IS 3025 (Part 47) : 1994
(Reaffirmed 2009)
(Reaffirmed 2014)
(Reaffirmed 2019)

भारतीय मानक

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

भाग 47 सीसा

(पहला पुनरीक्षण)

Indian Standard

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

PART 47 LEAD

(First Revision)

Fourth Reprint SEPTEMBER 2007
(Including Amendment No. 1 & 2)

UDC 628.1032 : 628.3 : 543.3 [546.815]

BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

October 1994

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.

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

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

In the preparation of this standard, considerable assistance has been derived from American Standard Test Methods (ASTM Annual Book Section 11, 1983) and Analytical Chemical Acta, 164 (1984) 1-21.

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

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*)'.

AMENDMENT NO. 2 APRIL 2003 TO IS 3025 (PART 47) : 1994 METHODS OF SAMPLING AND TEST (PHYSICAL AND CHEMICAL) FOR WATER AND WASTEWATER

PART 47 LEAD

(*First Revision)*

(*Page* 1, *clause* **1**) — Substitute the following for the existing:

'1 SCOPE

This standard prescribes the following four methods for determination of lead:

a) Atomic absorption method (Direct);

b) Atomic absorption method (Chelation-Extraction);

c) Differential Pulse Anodic Stripping Voltammetry (DPAV); and

d) Dithizonc method.'

(*Page* 4, *clause* **9.6**) — Insert the following new clause after **9.6:**

'10 DITHIZONE METHOD

10.1 Principle

An acidified sample containing microgram quantities of lead is extracted with dithizone solution in chloroform. The extraction is carried out in the presence of strong ammoniacal citrate-cyanide reducing agent *(p*H 10 to 11.5). The quantity of lead present in the sample is determined spcctrophotometrically by measuring the absorbance at 510 nm of the chloroform extract containing the lead dithizonate complex.

10.2 Minimum Detection Limit

1.0 µg Pb/10 ml dithizonc solution (extract).

10.3 Interference

This method uses a high *p*H, mixed colour and single dithizone extraction. The method is without interference. In strongly ammoniacal citrate-cyanide solution *(p*H 10 to 11.5) dithizones of Sn (11) and T1 (1) are unstable and extracted only

partially. Further, a modification of the method allows for detection and elimination of these interferences.

10.4 Apparatus

10.4.1 Spectrophotometer for use at 510 nm with a path length of 1 cm or longer.

10.4.2 *p*H *meter*

10.4.3 *Standard Volumetric Glasswares*

10.4.4 *TEF Beaker,* 100 ml for Acid Digestion.

10.4.5 *Separatory Funnels,* 250 ml and 500 ml.

10.4.6 All glasswares are to be cleaned with 1:1 HNO_3, and rinsed thoroughly with distilled water.

10.5 Reagents

10.5.1 *Quality of Reagents*

Only analytical or equivalent grade reagents, unless specified otherwise, are to be used. All reagents are to be prepared in lead-free distilled water.

10.5.2 *Stock Lead Solution*

Dissolve 0.159 9 g lead nitrate [$(Pb(NO_3)_2$, minimum purity, 99.5 percent (*w/w*)] in about 200 ml of water. Add 10 ml concentrated HNO_3 and dilute to 1 000 ml with water, 1.0 ml of this solution will contain 100 µg of Pb.

10.5.3 *Standard Lead Solution*

Dilute 2.0 ml of stock lead solution to 100 ml with water, 1.0 ml of this solution will contain 2 µg of Pb.

10.5.4 *Nitric Acid* — Concentrated (18N).

10.5.5 *Nitric Acid — Dilute* —20 percent, *v/v*.

10.5.6 *Ammonium Hydroxide* — Concentrated (14N).

10.5.7 *Ammonium Hydroxide* — Dilute 10 percent, *v/v* and 1 percent, *v/v*.

10.5.8 *Citrate-Cyanide Reducing Solution*

Dissolve 200 g anhydrous ammonium citrate [$(NH_4)_2$ $HC_6H_5O_7$] 10 g anhydrous sodium sulphite (Na_2SO_3), 5 g hydroxylamine hydrochloride

($NH_2OH.HCl$), 20 g potassium cyanide (KCN) in water and dilute to 500 ml, and mix with one litre of concentrated NH_4OH.

CAUTION — KCN is a poisonous solution. Handle with extreme care and do not pipette by mouth.

10.5.9 *Stock Dithizone Solution*

Dissolve 25 mg dithizone in about 50 ml chloroform ($CHCl_3$) taken in a 200 ml beaker and filter through Whatman No. 42 (or equivalent) filter paper. Collect the filtrate and two washings (10 ml each) in a 250 ml conical flask. Transfer the combined filtrate to a 500 ml separatory funnel. Add about 100 ml 1 percent (*v/v*) NH_4OH solution, shake moderately for about 1 min. Transfer the $CHCl_3$ layer to another 250 ml separatory funnel retaining the orange-red aqueous layer in the 500 ml separatory funnel. Repeat the extraction (of the $CHCl_3$ layer) with 100 ml of 1 percent (*v/v*) NH_4OH solution, transfer the $CHCl_3$ layer to another 250 ml separatory funnel and the aqueous layer to the original 500 ml separatory funnel containing the first extract. One more repetition, of extraction and transferring to the main aqueous layer is carried out.

To the combined aqueous extract in the 500 ml separatory funnel add 1:1 HC1 in 2 ml portions, mixing after each addition, until dithizone precipitation is complete and the solution is no longer orange-red. Extract the precipitated dithizone with three 25 ml portions of $CHCl_3$. Dilute the combined extract to 250 ml with $CHCl_3$, 1 ml of this solution will contain 100 µg of dithizone.

10.5.10 *Working Dithizone Solution*

Dilute 100 ml stock dithizone solution to 250 ml in a standard volumetric flask with $CHCl_3$, 1 ml of this solution will contain 40 µg of dithizone.

10.6 Procedure

10.6.1 *Sample Digestion*

Digest all samples for dissolved and total lead as per standard digestion procedure using $HNO_3H_2SO_4$ and HNO_3 - $HClO_4$.

10.6.2 To 100 ml acidified sample *(p*H_2*)* add 20 ml of dilute (20 percent, *v/v*) HNO_3, filter if required through a filter paper (Whatman No. 41 or equivalent), and transfer it to a 250 ml separatory funnel. Add 60 ml ammoniacal citrate-cyanide solution, mix and cool to room temperature. Add 10 ml of dithizone working solution. Shake the stoppered funnel vigorously for about 30 s, allow to stand (to get two separate layers). Discard l-2 ml $CHCl_3$ layer and then fill the absorption

cell. Measure the absorbance at 510 nm using working dithizone solution as reagent blank.

10.7 Calibration Curve

Plot a calibration curve using at least five standard lead solutions, after adding 50 ml ammoniacal citrate-cyanide solution to the individual lead standard solutions and extracting the same with 10 ml of dithizone working solution.

10.8 Calculation

$$\text{mg Pb/litre} = \frac{\mu\text{g Pb (in 10 ml extract obtained from the calibration curve)}}{\text{Volume of sample (ml)}}$$

10.9 Precision and Accuracy

Using the dithizone method, lead at the level of 0.026 mg/l can be recovered with 4.8 percent relative standard deviation and 15 percent relative error.

(CHD 12)

Printed at Prabhat Offset Press, New Delhi-2

AMENDMENT NO. 1 OCTOBER 2000
TO
IS 3025 (PART 47) : 1994 METHODS OF SAMPLING AND TEST (PHYSICAL AND CHEMICAL) FOR WATER AND WASTEWATER

PART 47 LEAD

(First Revision)

(*Page* 1, *clause* **2**) — Insert the following at the appropriate place:

'3025 (Part 1) : 1986 Methods of sampling and test (physical and chemical) for water and wastewater : Part 1 Sampling'

(*Page* 2, *clause* **7.5.1,** *line* 6) — Insert the words 'using dilute nitric acid' after the words '100 ml.'

(Page 4, *clause* **9.6**) — Substitute the following for the existing formula:

$$C \text{ sample, mg/l} = \frac{V_{std} \times C_{std}}{V_{sample}} \times \frac{I_1}{I_2 - I_1}$$

(CHD 12)

Printed at Prabhat Offset Press, New Delhi-2

Indian Standard

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

PART 47 LEAD

(First Revision)

1 SCOPE

This standard prescribes the following three methods for determination of lead:

a) Atomic absorption method (Direct),

b) Atomic absorption method (Chelation-Extraction), and

c) Differential Pulse Anodic Stripping Voltammetry (DPASV).

2 REFERENCES

The following Indian Standards are necessary adjuncts to this standard:

IS No.	*Title*
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 2

3 TERMINOLOGY

For the purpose of this standard, definitions given in IS 7022 (Part 1) : 1973 and IS 7022 (Part 2) : 1979 shall apply.

4 APPLICATION

Depending upon the concentration range and interference levels, choice of the method is made. When the concentration levels are below 300 µg/1, pre-concentration is carried out either by chelation and extraction prior to atomic absorption spectrophotometer (AAS) or by deposition on a mercury drop electrode as in DPASV method. For dissolved lead content, filtration through 0.45 µm membrane filter is required.

5 SAMPLING AND PRESERVATION

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

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

6 PURITY OF REAGENTS

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

6.2 Lead free distilled water should be used for preparing standards, and reagent solution.

7 ATOMIC ABSORPTION METHOD (DIRECT)

7.1 Principle

The lead content of the sample is determined by directly aspirating the sample into the flame of an atomic absorption spectrophotometer.

This method is applicable in the range from 1.0 to 10.0 mg/1 of lead. However, the concentration range will vary with the sensitivity of the instrument used.

7.2 Interferences

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

7.3 Apparatus

7.3.1 Atomic absorption spectrophotometer with air-acetylene flame.

7.3.2 Hollow-cathode lamps or electrodeless discharge lamps for use at 283.3 nm.

7.4 Reagents

7.4.1 *Hydrochloric Acid* — Concentrated.

7.4.2 *Nitric Acid* — Concentrated.

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

7.4.4 *Lead Solutions*

7.4.4.1 *Stock lead solution*

Dissolve 1.599 9 g of lead nitrate in a mixture of 10 ml or concentrated nitric acid and 100 ml of water and dilute to 1 litre (1 ml = 1.0 mg of Pb).

7.4.4.2 *Standard lead solution*

Dilute 100 ml of lead stock solution to 1 litre with dilute nitric acid (1 : 499) (1 ml = 0.1 mg of Pb).

7.5 Procedure

7.5.1 To 100 ml portion of the acidified sample add 0.5 ml of nitric acid, 5 ml of concentrated hydrochloric acid and heat it not to boil but to reduce the volume to 20 ml in a well-ventilated hood. Cool and filter the sample and make up to 100 ml in a standard flask. Aspirate the sample solution and measure the absorbance at 283.3 nm. Aspirate nitric acid (1 : 499) prior to sample aspiration.

7.5.2 Prepare a reagent blank and sufficient standards containing 1.0, 2.5, 5.0, 7.5 and 10.0 mg/1 of lead by diluting suitable volume of the standard solution with nitric acid (1 : 499) and repeat as above. Aspirate the solutions and measure the absorbance.

7.6 Calculation

Construct a standard calibration graph by plotting the absorbance versus mg of lead concentration of each standard. Read the concentration of the sample from the graph.

$$\text{Lead, (mg/l)} = \frac{M}{V} \times 1000$$

where

M = mass o lead present in mg in the sample, and

V = volume of sample in ml.

8 ATOMIC ABSORPTION METHOD (CHELATION – EXTRACTION)

8.1 Principal

Lead is chelated with pyrrolidine dithiocarbamic acid and extracted with chloroform. The extract is treated with hot nitric acid after evaporating to dryness, treated with hot hydrochloric acid and diluted with water to a specified volume. An aliquot is aspirated into the air-acetylene flame of the spectrophotometer. For total recoverable lead an acid digestion procedure is done prior to chelation or aspiration.

This method is applicable for concentration range from 100-1 000 µg/1 of lead.

NOTE — The lower range of determination to the extent of 0.001 mg/1 may be obtained by graphite system.

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

8.3 Apparatus — Same as in **7.3.**

8.4 Reagents

8.4.1 *Hydrochloric Acid* — Concentrated.

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

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

8.4.4 *Nitric Acid* — Concentrated.

8.4.5 *Pyrrolidine Dithiocarbamic 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 two litres of chloroform and store in a cool and dark place. The reagent is stable for atleast six months.

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

8.4.6 *Sodium Hydroxide Solution*

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

8.4.7 *Chloroform*

8.4.8 *Bromophenol Blue Indicator Solution*

Dissolve 0.1 g of bromophenol blue in 100 ml of 50 percent ethanol or *iso*propanol.

8.4.9 *Lead Solutions*

8.4.9.1 *Stock lead solution*

Dissolve 1.5999 g of lead nitrate in a mixture of 10 ml of concentrated nitric acid and 100 ml of water and dilute to 1 litre (1 ml = 1.0 mg of Pb).

8.4.9.2 *Standard lead solution*

Dilute 100 ml of lead stock solution to 1 litre with dilute nitric acid (1 : 499) (1 ml = 0.1 mg of Pb).

8.4.10 *Water Saturated Chloroform*

Mix one part of chloroform with one part of water in a separatory funnel. Shake 30 times and let separate. Discard aqueous layer. Save chloroform layer.

8.5 Procedure

8.5.1 For dissolved lead, filter 100 ml of the sample through 0.45 µm membrane filter paper. For total lead, add 5 ml of concentrated hydrochloric 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 lead) 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 : 49) 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 pyrrolidine dithiocarbamic 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 the flask. Repeat the extraction till the chloroform layer becomes colourless with fresh 6 to 7 ml portion of chloroform; combine the extracts and make up the volume to 25 ml. Aspirate the organic extracts directly into the the flame (zeroing the instrument on a water saturated chloroform blank) and record absorbance. To avoid problems associated with instability of extracted metal complexes, determine immediately, after extraction. Alternatively evaporate the extracts 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.

8.5.2 Prepare a reagent blank and sufficient standards containing 100, 200, 400, 500, 700, 900 and 1 000 µg/l of lead 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.

8.6 Calculation

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

$$\text{Lead, µg/l} = \frac{M}{V} \times 1\,000$$

where

M = mass of lead present in µg in the sample, and

V = volume of sample in ml.

9 DIFFERENTIAL PULSE ANODIC STRIPPING VOLTAMMETRY (DPASV)

9.1 Principle

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

9.2 Interferences

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

9.3 Apparatus

9.3.1 Polarographic instrumentation capable of performing differential pulse work.

9.3.2 *Hanging Mercury Drop Electrode*

9.3.3 *Platinum Counter Electrode*

9.3.4 *Saturated Calomel Reference Electrode*

9.3.5 *Magnetic Stirrer Control Unit, Stirring Bar*

9.4 Reagents

9.4.1 *Hydrochloric Acid* — Concentrated.

9.4.2 *Nitric Acid* — Concentrated.

9.4.3 *Nitric Acid* — Diluted (1 : 1) .

9.4.4 *Lead Solutions*

9.4.4.1 *Stock lead solution*

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

9.4.4.2 *Intermediate lead solution*

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

9.4.4.3 *Standard lead solution*

Dilute 10 ml of lead intermediate solution and 1 ml of concentrated nitric acid to 100 ml with water (1 ml = 0.2 µg of Pb).

This solution should be prepared just before use for preparing the working standards.

9.4.5 *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.

9.4.6 *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 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)

9.5 Procedure

9.5.1 Clean all the glasswares and the voltammetric cells by soaking them overnight in concentrated nitric acid and rinse them thoroughly with distilled water. If total dissolved lead alone is to be determined, the sample should be filtered through 0.45 µm membrane filter paper. For total recoverable lead, digest the sample with 3 ml each concentrated hydrochloric acid and nitric acid. Evaporate the solution to 15 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 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 — 0.6 V versus SCE for 3 minutes. Stop the stirrer and wait for 30 seconds. Start the anodic scan with the following settings:

Initial potential	— 0.6 V vs SCE
Scan rate	5 mV/sec
Scan direction	+ ve
Modulation amplitude	25 mV
Current range	1 — 10 μA
Drop time	0.5 sec
Display direction	— ve
Low pass filter	Off position
Mode	Differential pulse
Scan range	–0.6 V to –0.15

Measure the current peak height (I_1). Add 20 µl of standard lead solution and deaerate for 5 minutes. Repeat as above. Measure the current peak height (I_2).

9.6 Calculation

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

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 lead in the sample.

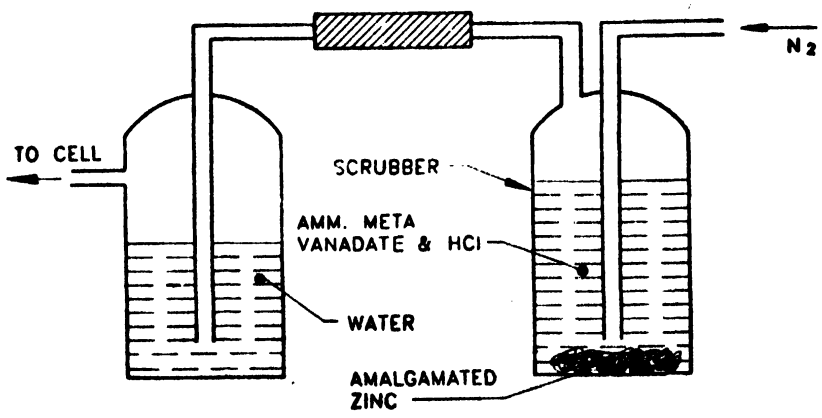

FIG. 1 SCRUBBER ASSEMBLY FOR NITROGEN PURIFICATION

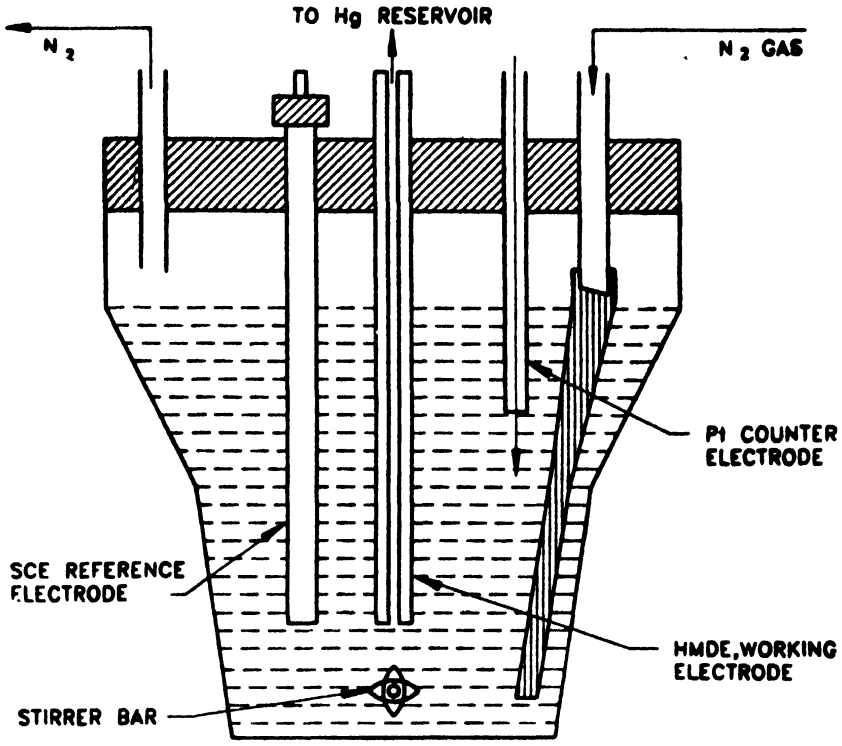

FIG. 2 VOLTAMMETRIC CELL ASSEMBLY

ANNEX A

(*Foreword*)

COMMITTEE COMPOSITION

Environmental Protection Sectional Committee, CHD 012

Chairman	*Representing*
PROF D. K. BISWAS	Central Pollution Control Board, Delhi
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(*Continued on page* 7)

(*Continued from page* 6)

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Member Secretary
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Joint Director (Chem), BIS

Water Environment Subcommittee, CHD 012 : 01

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This Indian Standard has been developed from Doc : No. CHD 012 (0170).

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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Printed at Prabhat Offset Press, New Delhi-2