the requirement of matter insoluble in sodium carbonate solution and that of difference between nitrite value and coupling value. The requirement of impurities, namely, Koch's acid content and chromotropic acid content along with their chromatographic test methods were introduced.

Considering to the development in analytical techniques in last one decade and use of more sophisticated instrument to determine purity and impurity profile, this revision has been undertaken. Test method for Purity and impurities like Chromotropic acid and Koach acid content using High Performance Liquid Chromatography (HPLC) are incorporated.

The composition of the Committee, responsible for the formulation of this standard is given at Annex D.

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 : 1960 'Rules for rounding off numerical values (*revised*)'. 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

H ACID — SPECIFICATION

(Second Revision)

1 SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for H acid.

2 NORMATIVE REFERENCES

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

IS No.	*Title*
797 : 1982	Common salt for chemical industries *(third revision)*
1070 : 1992	Reagent grade water *(third revision)*
2552 : 1989	Steel Drums (galvanized And Ungalvanized) — Specification *(third revision)*
5299 : 2001	Methods of sampling and tests for dye intermediates *(first revision)*

3 REQUIREMENTS

3.1 Description

The material shall be in the form of a paste or in the form of light grey to grey lumps or powder.

3.2 The material shall also comply with the requirements given in Table 1.

4 PACKING AND MARKING

4.1 Packing

The material shall be packed in steel drums (*see* IS 2552) lined with suitable polyethylene film or as agreed to between the purchaser and the supplier. Each container shall be securely closed.

4.2 Marking

Each container shall bear legibly and indelibly the following information:

a) Name of the material;

b) Name of the manufacturer and his recognized trade-mark, if any;

c) Batch number; and

d) Tare, net and gross mass.

4.2.1 The containers may also be marked with the Standard Mark

4.2.2 The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the BIS Act, 2016 and the Rules and Regulations framed thereunder, and the products may be marked with the standard mark.

Table 1 Requirements for H ACID

(*Clauses* 3.2,5.3.1, 5.3.2 *and* 6.1)

Sl No.	Characteristic	Requirement	Method of Test, Ref
(1)	(2)	(3)	(4)
i)	Purity (by nitrite value), percent by mass (on dry basis), *Min*	75.0	**A-2**
ii)	Difference between nitrite value and coupling value, percent by mass (on 100 percent basis), *Max*	1.5	**A-3**
iii)	Matter insoluble in sodium carbonate solution, percent by mass, *Max*	0.2	**A-4**
iv)	Purity (by HPCL), percent by mass (on dry basis), *Min*	75.0	Annex - B
v)	Chromotropic acid content, percent by mass (on 100 percent basis), *Max*	1.5	Annex - C
vi)	Koch's acid content, percent by mass (on 100 percent basis), *Max*	0.5	Annex - C

NOTES

1 Test for assay shall be conducted on each of the individual samples.

2 Tests for the determination of remaining characteristics, namely, difference between nitrite value and coupling value, matter insoluble in sodium carbonate solution, Koch's acid and chromotropic acid content shall be conducted on the composite sample.

5 SAMPLING

5.1 Representative samples of the material shall be drawn as prescribed in 4 of IS 5299.

5.2 Numbers of Tests

5.2.1 Tests for assay shall be conducted on each of the individual samples separately

5.2.2 Tests for the determination of remaining characteristics, namely, difference between nitrite value and coupling value, matter insoluble in sodium carbonate solution, Koch's acid and chromotropic acid content 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 of assay if each of the individual test results satisfies the relevant requirement 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 TEST METHODS

6.1 Tests shall be conducted according to methods prescribed in Annexes and IS Specifications as given in col 4 of Table 1.

6.2 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.

ANNEX A

(Table 1 *and Clause* 5.1 *)*

METHODS OF TEST FOR H ACID

A-1 PREPARED SAMPLE

A-1.1 Dry the material at 105±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.

A-2 ASSAY

A-2.1 Reagents

A-2.1.1 *Concentrated Hydrochloric Acid*

A-2-1.2 *Potassium Bromide*

A-2.1.3 *Standard Sodium Nitrite Solution* ─ 0.1 N.

A-2.1.4 *Potassium Starch Iodide Indicator Papers*

A-2.2 Procedure ─ Pipette 50 ml aliquot from volumetric solution prepared for the determination of insoluble in sodium carbonate solution (*see* **A-4.2.2**) in to a one-litre beaker. Add about 200 ml of water, 35 ml of hydrochloric acid, 5.0 g of potassium bromide and washed-ice. Cool to 10 to 15°C. Titrate, while stirring mechanically, with sodium nitrite solution using potassium starch iodide test paper. The end point is reached when a blue-colored ring appears which can be obtained repeatedly for a period of 10 min without further addition of nitrite solution.

A-2.3 Calculation

Assay (by nitrite value), percent by mass = $\frac{V_1 \times N_1 \times 319}{M}$
(Let this value be NV)

where

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

N_1 = normality of sodium nitrite solution, and

M = amass in g of the material taken for the test (*see* **A-4.2.1**).

A-3 DIFFERENCE BETWEEN NITRITE VALUE AND COUPLING VALUE

A-3.1 Determination of Coupling Value

A-3.1.1 *Reagents*

A-3.1.1.1 *Sodium carbonate solution* — 20 percent (*m/v)*.

A-3.1.1.2 *P-chloroanilinediazo solution* — 0.1 N.

A-3.1.1.3 *Common salt — see* IS 797.

A-3.1.2 *Procedure*

Pipette 50 ml aliquot from volumetric solution prepared for the determination of insoluble in sodium carbonate solution (*see* **A-4.2.2**) into a one-litre beaker. Add 100 ml of water, 15 ml of sodium carbonate solution and washed-ice. Cool to 0 to 5°C. Titrate, while stirring mechanically, at 0 to 5°C with aniline diazo solution which is run from a cold water-jacketed burette maintained at 0 to 5°C. Towards the end point, add 50 g of sodium chloride and continue titration until no red colour is obtained at the junction of the coupling solution and the aniline diazo when spotted on a piece of Whatmanpaper which should be considered as the end point.

NOTE — A yellow colour appears slowly in this region but may be distinguished from the red colour given by H acid in aniline diazo.

A-3.1.3 *Calculation*

Coupling value, percent by mass = $\frac{V_2 \times N_2 \times 319}{M}$

where

V_2 = volume in ml of the p-chloroaniline diazo solution used in the titration,

N_2 = normality of the p-chloroaniline diazo solution, and

M = mass in g of the material taken for the test (*see* **A-4.2.1**).

A-3.2 Difference Between Nitrite Value and Coupling Value

Difference between nitrite value and coupling value, = $\frac{D}{NV} \times 100$ percent by mass (on l00 percent basis)

where

D = difference between nitrite value and coupling value; and

NV = nitrite value, percent by mass (*see* **A-2.3**).

A-4 DETERMINATION OF MATTER INSOLUBLES IN SODIUM CARBONATE SOLUTION

A-4.1 Reagents

A-4.1.1 *Sodium Carbonate Solution* — 20 percent (*m/v)*.

A-4.1.2 *Brilliant Yellow Indicator Papers*

A-4.2 Procedure

A-4.2.1 Weigh accurately about 15 g of the *Prepared sample* (*see* **A-l.1)** and transfer to a 500-ml beaker. Paste well with about 100 ml of water. Add sodium carbonate solution till alkaline to brilliant yellow indicator paper. Add about 200 ml of water and heat to dissolve the material completely. Filter hot through counter-poised Whatman filter paper. Wash the residue with hot water till the washings are free from alkali. Dry the residue at 100 ± 5°C to constant mass.

A-4.2.2 Transfer quantitatively the filterate and washing together into a 500 ml volumetric flask and dilute with water up to the mark at room temperature. Mixwell.

A-4.3 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-B

[*Table 1 and Sl no. (*iv*)*]

TO DETERMINE PURITY OF H ACID BY HIGH PERFORMANCE LIQUID CHROMATOGRAPHY

B-0 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-1 OBJECTIVE

To determine Purity of H Acid by high performance liquid Chromatography

B-2 APPARATUS

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-3 COLUMN: C18 100A 250 x 4.6mm, 5µm or equivalent

B-4 REAGENT

a) Acetonitrile –HPLC grade
b) Water – HPLC Grade
c) Sodium di hydrogen orthophosphate – HPLC grade
d) Tetra n-Butyl Ammonium Bromide – HPLC grade
e) H Acid (Known Purity)

B-5 STANDARD PREPARATION

Weigh accurately 0.0500 g H Acid in 100 ml volumetric flask dissolve it in Water: Acetonitrile (20:80) & make up to the mark with Acetonitrile (20:80).

B-6 SAMPLE PREPARATION

Weigh accurately 0.0500 g Sample in 100 ml volumetric flask dissolve it in Water: Acetonitrile (20:80) and make up to the mark with Acetonitrile (20:80).

B-7 BUFFER PREPARATION

Take 5.000 g tetra n-butyl ammonium bromide and 1.666 g sodium Di-hydrogen ortho phosphate in 1 litre volumetric flask. Add 200ml HPLC grade water and complete dissolve it. Make total volume with HPLC grade water.

B-8 FLOW RATE: 1.00 ml/min

B-9 MOBILE PHASE

Acetonitrile	Buffer
35	65

B-10 COLUMN OVEN TEMPERATURE: 40^0C

B-11 INJECTION VOLUME: 5µl

B-12 RUN TIME: 20 minutes

B-13 WAVE LENGTH : 230nm

B-14 PEAK TIME: H Acid — 4.82 minute

B-15 CALCULATION:

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

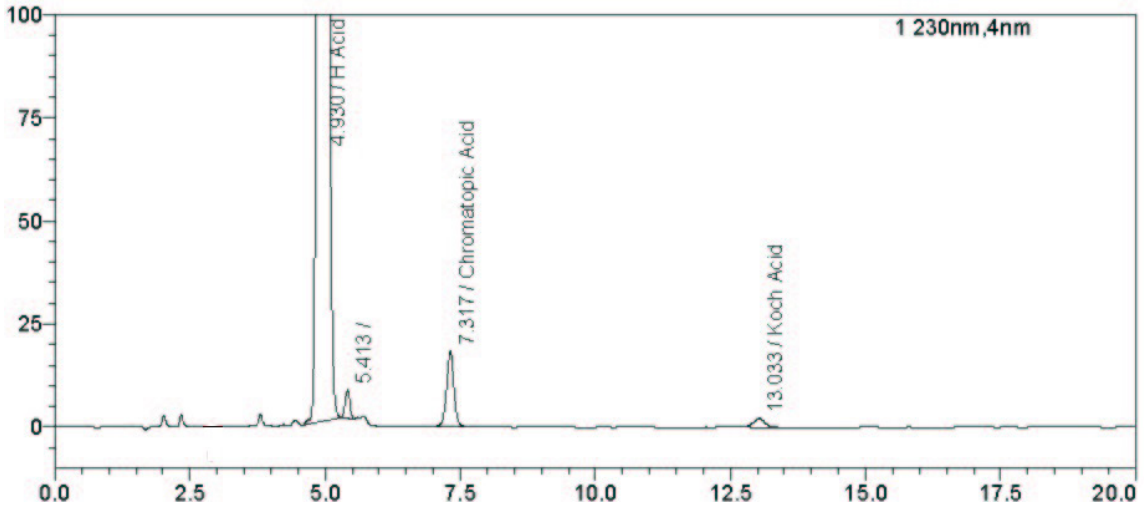


FIG. 1

$$\% \text{ of H Acid} = \frac{A_2 \times V_1 \times W_1 \times B_2}{A_1 \times V_2 \times W_2 \times B_1} \times 100$$

Where

A_1 → Area of standard H Acid

V_1 → Injection volume of standard H Acid

W_1 → Weight of standard H Acid

B_1 → Total volume of standard H Acid

A_2 → Area of H Acid peak in sample

V_2 → Injection volume of Sample

W_2 → Weight of sample

B_2 → Total volume of Sample

ANNEX-C

[*Table 1 and Sl No.(* v *and* vi *)*]

TO DETERMINE CHROMOTROPIC ACID & KOCH ACID CONTENT IN H ACID BY HIGH PERFORMANCE LIQUID CHROMATOGRAPHY

C-0 OBJECTIVE

To determine Chromotropic Acid & Koch Acid content in H Acid by high performance liquid Chromatography

C-1 APPARATUS

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.

Column: C18 100A 250 x 4.6mm, 5µm or equivalent

Reagent:

1 Acetonitrile –HPLC grade

2 Water – HPLC Grade

3 Sodium di hydrogen orthophosphate – HPLC grade

4 Tetra n-Butyl Ammonium Bromide – HPLC grade

5 Koch Acid – Known Purity

6 Chromatropic Acid – Known Purity

C-2 STANDARD PREPARATION

Weigh accurately 0.0500gm Chromatropic Acid in 100ml volumetric flask dissolve it in Water: Acetonitrile (20:80) & make up to the mark with Acetonitrile (20:80). ---- **Stock Solution A**

Weigh accurately 0.0500gm Koch Acid in 100 ml volumetric flask dissolve it in Water: Acetonitrile

(20:80) and make up to the mark with Acetonitrile (20:80). ----**Stock Solution B**

Take 1ml each of Solution A & Solution B in to 100 ml volumetric flask and make up to the mark with Acetonitrile (20:80). --- **Stock Solution C**

C-3 SAMPLE PREPARATION:

Weigh accurately 0.0500gm Sample in 100ml volumetric flask dissolve it in Water: Acetonitrile (20:80) & make up to the mark with Acetonitrile (20:80).

C-4 BUFFER PREPARATION:

Take 5.0000 gm Tetra n-Butyl Ammonium Bromide and 1.6666 gm Sodium Di-Hydrogen Ortho Phosphate in 1 litre volumetric flask. Add 200ml HPLC grade water & complete dissolve it. Make total volume with HPLC grade water.

Flow Rate: 1.00 ml/min

Mobile Phase:

Acetonitrile	Buffer
35	65

Column Oven Temperature	:	40^0C
Injection Volume	:	5µl
Run Time	:	20 minutes
Wave length	:	230 nm
Peak Time	:	H Acid — 4.82 minute Chromotropic Acid — 7.31 minute. Koch Acid — 13.03 minute

Calculation:

Calculate the peak area of individual constituent pertaining to Chromotropic Acid / Koch Acid on the chromatogram of the material. The concentration of the constituent may be obtained on the basis peak area on chromatogram obtained with known amount of pure Chromotropic Acid / Koch Acid.

$$\% \text{ of H Acid} = \frac{A_2 \times V_1 \times W_1 \times B_2}{A_1 \times V_2 \times W_2 \times B_1} \times 100$$

where

A_1 → Area of standard Chromotropic Acid / Koch Acid

V_1 → Injection volume of Standard Chromotropic Acid / Koch Acid

W_1 → Weight of standard Chromotropic Acid / Koch Acid

B_1 → Total volume of Standard Chromotropic Acid / Koch Acid

A_2 → Area of Chromotropic Acid / Koch Acid peak in Sample

V_2 → Injection volume of Sample

W_2 → Weight of Sample

B_2 → Total volume of Sample

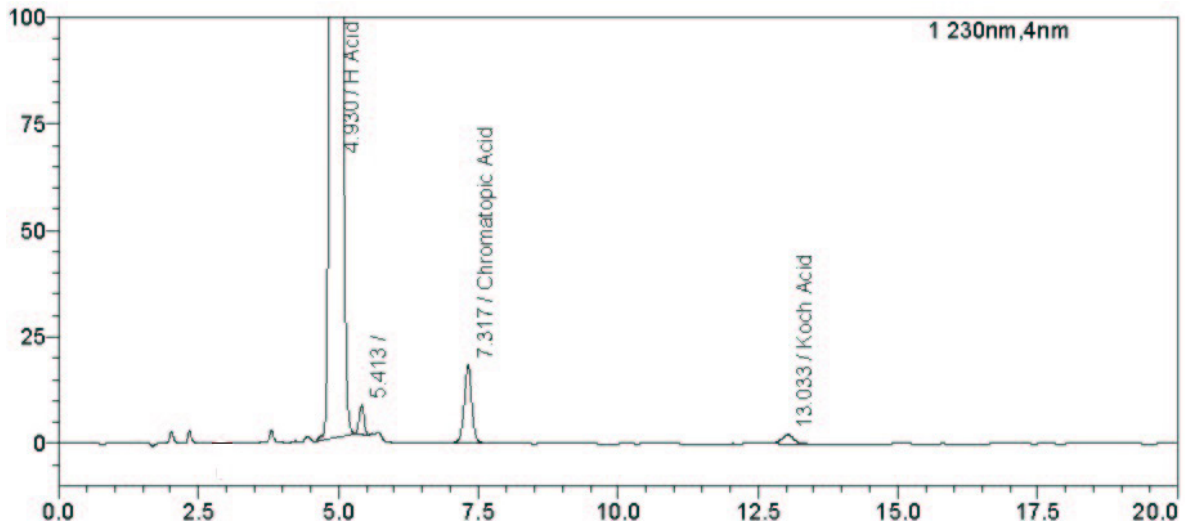


FIG. 2

ANNEX D

(*Foreword*)

COMMITTEE COMPOSITION

Dye Intermediates Sectional Committee, PCD 26

Organization	*Representative(s)*
ICT, Mumbai	Prof P. M. Bhate (***Chairman***)
Aarti Industries Limited, Mumbai	Shri Kirit H. Desai
Alkyl Amines Chemicals Limited, Mumbai	Shri Kirat Patel Shri S. V. Nikumbhe (*Alternate*)
Ankleshwar Research and Analytical Infrastructure Limited (ARAIL), Ankleshwar, Gujrat	Shri Mansukh H. Vekaria
Archroma, Thane	Dr Rajesh Ramamurthy Shri Hemant Mhadeshwar (*Alternate*)
Atul Limited, Valsad, Gujarat	Dr J. G. Desai Dr M. U. Rahman (*Alternate*)
BASF India Limited, Mumbai	Shri Uday Kulkarni
Central Revenue Control Laboratory (CRCL), New Delhi	Dr T. A. Sreenivasa Rao Shri Praful Dalal (*Alternate*)
Colourtex Industries Limited, Mumbai	Dr Pankaj Desai Shri Ismail Habibulla (*Alternate*)
Deepak Nitrite Limited, Vadodara	Shri Sailash Raval Dr Mangalya Kar (*Alternate* I) Dr J. K Astik (*Alternate* II)
Ecological and Toxicological Association of Dyes (ETAD), Vadodra	Dr Pariti Siva Rama Kumar
Gujarat Dyestuffs Manufacturers Association (GDMA), Ahmedabad	Shri R. S. Patel Shri Bansibhai Patal (*Alternate*)
Gujarat Pollution Control Board (GPCB), Ghandhinagar, Ahmedabad	Shri Y. A. Tai
Heubach Colour Private Limited, Mumbai	Shri J. I. Sevak Shri Vinod Madhukar (*Alternate*)
Indian Chemical Council, Mumbai	Shri P. S. Singh
Jay Chemical Industries, Ahmedabad	Shri Vilpesh Yadav Shrimati Maitri Vyas (*Alternate*)
Ministry of Chemicals & Fertilizers - Department of Chemicals & Petrochemicals - New Delhi	Shri Jasbir Singh
Ministry Of Defence (Research and Development), New Delhi	Dr Vijay Tak
National Test House (er), Kolkata	Shri P. K. Chakraborty Shri Y. C. Nijhawan (*Alternate*)
Sudarshan Chemical Industries Limited, Pune	Dr Jaychandran M. Nair
The Bombay Textile Research Association	Dr Ashok N. Desai
The Dyestuffs Manufacturers Association of India (DMAI), Mumbai	Shri V. R. Kanetkar
BIS Director General	Shri N. K. Bansal, Scientist 'F' and Head (PCD), [Representing Director General (*Ex-officio*)]

Member Secretary

Shri Chandrakesh Singh
Scientist 'D', BIS

Bureau of Indian Standards

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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 latest issue of 'BIS Catalogue' and 'Standards: Monthly Additions'.

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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 : Manak Bhavan, 9 Bahadur Shah Zafar Marg, NEW DELHI 110002 — 2323 7617, 2323 3841

Eastern : 1/14 C.I.T. Scheme VII M, V.I.P. Road, Kankurgachi, KOLKATA 700054 — 2337 8499, 2337 8561, 2337 8626, 2337 9120

Northern : Plot No. 4-A, Sector 27-B, Madhya Marg, CHANDIGARH 160019 — 265 0206, 265 0290

Southern : C.I.T. Campus, IV Cross Road, CHENNAI 600113 — 2254 1216, 2254 1442, 2254 2519, 2254 2315

Western : Manakalaya, E9 MIDC, Marol, Andheri (East), MUMBAI 400093 — 2832 9295, 2832 7858, 2832 7891, 2832 7892

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