**IS 3025 (Part 46) : 1994**
**(Reaffirmed 1999)**
**Edition 2.2**
**(2003-03)**

*भारतीय मानक*

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

## भाग 46 मैगनिशियम

*(पहला पुनरीक्षण)*

*Indian Standard*

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

## PART 46 MAGNESIUM

*( First Revision )*

(Incorporating Amendment Nos. 1 & 2)

UDC 628.1.032 : 628.3 : 543.3 [546.46]

**BUREAU OF INDIAN STANDARDS**
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NEW DELHI 110002

**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.

Magnesium ranks eighth among the elements in order of abundance and is a common constituent of natural water. 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, electrodialysis, or ion exchange reduces the magnesium and associated hardness to acceptable levels.

The technical committee responsible for formulation of IS 3025 and IS 2488 and its parts, decided to revise all the parts covered under them and publish it in separate parts.

This standard supersedes **34** of IS 3025 : 1964 and **6** of IS 2488 (Part 5) : 1976. In the preparation of this standard, considerable assistance has been derived from ‘Standard methods for the examination of water and wastewater’ published by the American Public Health Association, Washington, USA, 17th Edition, 1989.

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

This edition 2.2 incorporates Amendment No. 1 (September 2000) and Amendment No. 2 (March 2003) . Side bar indicates modification of the text as the result of incorporation of the amendments.

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* )’.

# *Indian Standard*

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

## PART 46 MAGNESIUM

## *( First Revision )*

### 1 SCOPE

This standard prescribes three methods for the determination of magnesium:

a) Gravimetric method as magnesium pyrophosphate;

b) Volumetric method using EDTA; and

c) Atomic absorption spectrophotometric method.

### 2 REFERENCES

The Indian Standards listed below are the necessary adjuncts to this standard:

| *IS No.* | *Title* |
|---|---|
| 264 : 1964 | Nitric acid ( *second revision* ) |
| 3025 (Part 1) : 1986 | Methods of sampling and test (physical and chemical) for water and wastewater: Part 1 Sampling ( *first revision* ) |
| 7022 (Part 1) : 1973 | Glossary of terms relating to water, sewage and industrial effluents, Part 1 |
| 7022 (Part 2) : 1979 | Glossary of terms relating to water, sewage and industrial effluents, Part 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 SAMPLING AND STORAGE

The sampling and storage shall be done as prescribed in IS 3025 (Part 1) : 1986.

### 5 GRAVIMETRIC METHOD AS MAGNESIUM PYROPHOSPHATE

#### 5.1 Principle

Diammonium hydrogen phosphate quantitatively precipitates magnesium in ammonical solution as magnesium ammonium phosphate. The precipitate is ignited and weighed as magnesium pyrophosphate. Below 1 mg/l, atomic absorption spectrophotometric method is desirable.

#### 5.2 Interference

The solution should be reasonably free from aluminium, calcium, iron, manganese, silica, strontium and suspended matter. It should not contain more than about 3.5 g ammonium chloride.

#### 5.3 Apparatus

**5.3.1** *Vacuum Pump or Other Source of Vacuum*

**5.3.2** *Filter Flasks*

**5.3.3** *Filter Crucibles* — medium porosity; 30 ml.

#### 5.4 Reagents

**5.4.1** *Methyl Red Indicator Solution*

Dissolve 100 mg of methyl red sodium salt in distilled water and dilute to 100 ml.

**5.4.2** *Hydrochloric Acid* — 1 : 1, 1 : 9 and 1 : 99.

**5.4.3** *Ammonium Oxalate Solution*

Dissolve 10 g $(NH_4)_2\ C_2O_4 + H_2O$ in 250 ml distilled water. Filter, if necessary.

**5.4.4** *Ammonium Hydroxide — Concentrated* — 1 : 19.

**5.4.5** *Nitric Acid* — Concentrated ( *see* IS 264 : 1964 ).

**5.4.6** *Diammonium Hydrogen Phosphate Solution*

Dissolve 30 g of diammonium hydrogen phosphate $(NH_4)_2\ HPO_4$ in distilled water and make up to 100 ml.

**5.4.7** *Urea*

#### 5.5 Procedure

**5.5.1** *Pre-treatment of Polluted Water and Wastewater Samples*

Mix the sample pretreated if so required and transfer a suitable volume (50 to 100 ml) to 250 ml conical flask or a beaker. Add 5 ml concentrated nitric acid and a few boiling chips or glass beads. Bring to a slow boil and evaporate on a hot plate to the lowest volume possible (about 10 to 20 ml) before precipitation or salting occurs. Add 5 ml concentrated nitric acid cover with a watch glass and heat to obtain a gentle refluxing action. Continue heating and adding concentrated nitric acid as necessary until digestion is complete as shown by a light-coloured clear solution. Do not let sample dry during digestion. Add 1 to 2 ml concentrated nitric acid and warm slightly to dissolve any remaining residue. Wash down beaker walls and watch glass with water and then filter, if necessary. Transfer filtrate to 100 ml volumetric flask, with two 5-ml portions of water adding these rinsings to the volumetric flask. Cool, dilute to mark and mix thoroughly. Take portions of this solution for the determination.

**5.5.2** *Removal of Calcium and Other Metals as Oxalates*

To 200 ml of the sample pretreated if so required containing about 50 mg of calcium, add a few drops of methyl red indicator and 1 : 1 hydrochloric acid. Sufficient acid must be present in the solution to prevent the precipitation of calcium oxalate when ammonium oxalate solution is added. Introduce 50 ml of ammonium oxalate solution and 15 g of urea. Boil the solution gently until the methyl red changes its colour to yellow. Filter the precipitate and wash with small volume of cold water until free from chloride.

**5.5.3** *Determination of Magnesium*

To the combined filtrate and washings from **5.5.2** containing not more than 60 mg magnesium add 50 ml of concentrated nitric acid and evaporate carefully to dryness on a hot plate. Do not let reaction become too violent during the later part of the evaporation stay in constant attendance to avoid losses through spattering. Moisten residue with 2 to 3 ml of concentrated hydrochloric acid, add 20 ml of distilled water, warm, filter and wash. To the filtrate add 3 ml of concentrated hydrochloric acid 2 to 3 drops of methyl red solution, and 10 ml of $(NH_4)_2HPO_4$ solution. Cool and add concentrated ammonium hydroxide drop by drop, stirring constantly until the colour changes to yellow, stir for 5 minutes add again 5 ml of concentrated ammonium hydroxide and stir vigorously for 10 minutes more. Let it stand overnight and filter through filter paper. Wash with 1 : 19 ammonium hydroxide. Transfer to an ignited, cooled and weighed crucible. Dry precipitate thoroughly and burn paper off slowly, allowing circulation of air. Heat at about 500°C until residue is white. Ignite for 30 minutes at 1 100°C to constant mass.

**5.5.4** *Calculation*

$$\text{Magnesium, mg/litre} = \frac{M}{V} \times 218.4 \times 10^3$$

where

$M$ = mass in mg of magnesium pyrophosphate, and

$V$ = volume in ml of sample.

### 5.6 Precision and Accuracy

The method is precise to ± 0.5 mg or better, and the accuracy is comparable if interferences are absent.

## 6 VOLUMETRIC METHOD USING EDTA

### 6.1 Principle

When water sample containing both calcium and magnesium is titrated with EDTA at *p*H 10, using Eriochrome black-T as indicator, which estimates values of calcium and magnesium. In a separate titration against EDTA at *p*H 12 to 13 range using murexide or Patton and Reeder's indicator, calcium is selectively estimated. From these two values magnesium content may be calculated. Below 1 mg/l concentration it is desirable to use atomic absorption spectrophotometric method.

### 6.2 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 addition of certain inhibitors to the water samples prior to titration with EDTA.

**6.3 Apparatus** — Hot plate.

### 6.4 Reagents

**6.4.1** *Indicator Solutions*

**6.4.1.1** *Patton and Reeder and indicator solution*

This indicator solution permits the direct titration of calcium in the presence of magnesium. It produces a sharp colour change from wine red to pure blue at the end point. It is prepared by mixing 1 g of Patton and Reeder reagent with 100 g of sodium chloride/ potassium chloride.

**6.4.1.2** *Murexide ( ammonium purpurate ) indicator solution*

This indicator changes from pink to purple at the end point. 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 to 300 microns. The tiration should be performed immediately after the addition of the indicator because it is unstable under alkaline conditions. End point recognition is facilitated by the preparation of colour comparison blank containing 2.0 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 to 0.10 ml) to produce an unchanging colour.

**6.4.1.3** *Eriochrome black T indicator ( EBT Indicator )*

Dissolve 0.5 g of EBT indicator and approximately 4.5 g of hydroxylamine hydrochloride in 100 ml of rectified spirit (ethanol or methanol).

**6.4.2** *Standard Zinc Solution* — 0.01 M.

Dissolve 0.653 8 g of pure zinc dust or granules of 99.9 percent purity in 20 ml of approximately 1 : 1 hydrochloric acid, warm if necessary. Cool and make up to 1 litre exactly in a volumetric flask. Preserve this solution in a tightly closed glass bottle.

**6.4.3** *Buffer Solution*

a) 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.

b) Sodium hydroxide 1 N — Dissolve 40 g of sodium hydroxide and dilute to 1 litre with distilled water.

**6.4.4** *Standard Ethylene Diamine Tetra Acetic Acid ( EDTA ) Solution* — 0.001 M.

Dissolve 3.75 g of disodium ethylene diamine tetra-acetate dihydrate in water and make up to 1 litre in a volumetric flask. Standardize this with standard zinc solution. Pipette out 25 ml of standard zinc solution in a 250 ml conical flask. Adjust the *p*H 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 standard zinc solution by exactly the same manner as given above. This should consume exactly 25.0 ml of standard EDTA solution.

**6.4.5** *Triethanolamine Solution* — 10 percent (m/v).

**6.4.6** *Potassium Cyanide Solution* — 10 percent (m/v).

NOTE — Preserve in a polyethylene bottle.

**6.4.7** *Hydroxylamine Hydrochloride Solution* — 10 percent (m/v).

**6.4.8** *Inhibitor and Complexing Agents* — For most waters there is no need to utilize an inhibitor or complexing agent like, Magnesium salt of 1, 2- cyclohexane diamine tetra acetic acid (MgCDTA). Occasionally water containing interfering ions other than $Ca^{++}$ and $Mg^{++}$ requires adding an appropriate inhibitor or complexing agents to give a clear, sharp change in colour to the end point. Avoid the use of sodium cyanide inhibitor.

### 6.5 Pre-treatment of Polluted Water and Waste-water Samples

As prescribed in **5.5.1**.

### 6.6 Determination of Calcium

**6.6.1** *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 to 10 mg. Analyse 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.

**6.6.2** Add 2.0 ml of sodium hydroxide solution or a volume sufficient to produce a *p*H of 12 to 13. Stir. Add 0.1 to 0.2 g of the indicator murexide-sodium chloride mixture selected (or 1 to 2 drops if a solution is used). Alternatively, approximately 1 g of the mixture of Patton and Reeder reagent and sodium chloride or potassium chloride may be 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. Let the volume used be $V_1$.

**6.6.3** *Determination of Magnesium*

Take a suitable aliquot of the solution, expected to contain approximately 10 to 30 mg of calcium and magnesium in a 500 ml conical flask. Add 10 ml of hydroxylamine hydrochloride solution, 2 ml of potassium cyanide solution and 25 ml of triethanolamine solution. Dilute to 150 to 200 ml and add sufficient quantity of buffer solution to bring the *p*H to 10.0 ± 0.1. Add 3 to 4 drops of EBT indicator solution and titrate with

0.01 M EDTA solution till the red colour changes to pure blue end point free from violet tinge. It is easier to note the end point by comparing the colour with that of previously titrated solution having a pure blue end point, where a slight excess of EDTA solution has been added.

NOTE — If magnesium is not present, it should be added as 1 : 1 magnesium EDTA complex, in order to get the proper colour change at the end point. This can be done by either adding 1 ml of 0.1 magnesium EDTA (1 : 1) complex before proceeding for titration, or, incorporating small amount of $Mg^{2+}$ ions in EDTA solution before its standardization.

**6.6.4** *Calculation*

$$\text{Magnesium (as Mg), mg/l percent by mass} = \frac{0.024\,35 \times 1\,000 \times (V_2 - V_1)}{V}$$

where

$V$ = volume in ml of the sample taken for the test;

$V_1$ = volume in ml of EDTA consumed in titration for calcium determination in the same aliquot of solution of sample; and

$V_2$ = volume in ml of EDTA solution consumed in titration.

## 7 ATOMIC ABSORPTION SPECTROPHOTOMETRIC METHOD

**7.1** This method is applicable to the analysis of raw and drinking waters and can be used for waters having a magnesium content up to 5 mg per litre. For samples containing higher concentration smaller volume of the sample must be taken for the analysis. In general nitrous oxide-acetylene flame should be used if the composition of the sample is complex or unknown.

### 7.2 Interferences

Chemical interferences are common. These can be overcome by the addition of a releasing agent, that is, lanthanum chloride [if air-acetylene (oxidizing) flame is used] or cesium chloride (if nitrous oxide-acetylene flame is used).

**7.3** 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.

### 7.4 Reagents

**7.4.1** *Hydrochloric Acid* — 1 N and 0.1 N.

**7.4.2** *Lanthanum Chloride* — 20 g/l of La.

To a 1 litre volumetric flask add 24 g of lanthanum oxide ($La_2O_3$) atomic absorption spectrometry grade). Slowly and cautiously add 50 ml of concentrated hydrochloric acid while stirring to dissolve the lanthanum oxide. Make up to the mark with water.

**7.4.3** *Cesium Chloride* — 20 g/l of Cs.

Dissolve 25 g of cesium chloride in 1 litre of 0.1 N hydrochloric acid.

**7.4.4** *Standard Magnesium Solution* — 1 000 mg/l

Dry a portion of magnesium oxide (MgO) at 180°C for 1 hour. Weigh 1.66 ± 0.01 g and dissolve in 1 N hydrochloric acid. Dilute with the same acid to 1 000 ml in a volumetric flask. Store the solution in a polyethylene bottle.

### 7.5 Procedure

**7.5.1** *Preparation of Test Solution*

Sample containing particulate matter after acidification shall be filtered to prevent clogging of the nebulizer and burner systems. To a 100 ml volumetric flask, add 10 ml of lanthanum chloride solution (**7.4.2**) if an air-acetylene flame is to be used, or 10 ml of cesium chloride solution (**7.4.3**) if a nitrous oxide-acetylene flame is to be used. Add 10 ml of the sample and make up to the mark with 0.1 N hydrochloric acid.

NOTE — If the concentration of the sample is higher, then appropriately smaller volume of the sample shall be used.

**7.5.2** *Preparation of Blank Solution*

Prepare a blank test simultaneously using the same reagents in the same quantities and following the same procedure, but replacing the volume of the test sample used in **7.5.1** by equal volume of water.

**7.5.3** *Preparation of Set of Calibration Standard Solutions*

To a series of 100 ml volumetric flasks with the aid of pipettes add 0, 2.5, 5, 10, 15, 20 and 25 ml of the magnesium standard solution (**7.4.4**), add either 10 ml of the lanthanum chloride solution or 10 ml of the cesium chloride solution as in **7.5.1** and make up to the mark with 0.1 N hydrochloric acid.

**7.5.4** *Preparation of Calibration Curve*

Aspirate the blank and calibration solutions with aspirating distilled water in between. Note down the absorbance value. Prepare calibration curve by plotting the absorbance readings against concentration of magnesium standard solution (**7.5.3**).

**7.5.5** Aspirate the test solution (**7.5.1**) and note the absorbance value and read the concentration of magnesium from the calibration curve against the absorbance value obtained.

### 7.6 Calculation

$$\text{Magnesium (as mg), mg/l} = C \times \frac{V_1}{V_0}$$

where

$C$ = concentration in mg/l of magnesium calculated from the calibration curve having taken into account the blank value,

$V_0$ = volume in ml of the original sample taken for analysis, and

$V_1$ = volume in ml to which the sample has been diluted.

## 8 PRECISION

In a single laboratory using distilled water with added calcium concentrations of 9.0 and 36 mg/l the standard deviations were 0.3 and 0.6 mg/l respectively. Recoveries at both these levels were 99 percent.

In an interlaboratory study with over 30 participating laboratories, Qs four waters with calcium contents in the range 100 to 300 mg/l and magnesium contents from 7 to 85 mg/l were analysed. The coefficients of variation varied from 3.5 to 4.6 percent for calcium and from 2.9 to 6.9 magnesium.

# ANNEX A

( *Foreword* )

## COMMITTEE COMPOSITION

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( *Continued on page* 7 )

( *Continued from page* 6 )

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| *Members* | |
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