IS 3025 (Part 60) : 2008

(Reaffirmed 2013)

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भारतीय मानक

जल एंव अपशिष्ट जल के नमूने लेना और परीक्षण (भौतिक एंव रासायनिक) की विधियाँ

भाग 60 फ्लोराइड

(पहला पुनरीक्षण)

Indian Standard

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

PART 60 FLUORIDE

(First Revision)

ICS 13.060.50

BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

April 2008 **Price Group 4**

Environment Protection and Waste Management Sectional Committee, CHD 32

FOREWORD

This Indian Standard (Part 60) (First 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.

As per IS 10500 : 1991 'Drinking water — Specification (*first revision*)', the desirable limit and permissible limit (in the absence of alternate source) for fluoride in the drinking water is 1.0 mg/l, *Max* and 1.5 mg/l, *Max* respectively. Beyond this level it causes fluorosis.

The Committee responsible for the formulation of IS 3025 : 1964 'Methods of sampling and test (physical and chemical) for water used in industry' had decided to revise the standard and publish it as separate parts. This standard is one of the different parts under the IS 3025 series of standards and supersede **23** of IS 3025.

Electrochemical probe method given in this standard is prepared based on the method specified in ISO 10359-1 'Water quality — Determination of fluoride — Part 1: Electrochemical probe method for potable and lightly pollute water'. It is technically equivalent except that measurement is made without filtration of the solution through membrane filter.

The composition of the Committee responsible for the formulation of this standard is given at Annex A.

In reporting the results 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 60 FLUORIDE

(First Revision)

1 SCOPE

1.1 This standard (Part 60) prescribes three methods of test for determination of fluoride content in water and wastewater.

a) Zirconium alizarin method without distillation.

b) Zirconium alizarin method with distillation, and

c) Electrochemical probe method.

1.2 The method without distillation (described under **5.6.1**) and electrochemical probe method is reliable for samples of potable and lightly polluted water in which the interfering substances are not in excess of the limits given below:

Chlorides (as Cl)	2 000 mg/l
Sulphates (as SO_4)	300 mg/l
Alkalinity (as $CaCO_3$)	400 mg/l
Iron (as Fe)	2 mg/l
Aluminium (as Al)	0.5 mg/l
Phosphates (as PO_4)	5 mg/l

Where the sample is highly coloured or turbid or has interfering substances in excess of the limits given above, the method with distillation (described under **5.6.2**) shall be used or the sample shall be appropriately diluted before this test. With samples of unknown composition or where greater accuracy is needed, the method with distillation shall be employed.

2 REFERENCES

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

IS No.	*Title*
265 : 1993	Hydrochloric acid (*fourth revision*)
266 : 1993	Sulphuric acid (*third 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) 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 ZIRCONIUM ALIZARIN METHODS

5.1 Principle

The colour (red to yellow with increasing concentration of fluoride) obtained with zirconium alizarin reagent is matched against that produced with a series of standard fluoride solutions.

5.2 Range and Applicability

This method is suitable for estimation of fluoride content up to 1.0 mg/l of fluoride as F. The minimum detection limit of this method is 0.05 mg/l fluoride as F.

5.3 Interference

Iron, alkalinity, phosphates interfere, if present above the values given in **1.2**. Interference of free residual chlorine can be removed by adding sodium arsenite. Aluminium gives negative error because of formation of Al-F complex which withdraws fluoride from the reaction of zirconium.

5.4 Apparatus

5.4.1 *Nessler Tubes*, 100 ml capacity.

5.4.2 *Distillation Apparatus* — The distillation apparatus shall consist of a Claisen flask of 100 ml capacity, a large flask for generating steam and an efficient condenser. The main neck of the Claisen flask shall be fitted with a two-holed rubber stopper through which shall pass a thermometer and a glass tube (for connecting with the steam supply), both the thermometer and the tube extending almost to the bottom of the flask. The side neck of the flask shall be closed with a rubber stopper and the side arm connected with the condenser. Steam shall be generated from water made alkaline with sodium hydroxide. Local overheating of the Claisen flask shall be avoided by use of an asbestos board with a hold which shall fit closely to the lower surface of the flask.

5.5 Reagents

5.5.1 *Sodium Thiosulphate Solution* — approximately 0.1 N.

5.5.2 *Standard Sodium Fluoride Solution* — Dissolve 0.221 g of dry sodium fluoride in distilled water and make up to 1 000 ml. Dilute 1 000 ml of the solution to 1 000 ml. One millilitre of this diluted solution contains 0.01 mg of fluoride (as F). The solution shall be kept in polyethylene or wax-lined glass bottles.

5.5.3 *Zirconium Alizarin Reagent*

a) Dissolve 0.3 g of zirconium oxychloride ($ZrOCl_2.8H_2O$), or 0.25 g of zirconium oxynitrate [$ZrO\ (NO_3)_2.2H_2O$] in 50 ml of distilled water. Dissolve 0.07 g of alizarin sodium monosulphonate (alizarin S) in another 50 ml quantity of distilled water and add the latter solution slowly to the zirconium solution with continuous stirring. The resulting solution clears on standing for a few minutes.

b) Dilute 112 ml of concentrated hydrochloric acid (*see* IS 265) to 500 ml with distilled water. Also add 37 ml of concentrated sulphuric acid (*see* IS 266) to 400 ml of distilled water and then dilute to 500 ml. Mix the two diluted acids when cool.

c) Dilute the clear zirconium solution prepared in (a) to 1 000 ml with the mixed acid solution prepared in (b). The reagent is at first red, but within an hour it changes to orange-yellow and is red for use. The solution shall be stored in the dark if kept in a refrigerator it is stable for 2 to 3 months. When 5 ml of this reagent are added to 100 ml of distilled water containing no fluorides, it soon turns pink. Fluorides discharge the pink colour of the lake so that the solution acquires a more yellow tint.

5.5.4 *Silver Sulphate*

5.5.5 *Perchloric Acid* — 60 percent.

5.5.6 *Phenolphthalein Indicator*

5.5.7 *Sodium Hydroxide Solution* — 10 percent *w/v*.

5.5.8 *Concentrated Sulphuric Acid* — *See* IS 266.

5.6 Procedure

5.6.1 *Method Without Distillation*

5.6.1.1 The sample shall not contain free chlorine; if necessary, it shall be dechlorinated with a slight excess of sodium thiosulphate solution before use.

5.6.1.2 Take 100 ml of the clear sample and a series of dilutions of standard sodium fluoride solution in 100 ml of distilled water in Nessler tubes and add 5.0 ml of the zirconium alizarin reagent to each. The sample and standards shall be at the same temperature to within 1 °C to 2°C. Mix and compare the colours after standing for 1h exactly. Note the volume of standard sodium fluoride solution contained in the tube with which a match with the sample under test is obtained.

5.6.2 *Method with Distillation*

5.6.2.1 Introduce into the Claisen flask a number of fragments of Pyrex glass or glass beads, 0.2 g of silver sulphate, 7 ml of distilled water and 15 ml of perchloric acid. Heat the flask until the temperature reaches 120°C to 125°C, connect to the steam supply and regulate the gas and steam so that the distillation proceeds at a temperature of 137°C to 140°C. Distil 150 ml in 25 to 35 min and steam out the condenser towards the end of the distillation. Discard the first distillate. Distil a further 150 ml and determine the fluorides in it by the method given in **5.6.1.2**. The figure for this blank shall not exceed 0.001 5 mg and shall be approximately constant for any further 150-ml fraction.

5.6.2.2 Make 150 ml of the sample alkaline to phenolphthalein indicator with sodium hydroxide solution, add a few drop in excess and concentrate to 20 ml. When cool, transfer quantitatively to the distillation flask and carefully add 15 ml of concentrated sulphuric acid. If the amount of chloride in the aliquot exceeds 5 mg, add about 5 mg of silver sulphate for each milligram of chlorine. Connect up the apparatus and distil 150 ml as in **5.6.2.1**. Determine the fluoride content of the total 150 ml of distillate as in **5.6.1.2**.

5.7 Calculation

5.7.1 *Method without Distillation*

$$\text{Fluoride (as F), mg/l} = \frac{1\,000\,W}{V}$$

where

W = weight of fluorides (as F) in the standard solution matched by the sample, in mg; and

V = volume of the sample taken for the test in **5.6.1.2**; in ml.

5.7.2 *Method with Distillation*

$$\text{Fluoride (as F), mg/l} = \frac{1\,000\,W}{V}$$

where

W = weight of fluorides (as F) in the standard solution matched by 150 ml of the distillate, in mg; and
V = volume of the sample taken for the test in **5.6.1.2**, in ml.

6 ELECTROCHEMICAL PROBE METHOD

6.1 Range and Applicability

The electrochemical technique method is directly suitable for measuring fluoride concentrations from 0.2 mg/l to 2.0 g/l. After the addition of a known amount of fluoride, concentrations as low as 0.02 mg/l can be detected (*see* **7.3**). The method is not suitable for waste waters and industrial effluents.

6.2 Interferences

The electrode will respond directly to hydroxide ions. The formation of HF under acidic conditions will reduce the measured fluoride concentration. Therefore, buffer all test aliquots to a *p*H between 5 and 7 to prevent such interference. Cations such as calcium, magnesium, iron and aluminium form complexes with fluoride or precipitates to which the electrode does not respond. Therefore the buffer solution also contains trans-1,2-diaminocyclohexane-N,N,N',N'-tetraacetic acid (CDTA) as a decomplexing agent to free bound fluoride. The boron tetrafluoride anion, is not decomplexed by the addition of buffer.

6.3 Principle

When a fluoride ion-selective electrode comes into contact with an aqueous solution containing fluoride ions, a potential difference develops between the measuring electrode and the reference electrode. The value of this potential difference is proportional to the logarithm of the value of the fluoride ion activity in accordance with the Nernst equation.

NOTES

1 Temperature and ionic strength may influence the potential difference. Accordingly, these parameters shall be the same during calibration and measurement and shall be kept constant throughout the procedure.

2 The activity of the fluoride ions is also *p*H-dependant. Values of *p*H between 5 and 7 have proved favourable for measurement. Special buffer solutions are used to fix the *p*H and the activity coefficient. On these assumptions, this method will no longer refer to activities, but to fluoride ion concentrations. Fluoride ion-selective electrodes operate between 0.2 mg/l and 2 000 mg/l, and show a linear relationship between the potential and the logarithm of the numerical value of the fluoride activity.

6.4 Apparatus

6.4.1 *Millivolt Meter* — A millivolt meter with an impedance of not less than $10^{12}\Omega$, capable of resolving potential differences of 0.1 mV or better.

6.4.2 *Fluoride Ion-selective Electrode* — The e.m.f. response, using standard solutions, shall not be less than 55 mV per decade change in fluoride concentration at 25°C.

6.4.3 *Reference Electrode*

Either a calomel electrode, filled with saturated potassium chloride (KCl) solution, or a silver/silver chloride electrode shall be used.

NOTE — Single junction, sleeve-type electrodes which reduce the liquid-liquid junction potential are preferable.

6.4.4 *Measuring Cells* — capacity 100 ml, made of polypropylene and fitted with a thermostated jacket.

6.4.5 *Water Bath* — capable of supplying water to the jacket of the measuring cell (*see* **6.4.4**) at a temperature of 25°C ± 0.2°C.

6.4.6 *Magnetic Stirrer*, with a polytetrafluoroethylene (PTFE)-coated stirring bar.

6.4.7 *Polyethylene Beaker*, of capacity 100 ml.

6.5 Reagents

6.5.0 *Purity of the Reagents* — Unless specified otherwise, only pure chemicals and fluoride free distilled water shall be used in tests.

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

6.5.1 *Sodium Hydroxide* 5 (M) — Dissolve cautiously 100 ± 0.5 g of sodium hydroxide in water, cool and dilute to 500 ml.

6.5.2 *Total Ionic Strength Adjustment Buffer (TISAB)* — Add 58 g of sodium chloride (NaCl) and 57 ml of glacial acetic acid [$\rho(CH_3COOH) = 1.05$ g/ml] to 500 ml of water in a 1 litre beaker. Stir until dissolved. Add 150 ml of the sodium hydroxide solution (**6.5.1**) and 4 g of CDTA (trans-1,2-diaminocyclohexane-N,N,N',N'tetraacetic acid). Continue stirring until all the solids have dissolved and adjust the solution to *p*H 5.2 with sodium hydroxide solution using a *p*H meter. Transfer to a 1 000 ml one-mark volumetric flask, make up to the mark with water and mix.

NOTES

1 The solution is stable for about 6 months, but do not use it, if a precipitate forms.

2 This solution is commercially available.

6.5.3 *Fluoride, Stock Solution*, 1 000 mg/l

Dry a portion of sodium fluoride (NaF) at 150°C for

4 h and cool in a desiccator. Dissolve 2.210 ± 0.001 g of the dried material in water contained in a 1 000 ml one-mark volumetric flask. Make up to the mark with water and mix. Store the solution in a screw-capped polyethylene container.

6.5.3.1 *Fluoride, working standard solution I,* 10 mg/l

Pipette 10 ml of the fluoride stock solution (*see* **6.5.3**) into a 1 000 ml one-mark volumetric flask. Make up to the mark with water and mix. All standard solutions should be stored in plastic bottles and are usable for one month.

6.5.3.2 *Fluoride, working standard solution II,* 5 mg/l

Pipette 5 ml of the fluoride stock solution (*see* **6.5.3**) into a 1 000 ml one-mark volumetric flask and make up to the mark with water.

6.5.3.3 *Fluoride, working standard solution III,* 1 mg/1

Pipette 100 ml of the working standard solution I (*see* **6.5.3.1**) into a 1 000 ml one-mark volumetric flask and make up to the mark with water.

6.5.3.4 *Fluoride, working standard solution IV,* 0.5 mg/l

Pipette 100 ml of the working standard solution II (*see* **6.5.3.2**) into a 1 000 ml one-mark volumetric flask and make up to the mark with water.

6.5.3.5 *Fluoride, working standard solution V,* 0.2 mg/1

Pipette 20 ml of the working standard solution I (*see* **6.5.3.1**) into a 1 000 ml one-mark volumetric flask and make up to the mark with water.

7 PROCEDURE

7.1 Preparation for Measurement

Since the electrode characteristics of a fluoride ion selective electrode generally vary with time, check the calibration curve on the day of use. To accelerate the establishment of the equilibrium potential, condition the electrode prior to measurement in the following way. Prior to measurement, immerse the electrode for 1 h in the cell (*see* **6.4.4**) which contains the reference solution 5 (*see* Table 1). After rinsing with the first solution to be measured, the electrode is ready for use.

7.2 Measurement

Pipette 25 ml of the buffer solution (*see* **6.5.2**), followed by 25 ml of the water sample, into a measuring cell (*see* **6.4.4**). Ensure that the *p*H is 5.2 ± 0.2; if necessary, adjust the *p*H with hydrochloric acid or sodium hydroxide solution, using as little as possible.

NOTE — If a precipitate is formed, perform the analysis with a diluted sample. Any dilution of the sample should be taken into account during the calculation of the results.

For a series of determinations, start the measurement with the lowest concentration and finish with the highest following the anticipated concentration of the samples. After measuring the high concentrations, recondition the electrode before measuring the low concentrations (*see* **7.1**). Measure all the solutions according to the following procedure. Wait until constant temperature (for example 25 ± 0.5°C) is reached and carry out all the measurements at this temperature. Put a stirring bar into the measuring cell (*see* **6.4.4**) and place it on the magnetic stirrer ((*see* **6.4.6**). Insert the electrodes (*see* **6.4.2**) into the solution and fix them in place. Adjust the stirring rate to about 180 min/1 to 200 min/1. When the potential does not change by more than 0.5 mV in 5 min, switch off the stirrer. After at least 15 s, record the value obtained. Rinse the stirring bar and the electrodes with the next solution to be measured, before starting the next measurement.

7.3 Measurement After Concentration Enhancement

If a water sample contains less than 0.2 mg/l F, proceed as follows:

Add 500 ml of the fluoride standard solution I (*see* **6.5.3.1**) to 25 ml of the sample using a piston pipette, and 25 ml of the buffer solution (*see* **5.4.2**) with a volumetric pipette; continue as described in **7.2**. When calculating the result, subtract the amount of fluoride ions added from the total result.

7.4 Calibration

Establish a calibration function using the five reference solutions in the corresponding concentration range. For the range 0.2 mg/l to 10 mg/l, proceed as follows:

a) Pipette 25.0 ml of the buffer solution (*see* **5.4.2**) into each of five measuring cells (*see* **6.4.4**), and
b) Pipette the respective volumes of the working standard fluoride solutions specified in Table 1 into the measuring flasks.

For the establishment of the calibration function proceed step by step from the most dilute solution to the most concentrated solution, rinsing after each measurement with the solution of the next highest concentration. After the above measurements have been completed, recondition the electrode for 5 min to 10 min, using the reference solution 5 (*see* Table 1) in order to eliminate memory effects.

8 CALCULATION AND EXPRESSION OF RESULTS

Plot the calibration values on semi-logarithmic paper,

Table 1 Preparation of Reference Solutions

(*Clauses* 7.1 *and* 7.4)

Sl No.	Reference Solution No. ml	Buffer Solution	Working Standard Solution		Fluoride Concentration mg/l
			No.	ml	
(1)	(2)	(3)	(4)	(5)	(6)
i)	1	25	I	25	10
ii)	2	25	II	25	5
iii)	3	25	III	25	1
iv)	4	25	IV	25	0.5
v)	5	25	V	25	0.2

Use the following order of measurement (the numbers refer to the reference solutions in this Table):

5 – rinse – 4 – rinse – 3 – rinse – 2 – rinse – 1 – rinse with 5 – recondition – repeat measuring run.

If the individual values of the parallel series vary from the first series by more than 1 ± 0.5 mV, repeat the measuring run. Regular checking of the calibration graph is essential. Ensure that the slope is not less than 55 mV, otherwise check the equipment and establish a new calibration graph.

NOTES

1 The term 'Concentration' refers to the concentration of the working standard solutions and the sample solution but not to the concentration of the measuring solutions after addition of the buffer.

2 *See* **6.5.3.1** to **6.5.3.5**.

with the fluoride concentrations, in milligrams per litre, on the abscissa and the cell potential, in millivolts, on the ordinate and establish the regression line. Read the value for the samples by using the regression line and express the mass concentration of fluoride in milligrams per litre.

NOTE — The evaluation may also be calculated using the Nernst equation.

9 PRECISION

An inter-laboratory trial, carried out in Germany in 1982, gave the results shown in Table 2.

Table 2 Precision Data

Sl No.	Sample	*l*	*n*	ρ mg/l	x mg/l	WFR %	σ_r mg/l	VC$_r$ %	σ_R mg/l	VC$_R$ %
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)	(10)	(11)
1	Drinking water	12	48	0.275	0.283	–	0.021	7.4	0,0068	2.4
2	Drinking water + F$^-$	13	52	2.4 + 0.275	2.596	–	0.103	4.0	0.0395	1.5
3	Drinking water + F$^-$									
	+ 15 mg Al	13	52	5.0 + 0.275	4.340		0.110	2.5	0.055 9	1.3
						–				
	100 mg Mg									
	500 mg Ca									
4	Distilled water + F$^-$									
	+ 100 mg Fe									
	15 mg Al	11	43	0.6	0.531	88.5	0.024	4.5	0.011 6	2.2
	100 mg Mg									
	500 mg Ca									
5	Distilled water + F$^-$									
	+ 100 mg Fe									
	15 mg Al	12	48	8.6	6.864	79.8	0.195	2.8	0.070 1	1.0
	100 mg Mg									
	500 mg Ca									
6	Same as 5 + F$^-$	12	48	60.6	46.351	76.5	1.560	3.4	0.643 3	1.4

l = number of laboratories

n = number of values

p = mass concentration

$\bar{x}$ = mean value

WFR = recovery rate

σ_r = repeatability standard deviation

VC$_r$ = repeatability variation coefficient

σ_R = reproducibility standard deviation

VC$_R$ = reproducibility variation coefficient

ANNEX A

(*Foreword*)

COMMITTEE COMPOSITION

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Review of Indian Standards

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Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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