भारतीय मानक

Indian Standard

IS 3025 (Part 47) : 2024

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

भाग 47 सीसा

(दूसरा पुनरीक्षण)

Methods of Sampling and Test (Physical and Chemical) for Water and Wastewater

Part 47 Lead

(Second Revision)

ICS 13.060.50

भारतीय मानक ब्यूरो
BUREAU OF INDIAN STANDARDS
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April 2024 **Price Group 6**

Water Quality Sectional Committee, CHD 36

FOREWORD

This Indian Standard (Part 47) (Second Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Water Quality Sectional Committee had been approved by the Chemical Division Council.

Lead is a serious cumulative body poison. Natural waters seldom contain more than 20 µg/1, although values as high as 400 µg/l have been reported. Lead in a water supply may come from industrial, mine and smelter discharges or from the dissolution of old lead plumbing. Tap waters that are soft, acidic and not suitably treated may contain lead resulting from an attack on lead service pipes. It is toxic and therefore, a stringent limit has been specified for lead in potable water.

Also, lead is to be specially tested when pollution/plumbo solvency is suspected. Therefore, the test for lead is essential. These tests serve to determine whether the lead content of potable water and waste water is within the acceptable limit or not.

The Committee responsible for formulation of IS 3025 : 1964 'Methods of sampling and test (physical and chemical) for water used in industry' decided to revise the standard and publish it in separate parts. This standard was one of the different parts published under IS 3025 series of standards and superseded **37** of IS 3025 : 1964. The first revision was published in 1994.

In this revision the following changes have been incorporated:

a) Amendments issued have been incorporated;

b) Dithizone method has been removed as the reagent such as KCN used in this method, are hazardous in nature;

c) Inductively coupled plasma (ICP) spectroscopy methods have been incorporated;

d) References, ICS No. have been updated; and

e) Other editorial changes have been done to bring the standard in the latest style and format of Indian Standards.

In the preparation of this standard, considerable assistance has been derived from the Method No. 3500-Pb A, 3111 B and C, and 3130 B of ―Standard methods for the examination of water and wastewater, published by the American Public Health Association, Washington, USA, 23rd edition, 2017.

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

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

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

PART 47 LEAD

(Second Revision)

1 SCOPE

This standard (Part 47) prescribes the following methods for determination of lead in water and wastewater:

a) Atomic absorption method (Direct) (method is applicable in range from 1.0 mg/l to 10.0 mg/l of lead);

b) Atomic absorption method (Chelation-Extraction) (method is applicable in range from 0.1 mg/l to 1.0 mg/l of lead);

c) Differential pulse anodic stripping voltammetry (DPASV) (method is applicable in range from 0.001 mg/l to 0.1mg/l of lead); and

d) Inductively coupled plasma (ICP) spectroscopy methods.

2 REFERENCES

The standards listed in Annex A contain provisions which, through reference in this text, constitute provisions of this standard. At the time of publications, 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.

3 TERMINOLOGY

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

4 APPLICATION

Depending upon the concentration range and interference levels, choice of the method is made. When the concentration levels are below 300 µg/l, pre-concentration is carried out either by chelation and extraction prior to atomic absorption spectrophotometer (AAS) or by deposition on a mercury drop electrode as in DPASV method. For dissolved lead content, filtration through 0.45 µm membrane filter is required.

5 SAMPLING AND PRESERVATION

Sampling and sample preservation shall be done as prescribed in IS 17614 (Part 1) and IS 17614 (Part 3). The sampling bottles should be cleaned thoroughly with dilute nitric acid (6 N) prior to final rinsing with water. The water samples should be collected and stored preferably in polypropylene or chemically resistant glass containers. For preservation, the samples should be acidified with concentrated nitric acid (2 ml of AR grade nitric acid in 1 litre of the sample just to bring down the *p*H to below 2). For dissolved lead filter the sample in the field and acidify the filtrate with nitric acid to a *p*H of 2 or lower.

NOTE — Avoid excess nitric acid. Add 5 ml of 0.1 N iodine solution to avoid losses of volatile organo-lead compounds during handling and digestion of samples.

6 DIRECT AIR-ACETYLENE FLAME ATOMIC ABSORPTION METHOD

6.1 Principle

The lead content of the sample is determined by directly aspirating the sample into the flame of an atomic absorption spectrophotometer. This method is applicable in the range from 1.0 mg/l to 10.0 mg/l of lead. However, the concentration range will vary with the sensitivity of the instrument used.

6.2 Interferences

Other metals usually do not interfere. However, high concentrations of calcium do interfere and give high values for lead. In these cases, the chelation-extraction procedure should be used. Background correction should be applied.

6.3 Apparatus

Atomic absorption spectrophotometer set up and equipped with an appropriate burner for air-acetylene flame and a hollow cathode lamp for lead with wavelength of 283.3 nm.

6.4 Reagents

6.4.1 Unless otherwise specified, only AR grade chemicals should be used for all the tests.

6.4.2 *Air* — clean, dried and free from oil, water and other foreign substances. The source may be a compressor or commercially bottled gas.

6.4.3 *Acetylene* — standard commercial grade.

CAUTION — Acetylene gas is an explosive threat in the laboratory. In plumbing and utilizing this gas, follow the manufacturer's instructions. Allow no gas contact with copper, brass containing more than 65 percent copper, silver or liquid mercury. Use copper or brass tubing, regulators, and fittings that contain more than 65 percent copper.

6.4.4 *Lead-Free Distilled Water* — should be used for preparing standards and reagent solution (*see* IS 1070).

6.4.5 *Nitric Acid* (*HNO_3*) — 2 percent (all *v/v*), 1 + 5, 1 + 1, and concentrated.

6.4.6 *Stock Lead Solution*

Completely dissolve 0.159 8 g of lead nitrate using the least possible quantity of 1 + 1 nitric acid solution. Then, add 10.0 ml of concentrated nitric acid and subsequently dilute the mixture with 1 000 ml of water (1.00 ml = 100 µg of Pb).

6.4.7 *Standard Lead Solution*

Prepare a series of standard lead solution by diluting stock lead solution 6.4.6 with water containing 1.5 ml of concentrated HNO_3/l. Stock standard solution can be taken from a number of commercial supplier. Alternatively, it can be prepared as described in 6.4.6.

6.5 Procedure

6.5.1 *Sample Preparation*

Samples containing particulate matter after acidification shall be filtered to prevent clogging of the nebulizer and burner systems.

6.5.2 *Instrument Operation*

6.5.2.1 It is difficult to formulate instructions as applicable to every instrument, because of differences between makes and models of atomic absorptions spectrometers. See manufacturer’s operating manual.

6.5.2.2 Install a hollow-cathode lamp for lead in the instrument and set the wavelength at 283.3 nm. Set the slit width as suggested by manufacturer, for lead being measured. Turn on the instrument and apply current to hollow cathode lamp for 10 min to 20 min to stabilize the energy source. After adjusting the wavelength, install burner head. Turn on the air 6.4.2 and adjust the flow rate as suggested by the manufacturer to give maximum sensitivity for lead being measured. Turn on acetylene 6.4.3 and adjust flow rate. Ignite the flame and let it stabilize for a few min.

6.5.2.3 Aspirate a blank consisting of distilled water 6.4.4 containing 1.5 ml of concentrated nitric acid. Set zero the instrument. Aspirate the lead standard solution. Check the aspiration rate of nebulizer and adjust it to obtain maximum sensitivity. Obtain maximum response by adjusting burner position vertically and horizontally. Aspirate the blank and re-zero the instrument. Aspirate standard lead solution near middle of linear range. Prepare a fresh same standard and with a new hollow cathode lamp, record the absorbance of this standard. Check consistency of instrument setup and aging of hollow-cathode lamp and standard referring to these data on subsequent determinations of lead.

6.5.2.4 The instrument is now ready to operate. At the end of analysis, first switch off acetylene than air.

6.5.3 *Standardization*

Prepare at least three standard lead solutions containing lower concentration 0.1 mg/l of lead to bracket the expected metal concentration of a sample. Prepare a reagent blank of 100 ml volume with distilled water 6.4.4 containing 1.5 ml of concentrated nitric acid 6.4.5. Aspirate the reagent blank and carry out zero adjustment. Aspirate blank and zero the instrument. Aspirate each standard solution in turn into the flame and record the absorbance readings. Most modern instruments are equipped with micro-processors and digital read-out which permit calibration in direct concentration terms.

6.5.4 *Analysis of Sample*

Rinse nebulizer by aspirating distilled water 6.4.4 containing 1.5 ml concentrated nitric acid/l and zero instrument. Aspirate a sample and determine its absorbance.

6.6 Calculation

Prepare a calibration curve on linear graph paper by plotting absorbance of the standard reading versus their original concentration of standards. Alternatively, read the concentration directly from the instrument read-out if the instrument is so equipped. If sample has been diluted, multiply by the appropriate dilution factor.

7 ATOMIC ABSORPTION METHOD (CHELATION-EXTRACTION)

7.1 Principle

The given method is suitable for the detection of low concentrations of lead in water and wastewater. This method uses ammonium pyrrolidine dithiocarbamate (APDC) as the chelating agent,followed by extraction into methyl isobutyl ketone after the aspiration into an air-acetylene flame.

7.2 Apparatus — *see* 6.3

7.3 Reagents

7.3.1 Unless otherwise specified, only AR grade chemicals should be used for all the tests.

7.3.2 *Air* — clean, dried and free from oil, water and other foreign substances. The source may be a compressor or commercially bottled gas.

7.3.3 *Acetylene* — standard commercial grade

7.3.4 Lead-free distilled water should be used for preparing standards and reagent solution.

7.3.5 *Methyl Isobutyl Ketone* (*MIBK*) — reagent grade

7.3.6 *Nitric Acid* — concentrated, ultrapure

7.3.7 *Sodium Sulphate* — anhydrous

7.3.8 *Ammonium Pyrolidine Dithiocarbamate Solution*

Dissolve 4 g of Ammonium pyrolidine dithiocarbamate in 100 ml water. If necessary purify the salt with an equal volume of MIBK. In a separating funnel, shake it for 30 sec. Withdraw a lower portion and discard MIBK layer.

7.3.9 *Water saturated MIBK*

In a separating funnel, mix one part of purified MIBK with 1 part of water. Shake it off for 30 sec, than allow it to settle. Save MIBK layer and discard aqueous layer.

7.3.10 *Stock Lead Solution*

Completely dissolve 0.159 8 g of lead nitrate using the least possible quantity of 1 + 1 nitric acid solution. Then, introduce 10.0 ml of concentrated nitric acid and subsequently dilute the mixture with 1 000 ml of water (1.00 ml = 100 µg of Pb).

7.3.11 *Standard Lead Solution*

Prepare a series of standard lead solution by diluting stock lead solution 7.3.10 with water containing 1.5 ml of concentrated HNO_3/l. Stock standard solution can be taken from a number of commercial supplier. Alternatively, it can be prepared as described in 7.3.10.

7.3.12 *Bromophenol Blue Indicator Solution*

Dissolve 0.1 g of bromophenol blue in 100 ml of 50 percent ethanol or isopropanol.

7.4 Procedure

7.4.1 *Instrument Operation*

7.4.1.1 It is difficult to formulate instructions as applicable to every instrument, because of differences between makes and models of atomic absorptions spectrometers. See manufacturer's operating manual.

7.4.1.2 Install a hollow-cathode lamp for lead in the instrument and set the wavelength at 283.3 nm. Set the slit width as suggested by manufacturer for lead being measured. Turn on the instrument and apply current to hollow cathode lamp for 10 min to 20 min to stabilize the energy source. After the adjustment of final position of the burner, aspirate the water-saturated MIBK Solution into the flame and then gradually reduce the flow of the fuel until the flame is similar to the pre-aspiration of the solvent.

7.4.2 *Standardization*

7.4.2.1 A minimum of three concentrations of standard lead solutions (*see* 7.3.11) are required to be selected to bracket the expected lead concentration and to be, in the optimum range of concentration of the instrument, after extraction. The *p*H of 100 ml of a lead-free water blank and 100 ml of standard is adjusted to a *p*H of (2.3 ± 0.2) by addition of 1 N Nitric acid or 1 N sodium hydroxide, as required.

7.4.2.2 Each type of standard solution and blank needs to be transferred into 200 ml of volumetric flasks, followed by addition of 1 ml of ammonium pyrolidine dithiocarbamate solution to each of the flasks and shaken well to mix. Now add 10 ml of MIBK to each of the flasks, followed by vigorous shaking, for about 30 sec. (The maximum volume ratio of sample to MIBK is 40). Let the content of every flask to settle and separate into organic and aqueous layers, then carefully add water down the side of each flask to bring the organic layer to the neck so that it is accessible to the aspirating tube. Set zero on the instrument at water-saturated MIBK blank. Now aspirate the organic extract into the flame directly and record the absorbance.

7.4.2.3 Prepare a calibration curve of absorbance vs concentrations before extraction on a linear graph paper.

7.4.3 *Sample Analysis*

7.4.3.1 Prepare the sample in a similar way, as the standards. Aspirate water saturated MIBK to rinse the atomizer. Now aspirate the organic extract as treated above into the flame directly and record the absorbance.

7.4.3.2 During the extraction, if any emulsion is formed at the interface of water-MIBK, add anhydrous sodium sulphate in order to obtain the homogeneous organic phase. In that case, add sodium sulphate needs to be added into all the blanks and standards. The lead need to be determined immediately after the extraction process in order to avoid the problems related to the instability of extracted complexes.

8 DIFFERENTIAL PULSE ANODIC STRIPPING VOLTAMMETRY (DPASV)

8.1 Principle

Lead is deposited on a hanging mercury drop at a negative (− ve) potential of − 0.6 V versus saturated calomel electrode (SCE). Then the lead is stripped back into the solution by applying a positive (+ ve) potential scan. The anodic current peak which is measured is representative of the lead concentration in the sample. For total dissolved lead the sample is filtered through a 0.45 µm membrane filter paper prior to acidification and analysis. This method is applicable in the concentration range 1.0 µg/l of lead to 100 µg/l of lead.

8.2 Interferences

Selenium interferes when it is present in excess of 50 µg/l. This may be overcome by adding ascorbic acid which reduces selenium (IV) to selenium metal. Iron (III) interferes when present at levels greater than lead. However, this may be overcome by warming the solution with hydroxylamine. Also, the presence of any other neighbouring stripping peaks which is less than 100 mV from that of the lead will interfere.

8.3 Apparatus

8.3.1 Polarographic instrumentation capable of performing differential pulse work.

8.3.2 *Hanging Mercury Drop Electrode*

8.3.3 *Platinum Counter Electrode*

8.3.4 *Saturated Calomel Reference Electrode*

8.3.5 *Magnetic Stirrer Control Unit, Stirring Bar*

8.4 Reagents

8.4.1 *Hydrochloric Acid* — concentrated

8.4.2 *Nitric Acid* — concentrated

8.4.3 *Nitric Acid* — diluted (1 : 1)

8.4.4 *Lead Solutions*

8.4.4.1 *Stock lead solution*

Dissolve 0.319 8 g of lead nitrate in water containing 1 ml of concentrated nitric acid. Dilute to one litre with water (1 ml = 200 µg of Pb).

8.4.4.2 *Intermediate lead solution*

Dilute 10 ml of lead stock solution and 1 ml of nitric acid to one litre with water (1 ml = 2 µg of Pb).

8.4.4.3 *Standard lead solution*

Dilute 10 ml of lead intermediate solution and 1 ml of concentrated nitric acid to 100 ml with water (1 ml = 0.2 µg of Pb). This solution should be prepared just before use for preparing the working standards.

8.4.5 *Amalgamated Zin*

Cover 10 g of granular zinc with water and add 2 drops of concentrated hydrochloric acid. Then add 5 to 8 drops of mercury with continuous shaking.

8.4.6 *Purified Nitrogen*

Boil 2 g of ammonium meta vanadate with 25 ml of concentrated hydrochloric acid. Dilute to 250 ml and transfer to the scrubber. Add 10 g to 15 g of amalgamated zinc. Pass nitrogen gas through the scrubber for removal of traces of oxygen and through distilled water for washing any traces of scrubber chemicals (Fig. 1).

8.5 Procedure

Clean all the glassware and the voltametric cells by soaking them overnight in concentrated nitric acid and rinse them thoroughly with distilled water. If total dissolved lead alone is to be determined, the sample should be filtered through 0.45 µm membrane filter paper. For total recoverable lead, digest the sample with 3 ml each concentrated hydrochloric acid and nitric acid. Evaporate the solution to 15 ml to 20 ml. Cool and make up to 100 ml in a volumetric flask. Take 10 ml of the sample in the polarographic cell and de-aerate for 15 min. The cell should be covered with nitrogen gas during the experiment (Fig. 2) Generate a new droplet of mercury and put the stirrer on. Connect the cell and deposit at − 0.6 V versus SCE for 3 min. Stop the stirrer and wait for 30 sec. Start the anodic scan with the following settings:

Initial potential	− 0.4 V *vs* saturated calomel electrode (SLE)
Scan rate	5 mV/s
Scan direction	+
Modulation amplitude	25 mV
Current range	1 µA to 10 µA
Droptime	5 sec
Display direction	‘—’
Low pass filter	Off position
Mode	Differential pulse
Scan range	0.75 V

Measure the current peak height (I_1). Add 20 µl of standard copper (II) solution and de-aerate for 5 min. Repeat as above. Measure the current peak height (I_2).

8.6 Calculation

$$C_{\text{sample}}, \frac{\text{mg}}{\text{l}} = \frac{I_1 * V_{\text{sample}} \times C_{\text{std}} * 1\,000}{I_2 v + (I_2 - I_1) V_{\text{sample}}}$$

where

I_1 = current peak height for sample;

I_2 = current peak height for sample + standard;

v = volume of standard added (20 µl);

V_{sample} = volume of the sample solution, in ml;

C_{std} = concentration of the standard solution added; and

C_{sample} = concentration of lead in the sample.

9 INDUCTIVELY COUPLED PLASMA SPECTROSCOPY

Lead can also be determined by inductively coupled plasma optical emission spectroscopy with reference to procedure given in IS 3025 (Part 2). Likewise, inductively coupled plasma mass spectroscopy with reference to procedure given in IS 3025 (Part 65) can also be used for the determination of lead in water and wastewater.

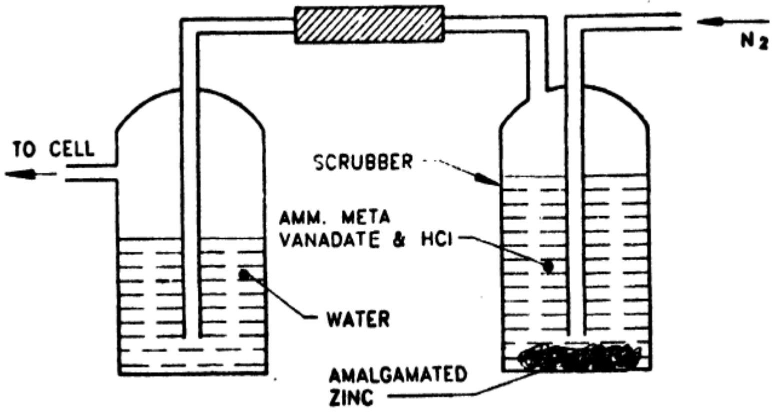

FIG. 1 SCRUBBER ASSEMBLY FOR NITROGEN PURIFICATION

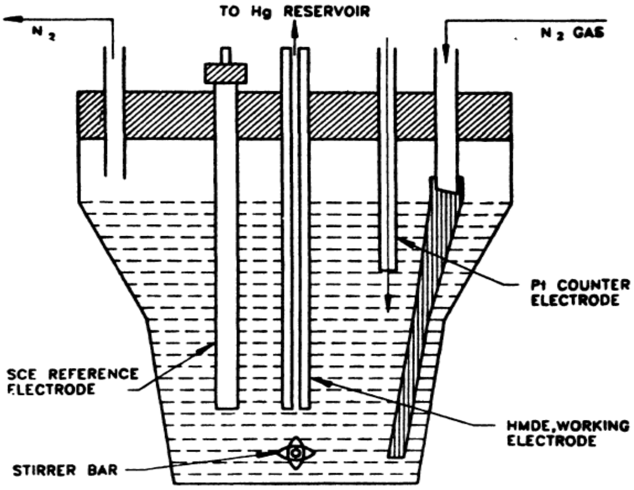

FIG. 2 VOLTAMMETRIC CELL ASSEMBLY

ANNEX A

(*Clause* 2)

LIST OF REFERRED STANDARDS

IS No.	*Title*
IS 1070 : 2023	Reagent grade water — Specification (*fourth revision*)
IS 3025	Methods of sampling and test (physical and chemical) for water and wastewater:
(Part 2) : 2019/ ISO 11885 : 2007	Determination of selected elements by inductively coupled plasma optical emission spectrometry (ICP-OES) (*first revision*)
(Part 65) : 2022/ ISO 17294-2) : 2016	Application of inductively coupled plasma mass spectrometry (ICP-MS) — Determination of selected elements including uranium isotopes (*first revision*)
IS 7022 (Part 1) : 1973	Glossary of terms relating to water, sewage and industrial effluents: Part 1
IS 7022 (Part 2) : 1979	Glossary of term relating to water, sewage and industrial effluents: Part 2
IS 17614	Water quality — Sampling:
(Part 1) : 2021/ ISO 5667-1) : 2020	Guidance on the design of sampling programmes and sampling techniques
(Part 3) : 2021/ ISO 5667-3) : 2018	Preservation and handling of water samples

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ANNEX B

(*Foreword*)

COMMITTEE COMPOSITION

Water Quality Sectional Committee, CHD 36

Organization	*Representative(s)*
Chief Scientist, Environment Protection Training and Research Institute, Hyderabad	Dr N. Raveendhar **(*Chairperson*)**
Andhra Pradesh Pollution Control Board, Vijayawada	Shrimati M. Sreeranjan Shrimati A. Sri Samyuktha (*Alternate*)
Bhabha Atomic Research Centre, Mumbai	Shri S. Jayakumar Shri Manoj Mohapatra (*Alternate*)
Bharat Heavy Electricals Limited, Haridwar	Shri Shailendra Kumar Shri Sudhir Bhartiya (*Alternate* I) Shri Avinash Kumar (*Alternate* II)
Bureau of Indian Standards, New Delhi	Ms Nitasha Doger
CII Triveni Water Institute, New Delhi	Shri Kapil K. Narula Dr Sipika Chauhan (*Alternate*)
Central Pollution Control Board, New Delhi	Shri P. K. Mishra Shri Vishal Gandhi (*Alternate*)
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CSIR - Indian Institute of Toxicology Research, Lucknow	Dr Satyakam Patnaik (*Alternate*)
CSIR - National Environmental Engineering Research Institute, Nagpur	Dr Noor Afshan Khan Dr Paras R. Pujari (*Alternate*)
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Envirocare Laboratories Private Limited, Thane	Dr Priti Amritkar Dr Nilesh Amritkar (*Alternate*)
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Haryana State Pollution Control Board, Panchkula	Shri Jatinfer Pal Singh
Himachal Pradesh Pollution Control Board, Shimla	Er Praveen Gupta Shri Praveen Sharma (*Alternate*)

Organization	*Representative(s)*
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Indian Chemical Council, Mumbai	Shri J. Sevak Shri Dhrumil Soni (*Alternate*)
Indian Water Works Association, Mumbai	Shri Vijay Carhate
Karnataka State Pollution Control Board, Bengaluru	Dr H. Rupadevi Ms Gouri Golsangi (*Alternate*)
Maharashtra State Pollution Control Board, Mumbai	Dr Vishwajeet Thakur Dr Smita N. Wagh (*Alternate*)
Ministry of Environment Forest and Climate Change, New Delhi	Dr Sonu Singh
Ministry of Jal Shakti, Department of Drinking Water and Sanitation, New Delhi	Shri Sumit Priyadarshi (*Alternate*)
NTPC Limited, New Delhi	Dr Vijay Prakash Shri Himat Desur Gojiya (*Alternate*)
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BIS Directorate General	Shri Ajay Kumar Lal, Scientist 'F'/Senior Director And Head Chemical (Chemical) [Representing Director General (*Ex-officio*)]

Member Secretary
Ms Shubhanjali Umrao
Scientist 'B'/Assistant Director
(Chemical), 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 website-www.bis.gov.in or www.standardsbis.in.

This Indian Standard has been developed from Doc No.: CHD 36 (23138).

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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Published by BIS, New Delhi