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IS 3550 (1965): Methods of test for routine control for water used in industry [CHD 32: Environmental Protection and Waste Management]



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“Knowledge is such a treasure which cannot be stolen”



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**IS : 3550 - 1965**  
( Reaffirmed 2009 )

*Indian Standard*  
**METHODS OF TEST FOR  
ROUTINE CONTROL FOR WATER  
USED IN INDUSTRY**

Ninth Reprint JANUARY 2006  
( Including Amendment No 1 )

UDC 543.3 : 663.63

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**BUREAU OF INDIAN STANDARDS**  
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NEW DELHI 110002

Gr 8

*January* 1967

**AMENDMENT NO. 2 OCTOBER 2000**  
**TO**  
**IS 3550 : 1965 METHODS OF TEST FOR ROUTINE**  
**CONTROL FOR WATER USED IN INDUSTRY**

*( Page 4, Foreword, clause 0.2, line 5 )* — Delete the word 'unqualified'.

*( Page 4, Foreword, clause 0.2, lines 7 and 8 )* — Substitute 'The reference methods, which require a well equipped laboratory, may be found in appropriate parts of IS 3025 series' *for* 'Additional tests, which require a well equipped laboratory, may be found in IS 3025:1964'.

( CHD 12 )

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Reprography Unit, BIS, New Delhi, India

**AMENDMENT NO. 1 JANUARY 2000**  
**TO**  
**IS 3550 : 1965 METHODS OF TEST FOR ROUTINE**  
**CONTROL FOR WATER USED IN INDUSTRY**

( *Page 10, clause 9.1.3* ) — Insert the following at the end of the clause:

'NOTE — The multiplication factor 0.667 may vary from 0.550 to 0.900 depending upon the nature of sample. Therefore, it is advisable to determine the factor for a particular sample, using gravimetric method [ *see IS 3025 ( Part 16 ) Methods of sampling and test (physical and chemical) for water and waste water : Part 16 Filterable residue ( total dissolved solids ) ( first revision ) . ]'*

( *Page 13, clause 11.2, Note 2, line 3* ) — Insert the following after the words 'buffer solution':

'(pH to be maintained between 10.0 to 10.1)'

( CHD 12 )

***Indian Standard***

**METHODS OF TEST FOR  
ROUTINE CONTROL FOR WATER  
USED IN INDUSTRY**

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## *Indian Standard*

# **METHODS OF TEST FOR ROUTINE CONTROL FOR WATER USED IN INDUSTRY**

### **0. FOREWORD**

**0.1** This Indian Standard was adopted by the Indian Standards Institution on 7 October 1965, after the draft finalized by the Water Sectional Committee had been approved by the Chemical Division Council.

**0.2** In this standard such tests for industrial water are covered which do not require a well-equipped laboratory and where the apparatus required is that which may be conveniently set up and used near the point of sampling. The tests are of a comparatively simple nature and are suitable for use by unqualified personnel after simple training. They are not intended for use in cases of dispute or for purposes of exhaustive investigation. Additional tests, which require a well equipped laboratory, may be found in IS : 3025-1964\*

**0.3** In the preparation of this standard, considerable assistance has been taken from U.S. 1427 : 1962 'Routine control methods of testing water used in industry', issued by the British Standards Institution.

**0.4** 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†.

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### **1. SCOPE**

**1.1** This standard lays down the methods for routine control testing of water used in industry.

### **2. SAMPLING**

**2.1** Representative test samples of the water to be tested shall be prepared as prescribed in IS : 3025-1964\*. However, in tests where specific sampling procedures are prescribed, these shall be followed.

\*Methods of sampling and test (physical and chemical) for water used in industry.

†Rules for rounding off numerical values (*revised*).

### 3. GENERAL PRECAUTIONS AND DIRECTIONS

**3.1 Quality of Reagents** — Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070-1960\*) shall be used in tests.

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

**3.2** Except where otherwise specified, before carrying out a determination, the sample shall be filtered to obtain a clear filtrate. For some tests, for example, free carbon dioxide, filtration is not permissible. In such cases, the sample may be allowed to stand for some time to allow the suspended matter to settle.

**3.3** Where tests are to be carried out on the unfiltered sample, as much as possible of the suspended matter shall be allowed to settle before removal of the portion required.

**3.4** Many analytical procedures given are subject to interference from other constituents that may be present. Whenever interference is encountered or suspected and no specific procedure is laid down for overcoming it, steps shall be taken to eliminate the interference without adversely affecting the analysis itself.

**3.5** Where the range of amount of the substance being determined is stated, it is usually given in either micrograms ( $\mu\text{g}$ ) or milligrams. The procedures laid down for the various tests cover the stated ranges, but may, if necessary, be modified either by taking a smaller volume of the sample and diluting with distilled water or in other cases by taking a larger volume of the sample and evaporating it.

**3.6** As some of the reagents are corrosive and some of the waters may be bacteriologically unsafe, the pipettes should not be filled by sucking with the mouth. The necessary suction may be obtained by a rubber ball-type pipette filler

**3.7** Unless otherwise stated, measurement of the portion of the sample required for a test shall be carried out by means of a measuring cylinder.

**3.8** Except where otherwise stated, measurement of reagents shall be carried out by means of a burette or a one-mark pipette.

**3.9** For determinations for which calibrated colour discs are available, these may be used provided instructions of the manufacturer are followed.

### 4. COLOUR

**4.1** A qualitative description of the colour of the sample, along with an indication of the amount and appearance of any suspended matter should be given, for example, slightly turbid, turbid, highly turbid, flocculent, etc.

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\*Specification for water, distilled quality (*revised*)

## 5. TURBIDITY

**5.0 Outline of the Method** — The sample is matched against standard suspensions of fullers' earth in water.

**5.1 Terminology** — For the purpose of this test, the following definition shall apply.

**5.1.1 Silica Scale Unit** — Turbidity imparted by 1 mg of fullers' earth when suspended in 1 000 ml of distilled water.

**5.2 Preparation of Turbidity Standards** — Mix slowly with constant stirring 5 000 g of fullers' earth, previously dried and sifted through 75-micron IS Sieve, with distilled water and dilute it to 1 000 ml. Agitate intermittently for one hour and then allow to stand for 24 hours. Withdraw the supernatant liquid without disturbing the sediment. Evaporate about 50 ml of the removed liquid, dry the residue at  $105 \pm 2^\circ\text{C}$  and weigh the residue to determine the amount of clay in suspension. Prepare turbidity standards with this standardized stock suspension with distilled water. A drop of saturated mercuric chloride solution may be added as preservative. The standards are stable for three months.

**5.3 Procedure** — Pour the sample, after thorough shaking, into a clear glass bottle of suitable capacity, say, one litre. Compare it with the turbidity standards contained in similar bottles, holding them against a suitable background and using a source of light which illuminates them equally and is placed so that no rays reach the eye directly. The sample and the standards shall be shaken simultaneously immediately before comparison. If the sample has turbidity over 100 units, dilute it with distilled water before testing and multiply the result with an appropriate factor.

NOTE — Comparison of turbidity may also be done with the help of suitable instruments

**5.3.1 Report** — Record the turbidity readings in accordance with Table 1.

TABLE 1 RECORDING OF TURBIDITY READINGS

SL No.	TURBIDITY		RECORD TO THE NEAREST (UNITS)
	Greater than (units)	Up to (units)	
(1)	(2)	(3)	(4)
i)	0.0	1.0	0.1
ii)	1	10	1
iii)	10	100	5
iv)	100	400	10
v)	400	700	50
vi)	700	—	100

## 6. pH VALUE

**6.0 Methods** — The determination shall be carried out either by the electrometric method (6.1) or the indicator method (6.2). The electrometric method shall be the referee method.

NOTE — Chemically resistant glass bottles shall be used for the sample and this test shall be done as soon as possible after the sample is drawn. Samples kept for a long time are likely to change in pH due to escape of dissolved gases such as carbon dioxide.

**6.1 Electrometric Method** — The determination may be made by any pH meter with glass electrode.

### 6.2 Indicator Method

#### 6.2.1 Apparatus

**6.2.1.1 Nessler tubes** — matched.

**6.2.2 Reagents** — A series of indicators and buffer solutions is required for this method. Methods of preparation of these are given in Appendix A.

**6.2.3 Procedure** — Take 10 ml of the sample in a glass tube and determine the approximate pH by using a universal indicator. Repeat using a solution of the indicator (about 1/20 of the volume of the liquid being tested) which corresponds to the approximate pH found above. Compare the colour produced with a series of buffer solutions of known pH, each containing the same proportion of the indicator. Report as pH, the pH of the buffer solution which matches with that of the sample.

## 7. ELECTRICAL CONDUCTANCE

**7.0 General** — The unit of conductance is the mho or reciprocal ohm. Specific conductivity is the conductance at a specified temperature across a column of liquid one square centimetre in area and one centimetre long, and is expressed in mhos per centimetre. This is an inconveniently large unit for water testing and it is usual to use the micromho per centimetre known as the 'dionic unit', which is one-millionth part of a mho per centimetre.

**7.1 Apparatus** — Several kinds of apparatus are available. They generally consist of two parts (7.1.1 and 7.1.2).

**7.1.1 Conductivity Cell** — containing a pair of electrodes. The solution to be tested is pouted into this cell. There are many forms of cell. One of the most convenient types is provided with a funnel for filling, a drain for emptying, and an overflow for maintaining constant level. Electrodes for use with samples having very low dissolved solids (such as condensates) should not be coated with platinum black. Platinum black which

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has been heated to redness until it is grey is suitable. Bright platinum or gold or heavily gold plated electrodes may be used.

**7.1.1.1** Some instruments are designed to work with a particular form of conductivity cell, and are then calibrated directly in conductivity units. Other instruments, primarily introduced for more general application, are calibrated on conductance units and their readings require multiplication by a factor known as the cell constant which shall be determined by experiment.

**7.1.2 Measuring Instrument** — for measuring the electrical conductance (or the resistance which is the inverse of conductance) between the electrodes of the cell. There are several satisfactory commercial models. Operators, unless they have adequate facilities, would be well advised to purchase a ready-made instrument.

## 7.2 Procedure

**7.2.1** Determine the cell constant, if necessary, either directly with a standard potassium chloride solution (say 0.002 N) or by comparison with a cell the constant of which is known accurately. (In the latter case, the concentration and nature of the electrolytes in the liquid which is used for the comparison should be the same as those of the liquids with which the cell is likely to be used in practice.)

**7.2.2** Use some of the sample to wash out the conductivity cell thoroughly. Fill the conductivity cell with the sample. Measure the conductivity in accordance with the instructions of the instrument manufacturer.

## 7.3 Calculation

### 7.3.1 For Instrument Reading Resistance

$$\begin{array}{l} \text{Dionic units (or micromhos} \\ \text{per centimetre)} \end{array} = \frac{1}{rk} \times 10^6$$

where

$r$  = resistance in ohms per centimetre, and

$k$  = cell constant.

### 7.3.2 For Instrument Reading Conductance

$$\begin{array}{l} \text{Dionic units (or micromhos} \\ \text{per centimetre)} \end{array} = ck$$

where

$c$  = conductance in micromhos per centimetre, and

$k$  = cell constant

## 8. SPECIFIC GRAVITY

### 8.1 Apparatus

**8.1.1 Hydrometer** — having a range of 0.998 0 to 1.010 0 at 15.5 /15.5°C.

NOTE — Do not dry the hydrometer with a cloth. Allow it to air-dry by placing it horizontally on a clean cloth laid on a flat surface.

**8.1.2 Hydrometer Jar** — of minimum internal diameter 65 mm and minimum internal depth 350 mm.

### 8.2 Procedure

**8.2.1** Since hydrometers are particularly sensitive to capillary conditions, it is recommended that a small amount of a wetting agent be added to the sample to wet thoroughly the hydrometer stem in the neighbourhood of the liquid meniscus.

**8.2.2** For checking the reliability of the hydrometer, the specific gravity of distilled water at 15.5°C should be determined. The instrument is satisfactory if the reading is between 0.999 6 and 1.000 4.

**8.2.3** Determine the specific gravity by means of the hydrometer. Correct the hydrometer reading to 15.5°C by using the temperature corrections supplied with the hydrometer.

## 9. DISSOLVED SOLIDS

**9.0 General** — A quick estimate of dissolved solids may be obtained from determination of either electrical conductance or specific gravity of the sample.

### 9.1 Dissolved Solids from Electrical Conductance

#### 9.1.1 Reagents

**9.1.1.1 Acetic acid** — 5.percent (v/v).

**9.1.1.2 Phenolphthalein indicator solution** — Dissolve 0.1 g of phenolphthalein in 60 ml of rectified spirit (conforming to IS: 323-1959\*) and dilute with distilled water to 100 ml (see IS: 2263-1962†).

**9.1.2 Procedure** — If the sample is alkaline to phenolphthalein, add acetic acid drop by drop and with careful mixing until the pink colour disappears. Determine the electrical conductivity of the neutralized sample in micromhos/cm.

\*Specification for rectified spirit (*revised*)

†Methods of preparation of indicator solutions for volumetric analysis

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**9.1.2.1** If the conductivity lies outside the range of the instrument, redetermine it after diluting the neutralized sample appropriately and apply the necessary correction in calculation.

### 9.1.3 Calculation

Dissolved solids, mg/l =  $0.667 \times$  electrical conductivity.

## 9.2 Dissolved Solids from Specific Gravity

**9.2.0** The measurement of the specific gravity of boiler waters provides, in many cases, an approximate method of determining the dissolved solids. It has been found that for a wide range of boiler waters, each 0.000 1 of specific gravity at 15.5°C is approximately equal to 110 mg/l of dissolved solids. This value shows a variation of the order of 10 per cent.

**9.2.1 Procedure** — Determine the specific gravity of the sample at 15.5 /15.5°C as prescribed in **8**.

### 9.2.2 Calculation

Dissolved solids, mg/l = (specific gravity at 15.5 /15.5°C–1.000 0)  $\times 1.1 \times 10^6$

NOTE — If the specific gravity of the sample is above the limit of the hydrometer being used, dilute a known volume of the sample correctly and correct for dilution as follows

Dissolved solids, mg/l = Dissolved solids in diluted

$$\text{sample} \left\{ 1 + \frac{\text{Volume of water added}}{\text{Volume of original sample}} \right\}$$

## 10. ALKALINITY

**10.0 Outline of the Method** — The alkalinity is determined by titration against standard acid. The different types of alkalinities are determined by using, separately, phenolphthalein and methyl orange as indicators in two titrations, and phenolphthalein after addition of barium chloride in the third titration.

### 10.1 Reagents

**10.1.1 Phenolphthalein Indicator Solution** — same as in **9.1.1.2**.

**10.1.2 Standard Hydrochloric Acid** — 0.02 N.

**10.1.3 Barium Chloride Solution** — 10 percent (w/v).

**10.1.4 Methyl Orange Indicator** — Dissolve 0.01 g in 100 ml of distilled water.



## 10.2 Procedure

**10.2.1** To 100 ml of the sample contained in a porcelain basin, add 1 ml of phenolphthalein indicator and if a pink colouration is obtained, titrate with standard hydrochloric acid, stirring with a glass rod, till the disappearance of the pink colour. Preserve the titrated solution.

**10.2.2** To the solution preserved in **10.2.1**, add 3 drops of methyl orange indicator and continue the titration until the colour changes from yellow to orange.

**10.2.3** Determine the alkalinity to phenolphthalein of another 100 ml aliquot of the sample after adding a crystal of sodium sulphate and 10 ml of barium chloride solution and stirring well for 2 minutes. Disregard any reappearance of the pink colour.

NOTE 1 — Organic matter often interferes with the methyl orange alkalinity, and the determination of this alkalinity by titration is therefore unreliable. This can be calculated if alkalinity to phenolphthalein and alkalinity to phenolphthalein after addition of barium chloride are known.

NOTE 2 — The indicator methods are not suitable for highly coloured waters and for these a potentiometric method is recommended. The *pH* values at which the titrations are stopped are 8.3 for determining alkalinity to phenolphthalein and 4.5 for determining alkalinity to methyl orange. In case a *pH* meter is not available, either the sample may be diluted to a point when the end point can be detected visually or drops of the respective indicator may be put in the depressions of a porcelain spotting tile, and a drop of the titrated sample put alongside the indicator from time to time until one of the spots on the tile shows a distinct change in colour.

## 10.3 Calculation

- Alkalinity to phenolphthalein (*P*) (as  $\text{CaCO}_3$ ),  $\text{mg/l} = 10 V_1$
- Alkalinity to methyl orange (*M*) (as  $\text{CaCO}_3$ ),  $\text{mg/l} = 10 V_2$
- Alkalinity to phenolphthalein after addition of barium chloride [*P*( $\text{BaCl}_2$ )] (as  $\text{CaCO}_3$ ),  $\text{mg/l} = 10 V_3$

where

$V_1$  = volume in ml of standard acid used in **10.2.1**,

$V_2$  = total volume in ml of standard acid used in **10.2.1** and **10.2.2**, and

$V_3$  = volume in ml of standard acid used in **10.2.3**.

## 10.4 Calculation of Hydroxide Alkalinity, Carbonate Alkalinity, Bicarbonate Alkalinity and Total Alkalinity

**10.4.1** *In the Absence of Alkali Phosphates* In the absence of any treatment with alkali phosphates, the various alkalinities may be calculated from values of *P*, *M* and *P* ( $\text{BaCl}_2$ ) with the help of Table 2.

TABLE 2 CALCULATION OF VARIOUS ALKALINITIES OF WATER

(Clause 10.4.1)

VALUES OF $P$ , $M$ AND $P(\text{BaCl}_2)$	HYDROXIDE ALKALINITY	CARBONATE ALKALINITY	BICARBONATE ALKALINITY	TOTAL ALKALINITY
(1)	(2)	(3)	(4)	(5)
$P = \text{Zero}$	Nil	Nil	$M$	$M$
$2P < M$	Nil	$2P$	$M - 2P$	$M$
$2P = M$	Nil	$2P$	Nil	$M$
$2P > M$	$2P - M$	$2(M - P)$	Nil	$M$
$P = M$	$M$	Nil	Nil	$M$
$P > P(\text{BaCl}_2)$	$P(\text{BaCl}_2)$	$2[P - P(\text{BaCl}_2)]$	Nil	$2P - P(\text{BaCl}_2)$
$P(\text{BaCl}_2) = \text{Zero}$	Nil	$2P$	No correla- tion	No correla- tion

**10.4.2 In the Presence of Alkali Phosphates** — In the presence of phosphate treatment and where the phosphate is present as a trisodium salt, part of the alkalinity to phenolphthalein will be due to the alkalinity combined with the phosphate ( $\text{PO}_4$ ). If the phosphate content (as  $\text{PO}_4$ ) is expressed as mg/l, the effect on  $P$  will be equal to one half of the content in mg/l of phosphate, and this shall be subtracted from  $P$  reading before using Table 2. The alkalinity to phenolphthalein after addition of barium chloride is not affected by the presence of phosphate.

**10.5 Range of Accuracy** — The method is suitable in the range of 0 to 500 mg/l (as  $\text{CaCO}_3$ ).

## 11. TOTAL HARDNESS

**11.0 Outline of the Method** — The method is based on the reaction of calcium and magnesium salts with ethylenediamine tetra-acetate (EDTA); eriochrome black T is used as the indicator.

### 11.1 Reagents

**11.1.1 Indicator Solution** — Dissolve 0.5, g of eriochrome black T in 100 ml of triethanolamine.

**11.1.2 Buffer Solution** — Dissolve 40 g of borax in approximately 800 ml of distilled water. Dissolve 10 g of sodium hydroxide, 10 g of sodium potassium tartrate and 5 g of sodium sulphide in 100 ml of distilled water. When cool, mix the two solutions and dilute to 1 000 ml with distilled water. The reagent should not be used for more than one month after preparation.

**11.1.3 Standard EDTA Solution** — (0.02N). Dissolve 3.72 g of disodium ethylenediamine tetra-acetate dihydrate per litre of the solution.

**11.2 Procedure** — Measure 50 ml of the sample into a 250-ml conical flask. Add 2 to 3 drops of indicator solution and mix; add 0.5 ml of buffer solution and mix. Titrate immediately with EDTA solution with continuous mixing until the colour changes from red to blue. As the end point is approached, the solution shows some blue colouration but a definite reddish tinge is observed and is discharged at the end point.

NOTE 1 — If the colour of the sample is too dark to observe the end point, it will be necessary to decolorize it by the following process

Add 1 g of decolorizing powder (activated charcoal powder giving a blank of less than 0.5 mg/l in terms of calcium carbonate) to 100 ml of distilled water containing 1 ml of approximately 4 N hydrochloric acid. Mix thoroughly and filter through a pad of paper pulp supported on a filter disc resting in a glass adapter. Use a Witts filter or similar apparatus and a moderate vacuum. Wash the bed of decolorizing powder with 100 ml of distilled water and discard the nitrate and washings. Take 100 to 500 ml of the filtered coloured sample, as appropriate, and adjust the pH with 4 N hydrochloric acid to 2.5 (a drop gives a yellow colour with bromophenol blue indicator). Filter through the bed of decolorizing powder, re-filtering if necessary, until the colour is satisfactory.

To the decolorized sample add a small piece of litmus paper and then add ammonia dropwise with constant stirring until the paper just turns blue. Use this sample for the determination of hardness.

NOTE 2 — It is recommended that for hardness below 100 mg/l, 100 ml of the sample be used with 7 to 9 drops of indicator solution and 1.0 ml of buffer solution whereas for hardness appreciably above 200 mg/l, a smaller volume than 50 ml of the sample be taken and diluted to 50 ml with distilled water. For samples of very low hardness, take 500 ml of the sample, add 6 drops of indicator solution and 2 ml of buffer solution. Titrate as in 11.2.

### 11.3 Calculation

$$\text{Total hardness (as CaCO}_3\text{), mg/l} = \frac{1000 \times V_1}{V_2}$$

where

$V_1$  = volume in ml of standard EDTA solution consumed, and

$V_2$  = volume in ml of the sample taken for the test.

**11.4 Range of Accuracy** — The method is applicable in the range 0.1 to 25 mg in terms of calcium carbonate. The accuracy depends on the interfering substances present.

## 12. SULPHATES

### 12.0 General

**12.0.1** The test is not applicable to waters containing appreciable amounts of calcium and magnesium as these are also titratable with EDTA. The test is generally applicable to boiler waters of hardness less than 20 mg/l as CaCO<sub>3</sub>.

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**12.0.2 Outline of the Method** — A measured excess of standard barium chloride solution is added to the sample and the excess of barium chloride estimated by titration against EDTA.

### 12.1 Reagents

**12.1.1 Dilute Hydrochloric Acid** – 0.5 N.

**12.1.2 Standard Barium Chloride Solution (0.04 N)** — Dissolve 4.886 g of barium chloride dihydrate in distilled water and make up to 1 litre. One millilitre of the solution is equivalent to 1.92 mg of sulphate (as SO<sub>4</sub>).

**12.1.3 Buffer Solution** — Dissolve 67.5 g of ammonium chloride in 570 ml of ammonium hydroxide (sp gr 0.92) and dilute with distilled water to 1 litre.

**12.1.4 Eriochrome Black T Indicator Solution** — same as in 11.1.1.

**12.1.5 Standard EDTA Solution (0.02N)** — same as in 11.1.3.

**12.2 Procedure** Pipette 10 ml of the sample into a porcelain dish and acidify with 1 ml of dilute hydrochloric acid. Add exactly 10 ml of standard barium chloride solution and stir well. Add 1 ml of buffer solution and several drops of eriochrome black T indicator solution. Titrate with standard EDTA solution to a blue colour which does not change on addition of further drops of indicator solution.

### 12.3 Calculation

$$\text{Sulphates (as SO}_4\text{), mg/l} = 192 \left( 10 - \frac{V}{2} \right)$$

where

$V$  = volume in ml of standard EDTA solution consumed.

NOTE – To obtain the result for sulphates in terms of sodium sulphate (Na<sub>2</sub>SO<sub>4</sub>), multiply the figure obtained in 12.3 by 1.48,

**12.4 Range of Accuracy** – The method is suitable in the range of 100 to 1350 mg/l (as SO<sub>4</sub>).

## 13. SULPHITES

**13.0 Outline of the Method** - The sample is titrated against standard iodate-iodide solution using starch indicator.

### 13.1 Reagents

**13.1.1 Dilute Hydrochloric Acid** — Add 200 ml of concentrated hydrochloric acid (conforming to IS : 265-1962\*) to 100 ml of distilled water.

\*Specification for hydrochloric acid (*revised*).

**13.1.2 Starch Indicator Solution** Intuiate 5 g of starch and 0.01 g of mercuric iodide with 30 ml of cold distilled water and slowly pour it with constant stirring into one litre of boiling distilled water. Boil for 3 minutes. Allow to cool and decant off the supernatant clear liquid.

**13.1.3 Standard Iodate-Iodide Solution** Weigh accurately 0.713 g of potassium iodate and dissolve in about 150 ml of distilled water. Add 7 g of potassium iodide and 0.5 g of sodium bicarbonate, when dissolved, dilute the solution to exactly 1 litre.

## 13.2 Procedure

**13.2.1** Since sulphite tends to be oxidized in contact with air, great care shall be exercised in sampling. The following procedure shall be adopted:

Collect the sample by running it to the bottom of a 250 ml bottle but allowing about eight to ten times the volume of the bottle to run to waste before taking the sample. For this test a filtered sample shall not be used unless suspended matter is present containing interfering substances for example, non oxide, in which case, insert a filter in the sampling line.

**13.2.2** Measure 2 ml of dilute hydrochloric acid into a 150 ml porcelain basin and add 100 ml of the sample (**13.2.1**) and 1 ml of starch indicator to it. Titrate immediately with standard iodate iodide solution, stirring continuously, until a faint permanent blue colour is obtained.

## 13.3 Calculation

Sulphites (as  $\text{SO}_3$ ), mg/l =  $8 \cdot 1$

where

$V$  = volume in ml of standard iodite iodide solution consumed

**13.4 Range of Accuracy** — The method is suitable in the range of 10 to 200 mg/l (as  $\text{SO}_3$ )

## 14. PHOSPHATES

**14.0 Outline of the Method** - The sample is reacted with ammonium molybdate and hydroquinone. The blue colour obtained is matched against that produced with a series of standard phosphate solutions.

### 14.1 Apparatus

**14.1.1 Nessler Tubes** — 50 ml capacity

### 14.2 Reagents

**14.2.1 Standard Phosphate Solution** — Dissolve 1.430 g of potassium dihydrogen phosphate in distilled water and make up to 1 litre. Dilute 10 ml of the solution to 100 ml. One millilitre of the diluted solution contains 0.1 mg of phosphate (as  $\text{PO}_4$ )

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**14.2.2 Carbonate-Sulphite Solution** — Dissolve 130 g of anhydrous potassium carbonate and 24 g of sodium sulphite heptahydrate in 500 ml of distilled water.

**14.2.3 Ammonium Molybdate Solution** — Dissolve 8.8 g of ammonium molybdate in about 100 ml of distilled water. Separately, add 38 ml of concentrated sulphuric acid slowly and carefully to about 300 ml of distilled water; mix, cool and then add the dilute acid to the molybdate solution. Dilute the mixture to 500 ml.

**14.2.4 Sulphuric Acid** — 6.5 percent (v/v).

**14.2.5 Hydroquinone Solution** — Dissolve 0.5 g of hydroquinone in 50 ml of 0.02 N sulphuric acid. Store in a dark bottle.

### 14.3 Procedure

**14.3.1** Into nine numbered Nessler tubes (No. 1 to 9), pipette out 0, 0.5, 1.0, 1.5, 2.0, 2.5, 3.0, 3.5 and 4.0 ml of standard phosphate solution and make up the volume in each to 5 ml by adding distilled water. Into a further two numbered Nessler tubes (No. 10 and 11) pipette out 5 ml of the filtered sample.

**14.3.2** Into each of a further eleven Nessler tubes (No. 12 to 22), measure 2 ml of carbonate-sulphite solution. Adjust the temperature of the standards, sample and all reagents to  $25 \pm 2^\circ\text{C}$ .

**14.3.3** Measure into Nessler tubes No. 1 to 10, two millilitres of ammonium molybdate solution and into Nessler tube No. 11, two millilitres of sulphuric acid. Mix, add 1 ml of hydroquinone solution to each of the tubes No. 1 to 11 and mix again.

**14.3.4** After allowing to stand for 5 minutes, pour the contents of each of the Nessler tubes No. 1 to 11 into a separate Nessler tube containing the carbonate-sulphite solution (No. 12 to 22) and then back again into the original Nessler tube. Compare the colour of Nessler tube No. 10 with the tubes No. 1 to 9.

NOTE 1 — If the solution in tube No 11 (blank) has more than a trace of colour, comparison may be difficult and a calibrated disc method is to be resorted to

NOTE 2 — Boiler waters are sometimes highly coloured. They may be tested only by the calibrated disc method wherein a blank is used for compensation.

### 14.4 Calculation

$$\text{Phosphates (as PO}_4\text{), mg/l} = 20 \times V$$

where

$V$  = volume in ml of standard phosphate solution matching with the sample.

**14.5 Range of Accuracy** — The method is suitable in the range of 0 to 80 mg/l (as PO<sub>4</sub>).

## 15. CHLORIDES

**15.0 Outline of the Method** — The sample, after neutralization, is titrated against standard silver nitrate solution using potassium chromate indicator.

### 15.1 Reagents

**15.1.1 Aluminium Hydroxide Suspension** — Dissolve 125 g of potassium or ammonium alum in one litre of distilled water. Precipitate the aluminium by adding ammonium hydroxide slowly and with stirring. Wash the precipitate by successive decantation with several portions of distilled water until free from sulphates.

**15.1.2 Hydrogen Peroxide** — 30 percent.

**15.1.3 Calcium Carbonate**

**15.1.4 Dilute Nitric Acid** — approximately 0.1 N.

**15.1.5 Potassium Chromate Solution** — Dissolve 5 g of potassium chromate in distilled water and make up to 100 ml. Add silver nitrate solution to produce a slight red precipitate and filter.

**15.1.6 Standard Silver Nitrate Solution** — Dissolve 4.791 g of silver nitrate, dried at 105 ± 2°C, in distilled water and make up to 1 000 ml. The solution shall be kept in the dark.

### 15.2 Procedure

**15.2.1** Use 100 ml of the sample, filtered if necessary, for the titration. If the sample is coloured, decolorize by adding 3 ml of aluminium hydroxide suspension. Stir thoroughly and after a few minutes filter and wash with 10 to 15 ml of distilled water. If sulphites are present, add 1 ml of hydrogen peroxide with stirring.

**15.2.2** Place the sample treated as in 15.2.1 in a porcelain basin. If the pH of the sample is less than 6.8, add a small amount of calcium carbonate to the sample in the basin so as to neutralize the acidity. If the pH is above 10, determine the amount of dilute nitric acid required to neutralize 100 ml of the sample, and add this amount of the acid to the portion used for the chloride determination, and then add a trace of calcium carbonate. Add 1 ml of potassium chromate solution and titrate with standard silver nitrate solution with constant stirring until there is perceptible reddish coloration. Subtract 0.2 ml from the titration figure to allow for the excess of reagent required to form silver chromate.

NOTE — If the sample requires more than 25 ml of silver nitrate solution, repeat the determination with a smaller quantity of the sample diluted to 100 ml with distilled water. If chlorides are present in very small quantities, use a larger volume of the sample.

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### **15.3 Calculation**

Chlorides (as Cl), mg/l –  $10 V$

where

$V$  = volume in ml of standard silver nitrate solution consumed.

**15.4 Range of Accuracy** — The method is suitable in the range of 0 to 500 mg/l (as Cl).

## **16. SILICA**

**16.0 Outline of the Method** — The silicates in the sample are precipitated in the form of a yellow silico-molybdate complex by treatment with ammonium molybdate. Reducing substances, for example, sulphites and hydrazine shall not be present in this test, otherwise a blue colour is produced. If reducing substances are present, these should be oxidized with a few drops of 0.1 percent potassium permanganate solution.

### **16.1 Apparatus**

**16.1.1 Nessler Tubes** — 50 ml capacity.

### **16.2 Reagents**

**16.2.1 Standard Silica Solution** - Dissolve **1.566** g of sodium fluoro-silicate in 400 ml of distilled water, heating until solution is complete. Cool and dilute to 500 ml. Just before use, dilute 50 ml of the solution to 1 000 ml. One millilitre of the diluted solution contains 0.05 mg of silica ( as  $\text{SiO}_2$  ).

**16.2.2 Ammonium Molybdate Solution** — 10 percent (v/v).

**16.2.3 Dilute Sulphuric Acid** — 2N.

**16.2.4 Sodium Citrate Solution** – 30 percent solution of sodium citrate dihydrate.

NOTE — Sodium citrate is added to prevent interference from phosphate in concentrations normally present in boiler water.

**16.3 Procedure** — Into ten numbered Nessler tubes, pipette out 0, 1.0, 2.5, 5.0, 7.5, 10.0, 12.5, 15.0, 17.5 and 20.0 ml of standard silica solution and make up to 50 ml. Into two more numbered tubes, measure 50 ml of the sample. The temperature should be between 25° and 35°C. Add 2 ml of ammonium molybdate solution in the first eleven tubes and 2 ml of water in the twelfth tube. Mix, add 4 ml of dilute sulphuric acid to all tubes and allow to stand for ten minutes at 25 to 35°C. Add to each tube 2 ml of sodium citrate solution, mix and compare the colour (a range of yellows) of the eleventh tube with the first ten standards.



NOTE — If the solution in the twelfth Nessler tube (blank) has more than a trace of colour, comparison may be difficult and a calibrated disc method shall be resorted to.

#### 16.4 Calculation

Silica (as SiO<sub>2</sub>), mg/l =  $V$

where

$V$  = volume in ml of standard silica solution matching with the sample.

**16.5 Range of Accuracy** — The method is applicable in the range of 0 to 20 mg/l (as SiO<sub>2</sub>).

### 17. ALUMINIUM

#### 17.0 General

**17.0.1 Outline of the Method** — The sample is treated with hæmatoxylin in the presence of ammonium carbonate; the solution is then acidified and the colour developed (yellow through brown to violet) is matched against that produced with a series of standard aluminium solutions.

**17.0.2** The method is unsuitable in the presence of more than 0.2 mg/l of manganese (as Mn) or more than 0.4 mg/l of iron (as Fe). With more than 0.2 mg/l of manganese the colour developed with hæmatoxylin is bleached within two minutes of its addition, that is, before the addition of acetic acid, and a golden yellow colour develops which is similar to that produced when aluminium is absent. More than 0.4 mg/l of iron produces 'off' shades usually greenish in colour. In such cases, the procedure prescribed in **31** of IS : 3025-1964\* shall be followed.

#### 17.1 Apparatus

**17.1.1 Nessler Tubes** — 50 ml capacity.

#### 17.2 Reagents

**17.2.1 Standard Aluminium Solution** — Dissolve 0.84 g of aluminium ammonium sulphate [Al<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub>·(NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub>·24H<sub>2</sub>O] in a little distilled water and dilute to 1 litre. Before use, dilute 10 ml of this solution to 100 ml. One millilitre of this solution contains 0.005 mg of aluminium (as Al).

**17.2.2 Ammonium Carbonate Solution** — 15 percent (w/v).

**17.2.3 Hæmatoxylin Solution** — Dissolve 0.1 g of hæmatoxylin by adding it to 20 ml of boiling distilled water and swirling gently. Add 25 ml of 0.02 N sulphuric acid and dilute to 100 ml in a cylinder. Preserve the solution in a dark bottle and reject it after a week.

\*Methods of sampling and test (physical and chemical) for water used in industry.

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**17.2.4 Acetic Acid** — 30 percent (w/w).

### 17.3 Procedure

**17.3.1** Into nine Nessler tubes, pipette out 0, 1.0, 2.0, 3.0, 4.0, 5.0, 6.0, 7.0 and 8.0 ml of standard aluminium solution and make up the volume to 50 ml in each tube. In a tenth Nessler tube, pipette out a suitable volume of the sample (10, 25 or 50 ml) and dilute to the 50 ml mark.

**17.3.2** Add to each Nessler tube, by means of a pipette, 2 ml of ammonium carbonate solution, mix, add 1 ml of hæmatoxylin solution and mix again. After allowing to stand for exactly ten minutes, add 2 ml of acetic acid to each tube and mix. Allow to stand for exactly 5 minutes and compare the colour of the sample with those of the standards

### 17.4 Calculation

$$\text{Aluminium (as Al), mg/l} = \frac{V_1}{V_2}$$

where

$V_1$  = volume in ml of standard aluminium solution matching the colour, and

$V_2$  = volume in ml of the sample taken for the test.

**17.5 Range of Accuracy** — The method is suitable in the range of 0 to 4 mg/l (as Al).

## 18. IRON

**18.0 Outline of the Method** — The purple colour developed with thioglycollic acid in ammoniacal solution is matched against that obtained with a series of standard iron solution.

### 18.1 Apparatus

**18.1.1 Nessler Tubes** — 50 ml capacity.

### 18.2 Reagents

**18.2.1 Standard Iron Solution** — Dissolve 0.702 g of ammonium ferrous sulphate  $[(\text{NH}_4)_2\text{SO}_4 \cdot \text{FeSO}_4 \cdot 6\text{H}_2\text{O}]$  in 100 ml of distilled water and 10 ml of concentrated sulphuric acid conforming to Analytical Reagent Grade of IS: 266-1961\*. Dilute to 1 000 ml. Before use, again dilute 10 ml of the solution to 100 ml. One millilitre of the final solution contains 0.01 mg of iron (as Fe).

**18.2.2 Citric Acid Solution** — 20 percent (w/v).

**18.2.3 Thioglycollic Acid**

**18.2.4 Ammonium Hydroxide** — 1:3 (v/v).

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\*Specification for sulphuric acid (revised).

### 18.3 Procedure

**18.3.1** Into ten Nessler tubes, pipette out 0, 0.5, 1.0, 1.5, 2.0, 2.5, 3.0, 4.0, 5.0 and 6.0 ml of standard iron solution, make up to the mark with distilled water and mix. Fill a further Nessler tube to the mark with the sample.

**18.3.2** To each of the eleven tubes, add 2 ml of citric acid solution and 0.1 ml of thioglycolic acid. Mix and add 2 ml of ammonium hydroxide. Allow to stand for 5 minutes and match the colour of the sample against those of the standards.

### 18.4 Calculation

$$\text{Iron (as Fe), mg/l} = 0.2 V$$

where

$V$  = volume in ml of standard iron solution which matches the colour obtained with the sample.

**18.5 Range of Accuracy** — The method is suitable in the range of ??? to 12 mg/l (as Fe).

## 19. CALCIUM

**19.0 Outline of the Method** — The sample is titrated against EDTA solution using murexide indicator.

### 19.1 Reagents

**19.1.1 Standard Hydrochloric Acid** — 0.02 N.

**19.1.2 Standard Sodium Hydroxide Solution** — 4 N.

**19.1.3 Triethanolamine** — 20 percent (v/v).

NOTE — Triethanolamine is added to suppress the effect of ferric iron and manganese that may be present in some waters.

**19.1.4 Murexide Indicator** — Mix 0.20 g of murexide and 100 g of sodium chloride and grind to a fine powder.

**19.1.5 Standard EDTA Solution (0.02 N)** — Dissolve 3.72 g of disodium ethylenediamine tetra-acetate dihydrate in 1 litre of the solution. One millilitre of this solution is equivalent to 1 mg of calcium (as CaCO<sub>3</sub>).

**19.2 Procedure** — Measure 100 ml (Note 1) of the sample into a porcelain basin, neutralize it by adding standard hydrochloric acid in a quantity equivalent to the alkalinity to methyl orange as determined in 10. Add 1 ml (Note 1) of standard sodium hydroxide solution and allow to stand for 5 minutes. Add 5 ml of triethanolamine and about 50 mg (Note 1)

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of murexide indicator and titrate the solution with standard EDTA solution until the colour changes from pink to purple.

NOTE 1 — For concentrations less than 10 mg/l of calcium (as CaCO<sub>2</sub>) use 500 ml of the sample, 4 ml of sodium hydroxide solution and 200 mg of murexide indicator.

NOTE 2 — Coloured samples shall be decolorized as prescribed in 11.2 Note 1. However, instead of neutralizing with ammonia, 4 N sodium hydroxide solution shall be used.

### 19.3 Calculation

$$\text{Calcium (as CaCO}_3\text{), mg/l} = \frac{1\ 000\ V_1}{V_2}$$

where

$V_1$  = volume in ml of standard EDTA solution consumed, and

$V_2$  = volume in ml of the sample taken for the test.

**19.4 Range of Accuracy** — The method is suitable in the range of 0 to 250 mg/l (as CaCO<sub>3</sub>).

## 20. MAGNESIUM

**20.1 Procedure** — The magnesium content in terms of CaCO<sub>3</sub> shall be calculated by subtracting the calcium content (mg/l in terms of CaCO<sub>3</sub>) from the total hardness content (mg/l in terms of CaCO<sub>3</sub>).

## 21. FREE CARBON DIOXIDE

**21.0 Outline of the Method** - The sample is titrated with standard sodium carbonate solution using phenolphthalein indicator.

### 21.1 Reagents

**21.1.1 Phenolphthalein Indicator Solution** — same as in 9.1.1.2.

**21.1.2 Standard Sodium Carbonate Solution** — 0.02 N.

**21.2 Procedure** - Take 100 ml of the unfiltered sample in a measuring cylinder avoiding undue agitation. Add 10 drops of phenolphthalein indicator, run in standard sodium carbonate solution 0.5 ml at a time, stirring the sample gently but thoroughly by raising and lowering a glass rod. The end point is reached when a definite pink colour persists for 5 minutes.

### 21.3 Calculation

Free carbon dioxide (as CO<sub>2</sub>), mg/l — 4.4  $V$

where

$V$  = volume in ml of standard sodium carbonate solution consumed.

**21.4 Range of Accuracy** — The method is suitable in the range of 0 to 200 mg/l (as CO<sub>2</sub>)

## 22. RESIDUAL CHLORINE

**22.0 Outline of the Method** — Residual chlorine reacts under acidic conditions with *o*-tolidine to give a yellow colour which is matched against standard colours

**22.0.1** The determination shall be carried out with the help of suitable calibrated discs

### 22.1 Reagent

**22.1.1 *o* Tolidine Reagent** — Dissolve 0.1 g of *o*-tolidine in 10 ml of concentrated hydrochloric acid and dilute with distilled water to 100 ml.

### 22.2 Procedure

**22.2.1 Free Residual Chlorine** — Fit the appropriate calibrated disc and cells into a comparator and measure the correct amount of the sample into the two cells (5, 10 or 20 ml according to the cell being used) Draw up into a squeeze bulb pipette a volume of *o*-tolidine reagent equal to 1 ml per 100 ml of sample Hold the comparator at eye level against a white background and insert the pipette into the appropriate cell until the end of the pipette is just clear of the bottom Quickly squeeze the teat so that the reagent is ejected and a few bubbles of air quickly follow to mix the solutions. Withdraw the pipette Match at once any yellow colour that may appear. Record the result as mg/l of free residual chlorine. Preserve the solution for test in **22.2.2**.

**22.2.2 Total Residual Chlorine** — Allow the solution preserved in **22.2.1** to stand for 20 minutes in dark or in subdued light and again match the colour. Record the result as mg/l of total residual chlorine.

**22.3 Range of Accuracy** — The method is suitable in the range 0 to 5 mg/l (as Cl).

## 23. AMMONIACAL NITROGEN

### 23.0 General

**23.0.1 Outline of the Method** — The sample is treated with Nessler reagent The brown colour obtained is matched against that produced with a series of standard ammonium chloride solutions.

**23.0.2** The determination shall be carried out in an atmosphere free from ammonia

### 23.1 Apparatus

**23.1.1 Nessler Tubes** — 50 ml capacity

## 23.2 Reagents

**23.2.1 Ammonia-Free Water** — This shall be prepared by redistillation of distilled water after the addition of a few drops of dilute sulphuric acid. The first runnings shall be tested for freedom from ammonia and rejected if necessary.

**23.2.2 Standard Ammonium Chloride Solution** — Dissolve 3.15 g of ammonium chloride in ammonia-free water and dilute to 1 litre. Dilute 50 ml of the solution to 500 ml. One millilitre of the diluted solution contains 0.1 mg of ammonia (as  $\text{NH}_3$ ).

**23.2.3 Potassium Sodium Tartrate Solution** — Dissolve 500 g of potassium sodium tartrate in 1 litre of ammonia-free water. Dilute to 1 000 ml with ammonia-free water.

**23.2.4 Nessler Reagent** — Dissolve 35 g of potassium iodide in 100 ml of distilled water. Add to it a cold saturated solution of mercuric chloride until, after thorough mixing, a slight red precipitate remains. Now add 120 g of sodium hydroxide and when dissolved, dilute to 1 litre. Finally, add a little more of mercuric chloride solution to produce red colour. Set aside to clear. The reagent shall be shaken occasionally.

**23.2.5 Cupric Sulphate Solution** — 10 percent (w/v).

**23.2.6 Sodium Hydroxide Solution** — 50 percent (w/v).

**23.2.7 Zinc Sulphate Solution** — 5 percent (w/v).

## 23.3 Procedure

**23.3.1** Into eight Nessler tubes, pipette out 0, 0.2, 0.4, 0.6, 0.8, 1.2, 1.6 and 2.0 ml of standard ammonium chloride solution and make up the volume in each tube to 50 ml. In a ninth Nessler tube, pipette out a suitable volume of the sample (10, 25 or 50 ml) (Note 1 and Note 2) and dilute to 50 ml mark. Add 1 ml of potassium sodium tartrate solution to each tube to prevent turbidity.

NOTE 1 — If the sample is turbid, add 1 ml of cupric sulphate solution for each 100 ml of the sample, mix thoroughly, add 1 ml of sodium hydroxide solution, again mix and allow to settle. If settling is not satisfactory, repeat, adding sodium hydroxide first, and then cupric sulphate.

NOTE 2 — If the sample contains hydrogen sulphide, use 1 ml of zinc sulphate solution instead of cupric sulphate in the procedure given in Note 1 above.

**23.3.2** Add to each Nessler tube 2 ml of Nessler reagent and mix. After allowing them to stand for 10 minutes for the colour to develop, compare the sample with the standards.

### 23.4 Calculation

$$\text{Ammoniacal nitrogen (as NH}_3\text{), mg/l} = \frac{10 V_1}{V_2}$$

where

$V_1$  = volume in ml of standard ammonium chloride solution required for matching the colour, and

$V_2$  = volume in ml of the sample taken for the test.

**23.5 Range of Accuracy** — The method is suitable in the range of 0 to 2 mg/l (as NH<sub>3</sub>).

## 24. NITRITE NITROGEN

**24.0 Outline of the Method** — The nitrite in the sample is treated with sulphanilic acid; the diazo salt formed couples with an amine to give a red colour which is matched against that produced with a series of standard nitrite solutions.

### 24.1 Apparatus

**24.1.1 Nessler Tubes** - 50 ml capacity.

### 24.2 Reagents

**24.2.1 Stock Nitrite Solution** — Dissolve 0.246 g of anhydrous sodium nitrite in nitrite-free distilled water and dilute to 1 litre. Preserve by adding 1 ml of chloroform. One millilitre of this solution contains 0.05 mg of nitrite nitrogen (as N).

**24.2.2 Standard Nitrite Solution** — Dilute 100 ml of stock nitrite solution to 1 litre with nitrite-free distilled water. Preserve by adding 1 ml of chloroform and store in a sterilized bottle. One millilitre of this solution contains 0.000 5 mg of nitrite nitrogen (as N).

**24.2.3 Sulphanilic Acid Reagent** — Dissolve 0.5 g of sulphanilic acid in a mixture of 30 ml of glacial acetic acid and 120 ml of distilled water. Filter if necessary.

**24.2.4 1-Naphthylamine Reagent** — Dissolve 0.1 g of 1-naphthylamine in 30 ml of glacial acetic acid, add 120 ml of distilled water, mix and filter.

### 24.3 Procedure

**24.3.1** Into nine Nessler tubes, pipette out 0, 0.1, 0.2, 0.4, 0.6, 0.8, 1.2, 1.6 and 2.0 ml of standard nitrite solution. In a tenth Nessler tube, pipette out a suitable volume of the sample (10, 25 or 50 ml). Make up the volume in each tube to 50 ml. Adjust the temperature of the solutions to about 20°C.

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**24.3.2** To each of the Nessler tubes, add 2 ml of sulphanilic acid reagent and 2 ml of 1-naphthylamine reagent. Mix the solutions by stirring with a glass rod and after allowing to stand for 15 minutes, match the colour obtained with the sample with those of the standards.

### 24.4 Calculation

$$\text{Nitrite nitrogen (as N), mg/l} = \frac{0.5 V_1}{V_2}$$

where

$V_1$  = volume in ml of standard nitrite solution matching with the sample, and

$V_2$  = volume in ml of the sample taken for the test.

**24.5 Range of Accuracy** — The method is suitable in the range of 0.001 to 1 mg/l (as N).

## 25. DISSOLVED OXYGEN

**25.0 General** — Two methods are prescribed. Method A is suitable when dissolved oxygen content is of the order of 0.05 mg/l. For low dissolved oxygen content (of the order of 0.007 mg/l), Method B shall be followed.

**25.0.1 Outline of the Methods** — The dissolved oxygen in the sample oxidizes manganous hydroxide to manganic hydroxide which, in turn, oxidizes iodide to free iodine in an acid medium. The iodine liberated is determined by titration.

### 25.1 Method A

#### 25.1.1 Reagents

**25.1.1.1 Manganous sulphate solution** — Dissolve 480 g of manganous sulphate ( $\text{MnSO}_4 \cdot 4\text{H}_2\text{O}$ ) in distilled water, filter, and dilute to 1 litre. The solution should liberate not more than a trace of iodine when added to an acidified solution of potassium iodide.

**25.1.1.2 Iodide-azide reagent** — Dissolve 500 g of sodium hydroxide (or 700 g of potassium hydroxide) and 135 g of sodium iodide (or 150 g of potassium iodide) in distilled water and dilute to 1 litre. The reagent should not give a colour with starch solution when diluted and acidified. Dissolve 10 g of sodium azide in 40 ml of distilled water and add to 950 ml of the first solution, with constant stirring.

**25.1.1.3 Concentrated sulphuric acid** - approximately 36 N. One millilitre of the acid is equivalent to about 3 ml of iodide-azide reagent.

**25.1.1.4 Standard sodium thiosulphate solution** — exactly 0.025 N, freshly standardized against potassium dichromate. One millilitre of this solution is equivalent to 0.2 mg of oxygen (as O).



**25.1.1.5 Starch indicator solution** — same as in 13.1.2.

**25.1.2 Procedure** — To the sample as collected in a 250- to 300-ml bottle, add 2 ml of manganous sulphate solution, followed by 2 ml of iodide-azide reagent well below the surface of the liquid. Stopper with care to exclude air bubbles completely, and mix by inverting the bottle several times. When the precipitate settles leaving a clear supernatant above the manganese hydroxide floc, repeat the shaking a second time. When settling has produced at least 100 ml of clear supernatant, carefully remove the stopper and immediately add 2.0 ml of concentrated sulphuric acid, allowing the acid to run down the neck of the bottle, re-stopper, and mix by gentle inversion until solution is complete. The iodine should be uniformly distributed throughout the bottle before decanting the amount needed for titration. This should correspond to 200 ml of original sample after correction has been made for the loss of sample by displacement with the reagents. Thus, when a total of 4 ml, 2 ml each of the manganous sulphate solution and the iodide-azide reagent, is added to a 300-ml bottle, the volume taken for titration should be:

$$200 \times \frac{300}{300-4} = 203 \text{ ml}$$

Titrate with standard sodium thiosulphate solution to a pale straw colour. Add 1 to 2 ml of starch solution and continue the titration to the first disappearance of the blue colour. Subsequent recolorations due to the catalytic effect of nitrites or to the presence of traces of ferric salts which have not formed fluoride complexes should be disregarded.

**25.1.3 Calculation****25.1.3.1 Dissolved oxygen, mg/l =  $V$** 

where

$V$  = volume in ml of standard sodium thiosulphate solution used in the titration.

**25.1.3.2** If the results are to be expressed in terms of millilitres of oxygen gas at 0°C and 760 mm pressure, the dissolved oxygen content in terms of mg/l should be multiplied by 0.698.

**25.1.3.3 Expression of results, in terms of percent saturation** — To express the results in terms of percent saturation at 760 mm pressure, the solubility value which is calculated by the method given below shall be used:

The solubility of dissolved oxygen in distilled water at an atmospheric pressure  $P$  mm, temperature  $t^\circ\text{C}$ , and saturated vapour pressure  $u$ , for the given temperature  $t^\circ\text{C}$ , shall be calculated between the temperatures

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0° and 30°C by equation (a) and between the temperatures 30° and 50°C by equation (b).

$$\text{a) Solubility (ml of dissolved oxygen per litre of water)} = \frac{0.678(P-u)}{(35 + t)}$$

$$\text{b) Solubility (ml of dissolved oxygen per litre of water)} = \frac{0.827(P-u)}{(49 + t)}$$

### 25.2 Method B

**25.2.0 Outline of the Method** — Dissolved oxygen is determined by comparing the colours produced with indigo-carmin which has been reduced with glucose, with a series of standard colours. These range through orange, pink and mauve to blue with increasing oxygen content.

**25.2.0.1** No interference has been found from nickel, copper or zinc in concentrations of 1 mg/l or from ferric iron at 3 mg/l. Ferrous iron interferes. Sulphite and hydrazine do not interfere at the low concentrations normally found in boiler feed water.

**25.2.1 Apparatus** — The sample is collected in a modified Nessler cylinder which is shown in Fig. 1. The cylinder fits into a Nesslerizer enabling the developed colour to be matched against the standard colours on a calibrated disc.

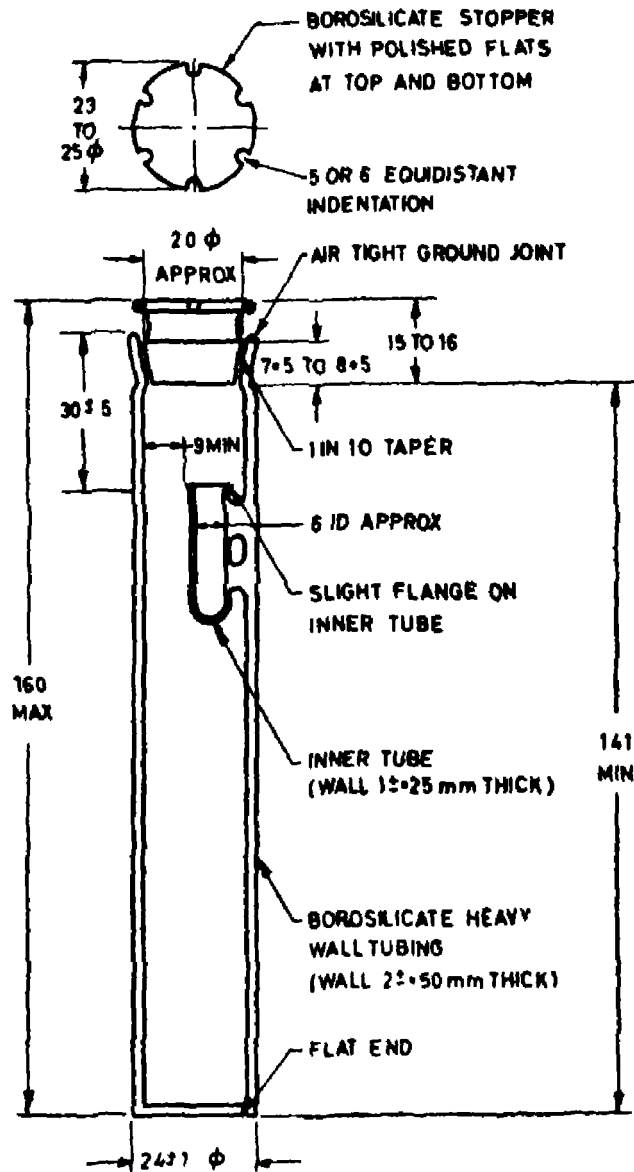
#### 25.2.2 Reagents

**25.2.2.1 Indigo carmine solution** — Dissolve 0.018 g of indigo carmine and 0.2 g of glucose in 5 ml of water and add 75 ml of glycerol. Mix well. This reagent is stable for 6 weeks if stored in the dark.

**25.2.2.2 Potassium hydroxide solution** — Dissolve 37.5 g of potassium hydroxide in 62.5 ml of water.

#### 25.2.3 Procedure

**25.2.3.1 Pipette** 8 ml of indigo carmine solution into a dry 25 g reagent bottle using a 10 ml graduated pipette. As this solution is very viscous, a pipette with an orifice of approximately 2 mm diameter should be used. The outside of the pipette shall be wiped to avoid more than 8 ml being added to the bottle. Next add 2 ml of the potassium hydroxide solution to the bottle by means of a safety pipette. Stopper the bottle and mix well. Keep undisturbed in the dark at a temperature of not less than 15°C until the initial dark red colour changes to lemon yellow. This will take approximately 30 minutes. This reduced indigo carmine, referred to as the 'leuco reagent', shall be prepared fresh each day.



All dimensions in millimetres.

FIG. 1 MODIFIED NESSLER CYLINDER

**25.2.3.2** Remove the glass stopper from the modified Nessler cylinder. Clean and dry the inner tube and glass ball and then fill the small inner tube with the leuco reagent. This is done by pipetting about 1 ml of the leuco reagent with a dropping pipette and then slowly forcing the reagent into the inner tube, avoiding as far as possible the introduction of any air bubbles. Wait until any minute air bubbles have risen to the surface. Then hold nearly vertically a length of clean, dry glass tubing over the

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top surface of the inner tube and roll the glass ball down inside the glass tubing so that it alights on the surface of the leuco reagent. Make certain that no air bubbles have been trapped.

**25.2.3.3** Take the modified Nessler cylinder to the cooling coil and with the water running at a rate of about 200 ml per minute raise the cylinder slowly until the bottom of the exit tube is close to the bottom of the vessel. Allow the water to flow until the sampling line and cylinder have been thoroughly flushed and a true sample is being obtained. In the meantime ensure that the glass stopper from the modified Nessler cylinder is clean and wet with the water being sampled. Slowly lower the modified Nessler cylinder, with the water still flowing until it is just free of the sampling line, and then immediately slide the clean, freshly wetted glass stopper across and into the top of the cylinder. Make certain that no air bubbles have been trapped.

**25.2.3.4** With the forefinger over the glass stopper, invert the Nessler cylinder so that the glass ball falls off the inner tube and the leuco reagent flows into the water. Invert the Nessler cylinder several times until it is clear from observation that there is no leuco reagent left in the inner tube. Five minutes after the first inversion of the cylinder, place it in the appropriate compartment of a Nessleriser and into the other compartment place a second modified Nessler cylinder filled with the sample. Rotate the disc until a colour match is obtained. The disc gives the oxygen directly in ml/l. The figure may be converted to mg/l by multiplying the result by 1.4.

## 26. HYDRAZINE

**26.0 Outline of the Method** — In acid medium, hydrazine reacts with *p*-aminobenzaldehyde to produce a yellow colour which is matched against that obtained with a series of standard hydrazine solutions.

### 26.1 Apparatus

**26.1.1** *Nessler tubes* — 50 ml capacity

### 26.2 Reagents

**26.2.1** *Dilute Hydrochloric Acid* — approximately 0.5 N.

**26.2.2** *Standard Hydrazine Solution* — Dissolve 0.328 g of hydrazine dihydrochloride in 50 ml of approximately 0.5 N hydrochloric acid and dilute with the same acid to 1 000 ml. Before use, dilute 10 ml of the solution to 1 000 ml with the same acid. One millilitre of the diluted solution contains 0.001 mg of hydrazine (N<sub>2</sub>H<sub>4</sub>).

**26.2.3** *p-Dimethylaminobenzaldehyde Solution* — Dissolve 4.0 g of *p*-dimethylaminobenzaldehyde (special grade for hydrazine determination) in a mixture of 200 ml of methanol and 20 ml of concentrated hydrochloric acid.

### 26.3 Procedure

**26.3.1** Hydrazine tends to be destroyed in neutral or alkaline solutions in contact with air. Hence it is essential that the sampling is done properly as given below and that the test is carried out immediately thereafter:

'Insert the outlet tube from an efficient stainless steel cooler to the bottom of a 250-ml bottle. Allow the water to flow until a representative sample is obtained, at a temperature ranging from 17 to 30°C. Withdraw the outlet tube slowly and immediately stopper the bottle, avoiding trapping any air-bubbles.'

**26.3.2** Into nine Nessler tubes, each containing 15 ml of dilute hydrochloric acid, add 0, 0.5, 1.0, 2.0, 3.0, 4.0, 6.0, 8.0 and 10.0 ml of standard hydrazine solution. Make the volumes to 40 ml by adding distilled water and mix. In the tenth Nessler tube containing 15 ml of dilute hydrochloric acid, pipette out a suitable volume of the sample (2, 10 or 25 ml) and dilute to 40 ml with distilled water and mix.

**26.3.3** Now add to each of the Nessler tubes 10 ml of  $\rho$ -dimethylamino-benzaldehyde solution. Mix and after 10 minutes compare the colour of the sample with those of the standards.

### 26.4 Calculation

$$\text{Hydrazine (as N}_2\text{H}_4\text{), mg/l} = \frac{V_1}{V_2}$$

where

$V_1$  = volume in ml of standard hydrazine solution required for matching the colour with the sample, and

$V_2$  = volume in ml of the sample taken for the test.

**26.5 Range of Accuracy** — The method is suitable in the range 0 to 5 mg/l of hydrazine.

## A P P E N D I X A

(Clause 6.2.2)

### PREPARATION OF INDICATORS AND BUFFER SOLUTIONS FOR DETERMINATION OF $\rho$ H

#### A-1. INDICATORS

**A-1.1** A list of suitable indicators is given in Table 3 together with their  $\rho$ H range, colour change and methods of preparation.

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**A-1.1.1 Universal Indicator** — Dissolve 0.05 g of methyl orange, 0.15 g of methyl red, 0.3 g of bromothymol blue and 0.35 g of phenolphthalein in one litre of ethyl alcohol (66 percent). The colour changes are:

pH	Colour
Up to 3	red
4	orange-red
5	orange
6	yellow
7	yellowish-green
8	greenish-blue
9	blue
10	violet
11	reddish-violet

**TABLE 3 INDICATORS**

(Clause A-1.1)

SL No.	NAME OF INDICATOR	pH RANGE	COLOUR CHANGE	METHOD OF PREPARATION
(1)	(2)	(3)	(4)	(5)
i)	Thymol blue (acid range)	1.2 to 2.8	Red to yellow	Triturate 0.10 g in 10.75 ml of N/50 sodium hydroxide solution and dilute with distilled water to 250 ml
ii)	Bromophenol blue	3.0 to 4.6	Yellow to blue violet	Triturate 0.10 g in 7.45 ml of N/50 sodium hydroxide solution and dilute with distilled water to 250 ml
iii)	Bromocresol green	3.8 to 5.4	Yellow to blue	Triturate 0.10 g in 7.15 ml of N/50 sodium hydroxide solution and dilute with distilled water to 250 ml
iv)	Methyl red	4.2 to 6.3	Red to yellow	Triturate 0.10 g in 18.60 ml of N/50 sodium hydroxide solution and dilute with distilled water to 250 ml
v)	Bromocresol purple	5.2 to 6.8	Yellow to blue violet	Triturate 0.10 g in 9.25 ml of N/50 sodium hydroxide solution and dilute with distilled water to 250 ml

(Continued)

TABLE 3 INDICATORS — *Contd*

SL No.	NAME OF INDICATOR	pH RANGE	COLOUR CHANGE	METHOD OF PREPARATION
(1)	(2)	(3)	(4)	(5)
vi)	Bromothymol blue	6.0 to 7.6	Yellow to blue	Triturate 0.10 g in 8.00 ml of N/50 sodium hydroxide solution and dilute with distilled water to 250 ml
vii)	Phenol red	6.8 to 8.4	Yellow to red	Triturate 0.10 g in 14.20 ml of N/50 sodium hydroxide solution and dilute with distilled water to 250 ml
viii)	Cresol red	7.2 to 8.8	Yellow to red	Triturate 0.10 g in 13.10 ml of N/50 sodium hydroxide solution and dilute with distilled water to 250 ml
ix)	Thymol blue (alkali range)	8.0 to 9.6	Yellow to blue	Triturate 0.10 g in 10.75 ml of N/50 sodium hydroxide solution and dilute with distilled water to 250 ml
x)	Thymolphthalein	9.3 to 10.5	Colourless to blue	Dissolve 0.10 g in 100 ml of rectified spirit (conforming to IS : 323-1959*)
xi)	Thymol violet	9.0 to 13.0	Yellow to green to violet	Dissolve 0.01 g of tropaeolin O in 100 ml of distilled water. Dissolve 0.04 g of thymolphthalein in a mixture of 50 ml of rectified spirit and 50 ml of water. Mix one part of tropaeolin O solution with 4 parts of thymolphthalein solution.

\*Specification for rectified spirit (*revised*).

## A-2. STANDARD BUFFER SOLUTIONS

**A-2.1** Standard buffer solutions prepared as given below shall be kept in bottles made of alkali-free glass (conforming to type 1 of IS : 2303-1963\*) or of polyethylene, and shall not be used later than three months after preparation:

- a) *Solutions from pH 1.2 to pH 2.2* shall be prepared by mixing 50 ml of M/5 potassium chloride solution with the specified volumes of

\*Method of grading glass for alkalinity.

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N/5 hydrochloric acid as given below and diluting with distilled water to 200 ml.

<i>pH</i>	<i>Volume (in ml) of N/5 hydrochloric acid</i>
1.2	64.5
1.4	41.5
1.6	26.3
1.8	16.6
2.0	10.6
2.2	6.7

- b) *Solutions from pH 2.2 to pH 3.8 shall be prepared by mixing 50 ml of M/5 potassium hydrogen phthalate solution with the specified volumes of N/5 hydrochloric acid as given below and diluting with distilled water to 200 ml.*

<i>pH</i>	<i>Volume (in ml) of N/5 hydrochloric acid</i>
2.2	46.7
2.4	39.6
2.6	33.0
2.8	26.4
3.0	20.3
3.2	14.7
3.4	9.9
3.6	6.0
3.8	2.6

- c) *Solutions from pH 4.0 to pH 6.2 shall be prepared by mixing 50 ml of M/5 potassium hydrogen phthalate solution with specified volumes of N/5 sodium hydroxide solution as given below and diluting with water to 200 ml.*

<i>pH</i>	<i>Volume (in ml) of N/5 sodium hydroxide solution</i>
4.0	0.4
4.2	3.7
4.4	7.5
4.6	12.2
4.8	17.7
5.0	23.8
5.2	30.0
5.4	35.4
5.6	39.8
5.8	43.0
6.0	45.4
6.2	47.0



- d) *Solutions from pH 5.8 to pH 8.0 shall be prepared by mixing 50 ml of M/5 potassium dihydrogen phosphate solution with the specified volumes of N/5 sodium hydroxide solution as given below and diluting with distilled water to 200 ml.*

<i>pH</i>	<i>Volume (in ml) of N/5 sodium hydroxide solution</i>
5.8	3.7
6.0	5.7
6.2	8.6
6.4	12.6
6.6	17.8
6.8	23.5
7.0	29.6
7.2	35.0
7.4	39.5
7.6	42.8
7.8	45.2
8.0	46.8

- e) *Solutions from pH 7.8 to pH 10.0 shall be prepared by mixing 50 ml of M/5 boric acid-potassium chloride solution (prepared by dissolving 12.3 g of boric acid and 14.9 g of potassium chloride in distilled water and making the final volume to 1 000 ml) with the specified volumes of N/5 sodium hydroxide solution as given below and diluting with distilled water to 200 ml.*

<i>pH</i>	<i>Volume (in ml) of N/5 sodium hydroxide solution</i>
7.8	2.6
8.0	4.0
8.2	5.9
8.4	8.5
8.6	12.0
8.8	16.3
9.0	21.3
9.2	26.7
9.4	32.0
9.6	36.8
9.8	40.8
10.0	43.9

- f) *Solutions from pH 8.4 to pH 12.8 shall be prepared by mixing X ml of M/10 glycine-sodium chloride solution (containing 7.505 g*

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of glycine and 5.850 g of sodium chloride per litre of the solution)  
and *Y* ml of N/10 sodium hydroxide solution as specified below:

<i>pH</i>	<i>X ml glycine-sodium chloride solution</i>	<i>Y ml sodium hydroxide solution</i>
8.4	9.5	0.5
8.8	9.0	1.0
9.2	8.0	2.0
9.6	7.0	3.0
10.0	6.0	4.0
10.3	5.5	4.5
10.9	5.1	4.9
11.1	5.0	5.0
11.4	4.9	5.1
11.9	4.5	5.5
12.2	4.0	6.0
12.5	3.0	7.0
12.7	2.0	8.0
12.8	1.0	9.0

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