भारतीय मानक **IS 3025 (Part 53) : 2024**

Indian Standard

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

भाग 53 लोहा

(दूसरा पुनरीक्षण)

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

Part 53 Iron

(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

March 2024 **Price Group 6**

Water Quality Sectional Committee, CHD 36

FOREWORD

This Indian Standard (Part 53) (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.

Pollution caused by substances, on which biotic and abiotic agencies of decomposition are ineffective, is a unique type of pollution. Toxic trace elements and heavy metals come under the category of non-degradable pollutants. The problem caused by these elements is in fact due to their concentration in the environment in the bio-available state and above a certain concentration become harmful to the living organism.

Iron (Fe) is naturally abundant in earth's crust. Amount of iron available in soluble from depends upon the concentration of the complex forming ions, *p*H and oxidation conditions. In the absence of the complex forming ions, ferric iron is not significantly soluble unless the *p*H is very low. Oxygenated surface waters seldom contain more than one milligram per litre of iron. Ground waters and the surface waters which are acidic, may, on the other hand, contain considerably more iron. In water samples, iron may be present, as free hydrated ions, in the form of organic/inorganic complex ions, in a colloidal state or as relatively coarse suspended particles. On exposure to air or addition of oxidants, ferrous iron is oxidized to the ferric state (Fe^{3+}) and may hydrolyze to form red, insoluble hydrated ferric oxide. Ferric iron is not significantly soluble unless the *p*H is very low.

The Committee responsible for the formulation of IS 3025 : 1964 had decided to revise the standard and publish it as separate parts. This standard supersedes **32** of IS 3025 : 1964. The first revision of this standard was published in 2003.

In this revision following changes have been incorporated:

a) Direct air-acetylene flame atomic absorption spectrometry (AAS) method has been added; and

b) Inductively coupled plasma spectroscopy methods have been added.

In the preparation of this standard, considerable assistance is derived from 'Standard methods for the examination of water and wastewater', 23rd edition-2017, published by the American Public Health Association, Washington, U.S.A.

The spectrometric method for determination of total dissolved iron and total (both dissolved and undissolved) iron using 1,10 phenanthroline are technically equivalent to ISO 6332 : 1988 'Water quality — Determination of iron — Spectrometric method using 1,10 phenanthroline'.

The composition of the Committee responsible for formulation of this 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 : 2022 'Rules for rounding off numerical values (*second revision*)'.

Indian Standard

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

PART 53 IRON

(Second Revision)

1 SCOPE

1.1 This standard (Part 53) prescribes following four methods for the determination of iron in water and wastewater:

a) 1,10 phenanthroline method (applicable in the range of 75 µg/1 to 500 µg/1 of iron);

b) Direct air-acetylene flame atomic absorption spectroscopy (applicable for 0.3 mg/l to 10 mg/l) method;

c) Extraction/air-acetylene flame atomic absorption spectroscopy method; and

d) Inductively coupled plasma spectroscopy method.

1.2 Depending upon the concentration range and interference levels, choice of the method is made.

2 REFERENCES

The standards listed below 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.

IS No.	*Title*
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 terms relating to water, sewage and industrial effluents: Part 1
IS 7022 (Part 2) : 1979	Glossary of terms relating to water, sewage and industrial effluents: Part 2
IS 17614	Water quality — Sampling:
(Part 1) : 2021/ISO 5667-1 : 2020	Guidance on the design of sampling programmes and sampling techniques
(Part 3) : 2021/ISO 5667-2018	Preservation and handling of water samples

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 STORAGE

The sampling 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 bottle or chemically resistant glass containers. For the determination of dissolved iron content, filter through 0.45 µm membrane filter, at the time of sampling. The analysis of such samples is to be carried out within 24 h of sampling. The samples should be acidified with concentrated nitric acid (2 ml of concentrated nitric acid in 1 litre sample, just to bring down the *p*H below 2). The acidified samples can be stored for a few days (up to 5 days) in a refrigerator.

Iron in water may vary in concentration and form with duration and degree of flushing before and at the time of sample collection. Shake bottle vigorously to obtain uniform suspension of precipitated iron. Use particular care for when colloidal iron adheres to the sampling bottle. The problem is more acute with plastic bottle.

5 PURITY OF THE REAGENTS

Unless specified otherwise, only pure chemicals and iron free distilled water shall be used in tests.

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

6 1,10 PHENANTHROLINE METHOD

6.1 Principle

Iron in the solution is reduced to the ferrous state by boiling with hydrochloric acid and hydroxylamine, and treated with 1,10 phenanthroline at *p*H to 3.3. Three molecules of phenanthroline chelate each atom of ferrous iron to form an orange-red complex. The coloured solution obeys Beer's law. Its colour intensity is independent of *p*H from 3 to 9. A *p*H between 2.9 and 3.5 insures rapid colour development in the presence of an excess of phenanthroline. This method is applicable in the range of 75 µg/1 to 500 µg/1 of iron.

6.2 Interference

Strong oxidizing agents, nitrite, cyanide, and phosphates (polyphosphates more so than orthophosphates), zinc, chromium are some of the interfering substances. The concentration of zinc 10 times more than iron, cobalt and copper in excess of 5 mg/l, and nickel in excess of 2 mg/1 may also lead to interferences.

Cadmium, bismuth, molybdate, mercury, and silver present in sample, precipitate phenanthroline. To avoid interference initially boil with acid to convert polyphosphates to orthophosphate and eliminates nitrite and cyanide that might interfere. Add of hydroxylamine to remove errors occurring due to excessive strong oxidizing reagents. Evaporate the sample, gently ash the residue and dissolve the residue in acid in case of the presence of interfering metals, if noticeable amount of colour or organic matter is present. Ashing is carried out in porcelain, silicon or platinum crucible that have been boiled for several hours in 6 N hydrochloric acid. Use a larger excess of phenanthroline to replace that complexed by the interfering metals. Before the extraction of iron, the presence of excess amount of organic matter may necessitate digestion of sample.

6.3 Apparatus

6.3.1 *Spectrophotometer* — for use at 510 nm, providing a light path of 1 cm or longer.

6.3.2 *Acid-Washed Standard Volumetric Glassware*

NOTE — Wash all glassware with concentrated hydrochloric acid (HCl) and rinse with distilled water before use to remove deposits of iron oxide.

6.4 Reagents

6.4.1 *Hydrochloric Acid* — concentrated solution containing less than 0.5 mg/l percent iron.

6.4.2 *Hydroxylamine Solution* (*$NH_2OH.HCl$*)

Dissolve 10 g of hydroxylamine hydrochloride in 100 ml water. This solution is stable for at least one week.

6.4.3 *Ammonium Acetate Buffer Solution*

Dissolve 250 g of ammonium ethanoate in 150 ml water and then add 700 ml of glacial acetic acid. Prepare new reference standards with each buffer preparation as even a good grade of ammonium ethanoate contains significant amount of iron.

6.4.4 *Sodium Acetate Solution*

Dissolve 200 g of sodium acetate ($NaC_2H_3O_2.3H_2O$) in 800 ml water.

6.4.5 1,10 *Phenanthroline Solution*

In 100 ml of solution, add 100 mg of 1,10 phenanthroline monohydrate ($C_{12}H_8N_2.H_2O$) by stirring and heating to 80 ºC. Do not boil the mixture. If the solution darkens, discard it. Heating is not necessary if two drops of concentrated hydrochloric acid are added to the water. The prepared solution is stable for at least one week.

NOTE — 1 ml of this reagent is sufficient up to 100 kg of Fe.

6.4.6 *Potassium Permanganate* — 0.02 M

To a reagent water, add 0.316 g of potassium permanganate and dilute the solution up to 100 ml.

6.4.7 *Stock Iron Solution*

With constant stirring carefully add 20 ml concentrated sulphuric acid (H_2SO_4) to 50 ml water contained in 250 ml beaker (cooled in ice water) and dissolve 1.404 g of ferrous ammonium sulphate [$Fe(NH_4)_2(SO_4)_2.6H_2O$]. Add 0.1 N potassium permanganate ($KMnO_4$) drop wise till a faint pink colour persists. Transfer quantitatively to a 1 000 ml volumetric flask and makeup the

volume up to the mark with water and mix well (1.0 ml = 200 µg of Fe).

6.4.8 *Standard Iron Solutions*

6.4.8.1 Pipette out 50.0 ml of stock solution into a 1 000 ml volumetric flask and make up the volume up to the mark with water and mix well such that (1.0 ml = 10.0 µg of Fe).

6.4.8.2 Pipette 5.0 ml of stock solution into a 1 000 ml volumetric flask and makeup the volume up to the mark with water and mix well (1.0 ml = 1.0 µg of Fe).

NOTE — Prepare standard iron solutions daily for use.

6.4.9 *Diisopropyl Ether*

CAUTION — Diispropyl ether is highly inflammable.

6.4.10 *Sulphuric Acid* — concentrated

6.4.11 *Nitric Acid* — concentrated

6.5 Procedure

6.5.1 *Determination of Total Iron*

Pipette out appropriate portions of standard iron solution into 125 ml conical flasks to contain from 10 µg of Fe to 100 µg of Fe. For the reagent blank, pipette out 10 ml of water to a separate conical flask. Dilute the contents of each conical flask to about 50 ml by adding water. To each flask, add 1 ml hydroxylamine solution ($NH_2OH.HCl$) solution and 2 ml concentrated HCl. Add a few boiling chips and boil the solution until the volume is reduced to about 20 ml. Cool to room temperature and quantitatively transfer to 100 ml volumetric flasks. Add 10 ml ammonium acetate buffer solution first, and then add 10 ml 1,10 phenanthroline solution to each flask. Dilute to 100 ml with water and mix thoroughly and allow to stand for 10 min to 15 min.

6.5.2 *Determination of Dissolved Iron*

Filter the sample right after as it is collected through a 0.45 µm membrane filter into a vacuum flask containing 1 ml of concentrated HCl/100 ml sample. For total iron analyze the filtrate (*see* 6.5.1) and for ferrous ion (*see* 6.5.3). This method of determination is applicable for laboratory analysis, as sample exposure to air might precipitate iron. Subtract dissolved iron from total iron to calculate the suspended iron in the sample.

6.5.3 *Determination of Ferrous Iron*

At the sampling site, determine the ferrous iron, because in acidic solution ferrous to ferric iron ratio may change with time. Acidify 2 ml of concentrated HCl/100 ml of sample for determination of ferrous ion only. Collect the sample from source, in stoppered bottle. In a 100 ml beaker, immediately add 50 ml portion of acidified solution followed by addition of 20 ml of 1,10 phenanthroline solution and 10 ml of ammonium acetate buffer solution. Stir the beaker vigorously. Add water to make up volume to 100 ml. Measure the color intensity within 5 min to 10 min. Avoid exposure to sunlight. Calculate ferric ion by subtracting ferrous ion from total iron.

NOTE — The amount of phenanthroline solution may be adjusted depending on the concentration of iron in the acidified solution. Large amount of iron requires, larger amount of phenanthroline solution for color development.

6.5.4 *Color Measurement*

Pipette out appropriate portions of standard iron solution into 125 ml conical flasks to contain from 10 µg of Fe to 100 µg of Fe. For the reagent blank, pipette out 10 ml of water to a separate conical flask. Dilute the contents of each conical flask to about 50 ml by adding water. To each flask, add 1 ml $NH_2OH.HCl$ solution and 2 ml concentrated HCl. Add a few boiling chips and boil the solution until the volume is reduced to about 20 ml. Cool to room temperature and quantitatively transfer to 100 ml volumetric flasks. Add 10 ml ammonium acetate buffer solution first and add 4 ml 1,10 phenanthroline solution to each flask. Dilute to 100 ml with water and mix thoroughly and allow to stand for 10 min to 15 min. Measure the absorbance of the iron complexes at 510 nm against the reagent blank. Construct a calibration curve by plotting absorbance values against micrograms of iron in 100 ml of the final solution.

6.5.5 *Samples Containing Organic Interferences*

If the sample contains an excessive amount of organic matter, then sample is digested with HNO_3 and H_2SO_4 and the iron present is separated by extraction process as described in 6.5.5.1.

6.5.5.1 *HNO_3.H_2SO_4, digestion*

Transfer a suitable volume of the homogenized sample to a beaker. (Sample volume depends upon the expected Fe content. If the expected Fe concentration is of the order of 1 mg/l, the sample volume will be about 800 ml. If Fe concentration is between 10 mg/l to 100 mg/1,

about 100 ml sample will be required.) Add 5 ml concentrated HNO_3 and a few boiling chips to both the solutions. Heat to boil the solutions, and concentrate carefully on a hot plate to lowest possible volume, if required cool the solutions and transfer quantitatively, to two separate beakers of smaller size (say 150 ml beaker). Add 5 ml concentrated HNO_3 and 10 ml concentrated H_2SO_4 to each of these. Heat up to the evolution of dense white fumes of SO_3. If the solution is not clear add 5 ml HNO_3 and re-heat until a clear solution (no evolution of brown fumes) results. Cool, transfer quantitatively to a 100 ml volumetric flask. Dilute up to the mark with water, and mix well. Use this solution for extraction of iron as described in 6.5.5.2.

6.5.5.2 *Extraction of iron*

Pipette out a portion of the solution obtained from acid digestion, containing 10 µg of iron to 100 µg of iron in a separator funnel. Add about 25 ml of concentrated HCl to this. Mix and allow to cool to room temperature. Extract the iron from the HCl solution in the separator funnels by shaking with 25 ml of diisopropyl ether. Draw off the lower HCl layer into another separator funnel; extract both again with 25 ml of diisopropyl ether. Continue the extraction process three times. Separate the acid layer and discard it. Transfer the combined ether layer to the original separator funnel. Shake the ether layer with 25 ml water; and transfer the aqueous layer to a 100 ml volumetric flask. Repeat the extraction of the ether layer with a second 25 ml portion of water, and transfer the aqueous layer to the volumetric flask. Use this solution for colour development, as described in 6.5.5.3.2. Prepare a reagent blank using a volume of water equal to that of the sample solution.

6.5.5.3 *Colour development and measurement*

6.5.5.3.1 *Soluble iron* (*for filtered sample direct determination*)

6.5.5.3.1.1 Pipette out a portion of filtered sample (filtered through 0.45 µm membrane filter), containing 50 µg of iron to 60 µg of iron in 150 ml conical flask. Dilute the contents of conical flask to about 50 ml by adding water. Add 1 ml $NH_2OH.HCl$ solution and 2 ml concentrated HCl. Add a few boiling chips and boil the solution until the volume is reduced to about 20 ml. Cool to room temperature and quantitatively transfer to 100 ml volumetric flasks.

6.5.5.3.1.2 Add 10 ml ammonium acetate buffer solution first and add 10 ml 1,10 phenanthroline solution to each flask.

6.5.5.3.1.3 Dilute to 100 ml with water and mix thoroughly and allow to stand for 10 min to 15 min. Measure the absorbance of the iron complexes at 510 nm against the reagent blank.

6.5.5.3.2 *For samples after digestion and extraction*

Add 1 ml of $NH_2OH.HC1$ solution, add 10 ml ammonium acetate buffer solution and then add 10 ml 1,10 phenanthroline solution to each flask. 10 ml of phenanthroline solution in to the volumetric flasks containing extracted iron (*see* 6.5.5.1). Dilute to 100 ml with water, mix thoroughly and allow to stand for 10 min. Measure the absorbance of the iron-complex at 510 nm against the reagent blank prepared in an identical manner (using water instead of the sample solution). Determine micrograms of iron in the solution from the absorbance reading, by referring to the calibration curve.

6.6 Calculation

6.6.1 *Soluble Iron* (*Direct Determination Without the Digestion Step*)

$$\text{mg of Fe/l} = \frac{\text{(µg of Fe (in 100 ml of the final solution)}}{V}$$

where

V = *volume* of the sample used, in ml.

6.6.2 *Total Iron* (*When the Digestion is Carried Out*)

$$\text{mg of Fe/l} = \frac{\text{(µg of Fe (in 100 ml of the final solution)}}{(V_1 \times V_2)} \times 100$$

where

V_1 = volume of the sample used, in ml; and

V_2 = total volume of digested solution used for Fe determination, in ml.

6.6.3 *Precision and Accuracy*

Precision and accuracy of this method depends upon the methods of sampling, methods of colour measurements (visual colorimetry or spectrophotometry), the iron concentration and the presence of interfering colour, turbidity and foreign ions. The relative standard deviation reported in the literature for iron in the 300 µg/1 concentration range is 25.5 percent.

7 DIRECT AIR-ACETYLENE FLAME ATOMIC ABSORPTION SPECTROPHOTOMETRY METHOD

7.1 Principle

The iron content of the sample is determined by atomic absorption spectrophotometry. For dissolved iron, the filtered sample is directly aspirated to the atomizer. For total recoverable iron, a pre-treatment with hydrochloric acid (HCl), nitric acid, and calcium solution is carried out prior to aspiration of the sample. This method is applicable in the range from 0.3 mg/l to 10 mg/l of iron. However, the concentration range will vary with the sensitivity of the instrument used.

7.2 Interferences

Interferences of cobalt, copper, nickel can be controlled by using a very lean (hot) flame. Silicons depressed the iron response, and can be overcome by the addition of 0.2 percent calcium chloride.

7.3 Apparatus

7.3.1 *Atomic Absorption Spectrometer and Associated Equipment*

7.3.2 Use burner head as recommended by the manufacturer.

7.3.3 *Standard Volumetric Glassware*

7.4 Reagents

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

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

7.4.3 *Acetylene* — standard commercial grade

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

7.4.5 *Calcium Solution*

Dissolve 630 mg calcium carbonate ($CaCO_3$), in 50 ml of 1 + 5 HCl. Boil gently to obtain complete solution. Cool and dilute to 1 000 ml with water.

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

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

7.4.8 *Stock Iron solution*

Dissolve 0.100 g iron wire in a mixture of 10 ml 1 + 1 HCl and 3 ml concentrated HNO_3. Add 5 ml of concentrated HNO_3 and dilute to 1 000 ml with water (1.00 ml = 100 µg of Fe).

7.4.9 *Standard Iron Solution*

Prepare a series of standard solution in optimum concentration by dilution of stock iron solution with water containing 1.5 ml concentrated HNO_3/l.

7.5 Procedure

7.5.1 *Calibration Curve*

Prepare sufficient standard solutions containing 0 µg/1 to 60 µg/1 of iron by diluting suitable volumes of standard iron solution with nitric acid (1 : 499) to 100 ml in volumetric flasks. Transfer the contents of the volumetric flasks to 150 ml beakers. Add 25 ml of calcium solution to each of the volumetric flasks. Prepare a reagent blank using 10 ml of water in a similar manner. Aspirate the reagent blank and carry out zero adjustment. Aspirate sequentially the standard solutions and measure the absorbance at 248.3 nm. Construct a calibration curve by plotting absorbance values against micrograms of Fe in 100 ml final volume. Ensure that the calibration curve is linear, make the necessary changes in the volume of the standard solution used. (Increase in the final volume due to the calcium solution is not to be considered for calculation of micrograms of Fe).

7.5.2 *Determination of Iron*

For the determination of dissolved iron content, filter the sample through 0.45 pm membrane filter. For total recoverable iron, digest the sample with HNO_3-H_2SO_4 (*see* **6.5.2**).

Add 0.5 ml of concentrated nitric acid to a suitable volume of the sample taken (or the solution obtained after digestion, which contains 50 µg of iron to 60 µg of iron) in a 100 ml volumetric flask. Make up to the mark. Transfer the contents of the volumetric flask to a 150 ml beaker. Add 25 ml calcium solution to this. Prepare a reagent blank with 100 ml water. Rinse the nebulizer by aspirating water containing 1.5 ml concentrated HNO_3/l. Aspirate the reagent blank and carry out zero adjustment. Aspirate the sample solution and measure the absorbance at 248.3 nm. Determine

micrograms of iron in the solution from the absorbance reading, by referring to the calibration curve.

7.6 Calculation

7.6.1 *Soluble Iron* (*Direct Determination Without the Digestion Step*)

mg of Fe/l =

$$\frac{(\mu g \text{ of Fe (in 100 ml of the final solution)}}{V}$$

where

V = volume of the sample used, in ml.

7.6.2 *Total Iron* (*When the Digestion is Carried Out*)

mg of Fe/l =

$$\frac{(\mu g \text{ of Fe (in 100 ml of the final solution)}}{V_1 \times V_2} \times 100$$

where

V_1 = volume of the sample used, in ml; and

V_2 = total volume of the digested solution used for Fe estimation, in ml.

8 EXTRACTION/AIR-ACETYLENE FLAME ATOMIC ABSORPTION SPECTROSCOPY METHOD

8.1 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.

8.2 Apparatus

8.2.1 *Atomic Absorption Spectrometer and Associated Equipment*

8.2.2 *Use Burner Head as Recommended by the Manufacturer*

8.3 Reagents

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

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

8.3.3 *Acetylene* — standard commercial grade

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

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

8.3.6 *Nitric Acid* — concentrated, ultrapure

8.3.7 *Sodium Sulphate* — anhydrous

8.3.8 *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.

8.3.9 *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.

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

8.4 Procedure

8.4.1 *Instrument Operation*

8.4.1.1 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.

8.4.1.2 Install a hollow-cathode lamp for iron in the instrument and set the wavelength at 248.3 nm. Set the slit width as suggested by manufacturer for iron 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.

8.4.2 *Standardization*

8.4.2.1 A minimum of three concentrations of standard iron solutions (*see* 7.4.9) are required to be selected to bracket the expected iron concentration and to be, in the optimum range of concentration of the instrument, after extraction. The *p*H of 100 ml of iron free water blank and 100 ml of standard is adjusted to a *p*H of 2 to 5 by addition of 1 N Nitric acid or 1 N sodium hydroxide, as required.

8.4.2.2 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.

8.4.2.3 Prepare a calibration curve of absorbance versus concentrations before extraction on a linear graph paper.

8.4.3 *Sample Analysis*

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.

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

9 INDUCTIVELY COUPLED PLASMA SPECTROSCOPY

Iron 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 iron in water and wastewater.

ANNEX A

(*Foreword*)

COMMITTEE COMPOSITION

Water Quality Sectional Committee, CHD 36

Organization	*Representative(s)*
Chief Scientist, EPTRI, Hyderabad	SHRI N. RAVEENDHAR **(*Chairperson*)**
Andhra Pradesh Pollution Control Board, Vijaywada	SHRIMATI M. SREERANJANI SHRIMATI A. SRI SAMYUKTHA (*Alternate*)
Bhabha Atomic Research Centre, Mumbai	DR S. K. SAHU SHRI I. V. SARADHI (*Alternate*)
Bureau of Indian Standards, New Delhi	MS NITASHA DOGER
Central Institute of Mining and Fuel Research, Dhanbad	DR ABHAY KUMAR SINGH
Central Pollution Control Board, New Delhi	SHRI P. K. MISHRA SHRI VISHAL GANDHI (*Alternate*)
Confederation of Indian Industry, New Delhi	DR KAPIL K. NARULA DR SIPIKA CHAUHAN (*Alternate*)
Envirocare Laboratories Pvt Ltd, Thane	DR PRITI AMRITKAR SHRI NILESH AMRITKAR (*Alternate*)
Gujarat Pollution Control Board, Gandhinagar	DR D. N. VANSADIA SHRI K. B. VAGHELA (*Alternate*)
Haryana State Pollution Control Board, Panchkula	SHRI JATINDER PAL SINGH
Himachal Pradesh State Pollution Control Board, Government of Himachal Pradesh, Shimla	ER PRAVEEN GUPTA SHRI PRAVEEN SHARMA (*Alternate*)
Indian Agricultural Research Institute – Water Technology Centre, New Delhi	DR KHAJANCHI LAL DR RAVINDER KAUR (*Alternate*)
Indian Chemical Council, Mumbai	SHRI J. I. SEVAK DR MRITUNJAY CHAUBEY (*Alternate* I) DR N. D. GANGAL (*Alternate* II)
Indian Institute of Chemical Technology, Hyderabad	DR SUNDERGOPAL SRIDHAR DR NIVEDITA SAHU (*Alternate*)
Indian Institute of Toxicology Research, Lucknow	DR S. C. BARMAN DR SATYAKAM PATNAIK (*Alternate*)
Indian Water Works Association, New Delhi	SHRI VIJAY CHARHATE
Karnataka State Pollution Control Board, Bengaluru	DR H. RUPADEVI MS GOURI GOLSANGI (*Alternate*)
Maharashtra State Pollution Control Board, Mumbai	DR V. R. THAKUR SHRI AMAR SUPATE (*Alternate*)

Organization	*Representative(s)*
Ministry of Environment and Forest & Climate Change, New Delhi	DR SONU SINGH
Ministry of Jal Shakti Department of Drinking Water and Sanitation, New Delhi	SHRI D. A. RAJASEKHAR SHRI SUMIT PRIYADARSHI (*Alternate*)
National Environmental Engineering Research Institute, Nagpur	DR NOOR A. KHAN
National Institute of Oceanography, Vizag	DR V. V. S. S. SARMA DR DURBAR RAY (*Alternate*)
NTPC Ltd, New Delhi	DR VIJAY PRAKASH SHRI HARIKRISHNA SURARAPU (*Alternate*)
Shriram Institute for Industrial Research, New Delhi	DR VIVEK NARAYAN SINGH DR JAGDISH KUMAR (*Alternate*)
Telangana State Pollution Control Board, Hyderabad	DR M. S. SATYANARAYANA RAO
Uttar Pradesh Pollution Control Board, Lucknow	DR ARUNIMA BAJPAI
In Personal Capacity (*1221, Mahatma Gandhi Road, P. O. - Haridevpur, Kolkata - 700082*)	SHRI SANJIB KUMAR GOSWAMI
In Personal Capacity (*S-168 A- Uppal Sothend, Sector 49, Sohna Road, Gurugram - 122018*)	SHRI RAKESH MALHOTRA
In Personal Capacity (*H. No. 1-78/2/S/121/1, Sathi Reddy Nagar Colony, Boduppal, Hyderabad - 500092*)	SHRI N. MURALI MOHAN
In Personal Capacity (*Z-7, 1st Floor, Sector 12, Noida - 201301*)	SHRI S. N. A. RIZVI
BIS Directorate General	SHRI AJAY KUMAR LAL, SCIENTIST 'F'/SENIOR DIRECTOR AND HEAD (CHEMICAL) [REPRESENTING DIRECTOR GENERAL (*Ex-officio*)]

Member Secretary
MS SHUBHANJALI UMRAO
SCIENTIST 'B'/ASSISTANT DIRECTOR
(CHEMICAL), BIS

Bureau of Indian Standards

BIS is a statutory institution established under the *Bureau of Indian Standards Act*, 2016 to promote harmonious development of the activities of standardization, marking and quality certification of goods and attending to connected matters in the country.

Copyright

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.

This Indian Standard has been developed from Doc No.: CHD 36 (22402).

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

BUREAU OF INDIAN STANDARDS

Headquarters:

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

Regional Offices:		*Telephones*
Central	: 601/A, Konnectus Tower -1, 6th Floor, DMRC Building, Bhavbhuti Marg, New Delhi 110002	2323 7617
Eastern	: 8th Floor, Plot No 7/7 & 7/8, CP Block, Sector V, Salt Lake, Kolkata, West Bengal 700091	2367 0012 2320 9474
Northern	: Plot No. 4-A, Sector 27-B, Madhya Marg, Chandigarh 160019	265 9930
Southern	: C.I.T. Campus, IV Cross Road, Taramani, Chennai 600113	2254 1442 2254 1216
Western	: Plot No. E-9, Road No.-8, MIDC, Andheri (East), Mumbai 400093	2821 8093

Branches : AHMEDABAD. BENGALURU. BHOPAL. BHUBANESHWAR. CHANDIGARH. CHENNAI. COIMBATORE. DEHRADUN. DELHI. FARIDABAD. GHAZIABAD. GUWAHATI. HIMACHAL PRADESH. HUBLI. HYDERABAD. JAIPUR. JAMMU & KASHMIR. JAMSHEDPUR. KOCHI. KOLKATA. LUCKNOW. MADURAI. MUMBAI. NAGPUR. NOIDA. PANIPAT. PATNA. PUNE. RAIPUR. RAJKOT. SURAT. VISAKHAPATNAM.

Published by BIS, New Delhi