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मानक



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Mazdoor Kisan Shakti Sangathan

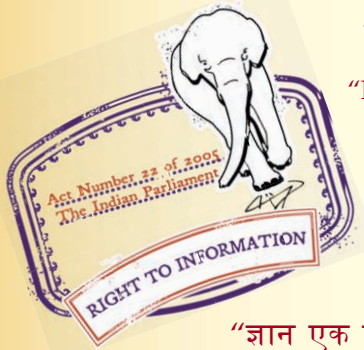
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IS 3025-21 (2009): Methods of sampling and test (physical and chemical) for water and wastewater, Part 21: Total hardness [CHD 32: Environmental Protection and Waste Management]



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Bhartrhari—Nitiśatakam

“Knowledge is such a treasure which cannot be stolen”

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भारतीय मानक
जल और अपशिष्ट जल के नमूने लेने तथा परीक्षण
(भौतिक एवं रसायन) की पद्धतियाँ
भाग 21 कठोरता
(दूसरा पुनरीक्षण)

Indian Standard
METHODS OF SAMPLING AND TEST (PHYSICAL AND
CHEMICAL) FOR WATER AND WASTEWATER
PART 21 HARDNESS
(*Second Revision*)

ICS 13.060.50

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BUREAU OF INDIAN STANDARDS
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NEW DELHI 110002

FOREWORD

This Indian Standard (Part 21) (Second Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Environment Protection and Waste Management Sectional Committee had been approved by the Chemical Division Council.

In most natural waters, hardness is mainly due to calcium and magnesium ions. In some waters, measurable concentrations of iron, aluminium, manganese, barium, zinc and other metals may be present. Total hardness of water is the sum of the concentrations of all the metallic cations other than cations of alkali metals, expressed as equivalent calcium carbonate concentration. When the hardness is numerically greater than the sum of carbonate alkalinity and bicarbonate alkalinity; the amount of hardness which is equivalent to total alkalinity is called 'carbonate hardness' and the amount of hardness in excess of this is called 'non-carbonate hardness'. Some waters containing high concentrations of borates, phosphates, silicates, may contribute to total alkalinity.

There is no ISO Standard on the subject. The first revision of this standard was published in 1983 by superseding **16** of IS 3025 : 1964 'Methods of sampling and test (physical and chemical) for water used in industry'. In this revision methods for determination of carbonate and non-carbonate hardness have been incorporated based on the relevance of these requirements in this field. Accordingly, the title of this standard is changed to 'Hardness'.

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 : 1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standard

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

PART 21 HARDNESS

(Second Revision)

1 SCOPE

1.1 This standard prescribes two methods for determination of total hardness, namely (a) Ethylenediamine tetraacetic acetate acid (EDTA) method, and (b) Method based on analytical data and also the methods for determination of carbonate and non-carbonate hardness.

1.2 The EDTA method is based on the reaction of calcium and magnesium salts with ethylenediamine tetraacetic acid or its disodium salt and is applicable to all types of water but not applicable to wastewater. The analytical data method is based on computation from analytical results of the sample and is applicable to water and waste water. In case of dispute, the method based on analytical data shall be used.

2 REFERENCES

The Indian 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 the standards indicated below:

<i>IS No.</i>	<i>Title</i>
3025	Methods of sampling and test (physical and chemical) for water and waste water:
(Part 1) : 1987	Sampling (<i>first revision</i>)
(Part 23) : 1986	Alkalinity (<i>first revision</i>)
7022	Glossary of terms relating to water, sewage and industrial effluents:
(Part 1) : 1973	Part 1
(Part 2) : 1979	Part 2

3 TERMINOLOGY

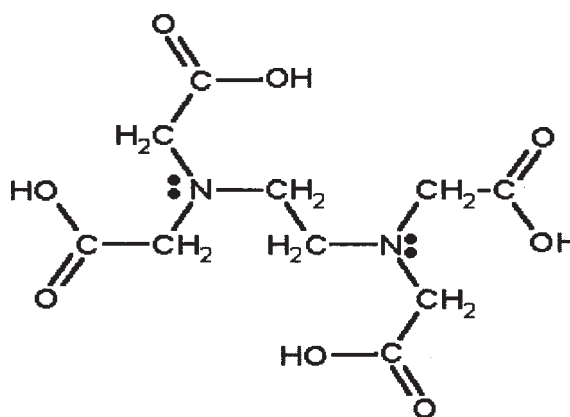
For the purpose of this standard, definitions given in IS 7022 (Part 1) and IS 7022 (Part 2) shall apply.

4 SAMPLING AND PRESERVATION

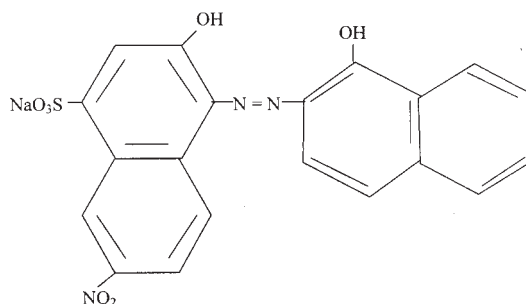
Sampling and sample preservation shall be done as prescribed in IS 3025 (Part 1).

5 EDTA METHOD FOR DETERMINATION OF TOTAL HARDNESS**5.1 Principle**

This method depends on ability of ethylenediamine tetraacetic acid ($C_{10}H_{16}O_8N_2$)

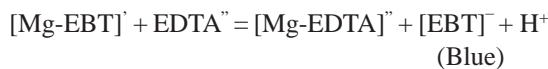
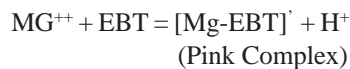


or its disodium salt to form stable complexes with calcium and magnesium ions. When the dye eriochrome black T (EBT) ($C_{20}H_{13}N_3O_7S$) is added to a solution containing calcium and



magnesium ions at pH 10.0 a wine red complex is formed. This solution is titrated with standard solution of disodium salt of EDTA, which extracts calcium and

magnesium from the dye complex and the dye is changed back to its original blue colour. Eriochrome black T is used to indicate the end-point for the titration of calcium and magnesium together.



5.2 Interferences

5.2.1 The EDTA forms stable complexes with iron, manganese, copper, lead, cobalt, zinc and nickel. Heavy metal interferences can be eliminated by complexing the metals with cyanide. In the presence of cyanide, the procedure may be used even when iron, copper, zinc or lead concentrations are as high as 10 mg/l.

5.2.2 The higher oxidation states of manganese above Mn^{++} react rapidly with the indicator to form discoloured oxidation products. Hydroxylamine hydrochloride reagent may be used to reduce manganese to divalent state. The divalent manganese interference can be removed by addition of one or two small crystals of potassium ferrocyanide.

5.2.3 In presence of high aluminium concentrations, the blue colour near end point starts disappearing and reverts to red.

5.2.4 Phosphate and carbonate ions may precipitate calcium at the pH of titration.

5.3 Reagents

5.3.1 Purity of the Reagents

Unless specified otherwise, only pure chemicals and tannin 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.

5.3.2 Buffer Solution

Dissolve 16.9 g ammonium chloride (NH_4Cl) in 143 ml concentrated ammonium hydroxide (NH_4OH), add 1.25 g of magnesium salt of EDTA and dilute to 250 ml with distilled water. Store the solution in a polyethylene bottle tightly stoppered to prevent loss of ammonia or pick-up of carbon dioxide for no longer than 1 month. Dilute 10 ml of the solution to 100 ml with distilled water and check that the pH value is 10.0 ± 0.1 .

5.3.3 In the absence of magnesium salt of EDTA, dissolve 1.179 g disodium salt of EDTA (AR quality) and 780 mg magnesium sulphate ($\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$) or

644 mg magnesium chloride ($\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$) in 50 ml of distilled water. Add this solution to 16.9 g ammonium chloride and 143 ml concentrated ammonium hydroxide with mixing and dilute to 250 ml with distilled water. To attain the highest accuracy, adjust to exact equivalence through appropriate addition of a small amount of EDTA or magnesium sulphate or chloride. The exact amount can be determined by taking an appropriate aliquot of buffer and titrate it with disodium salt of EDTA as in **5.4.1**. Keep the solutions tightly stoppered to prevent loss of ammonia or absorbance of carbon dioxide and do not store for more than a month. Dilute 10 ml of the solution to 100 ml with distilled water and check that the pH value is 10.0 ± 0.1 .

5.3.4 Standard Calcium Solution

1.00 ml = 1.00 mg calcium carbonate (CaCO_3). 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 AR quality, dropwise 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.

5.3.5 Eriochrome Black T Indicator Solution

Dissolve 0.40 g eriochrome black T and 4.5 g hydroxylamine hydrochloride ($\text{NH}_2\text{OH HCl}$) in 100 ml 95 percent ethanol. This indicator is stable for more than 2 months. Alternatively, dissolve 0.5 g eriochrome black T in 100 ml triethanolamine or 2-methoxyethanol or mixed 0.5 g EBT dye and 100 g sodium chloride in a pestle and mortar. Store in a tightly stoppered bottle. All indicator formulations tend to deteriorate especially when exposed to moisture. If the end point colour change is not sharp enough it is either due to the presence of some interfering ions or due to deterioration of the indicator. In the latter case, addition of inhibitor sodium cyanide or sodium sulphide (NaCN or Na_2S) does not sharpen the end point colour change.

5.3.6 Inhibitors

For most waters inhibitors are not necessary. If interfering ions are present, inhibitors given in **5.3.6.1** to **5.3.6.4** may be used.

5.3.6.1 Hydroxylamine hydrochloride solution

Dissolve 45 g hydroxylamine hydrochloride ($\text{NH}_2\text{OH.HCl}$) in demineralised water and dilute to 1 litre, or dissolve 4.5 g hydroxylamine hydrochloride in 100 ml of 95 percent ethanol or isopropanol.

5.3.6.2 Potassium ferrocyanide crystals**5.3.6.3 Sodium sulphide inhibitor**

Dissolve 5.0 g sodium sulphide ($\text{Na}_2\text{S}\cdot 9\text{H}_2\text{O}$) or 3.7 g $\text{Na}_2\text{S}\cdot 5\text{H}_2\text{O}$ in 100 ml distilled water. Tightly stopper so as to avoid excessive contact with air.

5.3.6.4 Sodium cyanide solution

Dissolve 2.5 g sodium cyanide (NaCN) in demineralised water and dilute to 100 ml. As sodium cyanide is extremely poisonous, it should be handled with care. The solution should not be made acidic and should be flushed down the drain with large amounts of water when it is to be disposed off.

5.3.7 Standard EDTA Solution

Dissolve 3.723 g EDTA ($\text{Na}_2\text{H}_2\text{C}_{10}\text{H}_{12}\text{ON}_2\cdot 2\text{H}_2\text{O}$) which has been dried overnight in a sulphuric acid desiccator, in demineralized water and dilute to 1 000 ml. The reagent is stable for several weeks and large volume is usually prepared. Check the reagent by titrating 25 ml of standard calcium solution as described in 5.3.4. Store in polyethylene bottles.

5.4 Procedure**5.4.1 Standardization**

Pipette 25.0 ml of standard calcium solution in a porcelain basin and adjust the volume to 50 ml with distilled water. Add 1 ml buffer solution (5.3.2). Add 1 to 2 drops of indicator (5.3.5), titrate slowly with continuous stirring until the reddish tinge disappears, adding last few drops at 3 to 5 interval. At the end point the colour is sky blue.

5.4.2 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 ($\text{NH}_2\text{OH}\cdot\text{HCl}$), solution. Add 1 to 2 ml buffer solution so as to achieve pH of 10.0 to 10.1. If the end point is not sharp (as determined by practice) add 2 ml sodium cyanide or sodium sulphide inhibitor solution. The addition of sodium cyanide or 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. If manganese is present, add 1 or 2 small crystals of potassium ferri cyanide [$\text{K}_4\text{Fe}(\text{CN})_6\cdot 3\text{H}_2\text{O}$]. Stir and wait for at least 5 min until sodium ferri cyanide precipitates. 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.

5.4.3 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 hot-plate 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 5.4.2.

NOTES

- 1 For water with very low hardness (less than 5 mg/l) micro burette may be used.
- 2 Selection of sample size may be made such that the result lies between 200 to 300 mg/l of hardness (as CaCO_3).

5.5 Calculation

Calculate the hardness as follows:

$$\text{Total hardness as } (\text{CaCO}_3), \text{ mg/l} = [1\ 000 (V_1 - V_2) / V_3] \times CF$$

where

- V_1 = volume in ml of the EDTA standard solution used in the titration for the sample,
- V_2 = volume in ml of the EDTA solution used in the titration for blank,
- V_3 = volume in ml of the sample taken for the test,
- $CF = X_1/X_2$ = correction factor for standardization of EDTA,
- X_1 = volume in ml of standard calcium solution taken for standardization, and
- X_2 = volume of ml of EDTA solution used in the titration.

5.6 Report

Report hardness in mg/l as CaCO_3 rounded to the first decimal place when the value is less than 10 mg/l and to the nearest unit if the value is more than 10 mg/l.

5.7 Precision and Accuracy

The precision is within 1 mg/l. The accuracy depends on the interfering substances present. In the absence of any interference, it is within 1 mg/l.

NOTE — Standard deviation of 1.2 mg/l has been reported.

6 METHOD BASED ON ANALYTICAL DATA FOR DETERMINATION OF TOTAL HARDNESS

6.1 Principle

Total hardness computed from the concentration of the different metallic cation (other than alkali metals) in the sample but most often the cations taken into account are calcium, magnesium, iron, aluminium, zinc, strontium, barium and manganese.

6.2 Calculation

$$\text{Total hardness (as CaCO}_3\text{), mg/l} = (2.497 \times \text{mg/l Ca}) + (4.116 \times \text{mg/l Mg}) + (2.69 \times \text{mg/l Fe}) + (5.567 \times \text{mg/l Al}) + (1.531 \times \text{mg/l Zn}) + (1.822 \times \text{mg/l Mn}) + (0.894 \times \text{mg/l Ba}) + (1.319 \times \text{mg/l Sr})$$

6.3 Expression of Values of Hardness in Different Units

Hardness of water shall be expressed in terms of mg/l of calcium carbonate. However, for the sake of convenience, factors for converting the value of

hardness into different-units that are in use are given in Table 1.

7 CARBONATE HARDNESS AND NON-CARBONATE HARDNESS

7.1 Procedure

Determine the total alkalinity as per IS 3025 (Part 23), total hardness as in 6 and calculate the carbonate and non-carbonate hardness as given in 7.2.

7.2 Calculation

7.2.1 Carbonate Hardness

- When total hardness is greater than total alkalinity, carbonate hardness (as CaCO_3 mg/l) is equal to total alkalinity (as CaCO_3).
- When total hardness is equal to or less than total alkalinity, carbonate hardness (as CaCO_3 mg/l) is equal to total hardness (as CaCO_3).

7.2.2 Non-carbonate Hardness

Non-carbonate hardness (as CaCO_3), mg/l = total hardness – total alkalinity

Table 1 Conversion Factors for Different Units for Expressing Hardness
(Clause 6.3)

SI No.	Unit of Hardness	Mg/l (as CaCO_3)	British Degree, Grains per Imperial Gallon (as CaCO_3)	American Degree, Grains per US Gallon (as CaCO_3)	French Degree, Parts per 100 000 (as CaCO_3)	German Degree, Parts per 100 000 (as CaO)	Russian Degree, Parts per Million (as Ca)
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)
i)	mg/l as CaCO_3	1.00	0.07	0.058	0.10	0.056	0.40
ii)	British Degree	14.29	1.00	0.83	1.43	0.79	5.72
iii)	American Degree	17.16	1.20	1.00	1.72	0.96	6.86
iv)	French Degree	10.00	0.70	0.58	1.00	0.56	4.00
v)	German Degree	17.86	1.25	1.04	1.79	1.00	7.14
vi)	Russian Degree	2.50	0.18	0.15	0.25	0.14	1.00

ANNEX A

(Foreword)

COMMITTEE COMPOSITION

Environment Protection and Waste Management Sectional Committee, CHD 32

<i>Organization</i>	<i>Representative(s)</i>
In personal capacity (40/14, C.R. Park, New Delhi 110019)	PROF DILIP BISWAS (<i>Chairman</i>)
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Gujarat Pollution Control Board, Gandhinagar	DR G. K. TRIVEDI
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Industrial Toxicology Research Centre, Lucknow	DR S. K. BHARGAVA
Ministry of Defence, DRDO, New Delhi	SHRI J. C. KAPOOR DR P. K. RAI (<i>Alternate</i>)

IS 3025 (Part 21) : 2009

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Municipal Corporation of Delhi, Delhi	SHRI RAVI DASS SHRI P. K. KHANDELWAL (<i>Alternate</i>)
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Scientist 'E' (CHD), BIS

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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 latest issue of 'BIS Catalogue' and 'Standards : Monthly Additions'.

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Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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