भारतीय मानक

IS 3025 (Part 42) : 2024

Indian Standard

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

भाग 42 ताँबा

(दूसरा पुनरीक्षण)

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

Part 42 Copper

(Second Revision)

ICS 13.060.50

भारतीय मानक ब्यूरो
BUREAU OF INDIAN STANDARDS
मानक भवन, 9 बहादुर शाह ज़फर मार्ग, नई दिल्ली - 110002
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI - 110002
www.bis.gov.in www.standardsbis.in

July 2024 **Price Group 7**

Water Quality Sectional Committee, CHD 36

FOREWORD

This Indian Standard (Part 42) (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.

Copper is found mainly as a sulphide, oxide or carbonate in the minerals. Copper enters the water system through mineral dissolution, industrial effluents, because of its use as algicide and insecticide and through corrosion of copper alloy water distribution pipes. It may occur in simple ionic form or in one of many complexes with groups, such as cyanides, chlorides, ammonia or organic ligands. The test for copper is essential because dissolved copper salts even in low concentrations are poisonous to some biota. Desirable limit for copper in potable water is 0.05 mg/l maximum which can be relaxed in the absence of better alternate source to 1.5 mg/l.

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 This standard was one of the different parts published under IS 3025 series of standards and superseded **36** of IS 3025 : 1964. The first revision was published in 1992.

In this second revision the following changes have been incorporated:

a) Amendment issued has been incorporated;

b) Bathocuproine method has been incorporated;

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-Cu A, B, C and 3111 B and C 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 42 COPPER

(Second Revision)

1 SCOPE

1.1 This standard (Part 42) prescribes the following methods for determination of copper in water and wastewater:

a) Neocuproine method (method is applicable in the concentration range of 0.05 mg/l to 5 mg/l of copper);

b) Bathocuproine method (method is applicable in the concentration range of 0.02 mg/l to 5 mg/l of copper);

c) Atomic absorption method (direct) (method is applicable in the concentration range of 0.02 mg/l to 5 mg/l of copper);

d) Atomic absorption method (chelation extraction) (method is applicable in the concentration range of 0.002 mg/l to 0.5 mg/l of copper);

e) Differential pulse anodic stripping voltammetry method; and

f) Inductively coupled plasma spectrometry method.

1.2 The choice or the method depends upon the concentration range and interference. When the concentration levels are below 500 g/l, pre-concentration is carried out either by chelation and extraction prior to atomic absorption spectrophotometer (AAS) or by depositing as in differential pulse anodic stripping voltammetry method. For dissolved copper 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 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 these standards.

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). Unacidified samples should be analyzed on the same day while the acidified samples may be stored for a few days in a refrigerator.

5 QUALITY OF REAGENTS

5.1 Unless specified otherwise, pure chemicals shall be used.

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

5.2 Double distilled water (*see* IS 1070) shall be used for preparing the standard and reagent solutions.

6 NEOCUPROINE METHOD

6.1 Principle

Copper (II) is reduced to copper (I) by hydroxyl amine hydrochloride and the *p*H of the solution is adjusted to 5 by sodium citrate solution. Copper (I) forms a soluble yellow complex with 2, 9-dimethyl-1,10-phenanthroline (neocuproine) suitable for spectrophotometric measurement. This method is applicable in the concentration range of 0.05 mg/l to 5 mg/l of copper.

6.2 Interferences

Chromium interferes when its concentration exceeds 5 times that of copper. The interference from organic matter, suspended solids, sulphide, cyanide and chromium can be eliminated by a preliminary sample treatment as given in **6.5.1**. The other commonly present ions do not interfere.

6.3 Apparatus

6.3.1 *Spectrophotometer* — for use at 457 nm with 1 cm cell

6.3.2 *Separatory Funnels* — 125 ml

6.4 Reagents

6.4.1 *Ammonium Hydroxide* — 5 N

Dilute 330 ml concentrated ammonium hydroxide (28 percent to 29 percent) to 1 000 ml with water. Store the mixture in polyethylene water.

6.4.2 *Chloroform* — AR grade

6.4.3 *Hydrochloric Acid* — concentrated

6.4.4 *Hydroxylamine Hydrochloride Solution*

Dissolve 50 g of hydroxylamine hydrochloride ($NH_2OH.HCl$) in 450 ml of water.

6.4.5 *Methanol* — reagent grade

6.4.6 *Neocuproine Solution*

Dissolve 0.1 g of neocuproine in 100 ml of methanol. This solution can be stored for a month under normal condition.

6.4.7 *Nitric Acid* — concentrated

6.4.8 *Sulphuric Acid* — concentrated

6.4.9 *Sodium Citrate Solution*

Dissolve 150 g of hydrated sodium citrate ($Na_3C_6H_6O_7.2H_2O$) in 400 ml of water. Add 10 ml each of neocuproine solution and 5 ml of hydroxylamine hydrochloride solution. Any impurities of copper can be removed by extraction with chloroform, discarding the chloroform layer.

6.4.10 *Copper* (II) *Solutions*

6.4.10.1 *Stock copper* (II) *solution*

Dissolve 0.2 g of pure copper metal by warming with 5 ml of 1 : 1 nitric acid. Add 1 ml of concentrated sulphuric acid and evaporate the solution to dryness. Dilute the solution to 1 litre with distilled water [1 ml = 200 µg of copper (II)].

6.4.10.2 *Standard copper* (II) *solution*

Dilute 100 ml of stock solution to 1 litre with distilled water [1 ml = 20 µg of copper (II)].

6.4.11 *Congo Red Paper*

6.5 Procedure

6.5.1 If interfering substances are present add 1 ml of concentrated sulphuric acid and 5 ml of concentrated nitric acid. Evaporate the sample to dense white sulphur trioxide fumes on a hot plate. Repeat the treatment with 5 ml or concentrated nitric acid and 5 ml of hydrogen peroxide and evaporate the solution to complete dryness. Dissolve the residue with 80 ml of water, boil, cool and filter. Adjust the *p*H with dropwise addition or ammonium hydroxide to 4 to 6. Add 0.2 ml of hydrochloric acid and dilute to 100 ml. If no interfering substance is present, just boil the acidified sample and cool.

6.5.2 *Extraction*

Transfer 50 ml of the acidified sample or filtrate (6.5.1) to a 125 ml separating funnel. Add 5 ml of hydroxylamine-hydrochloride solution and 10 ml of sodium citrate solution and 10 ml of neocuproine solution. Shake and mix it thoroughly. Adjust *p*H to 4 by adding ammonium hydroxide till congo red paper completely turns red. Add 10 ml of neocuproine reagent and 10 ml of chloroform and shake it for 30 s. Allow the mixture to separate into two layers. Collect the chloroform layer in a 25 ml dry flask. Repeat with separate 10 ml aliquot of chloroform. Combine the extracts and dilute to 50 ml with methanol. Prepare a reagent blank by treating 50 ml of double distilled water in the same way as described above.

6.5.3 Measure the optical density of the sample solution at 457 nm against the reagent blank. Treat 50 ml portions of standard solutions containing 0.02 mg to 0.2 mg of copper as above. Plot absorbance versus copper concentration (mg/l) for the standards to get a calibration graph. Read the concentration of copper in the sample from the calibration graph.

6.6 Calculation

$$\text{Concentration of copper,} \frac{\text{mg}}{\text{l}} = \frac{M}{V} \times 1\,000$$

where

M = mass of copper in the sample, in mg; and

V = volume of sample, in ml.

7 BATHOCUPROINE METHOD

7.1 Principle

The cuprous ion readily forms a water-soluble, orange-colored chelate when combined with bathocuproine disulphonate. This coloration occurs within a *p*H range of 3.5 to 11, but it is specifically recommended to maintain the *p*H between 4 to 5 for optimal results. To achieve this, the solution is buffered at a *p*H of 4.3. Once appropriately buffered, the cuprous ion is then reduced using hydroxylamine hydrochloride. This method is applicable in range of 0.02 mg/l to 5 mg/l of copper.

7.2 Apparatus

7.2.1 *Spectrophotometer* — for use at 484 nm with 1 cm cell.

7.2.2 *Nessler Tubes* — 100 ml

7.3 Reagents

7.3.1 *Stock Copper* (II) *Solution*

Dissolve 0.02 g of pure copper metal by warming with 5 ml of 1 : 1 nitric acid. Add 1 ml of concentrated sulphuric acid and evaporate the solution to dryness. Dilute the solution to 1 litre with distilled water [1 ml = 20 µg of copper (II)].

7.3.2 *Standard Copper* (II) *Solution*

Dilute 250 ml of stock solution to 1 litre with distilled water [1 ml = 5.0 µg of copper (II)].

7.3.3 *Hydrochloric Acid* — 1 + 1

7.3.4 *Hydroxylamine Hydrochloride Solution*

Dissolve 50 g of hydroxylamine hydrochloride ($NH_2OH.HCl$) in 450 ml of water.

7.3.5 *Sodium Citrate Solution*

Dissolve 300 g of hydrated sodium citrate ($Na_3C_6H_6O_7.2H_2O$) in 1 000 ml of water.

7.3.6 *Disodium Bathocuproine Disulphonate Solution*

Dissolve 1.00 g of disodium bathocuproine disulphonate in water and make up to 1 000 ml.

7.4 Procedure

7.4.1 First, pipet a 50 ml sample or an appropriate portion diluted to 50.0 ml into a 250 ml Erlenmeyer flask. In separate 250 ml Erlenmeyer flasks, prepare a 50 ml water blank and a series of 50 ml copper standard solutions containing 5 µg, 10 µg, 15 µg, 20 µg, and 25 µg of copper.

7.4.2 Next, for each sample, blank, and standard, add the following reagents one by one, ensuring proper mixing after each addition of 1 ml of HCl, 5.00 ml of hydroxylamine hydrochloride solution, 5.00 ml of sodium citrate solution, and 5 ml of disodium bathocuproine disulphonate.

7.4.3 Transfer each prepared solution to a suitable cell and measure the sample's absorbance against the blank at a wavelength of 484 nm.

7.4.4 Finally, plot the absorbance values against the micrograms of Cu in the standard solutions to create the calibration curve. This curve will help determine the copper concentration in future samples based on their absorbance readings.

7.5 Calculation

$$\text{Concentration of copper}, \frac{\text{mg}}{\text{l}} = \frac{A}{B} \times 1\,000$$

where

A = mass of copper in the sample, in mg; and

B = volume of sample, in ml.

8 ATOMIC ABSORPTION METHOD (DIRECT)

8.1 Principle

The copper content of the sample is determined by atomic absorption spectrophotometry. For dissolved copper, the filtered sample is directly aspirated into the atomizer. For total recoverable copper, an acid digestion procedure is carried out prior to aspiration of the sample. This method is applicable in the range (0.02 mg/l to 5 mg/l).

8.2 Interferences

Cadmium, lead, nickel, cobalt, manganese and chromium up to 10 mg/l do not interfere. Alkali and alkaline earth metals can be tolerated up to 5 000 mg. Iron does not interfere up to 1 000 mg/l.

8.3 Apparatus

8.3.1 *Atomic Absorption Spectrophotometer* — with air-acetylene flame

8.3.2 *Copper Hollow Cathode Lamp* — for use at 324.7 nm

8.4 Reagents

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

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

8.4.3 *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 copper.

8.4.4 *Copper-Free Distilled Water* — should be used for preparing standards and reagent solution.

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

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

8.4.7 *Stock Copper Solution*

Dissolve 0.100 g copper metal in 2 ml concentrated nitric acid. Add 10 ml of concentrated nitric acid and dilute to 1 000 ml with water (1.00 ml = 100 µg of Cu).

8.4.8 *Standard Copper Solution*

Prepare a series of standard copper solution by diluting stock copper solution (**8.4.7**) 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 **8.4.7**.

8.5 Procedure

8.5.1 *Sample Preparation*

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

8.5.2 *Instrument Operation*

8.5.2.1 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.

8.5.2.2 Install a hollow-cathode lamp for copper in the instrument and set the wavelength at 324.7 nm. Set the slit width as suggested by manufacturer for copper 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 (**8.4.2**) and adjust the flow rate as suggested by the manufacturer to give maximum sensitivity for copper being measured. Turn on acetylene (**8.4.3**) and adjust flow rate. Ignite the flame and let it stabilize for a few min.

8.5.2.3 Aspirate a blank consisting of distilled water (**8.4.4**) containing 1.5 ml of concentrated nitric acid. Set zero the instrument. Aspirate the copper 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 copper 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 copper.

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

8.5.3 *Standardization*

Prepare at least three standard copper solutions containing lower concentration 0.1 mg/l copper to bracket the expected metal concentration of a sample. Prepare a reagent blank of 100 ml volume with distilled water (**8.4.4**) containing 1.5 ml of concentrated nitric acid (**8.4.6**). 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.

8.5.4 *Analysis of Sample*

Rinse nebulizer by aspirating distilled water (**8.4.4**) containing 1.5 ml concentrated nitric acid/l and zero instrument. Aspirate a sample and determine its absorbance.

8.6 *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.

9 ATOMIC ABSORPTION METHOD (CHELATION-EXTRACTION)

9.1 General

The given method is suitable for the detection of low concentrations of iron 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.

9.2 Apparatus — *see* 8.3

9.3 Reagents

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

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

9.3.3 *Acetylene* — standard commercial grade

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

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

9.3.6 *Nitric Acid* — concentrated, ultrapure

9.3.7 *Sodium Sulphate* — anhydrous

9.3.8 *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 MBK. In a separating funnel, shake it for 30 s. Withdraw a lower portion and discard MBK layer.

9.3.9 *Water Saturated MIBK*

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

9.3.10 *Stock Copper Solution*

Dissolve 0.100 g copper metal in 2 ml concentrated nitric acid. Add 10 ml of concentrated nitric acid and dilute to 1 000 ml with water (1.00 ml = 100 µg of Cu).

9.3.11 *Standard Copper Solution*

Prepare a series of standard copper solution by diluting stock copper solution (**9.3.10**) with water containing 1.5 ml of concentrated HNO_3/l. Stock standard solution can be taken from a number of commercials supplier. Alternatively, it can be prepared as described in **9.3.10**.

9.3.12 *Bromophenol Blue Indicator Solution*

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

9.4 Procedure

9.4.1 *Instrument Operation*

9.4.1.1 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.

9.4.1.2 Install a hollow-cathode lamp for copper in the instrument and set the wavelength at 324.7 nm. Set the slit width as suggested by manufacturer for copper 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.

9.4.2 *Standardization*

9.4.2.1 A minimum of three concentrations of standard copper solutions (*see* **9.3.11**) are required to be selected to bracket the expected copper concentration and to be, in the optimum range of concentration of the instrument, after extraction. The *p*H of 100 ml of a copper free water blank and 100 ml of standard is adjusted to a *p*H of 2 to 10 by addition of 1 N Nitric acid or 1 N sodium hydroxide, as required.

9.4.2.2 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 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.

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

9.4.3 *Sample Analysis*

9.4.3.1 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.

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

10 DIFFERENTIAL PULSE ANODIC STRIPPING VOLTAMMETRY

10.1 Scope and Application

10.1.1 Copper is deposited on a hanging mercury drop at a negative potential of − 0.4 V versus saturated calomel electrode. Then the copper is stripped back into the solution by applying a positive potential scan. The anodic current peak which is measured represents the copper concentration in the sample. For total dissolved copper the sample is filtered through a Whatman No.40 filter paper prior to acidification analysis.

10.1.2 This method is applicable in the concentration range of 0.1 µg/1 to 100 µg/1 of copper.

10.2 Interferences

Iron interferes when present at levels greater than the copper. This can be overcome by subtracting the anodic peak current for a voltammogram without deposition from that for the stripping voltammogram. The presence or any neighboring stripping peaks which is less than 100 mV from that of copper will interfere.

10.3 Apparatus

10.3.1 Polarographic instrumentation capable of performing differential pulse work.

10.3.2 *Hanging Mercury Drop Electrode*

10.3.3 *Platinum Counter Electrode*

10.3.4 *Saturated Calomel Reference Electrode*

10.3.5 *Magnetic Stirrer Control Unit, Stirring Bar*

10.4 Reagents

10.4.1 *Hydrochloric Acid* — concentrated; Spectro grade

10.4.2 *Nitric Acid* — concentrated; Spectro grade

10.4.3 *Dilute Sulphuric Acid* — 1 : 1

10.4.4 *Dilute Nitric Acid* — 1 : 1

10.4.5 *Copper* (*II*) *Solutions*

10.4.5.1 *Stock Copper* (*II*) *Solution*

Dissolve 1.0 gm of pure copper metal in 30 ml of (1 : 1) nitric acid and add 4 ml of (1 : 1) sulphuric acid and heat until sulphur trioxide fumes evolve. Cool the solution and dilute to 1 litre with distilled water (1ml = 1.0 mg of copper).

10.4.5.2 *Intermediate copper* (II) *solution*

Dilute 100 ml of stock solution to be 1 litre with distilled water.

10.4.5.3 *Standard copper* (II) *solution*

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

10.4.6 Amalgamated Zinc

Cover 100 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.

10.4.7 Purified Nitrogen

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

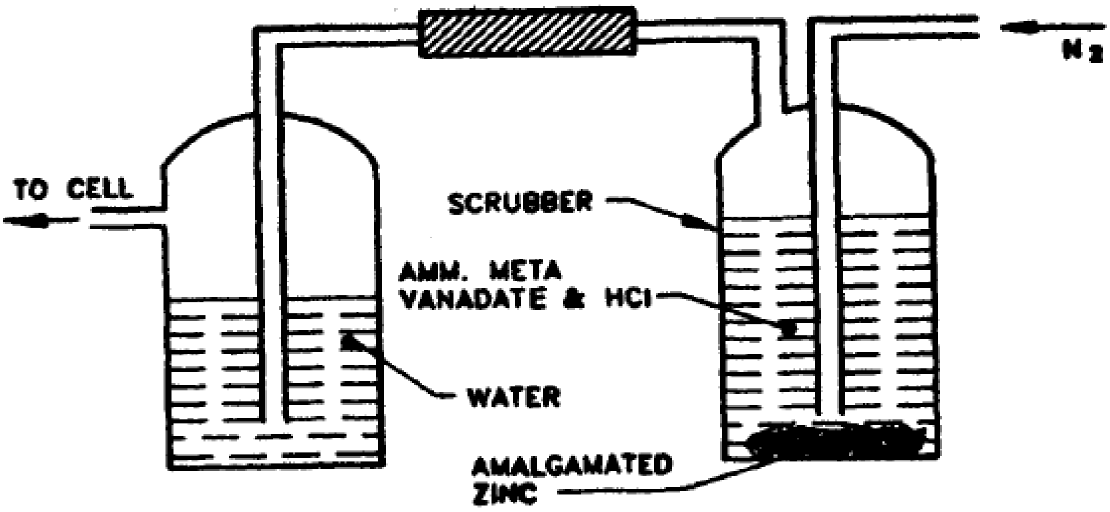

FIG. 1 SCRUBBER ASSEMBLY FOR NITROGEN PURIFICATION

10.5 Procedure

Clean all the glassware and the voltammetric cells by soaking them overnight in concentrated nitric acid and rinsing them thoroughly with distilled water. It the total dissolved copper alone is to be determined, the sample should be filtered through Whatman No. 40 filter paper. For total recoverable copper, 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 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 – 0.40 V versus saturated calomel electrode for 3 min. Stop the stirrer and wait for 30 s. Start the anodic scan with the followings 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).

10.6 Calculation

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

where

I_1 = current peak height for sample;

V = volume of the sample solution in ml;

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

I_2 = current peak height for sample + standard; and

v = volume of standard added (20 µl).

11 INDUCTIVELY COUPLED PLASMA SPECTROSCOPY

Copper 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 copper in water and wastewater.

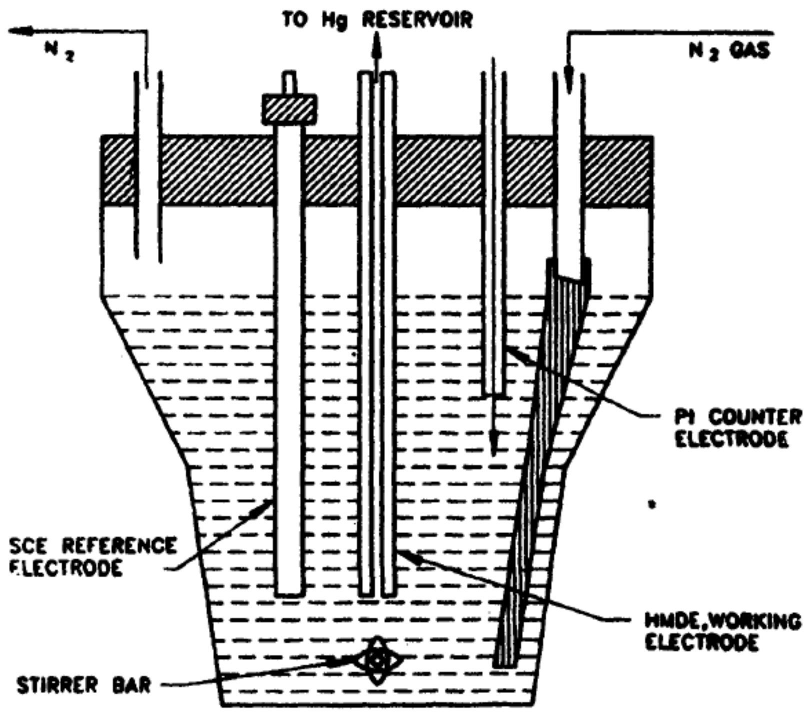

FIG. 2 VOLTAMMETRIC CELL ASSEMBLY

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 term 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

To access Indian Standards click on the link below:

https://www.services.bis.gov.in/php/BIS_2.0/bisconnect/knowyourstandards/Indian_standards/isdetails/

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)
Central Pollution Control Board, New Delhi	SHRI P. K. MISHRA SHRI VISHAL GANDHI (*Alternate*)
CII Triveni Water Institute, New Delhi	SHRI KAPIL K. NARULA DR SIPIKA CHAUHAN (*Alternate*)
CSIR - Central Institute for Mining and Fuel Research, Dhanbad	DR ABHAY KUMAR SINGH
CSIR - Indian Institute of Chemical Technology, Hyderabad	DR S. SRIDHAR DR NIVEDITA SAHU (*Alternate*)
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Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110002
Telephones: 2323 0131, 2323 3375, 2323 9402 *Website*: www.bis.gov.in

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