

जल एवं अपशिष्ट जल के नमूने लेने तथा
परीक्षण (भौतिक एवं रसायन) की पद्धतियाँ
भाग 49 जस्ता
(दूसरा पुनरीक्षण)

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

Part 49 Zinc
(Second Revision)

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FOREWORD

This Indian Standard (Part 49) (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.

Zinc is an essential and beneficial element in body growth. Concentration above 5 mg/l may cause a bitter astringent taste and opalescence in alkaline water. Zinc most commonly enters the domestic water supply from deterioration of galvanized iron and dezincification of brass. Zinc in water may also come from industrial water pollution.

The Committee responsible for formulation of IS 3025 : 1964 'Methods of sampling and test (physical and chemical) for water used in industry' and IS 2488 (Part 2) : 1968 'Methods of sampling and test for industrial effluents, Part II' decided to revise the standards and publish it in separate parts. This standard was one of the different parts published under IS 3025 series of standards and superseded **17** of IS 2488 (Part 2) : 1968 and **39** of IS 3025 : 1964.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) Zincon method has been deleted, as the reagents involved in this method, such as potassium cyanide (KCN), is a hazardous chemical and deadly poison;
- 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-Zn 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 49 ZINC***(Second Revision)***1 SCOPE**

1.1 This standard (Part 49) prescribes the following methods for determination of zinc in water and wastewater:

- a) Atomic absorption method (direct) (method is applicable in range from 0.01 mg/l of Zn to 2.0 mg/l of Zn);
- b) Atomic absorption method (chelation-extraction) (method is applicable in range from 1 µg/l of Zn to 200 µg/l of Zn);
- c) Differential pulse anodic stripping voltammetry (DPASV) (method is applicable in range from 1.0 µg/l of Zn to 100 µg/l of Zn); and
- d) Inductively coupled plasma (ICP) spectroscopy methods.

1.2 Depending upon the concentration range and interference levels, choice of the method is made. When the concentration levels are below 200 µg/l, preconcentration 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 zinc content, filtration through 0.45 µm membrane filter is required.

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 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 shall be cleaned thoroughly with dilute nitric acid (6 N) prior to the 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 of nitric acid in 1 litre just to bring down the pH below 2). Unacidified samples should be analyzed the same day while the acidified samples can be stored for a few days (5 days) in a refrigerator.

5 ATOMIC ABSORPTION METHOD (DIRECT)**5.1 Principle**

The zinc content of the sample is determined by atomic absorption spectrophotometry. For dissolved zinc, the filtered sample is directly aspirated to the atomizer. For total recoverable zinc, an acid digestion procedure is done prior to aspiration of the sample. This method is applicable in the range from 0.01 mg/l to 2.0 mg/l. However, the concentration range will vary with the sensitivity or the instrument used.

5.2 Interferences

Cadmium, lead, copper, nickel, cobalt and chromium up to 10 mg/l do not interfere. Alkali and alkaline earth metals can be tolerated up to 4 000 mg/l. Iron interferes at concentrations of 50 mg/l and above by suppressing, the zinc absorption.

5.3 Apparatus

5.3.1 *Atomic Absorption Spectrometer with Air-Acetylene Flame*

5.3.2 Multi-element hollow-cathode lamps or electrodeless discharge lamps for use at 213.9 nm.

5.4 Reagents

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

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

5.4.3 Acetylene — standard commercial grade.

CAUTION – Acetylene gas is an explosive threat in the laboratory. In plumbing and utilising 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

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

5.4.5 Nitric Acid (HNO₃) — 2 percent (all v/v), 1 + 5, 1 + 1, and concentrated

5.4.6 Hydrochloric Acid — 1 percent, 10 percent, 20 percent, 1 + 5, 1 + 1, concentrated

5.4.7 Stock Zinc Solution

Completely dissolve 0.100 g of zinc metal in 20 ml of 1 + 1 HCl. subsequently dilute the mixture with 1 000 ml of water (1.00 ml = 100 µg of Zn).

5.4.8 Standard Lead Solution

Prepare a series of standard lead solution by diluting stock zinc solution (5.4.7) with water containing 1.5 ml of concentrated HNO₃/l. Stock standard solution can be taken from a number of commercial supplier. Alternatively, it can be prepared as described in 5.4.7.

5.5 Procedure

5.5.1 Sample Preparation

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

5.5.2 Instrument Operation

5.5.2.1 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.

5.5.2.2 Install a hollow-cathode lamp for zinc in the instrument and set the wavelength at 213.9 nm. Set the slit width as suggested by manufacturer, for zinc 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 (5.4.2) and adjust the flow rate as suggested by the manufacturer to give maximum sensitivity for zinc being measured. Turn on acetylene (5.4.3) and adjust flow rate. Ignite the flame and let it stabilize for a few minutes.

5.5.2.3 Aspirate a blank consisting of distilled water (5.4.4) containing 1.5 ml of concentrated nitric acid. Set zero the instrument. Aspirate the zinc 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 zinc 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 zinc.

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

5.5.3 Standardization

Prepare at least three standard zinc solutions containing lower concentration 0.1 mg/l of zinc to bracket the expected metal concentration of a sample. Prepare a reagent blank of 100 ml volume with distilled water (5.4.4) containing 1.5 ml of concentrated nitric acid (5.4.5). 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.

5.5.4 Analysis of Sample

Rinse nebulizer by aspirating distilled water (5.4.4) containing 1.5 ml concentrated nitric acid/l and zero instrument. Aspirate a sample and determine its absorbance.

5.6 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.

6 ATOMIC ABSORPTION METHOD (CHELATION – EXTRACTION)

6.1 Principle

6.1.1 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.

6.1.2 This method is applicable for the concentration range from 1 µg/l of Zn to 200 µg/l of Zn.

NOTE — The lower range of determination to the extent of 0.001 mg/l may be obtained by graphite system.

6.2 Interferences — same as in [5.2](#)

6.3 Apparatus — same as in [5.3](#)

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

6.4.5 Methyl Isobutyl Ketone (MIBK) — reagent grade

6.4.6 Nitric Acid — concentrated, ultrapure

6.4.7 Sodium Sulphate — anhydrous

6.4.8 Ammonium Pyrrolidine Dithiocarbamate Solution

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

6.4.9 Water Saturated MIBK

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

6.4.10 Stock Zinc Solution

Completely dissolve 0.100 g of zinc metal in 20 ml of 1 + 1 HCl. subsequently dilute the mixture with 1 000 ml of water (1.00 ml = 100 µg of Zn).

6.4.11 Standard Zinc Solution

Prepare a series of standard lead solution by diluting stock zinc solution ([6.4.10](#)) with water containing 1.5 ml of concentrated HNO₃/l. Stock standard solution can be taken from a number of commercial supplier. Alternatively, it can be prepared as described in [6.4.10](#).

6.4.12 Bromophenol Blue Indicator Solution

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

6.4 Procedure

6.4.1 Instrument Operation

6.4.1.1 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.

6.4.1.2 Install a hollow-cathode lamp for zinc in the instrument and set the wavelength at 213.9 nm. Set the slit width as suggested by manufacturer for zinc 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.

6.4.2 Standardization

6.4.2.1 A minimum of three concentrations of standard zinc solutions (*see* [6.4.11](#)) are required to be selected to bracket the expected zinc concentration and to be, in the optimum range of concentration of the instrument, after extraction. The pH of 100 ml of a zinc free water blank and 100 ml of standard is adjusted to a pH of 2 to 6 by addition of 1 N Nitric acid or 1 N sodium hydroxide, as required.

6.4.2.2 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 pyrrolidine 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 s. (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.

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

6.4.3 Sample Analysis

6.4.3.1 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.

6.4.3.2 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 zinc need to be determined immediately after the extraction process in order to avoid the problems related to the instability of extracted complexes.

7 DIFFERENTIAL PULSE ANODIC STRIPPING VOLTAMMETRY (DPASV)

7.1 Principle

7.1.1 Zinc is deposited on a hanging mercury drop at a negative potential of -1.2 V vs saturated calomel electrode (SCE). Then the zinc is stripped back into the solution by applying a + ve potential scan. The anodic current peak which is measured is representative of the zinc concentration in the sample. For total dissolved zinc the sample is filtered through a $0.45 \mu\text{m}$ membrane filter paper prior to acidification and analysis.

7.1.2 This method is applicable in the concentration range $1.0 \mu\text{g/l}$ of zinc to $100 \mu\text{g/l}$ of zinc.

7.2 Interferences

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

7.3 Apparatus

7.3.1 *Polarographic Instrumentation Capable of Performing Differential Pulse Work*

7.3.2 *Hanging Mercury Drop Electrode*

7.3.3 *Platinum Counter Electrode*

7.3.4 *Saturated Calomel Reference Electrode*

7.3.5 *Magnetic Stirrer Control Unit, Stirring Bar*

7.4 Reagents

7.4.1 *Hydrochloric Acid* — concentrated

7.4.2 *Nitric Acid* — concentrated

7.4.3 *Nitric Acid* — diluted (1 : 1)

7.4.4 *Zinc Solutions*

7.4.4.1 *Stock zinc solution* — prepare as in [6.4.10](#)

7.4.4.3 *Zinc standard solution* — prepare as in [6.4.11](#)

7.4.5 *Amalgamated Zinc*

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.

7.4.6 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).

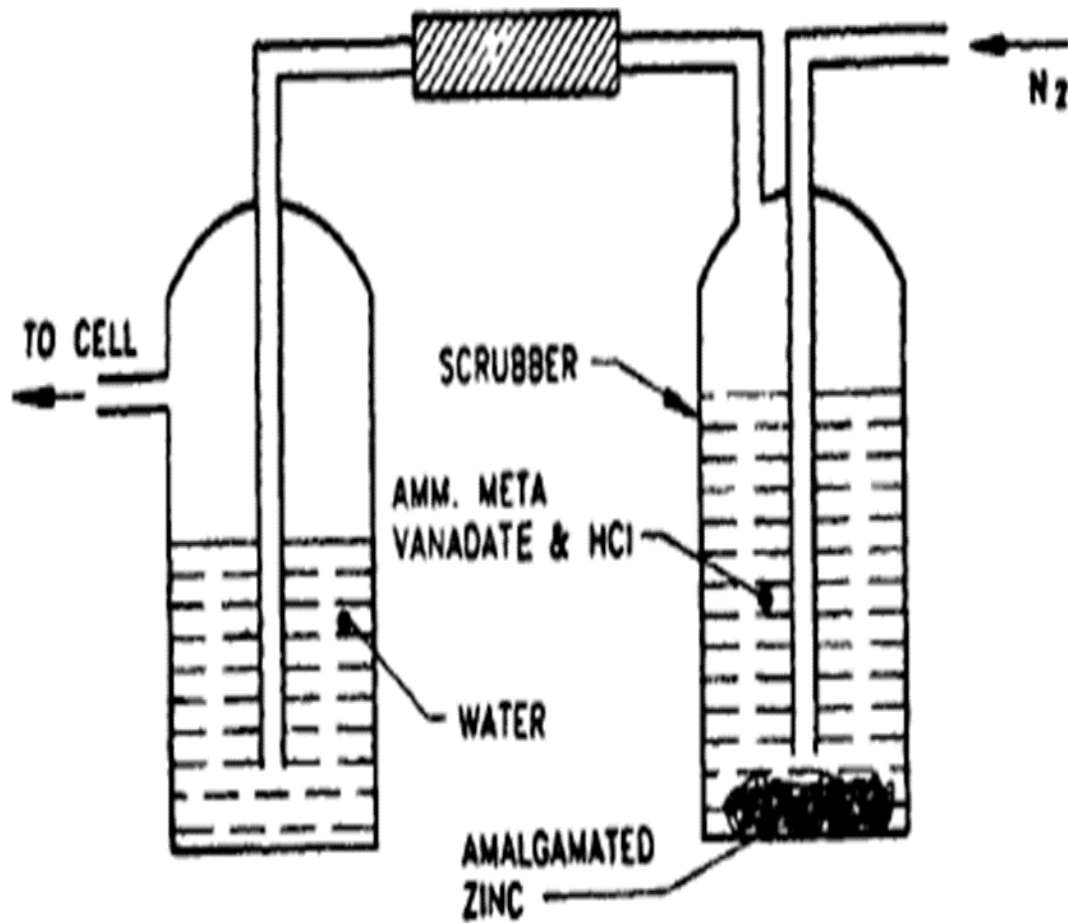


FIG. 1 SCRUBBER ASSEMBLY FOR NITROGEN PURIFICATION

7.5.1 Clean all the glassware and the voltammetric cells by soaking them overnight in concentrated nitric acid and rinsing them thoroughly with distilled water. If total dissolved zinc alone is to be determined, the sample should be filtered through 0.45 μm membrane filter paper. For total recoverable zinc, digest the sample with 3 ml each of concentrated hydrochloric acid and nitric acid.

7.5.2 Generate a new droplet of mercury and put the stirrer on. Connect the cell and deposit at -1.2 V versus SCE for 3 min. Stop the stirrer and wait for 30 s. Start the anodic scan with the following settings:

Initial potential	- 1.2 V versus SCE
Scan rate	5 mV/s
Scan direction	+ ve
Modulation amplitude	25 mV
Current range	1 μA to 10 μA
Drop time	0.5 s
Display direction	- ve
Low pass filter	off position
Mode	Differential pulse
Scan range	-1.2 to -0.6 V

Measure the current peak height (I). Add 20 μl of standard zinc solution and deaerate for 5 min. Repeat as above. Measure the current peak height (I_2).

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 deaerate for 15 min. The cell should be covered with nitrogen gas during the experiment (Fig. 2).

7.6 Calculation

$$C_{\text{sample}}, \frac{\text{mg}}{\text{l}} = \frac{I_1 V C_{\text{std}} \times 1000}{I_2 v + (I_2 - I_1) V}$$

where

C_{sample} = Concentration of zinc in the sample, mg/l.

I_1 = Current peak height for the sample;

V = Volume of the sample solution, in ml;

C_{std} = Concentration of the standard solution added.

I_2 = Current peak height for the sample + standard; and

v = Volume of standard added (20 μl);

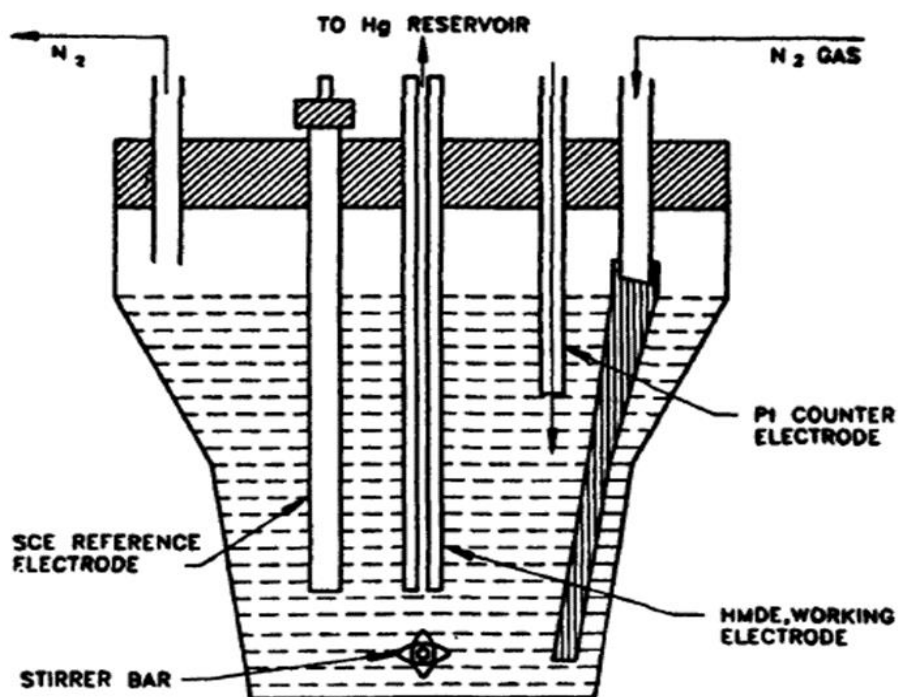


FIG. 2 VOLTAMMETRIC CELL ASSEMBLY

8 INDUCTIVELY COUPLED PLASMA SPECTROSCOPY

Zinc 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 zinc in water and wastewater.

ANNEX A

(Clause 2)

LIST OF REFERRED STANDARDS

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

ANNEX B

(Foreword)

COMMITTEE COMPOSITION

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Chief Scientist, Environment Protection Training and Research Institute, Hyderabad	DR N. RAVEENDHAR (<i>Chairperson</i>)
Andhra Pradesh Pollution Control Board, Vijayawada	SHRIMATI M. SREERANJAN SHRIMATI A. SRI SAMYUKTHA (<i>Alternate</i>)
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Bureau of Indian Standards, New Delhi	SHRIMATI NITASHA DOGER
Central Pollution Control Board, New Delhi	SHRI P. K. MISHRA SHRI VISHAL GANDHI (<i>Alternate</i>)
CII Triveni Water Institute, New Delhi	SHRI KAPIL K. NARULA DR SIPIKA CHAUHAN (<i>Alternate</i>)
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CSIR - Indian Institute of Chemical Technology, Hyderabad	DR S. SRIDHAR DR NIVEDITA SAHU (<i>Alternate</i>)
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CSIR - National Environmental Engineering Research Institute, Nagpur	DR NOOR AFSHAN KHAN DR PARAS R. PUJARI (<i>Alternate</i>)
CSIR - National Institute of Oceanography, Dona Paula	SHRI DURBAR RAY
Envirocare Laboratories Private Limited, Thane	DR PRITI AMRITKAR DR NILESH AMRITKAR (<i>Alternate</i>)
Federation of All India Packaged Drinking Water Manufacturers Associations (FIPMA), New Delhi	SHRIMATI APURVA NARENDRA DOSHI SHRI NAVEEN GOEL (<i>Alternate</i>)
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Haryana State Pollution Control Board, Panchkula	SHRI JATINFER PAL SINGH
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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.

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Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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