IS 3025 (Part 49) : 1994
(Reaffirmed 1999)
Edition 2.1
(2000-10)

भारतीय मानक

जल और अपशिष्ट जल (भौतिक एवं रसायनिक) के नमूने लेने तथा परीक्षण

भाग 49 जिंक

(पहला पुनरीक्षण)

Indian Standard

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

PART 49 ZINC

(First Revision)

(Incorporating Amendment No 1)

UDC 628.1.032 : 628.3 : 543.3 [546.47]

BUREAU OF INDIAN STANDARDS
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Price Group 4

Environmental Protection Sectional Committee, CHD 012

FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Environmental Protection 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. In the preparation of this standard considerable assistance has been derived from American Standard Test Methods (ASTM Annual Book Section 11, 1983) and Analytica Chimica Acta, 164 (1984) pp 1-21. The assistance so derived is thankfully acknowledged. This standard supersedes **17** of IS 2488 (Part 2) : 1968 and **39** of IS 3025 : 1964.

This edition 2.1 incorporates Amendment No. 1 (October 2000). Side bar indicates modification of the text as the result of incorporation of the amendment.

In reporting the result 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*)'.

The composition of the technical committee responsible for the formulation of this Indian Standard is given in Annex A.

Indian Standard

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

PART 49 ZINC

(First Revision)

1 SCOPE

This standard prescribes following four methods for determination of zinc:

a) Zincon Method,

b) Atomic Absorption Method (Direct),

c) Atomic Absorption Method (Chelation and Extraction), and

d) Differential Pulse Anodic Stripping Voltammetry (DPASV).

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

2.1 The Indian Standards listed below contain provisions which through reference in this text, constitute provision 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 standards are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	*Title*
3025 (Part 1) : 1986	Methods of sampling and test (physical and chemical) for water and wastewater : Part 1 Sampling (*first revision*)
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 II

3 SAMPLING AND PRESERVATION

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 analysed the same day while the acidified samples can be stored for a few days (5 days) in a refrigerator.

4 PURITY OF THE REAGENTS

4.1 Unless otherwise indicated, only AR/GR grade chemicals should be used for all the tests.

4.2 Double distilled water, with a specific conductivity of less than 1.0 μmho/cm should be used for preparing the standards and reagent solutions.

5 ZINCON METHOD

5.1 Principle

Zinc (II) forms a soluble blue complex with 2-carboxy-2-hydroxy-5-sulfoform-azyl benzene (zincon) at pH 9.0. The coloured complex obeys Beer's law and is suitable for spectrophotometric measurements.

This method is applicable in the range from 0.02 - 5 mg/l of zinc.

5.2 Interferences

Though many heavy metals react with zincon and interfere in the analysis, treatment of the sample as given in **5.5.1** with cyanide and chlorohexanone masks many of the interfering metal ions. The following ions interfere beyond the concentrations given against each:

Sl No.	*Ion*	*Concentration, mg/l*
i)	Cd^{2+}	1
ii)	Al^{3+}	5
iii)	Mn^{2+}	5
iv)	Fe^{3+}	7
v)	Fe^{2+}	9
vi)	Cr^{3+}	10

vii)	Ni^{2+}	20
viii)	Cu^{2+}	30
ix)	Co^{2+}	30
x)	CrO^4	50

5.3 Apparatus

5.3.1 *Spectrophotometer* — for use at 620 nm with 1 cm cells.

5.4 Reagents

5.4.1 *Sodium Hydroxide Solution* — 40 g/l.

Dissolve 40 g of sodium hydroxide (NaOH) in 500 ml of water and make up to 1 litre.

5.4.2 *Sodium Hydroxide Solution* — 240 g/l.

Dissolve 24 g of sodium hydroxide (NaOH) in 25 ml of water. Dilute to 100 ml with water.

5.4.3 *Potassium Cyanide Solution*

Dissolve 1 g of potassium cyanide (KCN) in 50 ml of water and dilute to 100 ml.

NOTE — Potassium cyanide is a deadly poison. Avoid skin contact or inhalation of vapours. Do not pipette by mouth or bring in contact with acids.

5.4.4 *Cyclohexanone Solution*

Dissolve 1 ml of purified cyclohexanone in 50 ml of water.

5.4.5 *Zincon Solution*

Dissolve 0.325 g of zincon reagent in 100 ml of methanol by heating gently. Cool. Dilute to 250 ml with methanol in a 250 ml standard flask. Store the reagent in a brown coloured bottle.

5.4.6 *Sodium Ascorbate*

Needed only when manganese content is more than 0.2 ppm.

5.4.7 *Borate Buffer Solution*

Dilute 213 ml of 1M NaOH solution to 500 ml with water and dissolve 37.3 g of potassium chloride and 31 g of boric acid. Make up to 1 litre in a standard flask.

5.4.8 *Hydrochloric Acid* — Concentrated.

5.4.9 *Zinc (II) Solutions*

Dissolve 0.274 5 g of zinc sulphate ($ZnSO_4 \cdot 7H_2O$) in 200 ml of water and dilute to 1 litre. 1 ml = 0.1 mg of Zn.

5.5 Procedure

5.5.1 For dissolved zinc, filter the sample through 0.45 μm membrane filter paper. For total zinc, add 1 ml of concentrated hydrochloric acid to 50 ml of sample and boil for 5 minutes. Cool the solution and adjust the *p*H to 7 with sodium hydroxide solution (**5.4.2**). Make up the solution to 50 ml in a standard flask. Take 10 ml of this solution or the filtrate (in the case of dissolved zinc) in an Erlenmeyer flask. Add 0.5 g of sodium ascorbate, 1 ml of cyanide solution, 5 m of buffer solution, 3 ml of zincon solution and 1 ml of cyclohexanone solution in the above order with sufficient precaution. Make up the solution to 500 ml. Prepare a reagent blank by treating 50 ml of double distilled water in the same way as described above.

5.5.2 Measure the optical density of the sample solution at 620 nm against the reagent blank containing added zinc and prepared in the same way except for the addition of 3 ml of chloral hydrate.

5.5.3 *Calibration*

Treat 50 ml portions of standard solutions containing 0.02, 0.05, 0.1, 0.5, 1.0, and 5.0 mg/l of zinc and treat as above and measure the absorbance. Plot absorbance versus milligram of zinc for the standards to get a calibration graph. Read the concentration of zinc in the sample from the calibration graph.

5.6 Calculation

$$\text{Zinc, mg/l} = \frac{M}{V} \times 1\,000$$

where

M = mass of zinc present in mg in the sample, and

V = volume of sample in ml.

6 ATOMIC ABSORPTION METHOD (DIRECT)

6.1 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 to 2.0 mg/l. However, the concentration range will vary with the sensitivity of the instrument used.

6.2 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.

6.3 Apparatus

6.3.1 *Atomic Absorption Spectrophotometer with Air-Acetylene Flame*

6.3.2 Multi-element hollow-cathode lamps or electrodeless discharge lamps for use at 213.8 nm.

6.4 Reagents

6.4.1 *Hydrochloric Acid* — Concentrated.

6.4.2 *Nitric Acid* — Concentrated.

6.4.3 *Nitric Acid* — Diluted (1 : 499).

6.4.4 *Zinc (II) Solutions*

6.4.4.1 *Stock zinc (II) solution*

Dissolve 1 g of acid washed and rinsed zinc granules or 1.245 g of zinc oxide (ZnO) in 20 ml of 1 : 1 nitric acid. Dilute to 1 : 1 with water. 1 ml = 1.0 mg of Zn.

6.4.4.2 *Standard zinc (II) solution*

Dilute 100 ml of zinc stock solution and 1 ml of nitric acid to 1 litre with water.

6.5 Procedure

6.5.1 Add 0.5 ml of nitric acid to 100 ml of the sample (filtered or unfiltered). If total recoverable zinc is to be determined, add 5 ml of concentrated hydrochloric acid and filter the sample through acid washed filter paper. Make up to 100 ml in a volumetric flask, aspirate the solution and measure the absorbance at 213.8 nm. Aspirate nitric acid (1 : 499) prior to sample aspiration.

6.5.2 Prepare a reagent blank and sufficient standards containing 0.01, 0.05, 0.1, 0.5, 1.0 and 2.0 mg/l of zinc by diluting suitable volume of the standard solution with nitric acid (1 : 499) and repeat as above. Aspirate the solutions and measure the absorbance.

6.6 Calculations

6.6.1 Construct a standard calibration graph by plotting the absorbance versus standard concentration for each standard. Read the concentration of the samples from the graph.

$$\text{Zinc, mg/l} = \frac{M}{V} \times 1\,000$$

where

M = mass of zinc present in mg in the sample, and

V = volume of sample in ml.

7 ATOMIC ABSORPTION METHOD (CHELATION — EXTRACTION)

7.1 Scope and Application

Zinc is chelated with pyrrolidine dithio carbamic acid and extracted with chloroform. The extract is treated with hot nitric acid after evaporating to dryness, dissolved in hydrochloric acid and diluted with water. An aliquot is aspirated into the air-acetylene flame of the spectrophotometer. For total recoverable zinc an acid digestion procedure is done prior to aspiration of the sample.

This method is applicable for the concentration range from 1-200 μg/l.

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

7.2 Interferences — Same as in **6.2**.

7.3 Apparatus — Same as in **6.3**.

7.4 Reagents

7.4.1 *Hydrochloric Acid* — Concentrated.

7.4.2 *Hydrochloric Acid* — Diluted (1 : 2).

7.4.3 *Hydrochloric Acid* — Diluted (1 : 49).

7.4.4 *Nitric Acid* — Concentrated.

7.4.5 *Pyrrolidine Dithio Carbamic Acid* — Chloroform reagent:

36 ml of pyrrolidine is mixed with 1 litre of chloroform. The solution is cooled and 30 ml of carbon disulphide is added in small fractions with continuous stirring. Dilute with 1 litre of chloroform and store in a cool and dark place. The reagent is stable for at least six months.

NOTE — As components of this mixture are highly toxic and flammable, prepare and use in a fume hood.

7.4.6 *Sodium Hydroxide Solution*

Dissolve 100 g of sodium hydroxide in water and dilute to 1 litre with water.

7.4.7 *Chloroform*

7.4.8 *Bromophenol Blue Indicator Solution*

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

7.4.9 *Stock Zinc (II) Solution*

Dissolve 1 g of acid washed and rinsed zinc granules or 1.245 g of zinc oxide (ZnO) in 20 ml of 1 : 1 HNO_3. Dilute to 1 litre with water. 1 ml = 1.0 mg of zinc.

7.4.10 *Intermediate Zinc Solution*

Dilute 100 ml of zinc stock solution and 1 ml of nitric acid to 1 litre with water.

7.4.11 *Standard Zinc Solution*

Dilute 10 ml of zinc intermediate solution and 1 ml of nitric acid to 1 litre with water. This solution should be prepared just before use.

7.5 Procedure

7.5.1 For dissolved zinc, filter 100 ml of the sample through 0.45 μm membrane filter paper. For total zinc, add 5 ml of concentrated nitric acid and evaporate the solution to 15 to 20 ml. Cool and filter the sample through acid washed filter paper. Make up to 100 ml in a volumetric flask. Add to this solution or the filtrate (in case of dissolved zinc) 2 drops of bromophenol blue indicator solution and mix. Adjust the *p*H by adding sodium hydroxide solution till a blue colour persists. Add diluted hydrochloric acid (1 : 50) drop by drop until the colour just disappears; then add 2.5 ml in excess to bring the *p*H to 2.3-2.5. Add 10 ml of pyrroline dithio carbamic acid — chloroform reagent and shake well. After the phases separate out, collect the chloroform phase by taking care to avoid any trace of water in a flask. Repeat the extraction till the chloroform layer becomes colourless with fresh 10 ml portions of chloroform and combine the extracts. Evaporate the solution just to dryness and dissolve the residue by dropwise addition of 2 ml of concentrated nitric acid by holding the beaker at an angle. Again evaporate to dryness and add 2 ml of hydrochloric acid (1 : 2) and heat for 1 minute. Cool and make up the solution in a 10 ml standard flask. Aspirate the sample and measure the absorbance.

7.5.2 Prepare a reagent blank and sufficient standards containing 10, 20, 50, 100 and 200 µg/l of zinc by diluting a suitable volume of the standard solution with 100 ml of water and repeat as above. Aspirate the solution and measure the absorbance.

7.6 Calculation

7.6.1 Construct a standard calibration graph by plotting the absorbance versus the micrograms of zinc. Read the concentration of the samples from the curve.

$$\text{Zinc, } \mu\text{g/l} = \frac{M}{V} \times 1\,000$$

where

M = mass of zinc present in the sample (in µg), and

V = volume of sample in ml.

8 DIFFERENTIAL PULSE ANODIC STRIPPING VOLTAMMETRY (DPASV)

8.1 Principle

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 µm membrane filter paper prior to acidification and analysis.

This method is applicable in the concentration range 1.0-100 µg/l of zinc.

8.2 Interferences

Selenium interferes when it is present in excess of 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 zinc. However this can 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 zinc 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 *Zinc Solutions*

8.4.4.1 *Stock zinc solution* — Prepare as in **7.4.9**.

8.4.4.2 *Intermediate zinc solution* — Prepare as in **7.4.10**.

8.4.4.3 *Zinc standard solution* — Prepare as in **7.4.11**.

8.4.5 *Amalgamated Zinc*

Cover 10 g of granular zinc with water and add 2 drops of concentrated hydrochloric acid. Then add 5-8 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 10-15 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

8.5.1 Clean all the glasswares 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. Evaporate the solution to 15-20 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 minutes. 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 – 1.2 V versus SCE for 3 minutes. Stop the stirrer and wait for 30 seconds. Start the anodic scan with the following settings:

Initial potential	– 1.2 V vs SCE
Scan rate	5 mV/s
Scan direction	+ ve
Modulation amplitude	25 mV
Current range	1-10 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_1). Add 20 μl of standard zinc solution and deaerate for 5 minutes. Repeat as above. Measure the current peak height (I_2).

8.6 Calculation

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

where

I_1 = current peak height for the sample,

I_2 = current peak height for the sample + standard,

v = volume of standard added (20 μl),

V = volume of the sample solution,

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

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

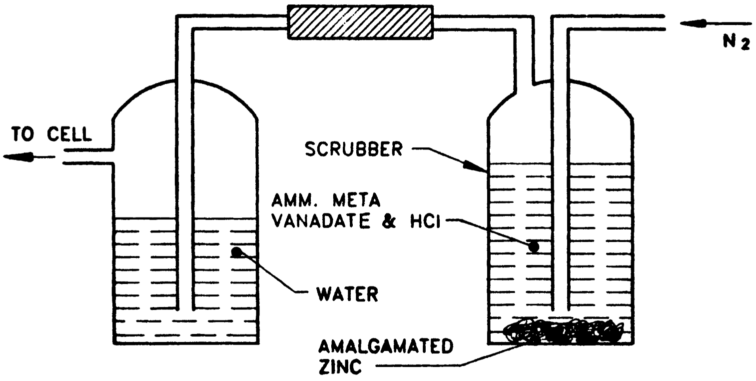

FIG. 1 SCRUBBER ASSEMBLY FOR NITROGEN PURIFICATION

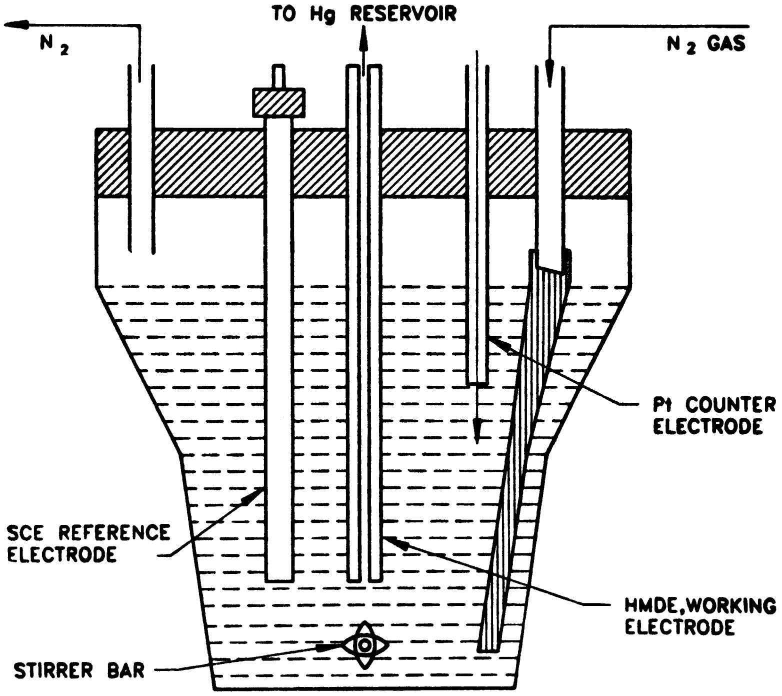

FIG. 2 VOLTAMMETRIC CELL ASSEMBLY

ANNEX A

(*Foreword*)

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(*Continued on page* 8)

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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'.

This Indian Standard has been developed from Doc : No. CHD 012 (172).

Amendments Issued Since Publication

Amend No.	Date of Issue
Amd. No. 1	October 2000

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