भारतीय मानक

Indian Standard

IS 3025 (Part 41) : 2023

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

भाग 41 कैडमियम

(दूसरा पुनरीक्षण)

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

Part 41 Cadmium

(Second Revision)

ICS 13.060.50

भारतीय मानक ब्यूरो
BUREAU OF INDIAN STANDARDS
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July 2023 **Price Group 6**

Water Sectional Committee, CHD 36

FOREWORD

This Indian Standard (Part 41) (Second Revision) was adopted by the Bureau of Indian Standards after the draft finalized by the Water Quality Sectional Committee and approved by the Chemical Division Council.

Cadmium is highly toxic and has been implicated in some cases of poisoning through food. Minute quantities of cadmium are suspected of being responsible for adverse changes in arteries of human kidneys. Cadmium may enter water as a result of industrial discharges or the deterioration of galvanized water supply pipe.

The Technical 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 among the different parts published under IS 3025 series of standards. The first revision of this standard was published in 1992.

In this second revision the following changes have been incorporated:

a) Inductively coupled plasma method has been incorporated;
b) References, ICS No. have been updated; and
c) 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 methods 3111 B, 3111 C of — Standard Methods for the Examination of Water and Wastewater, published by the American Public Health Association, Washington, USA, 23nd Edition, 2017.

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

In reporting the results of a test or analysis in accordance with this standard, if the final valueobserved 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 41 CADMIUM

(Second Revision)

1 SCOPE

This standard (Part 41) describes the following four methods for the determination of cadmium in water and wastewater:

a) Direct air-acetylene flame method;
b) Extraction/air-acetylene flame method;
c) Differential pulse anodic stripping voltammetry; and
d) Inductively coupled plasma method.

2 REFERENCES

The following standards given below 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 editions of these standards:

IS No.	*Title*
IS 3025	Methods of sampling and test (Physical And Chemical) for water and wastewater
(Part 2) : 2019	Determination of selected elements by inductively coupled plasma optical emission spectrometry (ICP-OES)
(Part 65) : 2022	Application of inductively coupled plasma mass spectrometry (ICP-MS) Determination of 62 elements
IS 7022 (Part 1) : 1973	Glossary of terms relating to water, sewage and industrial effluents: Part 1
IS 7022 (Part 2) : 1979	Glossary of terms relating to water, sewage and industrial effluents: Part 2
IS 17614	Water Quality — Sampling:
(Part 1) : 2021	Guidance on the design of sampling programmes and sampling techniques
(Part 3) : 2021	Preservation and handling of water samples

3 TERMINOLOGY

For the purpose of this standard, the 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 17614 (Part 1) and IS 17614 (Part 3). The sample bottles should be cleaned thoroughly with dilute nitric acid (6 N) prior to the final rinsing with water. The water samples should be collected and stored for 24 h 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 to 1 litre just to bring down the *p*H below 2). Un-acidified samples should be analysed on the same day while the acidified samples may be stored for a few days in a refrigerator.

5 QUALITY QF REAGENTS

5.1 Unless specified otherwise, pure chemicals shall be used.

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

5.2 Double distilled water with a specific conductivity less than 1 µS/cm shall be used for preparing the standard and reagent solutions.

6 DIRECT AIR-ACETYLENE FLAME METHOD

6.1 Principle

The cadmium content of the sample is determined by atomic absorption spectrophotometry. For dissolved cadmium, the filtered sample is directly aspirated to the atomizer. For total recoverable cadmium, a pre-treatment with HCl is carried out prior to aspiration of the sample. This method is applicable in the range from 0.05 mg/l to 2 mg/l of cadmium. However, the concentration range will vary with the sensitivity of the instrument used.

6.2 Interferences

Nickel, lead, copper zinc, cobalt and chromium do not interfere up to 10 mg/l. Alkali and alkaline earth metals can be tolerated up to 5 000 mg/l. Iron does not interfere up to 4 000 mg/l.

6.3 Apparatus

6.3.1 Atomic absorption spectrometer and associated equipment.

6.3.2 Cadmium hollow-cathode lamp or multi-element hollow-cathode lamp for use at 228.8 nm.

6.3.3 Use burner head as recommended by the manufacturer.

6.3.4 Standard volumetric glassware.

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.

6.4.4 *Cadmium*, free distilled water should be used for preparing standards and reagent solution.

6.4.5 *Hydrochloric Acid* (HCl), 1 percent, 10 percent, 20 percent (all v/v), 1 + 5, 1 + 1, and concentrated.

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

6.4.7 *Stock Cadmium Solution*

Dissolve 0.100 g cadmium metal in 4 ml concentrated HNO_3. Add 8.0 ml concentrated HNO_3, dilute to 1 000 ml with the water (1.00 ml = 100 µg Cd).

6.4.8 *Standard Cadmium Solution*

Prepare a series of standard cadmium solutions in the optimum concentration range by appropriate dilutions of stock cadmium solution **6.4.7** with distilled water **6.4.4** containing 1.5 ml of conc. nitric acid.

6.5 Procedure

6.5.1 *Instrument Operation*

6.5.1.1 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.

6.5.1.2 Install a hollow-cathode lamp for cadmium in the instrument and set the wavelength at 228.8 nm. Set the slit width as suggested by manufacturer for cadmium 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 and adjust the flow rate as suggested by the manufacturer to give maximum sensitivity for cadmium being measured. Turn on acetylene and adjust flow rate as specified by the manufacturer. Ignite the flame and let it stabilize for a few min.

6.5.1.3 Aspirate a blank consisting of distilled water **6.4.4** containing 1.5 ml of concentrated nitric acid. Set zero the instrument. Aspirate the standard cadmium 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 cadmium 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 cadmium.

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

6.5.2 *Standardization*

Prepare at least three standards cadmium solutions containing lower concentration 0.1 mg/l Cd to bracket the expected metal concentration of a sample. Prepare a reagent blank of 100 ml volume with distilled water containing 1.5 ml of concentrated nitric acid. 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.

6.5.3 *Analysis of Sample*

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

6.6 Calculation

Prepare a calibration curve on linear graph paper by plotting absorbance reading versus their reading. 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 EXTRACTION/AIR-ACETYLENE FLAME METHOD

7.1 General

Cadmium is chelated with pyrolidine dithiocarbamic acid and extracted with methyl isobutyl ketone (MIBK). The extract is treated with hot nitric acid after evaporating to dryness, then dissolved in hydrochloric acid and diluted with water. An aliquot is aspirated into the air-acetylene flame of the spectrophotometer. For total recoverable cadmium, an acid digestion procedure is carried out prior to aspiration of the sample.

This method is applicable in the concentration range of 5 µg/l to 200 µg/l cadmium.

7.2 Interferences — Same as in **6.2**

7.3 Apparatus

7.3.1 Atomic absorption spectrometer and associated equipment.

7.3.2 Use burner head as recommended by the manufacturer.

7.4 Reagents

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

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

7.4.3 *Acetylene*, standard commercial grade

7.4.4 *Cadmium*, free distilled water should be used for preparing standards and reagent solution

7.4.5 *Methyl Isobutyl Ketone* (*MIBK*), *Reagent Grade*

For trace analysis purify MIBK by re-distillation or by sub-boiling distillation.

7.4.6 *Nitric Acid*, concentrated, ultrapure

7.4.7 *Sodium Sulphate*, anhydrous

7.4.8 *Ammonium Pyrolidine Dithiocarbamate (APDC) 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.

7.4.9 *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.

7.4.10 *Potassium Permanganate Solution*, 5 percent (w/v), aqueous

7.4.11 *Stock Cadmium Solution*, Prepare as in **6.4.7**

7.4.12 *Intermediate Cadmium Solution*

Add 1 ml of concentrated nitric acid to 50 ml of stock solution and dilute to 1 liter with water (1 ml = 50 µg of cadmium).

7.4.13 *Standard Cadmium Solution*

To 10 ml of cadmium intermediate solution add 1 ml of concentrated nitric acid and dilute to 1 liter with water (1 ml = 0.5 µg of cadmium).

7.5 Procedure

7.5.1 *Instrument Operation*

7.5.1.1 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.

7.5.1.2 Install a hollow-cathode lamp for cadmium in the instrument and set the wavelength at nm. Set the slit width as suggested by manufacturer for cadmium 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.

7.5.2 *Standardization*

7.5.2.1 A minimum of three concentrations of standard cadmium solutions **6.4.8** are required to be selected to bracket the expected cadmium concentration and to be, in the optimum range of concentration of the instrument, after extraction. The *p*H of 100 ml of a cadmium free water blank and 100 ml of standard is adjusted to a *p*H of 1 to 6 by addition of 1 N Nitric acid or 1 N sodium hydroxide, as required.

7.5.2.2 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.

7.5.2.3 Set zero on the instrument at water-saturated MIBK blank. Now aspirate the organic extract into the flame directly and record the absorbance.

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

7.5.3 *Sample Analysis*

7.5.3.1 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.

7.5.3.2 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 cadmium 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

8.1 Principle

Cadmium is deposited on a hanging mercury drop electrode at a negative potential of 0.8 V versus saturated calomel electrode. Then the cadmium is stripped back into the solution by applying a positive potential scan. The anodic current peak which is measured is representative of the cadmium concentration in the sample. For total dissolved cadmium the sample is filtered through Whatman No. 40 filter paper prior to acidification and analysis. This method is applicable in the concentration range of 0.1 µg/l to 100 µg/l.

8.2 Interferences

Selenium interferes when its concentration exceeds 50 µg/l. This can be overcome by adding ascorbic acid which reduces selenium (IV) to selenium metal. Iron (III) interferes when present at levels greater than cadmium. However, this can be overcome by warming the solution with hydroxylamine. The presence of any other neighboring stripping peaks which is less than 100 mV from that of cadmium 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 *PIatinum Counter Electrode*

8.3.4 *Saturated Calomel Reference Electrode (SCE)*

8.3.5 *Magnetic Stirrer Control Unit*, Stirring bar

8.4 Reagents

8.4.1 Hydrochloric Acid, Concentrated; spectrograde

8.4.2 *Nitric Acid*, Concentrated; spectrograde

8.4.3 *Hydroxylamine Solution*, 100 g/l

Dissolve 5 g of hydroxylamine hydrochloride in 50 ml of water

8.4.4 *Dilute Nitric Acid*, 1 : 1

8.4.5 *Ascorbic Acid*, 100 mg/l

Dissolve 10 g of L-ascorbic acid in 100 ml of water.

8.4.6 *Cadmium Solutions*

8.4.6.1 *Stock cadmium solution*

Dissolve 1.0 of pure cadmium metal by warming with 10 ml of concentrated hydrochloric acid and 30 ml of 1 : 1 nitric acid. Dilute to 1 litre with distilled water.

8.4.6.2 *Intermediate cadmium solution*

Add 1 ml of concentrated nitric acid to 10 ml of stock solution and dilute to 1 liter with distilled water.

8.4.6.3 *Standard cadmium solution*

Dilute 10 ml of cadmium intermediate solution to 100 ml with distilled water. Prepare daily for use. (1 ml = 10 µg of cadmium).

8.4.7 *Amalgamated Zinc*

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

8.4.8 *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).

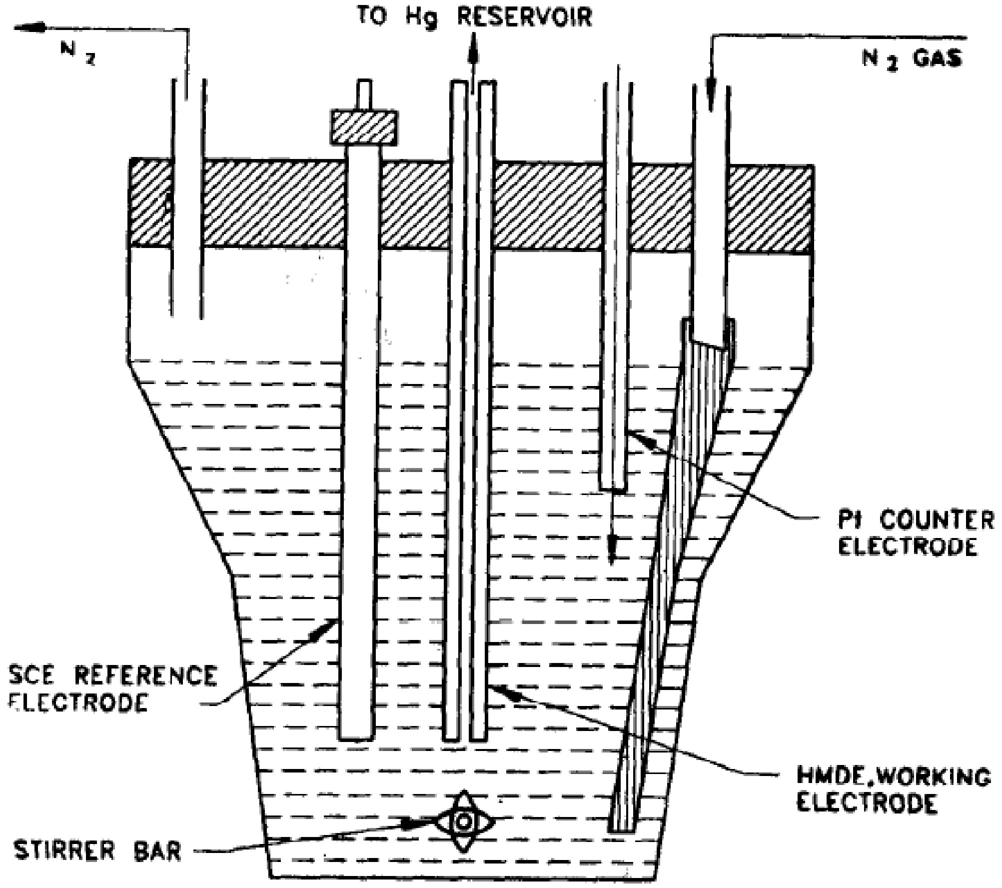

FIG. 1 SCRUBBER ASSEMBLY FOR NITROGEN PURIFICATION

8.5 Procedure

Clean all the glassware and the voltammetric cells by soaking them overnight in concentrated nitric acid and rinsing them thoroughly with water. Add 1 ml of hydroxylamine solution. Warm for 15 min to reduce the ferric concentration. Add 1 ml of ascorbic acid. Evaporate gently. Cool and make up to 100 ml. If total dissolved cadmium alone is to be determined, the sample should be filtered through 0.45 µm membrane filter paper. For total recoverable cadmium, digest the sample with 3 ml each of concentrated hydrochloric acid and nitric acid. Evaporate the solution to 15 ml to 20 ml. Cool and make up to 100 ml in a standard 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 - 0.80 V versus saturated calomel electrode for 3 min. Stop the stirrer and wait for 30 sec. Start the anodic scan with the following settings:

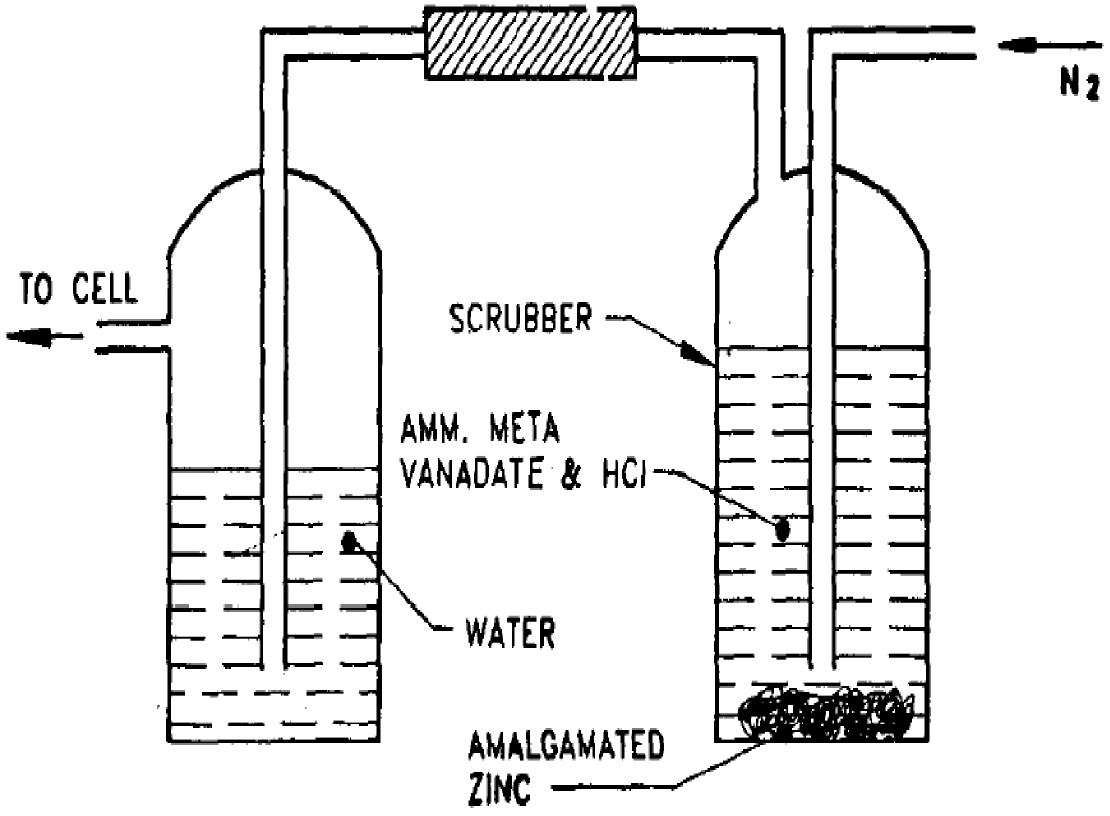

FIG. 2 VOLTAMMETRIC CELL ASSEMBLY

Initial potential	-0.80 V vs saturated calomel electrode (SCE)
Scan rate	5 mV
Scan direction	(+)
Modulation amplitude	25 mV
Current range	1 µA to 10 µA
Droptime	5 sec
Display direction	(–)
Law pass filter	Off position
Mode	Differential pulse
Scan range	0.75 V

Measure the current peak height (I_1). Add 20 µl of standard cadmium solution and deaerate for 5 min. Repeat as above, Measure the current peak height (l_2)

8.6 Calculation

$$\text{Cadmium, } \frac{\mu g}{l} = \frac{I_1 V C s t d}{I_2 v + (I_2 - I_1) V} \times 1\,000$$

where

I_1 = current peak height for sampl;

V = volume of the sample solution in ml;

$Cstd$ = concentration of the standard solution added;

I_2 = current peak height for sample and standard; and

v = volume of standard added (20 µl).

9 INDUCTIVELY COUPLED PLASMA SPECTROSCOPY

Cadmium 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 cadmium in water and wastewater.

ANNEX A

(*Foreword*)

COMMITTEE COMPOSITION

Water Quality Sectional Committee, CHD 36

Organization	*Representative(s)*
Chief Scientist, EPTRI, Hyderabad	Shri N. Raveendhar **(*Chairperson*)**
Andhra Pradesh Pollution Control Board, Vijaywada	Shrimati M. Sreeranjani Shrimati A. Sri Samyuktha (*Alternate*)
Bhabha Atomic Research Centre, Mumbai	Dr S. K. Sahu Shri I. V. Saradhi (*Alternate*)
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Ministry of Jal Shakti Department of Drinking Water and Sanitation	Shri D. A. Rajasekhar Shri Sumit Priyadarshi (*Alternate*)
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In Personal Capacity (*H. No. 1-78/2/S/121/1, Sathi Reddy Nagar colony, Boduppal, Hyderabad, - 500092*)	Shri N. Murali Mohan
BIS Directorate General	Shri Ajay Kumar Lal, Scientist 'F'/Senior Director and Head (Chemical) [Representing Director General (*Ex-officio*)]

Member Secretary
Ms Shubhanjali Umrao
Scientist 'B'/Assistant Director
(Chemical), BIS

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Review of Indian Standards

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