भारतीय मानक **IS 3025 (Part 60/Sec 1) : 2023**

Indian Standard

जल और अपशिष्ट जल के नमूने लेने और परीक्षण (भौतिक और रासायनिक की पद्धतियाँ)

भाग 60 फ्लोराइड

अनुभाग 1 आयन - सेलेकटिव इलेक्ट्रोड , सपअनडस और तरल क्रोमैटोग्राफी विधियाँ

(दूसरा पुनरीक्षण)

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

Part 60 Fluoride

Section 1 Ion-Selective Electrode, SPANDS and Liquid Chromatography Methods

(Second Revision)

ICS 13.060.50

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January 2023 **Price Group 6**

Water Quality Sectional Committee, CHD 36

FOREWORD

‘This Indian Standard (Part 60/Sec 1) (Second Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Water Quality Sectional Committee had been approved by the Chemical Division Council’.

Fluoride may be present in water or it may be added in limited amount. Some fluorosis can possibly occur if the concentration of fluoride exceeds the recommended limits. In rare circumstances, the fluoride concentration that occurs naturally can approach 10 mg/l.

The determination of fluoride has increased its importance with rise in the practice of fluoridation of water supplies as a measure of public health. Maintenance of concentration of fluoride at an optimal level is quite essential for maintaining the safety and effectiveness of the fluoridation procedure.

As per IS 10500 : Drinking water-Specification, the desirable limits 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 Technical Committee responsible for formulation of IS 3025 : 1964 ‘Methods of sampling and test (physical and chemical) for water used in industry’ decided to revise the standard and publish it in separate parts. This standard superseded **23** of IS 3025 : 1964 'Methods of sampling and test (physical and chemical) for water used in industry' and was one among the different parts published under IS 3025 series of standards.

The first revision of this standard was published in 2008 which prescribed following three methods for determination of fluoride content in water and wastewater:

a) Zirconium alizarin method with distillation;
b) Zirconium alizarin method without distillation; and
c) Electrochemical probe method.

In this second revision the following changes have been incorporated:

a) This standard has been renumbered as Part 60/Sec 1 as other sections will be developed for instrument based methods;
b) The zirconium alizarin method with and without distillation have been deleted;
c) Electrochemical probe method has been deleted;
d) SPANDS method has been added; and
e) Liquid Chromatography method has been added.

In the preparation of this standard, considerable assistance has been derived from the method No. 4500 - F^- A, B, C & D of Standard Methods for the Examination of Water and Wastewater, published by the American Public Health Association, Washington, USA, 23rd Edition, 2017.

The composition of the committee responsible for the formulation of this standard is listed in Annex A.

In reporting the results of a test or analysis in accordance with this standard, if the final value observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 2022 ‘Rules for rounding off numerical values (*second revision*)’.

Indian Standard

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

PART 60 FLUORIDE

Section 1 Ion-selective electrode, SPANDS and Liquid Chromatography methods

(Second Revision)

1 SCOPE

This standard (Part 60/Sec 1) prescribes three methods of test for determination of fluoride content in water and wastewater:

a) Ion-selective electrode method;
b) SPADNS Method; and
c) Liquid Chromatography method.

2 REFERENCES

The standards listed in Annex A, contain provisions which, through reference in this text, constitute provisions of this standard. At the tie 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 these standards.

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

Make use polyethylene bottles for the collection and storing the samples for the determination of fluoride. Glass bottles can also be used if it did not contained high fluoride solutions initially. The bottle should always be rinsed with a portion of a sample. For the SPADN method, do not use excess of de-chlorinating agent. To carry out de-chlorination, make use of sodium arsenate instead of sodium thiosulphate when using the SPADNS method as the latter may produce turbidity that can lead to errors in the readings.

5 PRELIMINARY DISTILLATION STEP

5.1 Discussion

5.1.1 The most satisfactory methods for determining the fluoride ion in water are the calorimetric methods and electrode method. In order to separate the fluoride from other nonvolatile constituents in water, carry out the conversion to hydrofluoric or fluosilicic acid followed by its distillation. The conversion can be completed by using a strong, high boiling acid. In order to protect against glassware etching, hydrofluoric acid is converted to fluosilicic acid by use of soft glass beads. Quantitative recovery of fluoride is done by using a large sample. In order to minimize the acid and sulphate carryover, the distillation is carried out in controlled range of temperature.

5.1.2 By using distillation, fluoride can be separated from most of the water samples. Some of the tightly bound fluoride, like in case of biological materials, may require digestion before the process of distillation but water samples not often require such kind of treatment. Distillation process further produces a distillate volume which is equal to that of the original water sample, so usually it is not required to incorporate any kind of dilution factor when the analytical results are expressed. The distillate will be free of substances that can possibly interfere with the determination of fluoride if the type of apparatus used is appropriate and the process of distillation is carried out properly. One of the most common volatile constituent that can interfere with colometric analysis of distillate is chloride. If the concentration of chloride is quite high to cause interference, silver sulphate is added to sulphuric acid distilling mixture in order to minimize the volatization of hydrogen chloride. Use of a magnetic stirrer and a quartz heating mantle in the distillation apparatus can simplify the mixing step.

CAUTION — Heating of an acid-water mixture can be hazardous in nature if no precautions are taken. Mix the acid and water properly before the heating step

5.2 Apparatus

5.2.1 *Distillation Apparatus*

It consists of 1 000 ml round bottom long neck flask that is made from borosilicate glass, with a connecting tube, condenser and a thermometer adapter which is readable upto 200 °C. For all the connections, standard taper joints should be used in the direct vapour bath. Adjust the thermometer in a position so that it is immersed in a boiling mixture.

The apparatus should be able to disassemble easily to permit the addition of sample.

The alternative types of distillation apparatus may be used. Evaluate the apparatus for the recovery of fluoride and sulphate carryover carefully. Obstruction in the vapor path and trapping of liquid in the condenser and adapter are the critical points. (Minimum obstruction should be provided to the vapour path in the condenser. Condenser should be double jacketed with inner spiral tube and cooling water in the outer jacket. Other condensers are also acceptable if there are minimum obstructions. Use of Graham type condensers should be avoided). Avoid the use of open flame as a source of heat as the heat that is applied to the boiling flask above the level of liquid causes superheating of vapor and sulphate carryover.

CAUTION — With whatever apparatus used, confirm the thorough mixing of sample and acid, heating of a non-homogeneous acid water mixture may cause the bumping or a violent explosion.

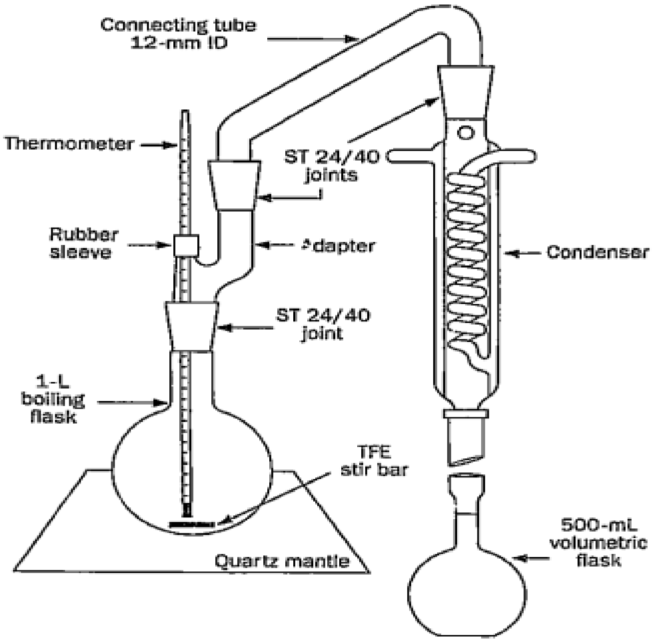

FIG 1. DIRECT DISTILLATION FOR FLUORIDE

5.2.2 *Quartz Hemispherical Heating Mantle* for full voltage operation

5.2.3 *Soft Glass Beads*

5.2.4 *Magnetic Stirrer*, with stirring bar coated with Tetrafluoroethylene (TFE)

5.3 Reagents

5.3.1 *Sulphuric Acid, Concentrated, Reagent Grade*

5.3.2 *Silver Sulphate, Crystals, Reagent Grade.*

5.4 Procedure

5.4.1 Take 400 ml of distilled water in a distillation flask and keep it on a magnetic stirrer. Then carefully add to it, 200 ml of sulphuric acid and, keep stirrer in operation throughout the process of distillation. Add some glass beads and then connect the apparatus as shown in Fig 1. Make sure that all the joints are tight. Start the process of heating and continue till the flask contents reach 180 °C (because of the retention of heat by the heating mantle, required to discontinue the heating if the temperature reached 178 °C so as to prevent overheating). Discharge the distillate and this process remove the contamination of fluoride and adjusts the acid water ratio for subsequent distillations.

5.4.2 After the mixture of acids has remained in the previous steps or when previous distillations has cooled to 80 °C or below, a 300 ml sample, continue stirring, distill it till the temperature reaches 180 °C. In order to prevent the carryover of sulphates, heat is turned off before the temperature reaches 178 °C. Retain the distillate for analysis.

5.4.3 Add silver sulphate to the distilling flask at the rate of 5 mg / mg Cl^- when the concentration of chloride is high enough to interfere.

5.4.4 Repeatedly use sulphuric acid solution in the flask till the contaminants from the samples accumulate to such an extent that its recovery is affected or the interferences are appeared in the distillate. Check the suitability of the acids after regular intervals of time by distilling the standard samples of fluoride and analyzing it for fluoride and sulfate. After the distillation of the samples that contain more than 3 mg F^-/l, flush by adding 300 ml of distilled water. Re-distill and combine the two fluoride distillates. If required, repeat flushing till the fluoride content of the last distillate is minimum. Mix the additional amount of fluoride that is recovered with that of the first distillation. After the periods of inactivity, likewise flush the still and discard the distillate.

5.5 Results

Fluoride recovery is quantitative within the accuracy of the methods used for the measurement.

6 ION-SELECTIVE ELECTRODE METHOD

6.1 General Discussion

6.1.1 *Principle*

6.1.1.1 The fluoride electrode acts like an ion selective sensor. The main element in the fluoride electrode is laser type doped lanthanum fluoride crystal, across which potential is established by fluoride solutions of different concentrations. The crystal is placed so that sample solution is on one face of crystal and reference solution on the other side.

$$Ag/AgCl,\ Cl^-\ (0.3\ M),\ F^-\ (0.001M)\ /LaF_3/\text{test Solution/reference electrode}$$

6.1.1.2 Along with the standard calomel electrode and with any pH meter with an expanded millivolt scale, the fluoride electrode can be used. Since the calomel electrodes contains both metallic and dissolved mercury, it is recommended to dispose them off only at approved sites or recycle them. Due to this reason, use of silver/silver chloride reference electrode is preferred.

6.1.1.3 Rather than the concentration, fluoride electrode measures the ion activity of fluoride. The ion activity of fluoride depends on the pH, fluoride complexing species and total ionic strength of the solution. The addition of an appropriate buffer can adjust the pH, breaks up complexes and provides uniform ionic strength background, so the electrode measures the concentration. The electrode based methods are suitable for the concentrations of fluoride from 0.1 to more than 10 mg/l.

6.1.2 *Interference*

Fluoride can form complexes with various polyvalent cations like aluminium and iron. The extent to which the complexation of ions can occur depends on the relative levels of fluoride, pH of the solution and the complexing species. However, cyclohexylene diamine tetraacetic acid (CDTA), part of the buffer can complex the interfering cations and can release the free fluoride ions. Most common interference, aluminium, present in a concentration of 3.0 mg/l can complex preferentially. If the solution is acidic, fluoride ion can form a poorly ionized HF. HF complex formation can be minimized if pH can be maintained above 5 with the help of buffer. Whenever the concentration of hydroxide ion is greater than one-tenth of the concentration of the fluoride ion hydroxide ion might interfere with the electrode response to fluoride ion in the alkaline solution. If the pH is maintained by the buffer, no interference by hydroxide occurs. Sample should also be distilled in case the concentration of dissolved solid exceeds 10 000 mg/l.

6.2 Apparatus

6.2.1 *Expanded Scale or Digital pH Meter or Ion Selective Meter*

6.2.2 *Sleeve Type Reference Electrode,* fibre tip reference electrode should not be used as they show erratic behavior in extremely dilute solutions.

6.2.3 *Timer*

6.2.4 *Magnetic Stirrer,* stirring bar coated with TFE

6.2.5 *Fluoride electrode*

6.2.6 *Magnetic Stirrer*

6.3 Reagents

6.3.1 *Stock Fluoride Solution*

Dissolve 221.0 mg of sodium fluoride (anhydrous) in distilled water and dilute the same to 1 000 ml (1.00 ml = 100 µg of F^-).

6.3.2 *Standard Fluoride Solution*

Dilute 100 ml of stock fluoride solution with 1 000 ml of distilled (1 ml = 10.0 µg F^-).

6.3.3 *Fluoride Buffer*

Place about 500 ml of distilled water in a 1 000 ml beaker and then add 57 ml of glacial acetic acid, 58 g sodium chloride and 4.0 g of cyclohexylene diamine tetraacetic acid. Stir the solution to dissolve the contents. Keep the beaker in a cool water bath and then gradually add 6N sodium hydroxide (about 125 ml) along with stirring, till the *p*H is between 5.0 and 5.5. Transfer it to a 1 000 ml volumetric flask and then add distilled water up to the mark.

6.3.4 *Sodium Fluoride, Anhydrous*

6.4 Procedure

6.4.1 *Calibration of the Instrument*

No major adjustments are required for the instrument in order to use the electrodes in the range of 0.2 to 2.0 mg F^-/l. In case of the instruments that have zero at the center scale, adjust the control of the calibration so that the 1.0 mg F^-/l standard reads at the central zero (100 mV) at times when the position of the meter is in the expanded scale position.

6.4.2 *Preparation of Fluoride Standards*

Series of standards are prepared by diluting with distilled water. Add 5.0 ml, 10.0 ml and 20.0 ml of standard fluoride solution to distilled water. These prepared standards should be equivalent to 0.5, 1.0 and 2.0 mg F^-/l.

6.4.3 *Treatment of Standards and Sample*

In 100 ml beakers or in other convenient containers, add 10 to 25 ml of standard or sample by using volumetric pipette. Bring the standards and sample to the same temperature, preferably to the room temperature. Add the equal amount of buffer and make sure that its total volume should be enough to immerse the electrodes and permit the operation of the stirring bar.

6.4.4 *Measurement with Electrode*

6.4.4.1 Immerse the electrodes in the each of the fluoride standard solutions and measure the potential developed while the solution is stirred on a magnetic stirrer. Presence of any air bubbles should be checked after immersing the electrodes as the air entrapped around the crystal can produce error in readings or fluctuations of needle. Let the electrodes remain in the solution for about 3 min (or until reading is constant) before taking the final reading on a millivolt. An insulating material layer that exists between the stirrer and the beaker minimizes the heating of the solution. Withdraw the electrodes and rinse it with distilled water and blot them dry between the readings. Repeat the measurements with the samples.

CAUTION — Electrode may get poisoned, if the blotting is not done properly and gently.

6.4.4.2 When the pH meter with an expanded scale or selective ion meter is used, recalibrate the electrode frequently by checking the potential reading of 1.00 mg F^-/L standard and adjust the control of the calibration if required, till the meter reads as before.

6.4.4.3 In case the direct reading of the instrument is not used, plot the measurement of potential for fluoride standards against concentration on two cycle semi-logarithmic axis (ordinate), by keeping the minimum concentration at the bottom of the graph. Plot the millivolts on the abscissa and from the potential measurement for each of the sample, read the corresponding concentration of fluoride from the standard curve.

6.4.4.4 Selective-ion meters may necessitate the use of slightly altered procedure, such as preparing the standards of concentration between 1.00 mg/l and 10.0 mg/l or some other concentration. Follow the directions of the manufacturer. Commercial standards that are often diluted with buffer are frequently supplied with meter. Verify the stated concentration of the fluoride standards by comparing them with the standards that are prepared by the analyst.

6.5 Calculation

$$\text{Concentration of fluoride, in mg /l} = \frac{\mu g\ F^-}{V_1} \qquad \ldots 1$$

where

V_1 = volume of sample, in ml.

7 SPADNS Method

7.1 General

7.1.1 *Principle*

7.1.1.1 The basis of the SPADNS colorimetric method is the reaction between a zirconium dye lake and fluoride. Fluoride can react with the dye lake by dissociating a portion of it into a dye and a colourless complex anion (ZrF_6^{2-}). As the amount of fluoride increases, the appeared colour becomes progressively lighter.

7.1.1.2 The rate of reaction between the zirconium ions and fluoride is greatly affected by the extent of acidity of the reaction mixture. If the proportion of the acid present in the reagent is increased, the reaction can be made almost instantaneous. Under these conditions, the effect of various type of ions differ from that in the conventional alizarin methods. The choice of dye for the rapid fluoride method is largely based on the resulting tolerance to these ions.

7.1.2 *Interference*

7.1.2.1 When any of the substance is present in sufficient quantity thereby causing an error of 0.1 mg/l or if at any time, there is a doubt in total interfering effect, distill the sample. Also distill the sample if it is turbid or colored. In some of the instances, addition of an appropriate amount of interfering substances to the standards or dilution of sample may be used to compensate for the interference effect. In case the alkalinity is the only form of significant interference, neutralize the solution by hydrochloric acid (HCl) or nitric acid (HNO_3). Interference for chlorine and provision for its removal is made.

7.1.2.2 For analytical accuracy volumetric measurement of the reagent and the sample is extremely important. Use the standards and the samples at the same temperature or at least within 2 °C. Maintain the constant temperature throughout the period of colour development. For different ranges of temperature, prepare different calibration curves.

7.2 Apparatus

7.2.1 *Colorimetric Equipment*

One of the following instrument is required for analysis:

7.2.1.1 *Spectrophotometer* — For use at a wavelength of 570 nm providing a light path of atleast 1 cm.

7.2.1.2 *Filter Photometer* — Providing a 1 cm light path and equipped with a greenish yellow filter having a maximum transmittance at 550 nm to 580 nm.

7.3 Reagents

7.3.1 *Stock Fluoride Solution*

Dissolve 221.0 mg of sodium fluoride (anhydrous) in distilled water and dilute the same to 1 000 ml (1.00 ml = 100 µg of F^-).

7.3.2 *Standard Fluoride Solution*

Dilute 100 ml of stock fluoride solution with 1 000 ml of distilled (1 ml = 10.0 µg F^-).

7.3.3 *SPADNS Solution*

Dissolve about 958 mg of SPADNS, sodium-2-(parasulpfophenylazo)-1,8-dihydroxy-3,6-napthalene disulfonate (it is also known as 4,5-dihydroxy-3-(parasulfophenylazo)-2,7-napthalenedisulfonic acid trisodium salt) in distilled water and dilute it to a volume of 500 ml. This solution is stable for a period of 1 year if protected from direct sunlight.

7.3.4 *Zirconyl-Acid Reagent*

Dissolve 133 mg of zirconyl chloride octahydrate in 25 ml of distilled water. To this, add 350 ml of concentrated hydrochloric acid and then dilute it to a volume of 500 ml by using distilled water.

7.3.5 *Acid Zirconyl-SPADNS Reagent*

The reagent is prepared by mixing equal amount of SPADNS solution and zirconyl-acid reagent.

7.3.6 *Reference Solution*

Add about 10 ml of SPADNS solution to 100 ml distilled water and dilute 7 ml of concentrated hydrochloric acid to 10 ml volume. Then add this to SPADNS solution. The resultant solution which is used for setting up the reference point of the instrument is stable for atleast 1 year. Alternatively, use a prepared standard of 0 mg F^-/l as a reference.

7.3.7 *Sodium Arsenite Solution*

Dissolve 5.0 g sodium arsenate ($NaAsO_2$) and dilute it to 1 000 ml with distilled water.

CAUTION — Avoid ingestion.

7.3.8 *Sodium Fluoride, Anhydrous*

7.4 Procedure

7.4.1 *Preparation of Standard Curve*

The fluoride standards are prepared in the range of 0 to 1.40 mg F^-/l by the dilution of appropriate quantities of standard fluoride solution to 50 ml by using distilled water. Pipet out 5.00 ml of zirconyl acid reagent and SPADNS solution or 10.00 ml of mixed acid-zirconyl–SPADNS reagent to each of the standard and mix it properly. Avoid any kind of contamination and set the photometer to zero absorbance by using the reference solution and obtain the readings of the standards for the absorbance value. Plot the milligrams of fluoride absorbance relationship curve and also prepare a new standard curve when a fresh reagent is prepared or some other standard temperature is desired. As an alternative of using a reference, photometer is set at some convenient point where the absorbance is 0.300 or 0.500 with the prepared 0 mg F^-/l standard.

7.4.2 *Pretreatment of Sample*

In case the sample contains some residual chlorine, remove it by adding a drop (which is approximately 0.05 ml) of sodium arsenate solution / 0.1 mg of residual chlorine and mix it well. (It is to be noted that concentrations of sodium arsinite around 1 300 mg/l can produce an error of 0.1 mg F^-/l)

7.4.3 *Development of Colour*

Use 50 ml of sample or a part of it that is diluted to the same volume with distilled water. Then adjust the temperature of the sample to that which is used for the preparation of standard curve. Add 5 ml of zirconyl acid reagent and SPADNS solution or 10.00 ml of acid zirconyl-SPADNS reagent. Mix it well and read the absorbance by first setting the reference point of the photometer as above. If the value of absorbance falls beyond the range of the standard curve, repeat the use of diluted sample.

7.5 Calculation

$$\text{Concentration of } F^-\text{, mg/l} = \frac{A*B}{V_1*C} \qquad \ldots 2$$

where

A = µg F^- determined from plotted curve;

B = final volume of diluted sample, in ml;

C = volume of diluted sample used for the development of color, in ml; and

V_1 = volume of sample, in ml.

If the prepared 0 mg F^-/l standard is used for setting up the photometer, calculate the concentration of fluoride alternatively as follows:

$$\text{Concentration of } F^-\text{, mg/l} = \frac{A_1-A_2}{A_1 - A_t} \qquad \ldots 3$$

where

A_1=absorbance of the prepared 0 mg F^-/l;

A_2= absorbance of the prepared sample; and

A_t= absorbance of a prepared 1.0 mg F^-/l standard.

8 LIQUID CHROMATOGRAPHY METHOD

Fluoride can be determined by liquid chromatography with reference to procedure given in IS 3025 (Part 75).

ANNEX A

(*Clause* 2)

LIST OF REFERRED STANDARDS

IS	*Title*
IS 3025 (Part 75) : 2022	Methods of sampling and test (physical and chemical) for water and wastewater: Part 75 Determination of dissolved anions by liquid chromatography of ions
IS 7022 (Part 1) : 1973	Glossary of terms relating to water, sewage and industrial effluents: Part 1
IS 7022 (Part2) : 1979	Glossary of terms relating to water, sewage and industrial effluents: Part 2
IS 17614 (Part 1) : 2021/ISO 5667 - 3 : 2018	Water Quality — Sampling: Part 1 Guidance on the design of sampling programmes and sampling techniques
IS 17614 (Part 3) : 2021/ISO 5667 - 3 : 2018	Water Quality — Sampling: Part 3 Preservation and handling of water samples

ANNEX B

(*Foreword*)

COMMITTEE COMPOSITION

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Confederation of Indian Industry, New Delhi	Dr Kapil K Narula Dr Sipika Chauhan (*Alternate*)
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