

इंटरनेट

मानक



Disclosure to Promote the Right To Information

Whereas the Parliament of India has set out to provide a practical regime of right to information for citizens to secure access to information under the control of public authorities, in order to promote transparency and accountability in the working of every public authority, and whereas the attached publication of the Bureau of Indian Standards is of particular interest to the public, particularly disadvantaged communities and those engaged in the pursuit of education and knowledge, the attached public safety standard is made available to promote the timely dissemination of this information in an accurate manner to the public.

“जानने का अधिकार, जीने का अधिकार”

Mazdoor Kisan Shakti Sangathan

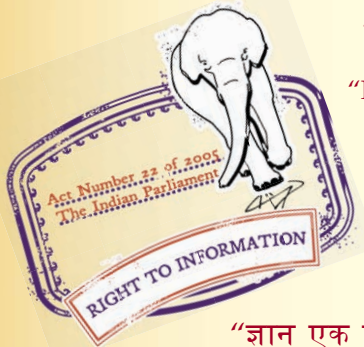
“The Right to Information, The Right to Live”

“पुराने को छोड़ नये के तरफ”

Jawaharlal Nehru

“Step Out From the Old to the New”

IS 3025-61 (2008): Methods of sampling and test (physical and chemical) for water and waste water, Part 61: Total and free carbon dioxide [CHD 32: Environmental Protection and Waste Management]



“ज्ञान से एक नये भारत का निर्माण”

Satyanarayan Gangaram Pitroda

“Invent a New India Using Knowledge”



“ज्ञान एक ऐसा खजाना है जो कभी चुराया नहीं जा सकता है”

Bhartrhari—Nitiśatakam

“Knowledge is such a treasure which cannot be stolen”



BLANK PAGE



भारतीय मानक

जल और अपशिष्ट जल के नमूने लेने तथा
परीक्षण (भौतिक एवं रसायन) की पद्धतियाँ

भाग 61 सकल और मुक्त कार्बन डाइऑक्साइड
(पहला पुनरीक्षण)

Indian Standard

**METHODS OF SAMPLING AND TEST
(PHYSICAL AND CHEMICAL) FOR WATER AND
WASTEWATER**

PART 61 TOTAL AND FREE CARBON DIOXIDE

(First Revision)

ICS 13.060.50

© BIS 2008

**BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002**

FOREWORD

This Indian Standard (Part 61) (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Environment Protection and Waste Management Sectional Committee had been approved by the Chemical Division Council.

The committee responsible for the formulation of IS 3025 : 1964 'Methods of sampling and test (physical and chemical) for water used in industry' had decided to revise the standard and publish it in separate parts. This standard is one of the different parts under the IS 3025 series of standards and supersedes clauses 42 and 43 of IS 3025.

Surface water normally contains less than 10 mg free carbon dioxide per litre, while ground water may exceed that concentration. The carbon dioxide content of water may contribute significantly to corrosion.

The composition of the Committee responsible for the formulation of this standard is given at 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 : 1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standard

**METHODS OF SAMPLING AND TEST
(PHYSICAL AND CHEMICAL) FOR WATER AND
WASTEWATER**

PART 61 TOTAL AND FREE CARBON DIOXIDE

(First Revision)

1 SCOPE

This standard (Part 61) prescribes methods of test for determination of total carbon dioxide and free carbon dioxide in water and wastewater.

2 REFERENCES

The standards listed below contain provisions which, through reference in this 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 agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

<i>IS No.</i>	<i>Title</i>
3025 (Part 1) : 1987	Methods of sampling and test (physical and chemical) for water and wastewater : Part 1 Sampling (<i>first revision</i>)
7022 (Part 1) : 1973	Glossary of terms relating to water, sewage and industrial effluents, Part 1
7022 (Part 2) : 1979	Glossary of terms relating to water, sewage and industrial effluents, Part 2

3 TERMINOLOGY

For the purpose of this standard, definitions given in IS 7022 (Part 1) and IS 7022 (Part 2) shall apply.

4 SAMPLING AND PRESERVATION

Sampling and sample preservation shall be done as prescribed in IS 3025 (Part 1).

5 DETERMINATION OF TOTAL CARBON DIOXIDE

5.1 Principle

Carbon dioxide is liberated by acidifying and heating the sample in a closed system. The carbon dioxide is absorbed in an alkaline barium chloride

solution and the barium carbonate formed is determined by titration.

5.2 Range and Applicability

This method is applicable for determination of total carbon dioxide in water and wastewater for the range from 11 mg/l to 4 000 mg/l in terms of CO₂.

5.3 Apparatus

5.3.1 Distillation Apparatus, assembled as shown in Fig. 1.

5.3.2 Apparatus for Storing Barium Chloride Solution, assembled as shown in Fig. 2.

5.4 Reagents

5.4.0 Purity of the Reagents

Unless specified otherwise, only pure chemicals and carbonate free distilled water shall be used in tests.

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

5.4.1 Dilute Hydrochloric Acid — 1 : 3 (v/v).

5.4.2 Phenolphthalein Indicator Solution

5.4.3 Barium Chloride Solution — 0.5 M.

5.4.4 Methyl Orange Indicator Solution

5.4.5 Standard Sodium Hydroxide Solution — 1 M and 0.1 M, standardized with methyl orange as indicator.

5.4.6 Standard Hydrochloric Acid — 1 M and 0.1 M.

5.5 Procedure

5.5.1 Into the 500-ml Kjeldahl flask *D* of the distillation apparatus, add 50 ml of dilute hydrochloric acid. Into flask *E*, add 50 ml of distilled water and a few drops of phenolphthalein indicator solution and connect *B*, *D* and *E* as shown in Fig. 1 with the rubber cap *G* but without the soda lime tower *A*. Close tap *C* and evacuate by a water pump through tap *F* until the pressure is below 3 cm of mercury. Close tap *F*.

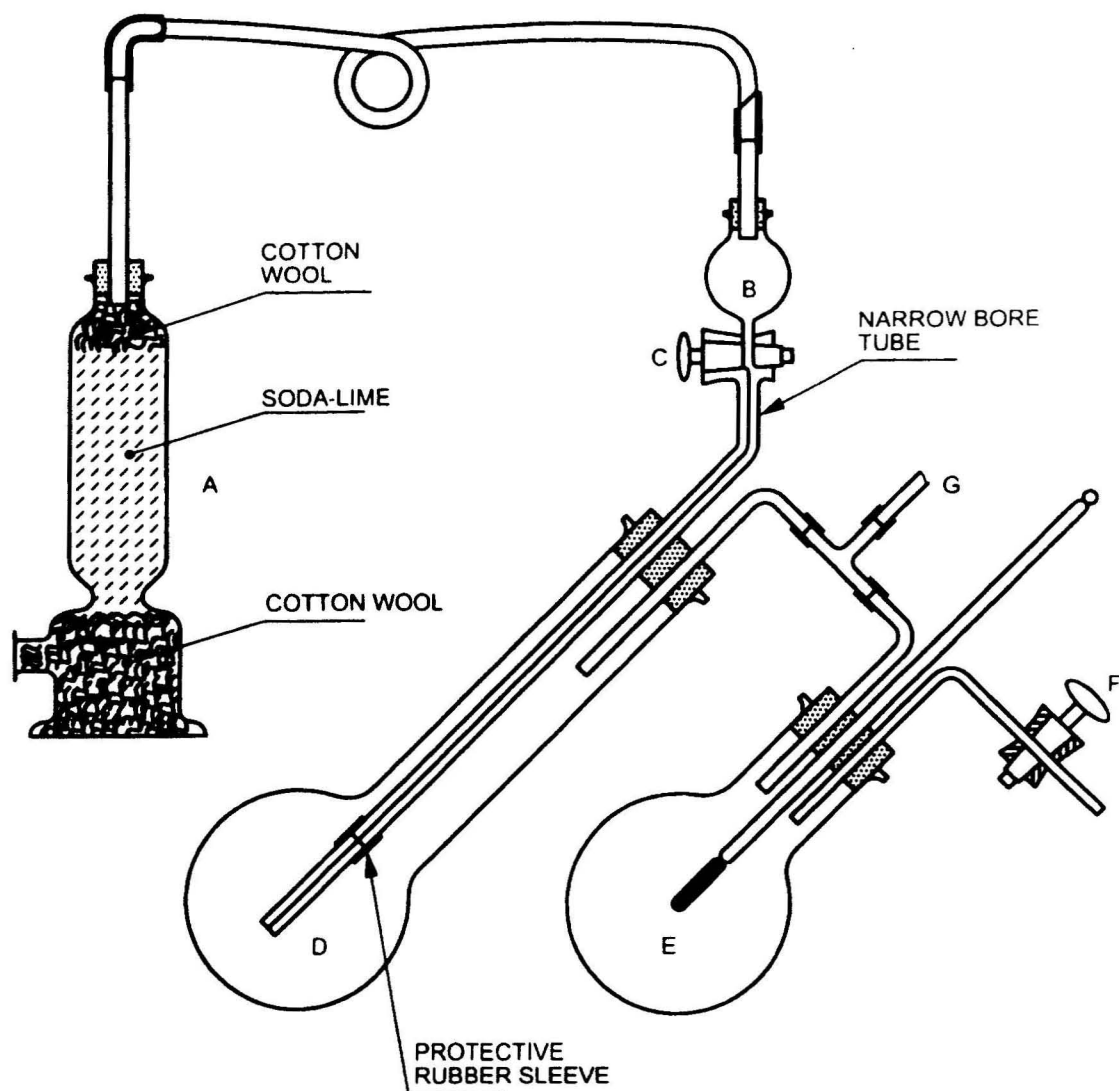


FIG. 1 DISTILLATION APPARATUS

5.5.2 In the aspirator *J* of the apparatus for storing barium chloride solution (see Fig. 2), add a mixture of equal volumes of barium chloride solution and standard sodium hydroxide solution (1 M). In the aspirator *H*, take distilled water. From aspirator *J*, run the liquid into a 45-ml bulb burette *K*, with the tap *L* shut, until the burette reads 0 ml. The volume of this solution to be used, which is approximately 0.5 M, should be equivalent to an excess of 10 ml of 0.5 M over the total alkalinity contained in the volume of the sample taken for the test. Connect the tap *F* in Fig. 1 to the bottom limb of the Y-piece below tap *L* in Fig. 2 by means of a short length of rubber tubing open taps *F* and *L* and allow the required volume of liquid to run from the burette. Shut tap *L*. Open the spring clip to allow distilled water from the aspirator *H* to flush out the connections and run into flask *E*. Close the spring clip and tap *F*. Disconnect the rubber tubing from the

bottom limb of the Y-piece. Connect tap *F*, while still shut, to a water pump running at full bore, open tap *F* and re-evacuate. Close tap *F*. Disconnect the water pump.

5.5.3 Into the funnel *B*, run 200 ml of the sample. Connect tower *A* to *B*. Open tap *C*. Run most of the sample from *B* into flask *D*. Close tap *C*, leaving about 0.5 ml of the sample in *B*. Disconnect the tower *A* from *B*. Run 5 ml of freshly boiled and cooled distilled water into *B*. Connect the tower *A* to *B* and open tap *C*. Run most of the solution from *B* into flask *D*, then close tap *C*, leaving about 0.5 ml of the solution in *B*. Repeat this rinsing of *B* in the same manner once more using 5 ml of freshly boiled and cooled distilled water. A small volume of the liquid should be left in *B* to prevent possible ingress of air. Heat flask *D* with a Bunsen flame

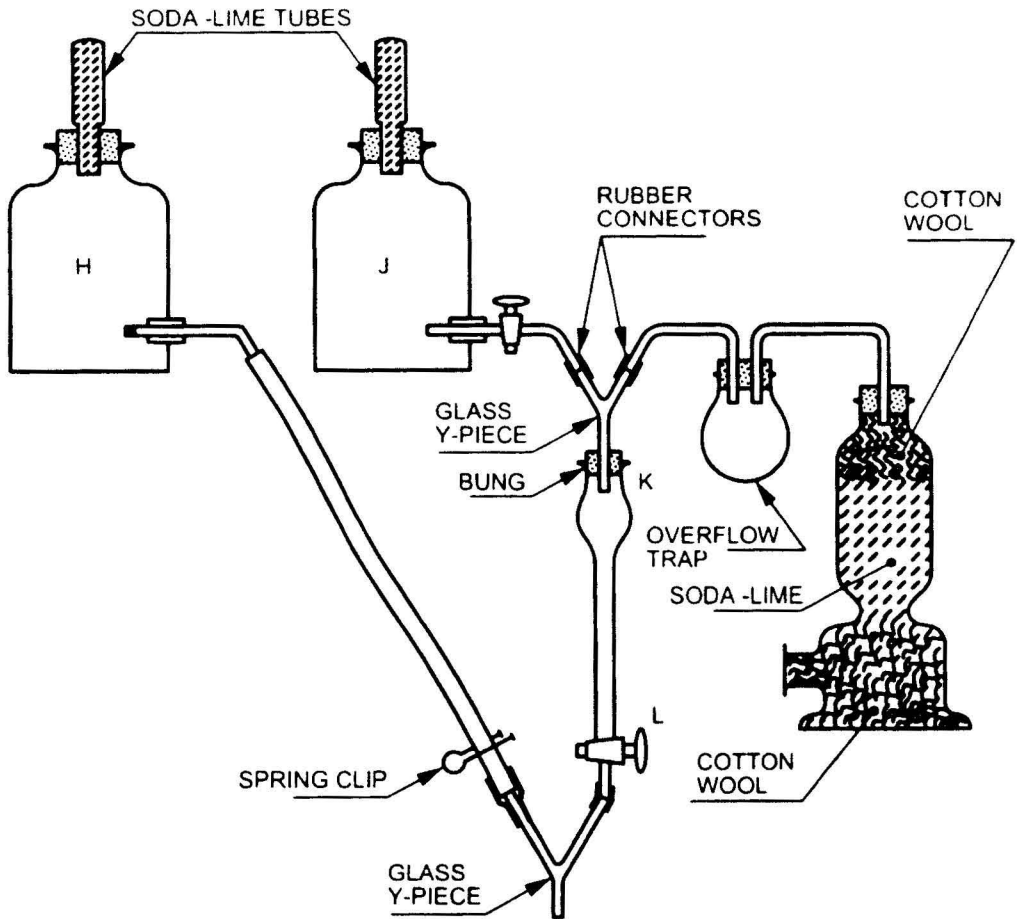


FIG. 2 APPARATUS FOR STORING BARIUM CHLORIDE SOLUTION

and shake the apparatus. This is simplified by having a felt pad below the retort stand. Continue heating until the thermometer in *E* reads 70°C. Remove the flame. Open tap *C* to allow air free from carbon dioxide to enter the apparatus. Let the apparatus cool.

5.5.4 Remove flask *E* and add a few drops of phenolphthalein indicator solution. Run standard hydrochloric acid (1 M) carefully into *E* until the phenolphthalein colour is nearly discharged. Complete the titration to the phenolphthalein end point using standard hydrochloric acid (0.1 M). To the contents of the flask, add standard hydrochloric acid (2 M or 0.1 M), according to the amount of carbonate present, with shaking until the precipitate dissolves. Add 2 ml in excess and note the reading. Add 2 drop of methyl orange indicator and titrate the excess acid with standard sodium hydroxide solution (1 M or 0.1 M) corresponding to the strength of acid previously used. Record as millilitres of 1 M acid and alkali respectively.

5.5.5 Carry out a blank determination on the reagents used. Subtract the number of millilitres of 1 M sodium

hydroxide required for the last titration from the millilitres of 1 M hydrochloric acid used to dissolve the barium carbonate, to obtain the number of millilitres of a normal solution equivalent to the carbon dioxide present. Let this figure be *X* and the corresponding blank be *Y*.

5.6 Calculation

$$\text{Total carbon dioxide (as CO}_2\text{), mg/l} = \frac{22\,000(X - Y)}{V}$$

where

X = volume, in ml, of 1 M hydrochloric acid added in 5.5.4 minus the volume, in ml, of 1 M sodium hydroxide solution used in 5.5.4;

Y = volume, in ml, of 1 M hydrochloric acid minus the volume, in ml, of 1 M sodium hydroxide solution used in the blank determination; and

V = volume of the sample taken for the test in ml.

6 DETERMINATION OF FREE CARBON DIOXIDE

6.1 Principle

6.1.1 The sample is titrated with standard sodium carbonate solution or standard barium hydroxide solution, using phenolphthalein indicator.

6.2 Range and Applicability

Two methods are prescribed for the determination of free carbon dioxide. Method A is applicable in the range 5 to 50 mg/l in terms of calcium carbonate and Method B is applicable up to 5 mg/l in terms of calcium carbonate.

6.3 Method A

6.3.1 Reagents

6.3.1.1 Phenolphthalein indicator solution

6.3.1.2 Standard sodium carbonate solution, 0.01 M.

6.3.1.3 Buffer solution — Dissolve 1.237 g of boric acid in carbon dioxide-free distilled water and make up to 100 ml (solution A). Dissolve 1.491 g of potassium chloride in carbon dioxide-free distilled water and make up to 100 ml (solution B). Mix 25 ml of solution A with 25 ml of solution B, add to the mixture 2.95 ml of an exactly 0.1 M sodium hydroxide solution and dilute with carbon dioxide-free distilled water to 100 ml. When required for use, dilute 10 ml of the solution to 100 ml with carbon dioxide-free distilled water.

6.3.2 Procedure

Pipette out 200 ml of the sample into a 250-ml glass stoppered bottle. Add 1 ml of phenolphthalein indicator solution and titrate in the bottle with standard sodium carbonate solution, using only gently agitation. Compare the tint with that obtained with 100 ml of buffer solution contained in a similar bottle to which 1 ml of indicator solution has been added. At the end point, the tint of the sample and the control shall be identical after one minute. The stopper shall be replaced after each addition of sodium carbonate solution.

6.3.3 Calculation

a) Free carbon dioxide (as CO_2), mg/l = $2.2 V$, and

b) Free carbon dioxide (as CaCO_3), mg/l = $5 V$.

where

V = volume of standard sodium carbonate solution used in the titration, in ml.

6.4 METHOD B

6.4.1 Apparatus

6.4.1.1 Sampling flask — A 500-ml conical flask

fitted with a rubber bung through which pass two glass tubes, the inlet tube reaching to the bottom of the flask and the outlet tube ending nearly flush with the bottom of the rubber bung. To each tube is connected a length of soft rubber tubing fitted with a spring clip.

6.4.1.2 Guard tube, containing soda-lime for fitting to the outlet of the sampling flask.

6.4.1.3 Semi-micro burette, capacity 1 ml. This is fitted with a fine immersion jet formed either by drawing out a length of glass tubing and connecting it to the tip of the burette with thick-walled rubber tubing, or by drawing out the tip of the burette.

6.4.2 Reagents

6.4.2.1 Nitrogen, Pure. If nitrogen supply is not available, air free from carbon dioxide may be used.

6.4.2.2 Phenolphthalein indicator solution

6.4.2.3 Standard sodium carbonate solution, 0.02 N.

6.5 Procedure

Collect the sample in the sampling flask, taking care to remove all bubbles and allowing about ten replacements of the sample to flow through the flask. Close the sample valve, then the clips. Wash a second 500-ml conical flask with distilled water and pass nitrogen through a bent glass tube hooked over the side of the flask and reaching nearly to the bottom. Fit the guard tube to the outlet of the sampling flask, hold the inlet tube so that it will act as a siphon, open both clips and allow about 100 ml of the sample to flow into the titration flask, taking care not to bubble nitrogen through the sample. Add 0.2 ml of phenolphthalein indicator solution and titrate with standard barium hydroxide solution, swirling gently during the titration and maintaining the flow of nitrogen about the sample. Note the reading of the burette when a distinct rose or pink colour develops. Pour the titrated sample into a measuring cylinder and note the volume.

6.6 Calculation

$$\text{a) Free carbon dioxide (as } \text{CO}_2\text{), mg/l} = \frac{440 V_1}{V_2}$$

$$\text{b) Free carbon dioxide (as } \text{CaCO}_3\text{), mg/l} = \frac{1\,000 V_1}{V_2}$$

where

V_1 = volume of standard barium hydroxide solution used in the titration, in ml; and

V_2 = volume of the sample taken for the titration, in ml.

ANNEX A

(Foreword)

COMMITTEE COMPOSITION

Environment Protection and Waste Management Sectional Committee, CHD 32

<i>Organization</i>	<i>Representative(s)</i>
In personal capacity (40/14, C.R. Park, New Delhi-110019) Bhabha Atomic Research Centre, Mumbai	PROF DILIP BISWAS (<i>Chairman</i>) DR (SMT) G. G. PANDIT DR I. V. SARADHI (<i>Alternate</i>)
Bharat Heavy Electricals Limited, Haridwar Cement Manufacturers' Association, New Delhi Central Fuel Research Institute, Dhanbad Central Leather Research Institute, Chennai Central Pollution Control Board, New Delhi	DR N. G. SHRIYASTAVA DR K. C. NARANG DR L. C. RAM DR S. RAJAMANI DR S. D. MAKHLANI DR C. S. SHARMA (<i>Alternate I</i>) DR S. K. TYAGI (<i>Alternate II</i>)
Confederation of Indian Industries, New Delhi	SHRI A. K. GHOSE SHRI R. P. SHARMA (<i>Alternate</i>)
Crop Care Federation of India, New Delhi Indian Institute of Technology, New Delhi	SHRI P. N. PARMESHAWARAN DR MUKESH KHARE DR ATUL MITTAL (<i>Alternate I</i>) DR ARVIND NEMA (<i>Alternate II</i>)
Delhi College of Engineering, Delhi	DR RAKESH MEHROTRA SHRI V. K. MINOCHA (<i>Alternate I</i>) DR (SMT) A. MANDAL (<i>Alternate II</i>)
Directorate General Factory Advice Service and Labour Institute, Mumbai Directorate General of Health Services, New Delhi Engineers India Limited, New Delhi	SHRI S. S. GAUTAM SHRI BRJ MOHAN (<i>Alternate</i>) DR (SMT) MADHURI SHARMA SHRI B. B. LAL SHRI SUSHEEL SADH (<i>Alternate</i>)
Envirotech Instruments Private Limited, New Delhi	SHRI RAKESH AGARWAL DR RAJENDRA PRASAD (<i>Alternate</i>)
Federation of Indian Chambers of Commerce & Industry (FICCI), New Delhi Gujarat Pollution Control Board, Ahmedabad Hindustan Lever Limited, Mumbai	KUMARI ANTARA ROY SHRI D. C. DAVE SHRI B. B. DAVE SHRI ADITYA JHAVAR (<i>Alternate</i>)
Indian Chemical Manufacturers' Association, Mumbai	SHRI V. N. DAS SHRI A. A. PANJWANI (<i>Alternate</i>)
Indian Council of Agricultural Research, New Delhi Indian Council of Medical Research, New Delhi Indian Oil Corporation Limited, Faridabad IPCL, Vadodara	DR R. C. MAHESHWARI SHRI H. N. SATYAD DR M. P. SINGH SHRI ANUPAM DESAI SHRI MIHIR BANERJI (<i>Alternate</i>)
Industrial Toxicology Research Centre, Lucknow Ministry of Defence (R & D), DRDO, New Delhi Ministry of Environment and Forests, New Delhi Ministry of Non-conventional Energy Sources, New Delhi Municipal Corporation of Greater Mumbai, Mumbai	DR S. K. BHARGAVA SHRI J. C. KAPOOR REPRESENTATIVE SHRI VINOD KUMAR JAIN DEPUTY CITY ENGINEER CIVIL (ENVT) DEPUTY EXECUTIVE ENGINEER (EMISSION INVENTORY GROUP) (<i>Alternate</i>)
National Institute of Occupational Health (ICMR), Ahmedabad	DR V. KRISHNA MURTHY DR A. K. MUKHERJEE (<i>Alternate</i>)
National Environmental Engineering Research Institute, Nagpur	DR V. I. PANDIT DR TAPAN NANDY (<i>Alternate</i>)
National Thermal Power Corporation Limited, New Delhi NCCBM, New Delhi	SHRI R. GOPAL SHRI M. S. BHAGWAT DR S. N. PATI (<i>Alternate</i>)

IS 3025 (Part 61) : 2008

<i>Organization</i>	<i>Representative(s)</i>
Reliance Industries Limited, Mumbai	DR E. SUNDERASAN DR U. K. SAROOP (<i>Alternate</i>)
Shriram Institute of Industrial Research, New Delhi	SHRI V. G. K. NAIR DR JAGDISH KUMAR (<i>Alternate</i>)
SGS India Limited, Chennai	SHRI S. RAVI
Steel Authority of India Limited, New Delhi	SHRI V. M. SHASTRI SHRI K. B. LAL (<i>Alternate</i>)
Thapar Centre for Industrial Research and Development, Patiala	DR MAHESHWAR ROY DR S. K. CHAKRABORTY (<i>Alternate</i>)
The Fertilizer Association of India, New Delhi	DR (SMT) B. SWAMINATHAN DR S. NAND (<i>Alternate</i>)
Town and Country Planning Organization, Ministry of Urban Development, New Delhi	SHRI K. K. JOADDER DR SANTOSH VIDHYA DHARAN (<i>Alternate</i>)
BIS Directorate General	DR U. C. SRIVASTAVA, Scientist 'F' and Head (CHD) [Representing Director General (<i>Ex-officio</i>)]

Member Secretary
SHRI N. K. PAL
Director (CHD), BIS

Bureau of Indian Standards

BIS is a statutory institution established under the *Bureau of Indian Standards Act, 1986* to promote harmonious development of the activities of standardization, marking and quality certification of goods and attending to connected matters in the country.

Copyright

BIS has the copyright of all its publications. No part of these publications may be reproduced in any form without the prior permission in writing of BIS. This does not preclude the free use, in the course of implementing the standard, of necessary details, such as symbols and sizes, type or grade designations. Enquiries relating to copyright be addressed to the Director (Publications), BIS.

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'.

This Indian Standard has been developed from Doc: No. CHD 32 (1299).

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.org.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 : SCO 335-336, Sector 34-A, CHANDIGARH 160022	{ 260 3843 260 9285
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
Branches : AHMEDABAD. BANGALORE. BHOPAL. BHUBANESHWAR. COIMBATORE. FARIDABAD. GHAZIABAD. GUWAHATI. HYDERABAD. JAIPUR. KANPUR. LUCKNOW. NAGPUR. PARWANOO. PATNA. PUNE. RAJKOT. THIRUVANANTHAPURAM. VISAKHAPATNAM.	