भारतीय मानक **IS 3025 (Part 46) : 2023**

Indian Standard

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

भाग 46 मैग्नीशियम

(दूसरा पुनरीक्षण)

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

Part 46 Magnesium

(Second Revision)

ICS 13.060.50

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July 2023 **Price Group 5**

Water Quality Sectional Committee, CHD 36

FOREWORD

This Indian Standard (Part 46) (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.

Magnesium salts are important contributors to the hardness of water which break down when heated forming scale in boilers. The magnesium concentration may vary from zero to several hundred milligrams. Chemical softening, reverse osmosis, electro dialysis, or ion exchange reduces the magnesium and associated hardness to acceptable levels.

The committee responsible for formulation of IS 3025 and IS 2488, decided to revise all the parts covered under them and publish it in separate parts. This standard supersedes **34** of IS 3025 : 1964 ‘Methods of sampling and test (physical and chemical) for water used in industry’ and **6** of IS 2488 (Part 5) : 1976 ‘Methods of Sampling and Test for Industrial Effluents: Part 5’. The first revision of his standard was published in 1994.

In this second revision following changes have been incorporated:

a) Gravimetric method as magnesium pyrophosphate has been deleted;
b) Volumetric method using EDTA has been updated;
c) Atomic absorption spectrophotometric method has been updated; and
d) Inductively coupled plasma spectroscopy method has been incorporated.

In preparation of this standard considerable assistance has been derived from the method No. 2340 C, 3111 B of ― Standard Methods for the Examination of Water and Wastewater, published by the American Public Health Association, Washington, USA, 23rd Edition, 2017.

The composition of the Committee responsible for the formulation of this standard is given in Annex 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 46 MAGNESIUM

(Second Revision)

1 SCOPE

This standard (Part 46) describes the following three methods for the determination of magnesium in water and wastewater:

a) Volumetric method using EDTA;
b) Atomic absorption spectrophotometric method; and
c) Inductively coupled plasma spectroscopy method.

2 REFERENCES

The following standards given below contain provisions which through reference in this text constitute provisions of this standard. At the time of publications, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of these standards :

IS No.	*Title*
IS 3025 (Part 2) : 2019	Methods of Sampling and Test (Physical and Chemical) for Water and Wastewater: Part 2 Determination of Selected Elements by Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES) (*first revision*)
IS 3025 (Part 65) : 2022	Methods of Sampling and Test (Physical and Chemical) for Water and Wastewater: Part 65 Application of Inductively Coupled Plasma Mass Spectrometry (ICP-MS) — Determination of 62 Elements (*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 (Part 1) : 2021	Water Quality — Sampling: Part 1 Guidance on the design of sampling programmes and sampling techniques
IS 17614 (Part 3) : 2021	Water Quality — Sampling: Part 3 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 and storage shall be done as prescribed in IS 17614 (Part 1) and IS 17614 (Part 3).

5 VOLUMETRIC METHOD USING ETHYLENEDIAMINETETRAACETIC ACID (EDTA)

5.1 Principle

Hardness is determined by the EDTA method in alkaline condition; EDTA and its sodium salts from a soluble chelated complex with certain metal ions. Calcium and magnesium ions develop wine red colour with eriochrome black T in aqueous solution at *p*H 10.0 ± 0.1. When EDTA is added as a titrant, calcium and magnesium divalent ions get complexed resulting in sharp change from wine red to blue which indicates end-point of the titration. The sharpness of the end point increases with increasing *p*H. However, the specified *p*H of 10.0 ± 0.1 is a satisfactory compromise. At a higher *p*H about 12.0 Mg^{2+} ions precipitate and only Ca^{2+} ions remain in solution. At this *p*H murexide (ammonium purpurate) indicator forms a pink colour with Ca^{2+}. When EDTA is added Ca^{2+} gets complexed resulting in a change from pink to purple which indicates end point of the reaction. From these two values, magnesium content may be calculated as the difference between hardness and calcium as $CaCO_3$. Below 1 mg/l concentration, it is desirable to use the atomic absorption spectrophotometric method.

5.2 Interference

Some metal ions form complexes with EDTA and interfere in the process of determination of calcium and magnesium by EDTA method by causing fading or indistinct end points. This interference is reduced by inhibitors to the water samples prior to titration with EDTA. When higher concentration of heavy metals are present, calcium and magnesium to be determined by non-EDTA method.

5.3 Apparatus

5.3.1 *Analytical grade Volumetric Glassware*

5.4 Reagents

5.4.1 *Eriochrome Black T (EBT) Indicator Solution*

Dissolve 0.40 g eriochrome black T solution and 4.5 g hydroxylamine hydrochloride ($NH_2OH.HCl$) in 100 ml 95 percent ethanol. This indicator is stable for more than 2 months.

5.4.2 *Murexide (Ammonium Purpurate) Indicator Solution*

This indicator changes from pink to purple at the endpoint. An indicator solution can be prepared by dissolving 150 mg of the dye in 100 g of absolute ethylene glycol. Water solutions of the dye are not stable for longer than a day. A ground mixture of the dye powder and sodium chloride provides a stable form of the indicator. It is prepared by mixing 200 mg of murexide with 100 g of solid sodium chloride and grinding the mixture to 355 µm to 300 µm. The titration should be performed immediately after the addition of the indicator because it is unstable under alkaline conditions. Endpoint recognition is facilitated by the preparation of a colour comparison blank containing 2 ml of sodium hydroxide solution, 0.2 g of solid indicator mixture (or 1 to 2 drops if a solution is used) and sufficient standard EDTA titrant (0.05 ml to 0.10 ml) to produce an unchanging colour.

5.4.3 *Inhibitors*

For most water inhibitors are not necessary. If interfering ions are present, inhibitors given below may be used.

5.4.3.1 *Hydroxylamine hydrochloride solution*

Dissolve 45 g hydroxylamine hydrochloride ($NH_2OH.HCl$) in demineralised water and dilute to 1 litre, or dissolve 4.5 g hydroxylamine hydrochloride in 100 ml of 95 percent ethanol or isopropanol.

5.4.3.2 *Sodium sulphide inhibitor*

Dissolve 5.0 g sodium sulphide ($Na_2S.9H_2O$) or 3.7 g $Na_2S.5H_2O$ in 100 ml distilled water. Tightly stopper to avoid excessive contact with air.

5.4.4 *Buffer Solution*

Dissolve 70 g of ammonium chloride and 570 ml of 30 percent ammonia solution (relative density 0.88 to 0.90) in water and make up to 1 litre.

5.4.5 *Sodium hydroxide*, 1 N

Dissolve 40 g of sodium hydroxide and dilute to 1 litre with distilled water.

5.4.6 *Standard Calcium Solution*

Dry analytical grade calcium carbonate ($CaCO_3$) in an oven at 180 °C for 1 h. Weigh 1.000 g, suspend it in distilled water and add 1 : 1 hydrochloric acid analytical reagent (AR) quality, drop wise slowly to dissolve the solid. Use minimum amount of acid. Boil for a few minutes, cool, add a few drops of methyl red indicator and adjust to orange colour with 3 N ammonium hydroxide or 1 : 1 hydrochloric acid. Dilute to 1 000 ml with distilled water. 1.00 ml = 1.00 mg calcium carbonate ($CaCO_3$).

5.4.7 *Standard EDTA Solution,* 0.01 M

Dissolve 3.723 g of analytical reagent grade disodium ethylenediaminetetraacetate dehydrate (EDTA)in water and make up to 1 litre in a volumetric flask. Standardize this with standard calcium solution. Pipette out 25 ml of standard calcium solution in 250 ml conical flask. Adjust the pH to approximately 10 with buffer solution. Dilute to about 100 ml and add 3 to 4 drops of EBT indicator solution. This will give a red colour. Titrate with 0.01 M of EDTA solution to a clear blue end point free from violet tinge. This solution will be slightly stronger than 0.01 M. Dilute the solution to exactly 0.01 M by adding calculated amount of water and recheck the strength by titrating 25 ml of standardized calcium solution by exactly the same manner as given above. This should consume exactly 25 ml of standard EDTA solution.

5.4.8 *Hydrochloric Acid*, 1 : 1

5.4.9 *Ammonium Hydroxide*, 3 N

5.5 Determination of Hardness

5.5.1 *Procedure for Drinking, Surface and Saline*

Waters Pipette an aliquot of water sample, maximum 50 ml, in a porcelain dish or 150 ml beaker and adjust the volume to approximately 50 ml. Add 1 ml hydroxylamine hydrochloride (NH2OH.HCl), solution. Add 1 ml to 2 ml buffer solution so as to achieve *p*H of 10.0 to 10.1. If the end point is not sharp sodium sulphide inhibitor solution. The addition of sodium sulphide may be omitted if copper, zinc, lead, cobalt and nickel are absent and if the sample contains less than 0.25 mg of iron and 0.025 mg of manganese. Add 2 ml eriochrome black T indicator solution, titrate with standard EDTA solution stirring rapidly in the beginning and slowly towards the end till end point is reached when all the traces of red and purple colour disappear and solution is clear sky blue in colour. Blank titration, carried out in a similar way as that for sample, may be used for comparison.

5.5.2 *Procedure for Waste Waters and Highly Polluted Waters*

Digest an aliquot of the sample with 3 ml of distilled concentrated nitric acid in a beaker on a hot plate and evaporate to near dryness cautiously making sure that the sample does not boil. Repeat digestion with nitric acid till the digestate is light in colour. Evaporate to near dryness and cool the beaker. Add a small quantity of 1 : 1 hydrochloric acid (5 ml) and warm on a hotplate or steam-bath to dissolve the residue. Cool, adjust to a suitable volume and take an aliquot of this digested sample. Proceed as given in **5.5.1**.

5.5.3 *Determination of Calcium*

5.5.3.1 *Sample preparation*

Because of the high *p*H used in this procedure, the titration should be performed immediately after the addition of the alkali and indicator. Use 50 ml of sample or a smaller portion diluted to 50 ml so that the calcium content is about 5 mg to 10 mg. Analyze hard waters with alkalinity higher than 300 mg/l $CaCO_3$ by taking a smaller aliquot and diluting to 50 ml, or by neutralization of the alkalinity with acid, boiling for one minute and cooling before beginning the titration.

5.5.3.2 Add 2 ml of sodium hydroxide solution or a volume sufficient to produce a *p*H of 12 to 13 and stir. Add 0.1 g to 0.2 g of the indicator murexide-sodium chloride mixture selected (or 1 to 2 drops if a solution is used). Add EDTA titrant slowly with continuous stirring to the proper end point. Check the end point by adding 1 to 2 drops of titrant in excess to make certain that no further colour change occurs.

5.5.4 *Determination of Magnesium*

5.5.4.1 *Calculation*

Total hardness as $CaCO_3$ mg/l, A =

$$\frac{\text{C x D x 1 000}}{\text{Volume of sample}} \qquad \ldots(1)$$

where

C = volume of EDTA required by sample, in ml; and

D = mass of $CaCO_3$ equivalent to 1ml EDTA titrant, in mg.

Calcium hardness as $CaCO_3$ mg/l, B =

$$\frac{\text{E x D x 1000}}{\text{Volume of sample taken for determination of calcium}} \qquad \ldots(2)$$

where

E = volume of EDTA used by sample, in ml; and

D = mass of $CaCO_3$ equivalent to 1ml EDTA titrant, in mg.

Magnesium hardness, as $CaCO_3$ mg/l = A-B ...(3)

Magnesium, as Mg mg/l = (A-B) x 0.243 …(4)

6 ATOMIC ABSORPTION SPECTROMETRIC METHOD

6.1 Principle

This method is applicable to the analysis of raw and drinking waters and can be used for waters having a magnesium content from 0.02 mg/l to 0.2 mg/l. For samples containing higher concentration smaller volume of the sample must be taken for the analysis.

6.2 Interferences

Chemical interferences are common. These can be overcome by the addition of a releasing agent, that is, lanthanum chloride when air-acetylene (oxidizing) flame is used.

6.3 Apparatus

Atomic absorption spectrophotometer set up and equipped with an appropriate burner for air-acetylene flame or nitrous oxide-acetylene flame and a hollow cathode lamp for magnesium with wavelength of 285.2 nm.

6.4 Reagents

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

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

6.4.3 *Acetylene*, standard commercial grade.

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

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

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

6.4.7 *Lanthanum Solution*

Dissolve 58.65 g lanthanum oxide (La_2O_3), in 250 ml concentrated hydrochloric acid. Add acid slowly until material is dissolved and dilute to 1 000 ml with water.

6.4.8 *Stock Magnesium Solution*

Dissolve 0.165 8 g magnesium oxide (MgO) in a minimum amount of nitric acid. Add 10.0 ml of concentrated nitric acid and dilute to 1 000 ml (1.00 ml = 100 µg).

6.4.9 *Standard Magnesium solution*

Prepare a series of standard magnesium solution by diluting stock magnesium solution (5.4.8) with water containing 1.5 ml of concentrated HNO3/l. Stock standard solution can be taken from a number of commercial supplier. Alternatively, it can be prepared as described in **5.4.8.**

6.5 Procedure

6.5.1 *Sample Preparation*

Mix 10 ml of lanthanum solution (**5.4.5**) into 100 ml of sample solution before aspiration.

6.5.2 *Instrument Operation*

6.5.2.1 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.

6.5.2.2 Install a hollow-cathode lamp for magnesium in the instrument and set the wavelength at 285.2 nm. Set the slit width as suggested by manufacturer for magnesium 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 (**6.4.2**) and adjust the flow rate as suggested by the manufacturer to give maximum sensitivity for magnesium being measured. Turn on acetylene (**6.4.3**) and adjust flow rate. Ignite the flame and let it stabilize for a few min.

6.5.2.3 Aspirate a blank consisting of distilled water (**6.4.4**) containing 1.5 ml of concentrated nitric acid and 10 ml lanthanum solution. Set zero the instrument. Aspirate the magnesium 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 magnesium 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 magnesium.

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

6.5.3 *Standardization*

Prepare at least three standard magnesium solutions containing lower concentration 0.1 mg/l Mg to bracket the expected metal concentration of a sample. Prepare a reagent blank of 100 ml volume with distilled water (**6.4.4**) containing 1.5 ml of concentrated nitric acid (**6.4.6**) and 10 ml lanthanum solution (**6.4.7**). Aspirate the reagent blank and carry out zero adjustment. Aspirate blank and zero the instrument. Mix 10 ml of lanthanum solution (**6.4.7**) into the standard solutions before aspiration. 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.

6.5.4 *Analysis of Sample*

Rinse nebulizer by aspirating distilled water (**6.4.4**) containing 1.5 ml concentrated nitric acid/l and zero instrument. Aspirate a sample and determine its absorbance.

5.6 Calculation

Prepare a calibration curve on linear graph paper by plotting absorbance of the standard reading versus their original concentration of standards before dilution with lanthanum solution. Alternatively, read the concentration directly from the instrument read-out if the instrument is so equipped. If sample has been diluted, multiply by the appropriate dilution factor.

6 INDUCTIVELY COUPLED PLASMA SPECTROSCOPY METHOD

Magnesium 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 magnesium.

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*)
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Confederation of Indian Industry, New Delhi	Dr Kapil K Narula Dr Sipika Chauhan (*Alternate*)
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Envirocare Laboratories Private Limited, Thane	Dr Priti Amritkar Shri Nilesh Amritkar (*Alternate*)
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Gujarat Pollution Control Board, Gandhinagar	Dr D. N. Vansadia Shri K. B. Vaghela (*Alternate*)
Haryana State Pollution Control Board	Shri Jatinder Pal Singh
Himachal Pradesh State Pollution Control Board, Government of Himachal Pradesh	Dr T. B. Singh Er Praveen Gupta
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Maharashtra State Pollution Control Board, Mumbai	Dr V. R. Thakur Shri S. C. Kollur (*Alternate*)
Ministry of Jal Shakti Department of Drinking Water and Sanitation	Shri D A Rajasekhar Shri Sumit Priyadarshi (*Alternate*)
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National Institute of Oceanography, Vishakhapatnam	Dr VVSS Sarma Dr Durbar Ray (*Alternate*)
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