भारतीय मानक
Indian Standard

IS 3025 (Part 60/Sec 2) : 2022
ISO/TS 17951-1 : 2016

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

भाग 60 फ्लोराइड

अनुभाग 2 ऑफ़लाइन आसवन के बाद प्रवाह इंजेक्शन विश्लेषण (एफआईए) तथा स्पेक्ट्रोमेट्रिक डिटेक्शन का उपयोग करने की विधि द्वारा

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

Part 60 Fluoride

Section 2 Method using flow injection analysis (FIA) and spectrometric detection after off-line distillation

(ICS 13.060.50)

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Water Quality Sectional Committee, CHD 36

NATIONAL FOREWORD

‘This Indian Standard (Part 60/Sec2) which is identical with ISO/TS 17951-1 : 2016 ‘Water quality — Determination of fluoride using flow analysis (FIA and CFA) — Part 1 : Method using flow injection analysis (FIA) and spectrometric detection after off-line distillation’ issued by the International Organization for Standardization (ISO) was adopted by the Bureau of Indian Standards on recommendation of the Water Quality Sectional Committee and approval of the Chemical Division Council’.

This Indian Standard (Part 60) has been issued in several sections. Other sections of this Indian Standards are:

> Part 60/Sec 1 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*)
>
> Part 60/Sec 3 Methods of Sampling and Test Physical and Chemical for Water and Wastewater Part 60 Fluoride Section 3 Method using continuous flow analysis (CFA) with automated in-line distillation

The text of the ISO Standard has been approved as suitable for publication as an Indian Standard without deviations. Certain conventions are, however, not identical to those used in Indian Standards. Attention is particularly drawn to the following:

a) Wherever the words 'International Standard' appear referring to this standard, they should be read as 'Indian Standard'

b) Comma (,) has been used as a decimal marker while in Indian Standards, the current practice is to use a point (.) as the decimal marker.

In this adopted standard, reference appears to certain International Standards where the standard atmospheric conditions to be observed are stipulated which are not applicable to tropical/subtropical countries. The applicable standard atmospheric conditions for Indian conditions are 27 ± 2°C and (65 ± 5) per cent relative humidity and shall be observed while using this standard.

In this adopted standard, references appear to certain International Standards for which Indian Standards also exist. The corresponding Indian Standards, which are to be substituted in their respective places, are listed below along with their degree of equivalence for the editions indicated:

International Standard	*Corresponding Indian Standard*	*Degree of Equivalence*
ISO 3696 Water for analytical laboratory use — Specification and test methods	IS 1070 : 1992 Reagent grade water — Specification (*third revision*)	Not Equivalent

The technical committee has reviewed the provisions of the following International Standards referred in this standard and has decided that they are acceptable for use in conjugation with this standard:

International Standard	*Title*
ISO 6353-2	Reagents for chemical analysis — Part 2 : Specifications — First series
ISO 8466-1	Water quality — Calibration and evaluation of analytical methods and estimation of performance characteristics — Part 1 : Statistical evaluation of the linear calibration function
ISO 8466-2	Water quality — Calibration and evaluation of analytical methods and estimation of performance characteristics — Part 2: Calibration strategy for non-linear second-order calibration functions

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 : 2022 ‘Rules for rounding off numerical values (second *revision*)’.

Contents

Introduction

Fluorine compounds in waters and effluents exist in various chemical forms, such as fluoride ion, complexes of iron, aluminium, boron and etc., as well as insoluble forms, such as calcium and magnesium fluorides. Excess fluoride can cause bone damage and fluorosis. The manual steam distillation method can be troublesome because of its complexity. It is necessary to ensure conversion of any insoluble fluorides into soluble fluoride for measurement.

Flow analysis with colourimetric detection is a rapid cost-effective method of determining of soluble (dissolved) fluoride in the method distillate.

This part of ISO 17951 describes FIA methods for flow analysis of fluoride.

Indian Standard

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

PART 60 FLUORIDE

Section 2 Method Using Flow Injection Analysis (FIA) and Spectrometric Detection After off-Line Distillation

WARNING — Persons using this part of ISO 17951 should be familiar with normal laboratory practice. This part of ISO 17951 does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any national regulatory conditions.

IMPORTANT — It is absolutely essential that tests conducted in accordance with this part of ISO 17951 be carried out by suitably qualified staff.

1 Scope

This part of ISO 17951 specifies a method for the determination of fluoride in waters, waste waters and effluents by flow injection analysis (FIA). Any insoluble or complexed fluoride is converted to fluoride ion by a manual distillation procedure from sulfuric/phosphoric acid. Fluoride ion in the distillate is measured using flow analysis with lanthanum alizarin complexone and spectrometric detection. This method is applicable to industrial waste waters, effluents, surface waters, ground waters, leachates. It is not recommended for drinking waters where a distillation step is not required. In this part of ISO 17951, two working ranges are described:

- working range I: 0,1 mg/l to 1 mg/l;
- working range II: 1 mg/l to 10 mg/l.

The specification of the calibration solutions are to be adapted accordingly.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

ISO 6353-2, *Reagents for chemical analysis — Part 2: Specifications — First series*

ISO 8466-1, *Water quality — Calibration and evaluation of analytical methods and estimation of performance characteristics — Part 1: Statistical evaluation of the linear calibration function*

ISO 8466-2, *Water quality — Calibration and evaluation of analytical methods and estimation of performance characteristics — Part 2: Calibration strategy for non-linear second-order calibration functions*

3 Principle

Prior to the introduction of sample solution into the FIA system, the sample solution is manually distilled (see Annex B). Then, the distillate is introduced into a continuous carrier stream (water) by means of injection valve and is mixed with a continuously streaming flow of lanthanum alizarin complexone solution. The formed blue colour is measured by spectrometric detection around 620 nm. Without the distillation, fluoride ion in water samples is determined.

4 Interferences

Without distillation, lanthanum alizarin complexone spectrometric method suffers from the interferences by aluminium, cadmium, cobalt, iron, nickel, beryllium, lead, etc. However, these interferences are removed by the distillation.

5 Reagents

Use only reagents of recognized analytical grade. The prepared solution is degassed, if necessary.

5.1 Water, grade 1, as specified in ISO 3696.

5.2 1,2-Dihydroxyanthraquinonyl-3-methylamine-*N*,*N*-diacetic acid dihydrate (alizarin complexone), $C_{19}H_{15}NO_8 \cdot 2H_2O$.

5.3 Ammonia solution I, $c(NH_3)$ = 15 mol/l, as specified in ISO 6353-2.

5.4 Hydrochloric acid I, $c(HCl)$ = 12 mol/l, as specified in ISO 6353-2.

5.5 Ammonium acetate, $C_2H_7NO_2$, as specified in ISO 6353-2.

5.6 Sodium acetate trihydrate, $C_2H_3O_2Na \cdot 3H_2O$, as specified in ISO 6353-2.

5.7 Acetic acid, $C_2H_4O_2$, as specified in ISO 6353-2.

5.8 Lanthanum(III) oxide, La_2O_3.

5.9 Acetone, C_3H_6O, as specified in ISO 6353-2.

5.10 Sodium fluoride, NaF.

5.11 Sulfuric acid, $c(H_2SO_4)$ = 18 mol/l.

5.12 Phosphoric acid, $c(H_3PO_4)$ = 14,6 mol/l.

5.13 Carrier solution, water (5.1).

5.14 Ammonia solution II.

Mix 10 ml of ammonia solution I (5.3) and 100 ml of water (5.1).

5.15 Ammonium acetate solution, $\rho(C_2H_7NO_2)$ = 200 g/l.

Dissolve 200 g of ammonium acetate (5.5) in about 800 ml of water (5.1). Make up to 1 000 ml with water (5.1).

5.16 Sodium acetate solution.

Dissolve 41 g of sodium acetate trihydrate (5.6) in 400 ml of water (5.1) and add 24 ml of acetic acid (5.7).

5.17 Hydrochloric acid II, $c(\mathrm{HCl}) = 2$ mol/l.

Mix 20 ml of hydrochloric acid (5.4) and 100 ml of water (5.1).

5.18 Lanthanum(III) solution, $c(\mathrm{La(III)}) = 0{,}1$ mol/l.

Add 0,163 g of lanthanum(III) oxide (5.8) to 10 ml of hydrochloric acid II (5.17) and dissolve it by heating of the solution.

5.19 Lanthanum-alizarin complexone solution.

Dissolve 0,192 g of alizarin complexone (5.2) to 4 ml of ammonia solution II (5.14) and 4 ml of ammonium acetate solution (200 g/l) (5.15). Add this solution into 425 ml of sodium acetate solution (5.16) with stirring, and add 400 ml of acetone (5.9) gradually. Then, add 10 ml of lanthanum(III) solution (5.18) to the solution and mix it. After cooling, adjust the pH of the solution to 4,7 with acetic acid (5.7) or ammonia solution I (5.3), then make it up to 1 000 ml with water (5.1).

Lanthanum-alizarin complexone solution (5.19) can be prepared by using alfusone.[1] In that case, after dissolving 1,2 g of alfusone to small amount of water (5.1), add 90 ml of acetone (5.9) and mix the solution. Make up the solution to 300 ml with water (5.1). The solution shall be prepared at the time of analysis.

5.20 Fluoride stock solution ion, $\rho(\mathrm{F}^-) = 100$ mg/l.

Take sodium fluoride (5.10) in a platinum plate and dry at 105 °C at least 1 h. Then cool it in a desiccator. Dissolve 0,221 g of NaF (5.10) in water in a 1 000 ml volumetric flask. Make up to volume with water (5.1).

This solution is stable for one month at room temperature in a polyethylene bottle.

5.21 Fluoride standard solution, $\rho(\mathrm{F}^-) = 10$ mg/l.

Take 10 ml of the fluoride stock solution (5.20) to a 100 ml volumetric flask and make up to volume with water (5.1).

This solution is stable for one week in the dark at 2 °C to 8 °C in a polyethylene bottle.

5.22 Calibration solutions.

Prepare at least five calibration solutions with fluoride concentrations roughly regularly distributed over the working range, by dilution of the appropriate fluoride standard solution or the fluoride stock solution. Examples of calibration solutions for two possible working ranges are given in 5.22.1. For other working ranges, prepare calibration solutions appropriate to cover a decade of concentrations, accordingly.

5.22.1 General

Prepare at least five calibration solutions to cover a decade of concentration, accordingly.

5.22.2 Calibration solutions I for working range 0,1 mg/l to 1 mg/l

For example, six calibration solutions should be prepared as follows.

Pipette, into 100 ml volumetric flasks, 1 ml, 2 ml, 4 ml, 6 ml, 8 ml and 10 ml respectively, of the fluoride standard solution I (5.21) and make up to volume with water (5.1).

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[1] Alfusone is a product commercially available. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of this product.

These solutions contain 0,1 mg/l, 0,2 mg/l, 0,4 mg/l, 0,6 mg/l, 0,8 mg/l and 1 mg/l fluoride, respectively.

5.22.3 Calibration solutions II for working range 1 mg/l to 10 mg/l

For example, six calibration solutions should be prepared as follows.

Pipette, into 100 ml volumetric flasks, 1 ml, 2 ml, 4 ml, 6 ml, 8 ml and 10 ml respectively, of the fluoride standard solution I (5.20) and make up to volume with water (5.1).

These solutions contain 1 mg/l, 2 mg/l, 4 mg/l, 6 mg/l, 8 mg/l and 10 mg/l fluoride, respectively.

6 Apparatus

Usual laboratory equipment and, in particular, the following.

6.1 Flow injection analysis system.

A suitable example of the system contains the components as specified in 6.1.1 to 6.1.5 (see Figure A.1).

6.1.1 Low pulsation pump.

Use a multichannel quantitative pump with a low pulsation.

6.1.2 Sample introduction system.

Use an injector for introducing samples. Select the appropriate amount of sample. Automatic sample introduction device can be used, if necessary.

6.1.3 Reaction manifold, consists of fluorocarbon polymer tubes, such as polytetrafluoroethylene (PTFE) with the internal diameter of 0,5 mm to 0,8 mm, plastics joints of chemically inert and small dead volume and a thermostat which is capable of heating at 70 °C.

6.1.4 Detection system.

Use a spectrometric detector with flow cell, which is capable of measuring at a wavelength of 620 nm ± 5 nm.

UV lamp and the light filter should be calibrated to ensure the repeatability of the results.

6.1.5 Recording system, which is capable of recording signals from the detector.

6.2 Distillation apparatus.

Use a distillation apparatus as described in Annex B as an example.

7 Sampling and sample preparation

Take the samples in polyethylene bottles which have been washed thoroughly and rinsed with fluoride-free water. Polypropylene, polystyrene and polycarbonate bottles can also be used. No preservative is necessary. Analyse the samples within one month. For further information on sample preservation, see ISO 5667-3.

8 Procedure

8.1 Distillation

Distill samples using a distillation apparatus as described in Annex B as an example.

8.2 Setting up the system

Set up the analytical apparatus and the detector ready for the analysis. Then change the water (5.1) to reagent solutions and wait until the base line is stable. Confirm that the drift of baseline does not interfere results and sufficient S/N ratio is obtained.

8.3 Reagent blank measurement

Set the analysis system in operation by first pumping water through the system. Wait for stabilization of the baseline and zero the baseline.

Run the reagent carrier solution [water (5.1)] and lanthanum alizarin complexone solution (5.19) respectively through the system and measure the increase in absorbance against water. If the absorbance per centimetre changes by more than 0,006 cm^{-1} of cell path length, it is possible that either the water or the reagent solutions are contaminated. Take appropriate measures to eliminate the interference.

NOTE If the metric detector does not give absorbance readings, the absorbance can be determined with an external absorbance-measuring spectrometer.

8.4 Adjustment of sensitivity

Adjust the sensitivity of detector to be appropriate for the response by analytical species in the sample. An appropriate path length should be used to achieve a minimum absorbance of 0,005 (absolute value) for a fluoride solution with concentration of the lower end (0,1 mg/l) of the working range.

8.5 Confirmation of repeatability

Analyse a standard solution five times at a concentration in the middle of the using working curve and confirm that repeatability coefficient of variation is not greater than 10 %.

8.6 Calibration

Prepare the calibration solutions for the working ranges by diluting the stock solution (5.20) or the standard solution (5.21) with water (5.1). At least five calibration solutions per working range are prepared. Measure each standard solution by the analytical conditions to be used for the analysis of samples.

Select the working mode of the flow system and calibrate by sequentially applying the calibration solutions (5.22) and the blank solution.

Prior to the calibration, zero the instrument, following the manufacturer’s instructions as long as they are in accordance with the specifications of this part of ISO 17951.

Determine the measured values from the calibration solutions.

The test conditions for the calibration and the measurement of samples (8.7) are the same. The magnitude of the measuring signal is proportional to the mass concentration of fluoride. Establish the regression line for the measuring series obtained.

Calibrate the flow system as specified in ISO 8466-1. In general, Formula (1) is appropriate (ISO 8466-1). If the linearity test described in ISO 8466-1 shows that the calibration curve is not linear, calculate the calibration curve as specified in ISO 8466-2.

The measured value for the calibration solutions, y, in terms of instrument related units (e.g. peak heights in centimetres or counts), is given by:

$$y = b\rho + a \tag{1}$$

where

b is the slope of the calibration function, expressed in instrument-related units per milligram per litre;

ρ is the mass concentration of the calibration solutions, expressed in milligrams per litre, mg/l;

a is the ordinate intercept, expressed in instrument-related units.

8.7 Measurement of samples

Measure samples by using the same conditions as the preparation of the calibration graphs. When the measurement value exceeds the range of the working curve, dilute the samples appropriately. In order to confirm the adequacy of the measurements, measure the calibration solutions for the working curve at the minimum and the maximum concentrations at an interval of 10 to 20 samples. In order to confirm that there is no hindrance to the results of the measurement, compare the response of the calibration solutions at this time with the response of the calibration solutions at the time of the preparation of the calibration graph. In addition, confirm that when each sample is measured, there is no abnormality in the peak shape and confirm that the variation of the baseline is within acceptable limits.

Analyse the samples in the same way as the calibration solutions with the flow injection system.

Make a new calibration, if necessary.

9 Calculation

For the calculation of concentration, use the working curve given in 8.6. Do not extrapolate the working curve.

Determine the mass concentrations of the samples using the measured values, obtained as specified in 8.6 for the calibration solutions.

Calculate ρ using Formula (2):

$$\rho = \frac{y - a}{b} \tag{2}$$

where ρ is the mass concentration of the calibration solutions, expressed in milligrams per litre, mg/l.

For an explanation of the symbols a and b, see 8.6.

10 Expression of results

Describe the concentration of fluoride determined in Clause 9 by using the unit of mg/l as F^-.

Report the results to two significant figures at most.

EXAMPLE 1 $\rho(F^-)$ 0,35 mg/l.

EXAMPLE 2 $\rho(F^-)$ 6,3 mg/l.

11 Test report

This test report shall contain at least the following information:

a) the test method used, together with a reference to this part of ISO 17951, i.e. ISO/TS 17951-1:2016;

b) all information necessary for identification of the sample;

c) the type of sample pretreatment;

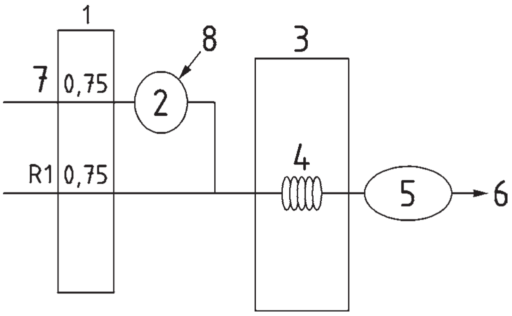
d) the fluoride concentration in milligrams per litre, expressed in accordance with Clause 10;

e) any special observations noted during the determination;

f) any deviations from this part of ISO 17951, which could have affected the result.

Annex A
(informative)

Example of flow injection analysis (FIA) system



Key

1	pump, flow rate ml/min
2	injector for introducing samples, sample volume 200 µl or 400 µl
3	thermostat, 70 °C
4	reaction coil, internal diameter 0,5 mm, length 10 m
5	detector 620 nm ± 5 nm
6	waste water
7	carrier solution (water)
8	sample
R1	lanthanum arizarin complexone solution (5.19)

NOTE Dimension of reaction coil is the value in Key 4 and the flow rate is 0,75 ml/min.

Figure A.1 — Example of a flow injection system (FIA) for the determination of fluoride in the working range 0,1 mg/l to 1 mg/l

Annex B
(informative)

Example of a distillation apparatus and procedure

B.1 Distillation

B.1.1 Additional reagents

B.1.2 Phenolphthalein solution, $\rho(C_{20}H_{14}O_4) = 5$ g/l.

Dissolve 0,5 g of phenolphthalein to 50 ml of ethanol (95) and make up to 100 ml with water.

B.1.3 Sodium hydroxide solution, $\rho(NaOH) = 100$ g/l.

Dissolve 10 g of sodium hydroxide to water and make up to 100 ml with water.

B.1.4 Silicon dioxide, SiO_2.

Use SiO_2 with a particle size of 100 µm to 150 µm.

B.2 Procedure

Take a suitable volume of sample (containing 30 µg or more amount of F^-) into a porcelain evaporating dish or a beaker. Add two or three drops phenolphthalein solution (B.1.2). Adjust the solution to slightly alkaline by adding sodium hydroxide solution (B.1.3) and concentrate the solution to about 30 ml by heating.

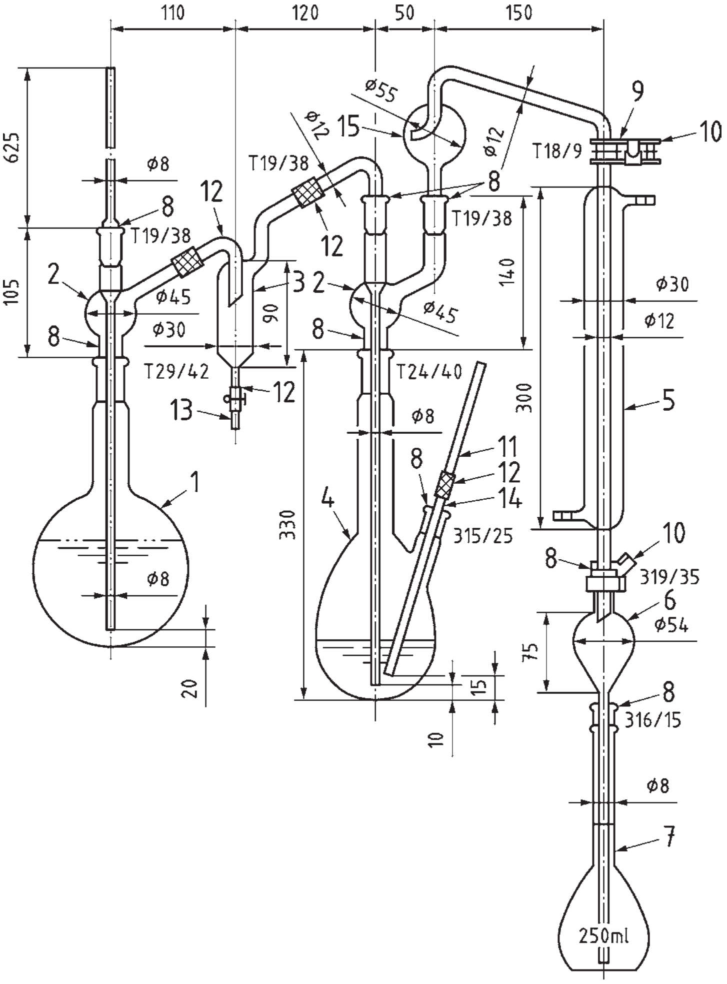
Quantitatively transfer the solution to the distillation flask (Figure B.1, Key 4). Add 1 g of silicon dioxide (B.1.4), 1 ml of phosphoric acid (5.12) and 30 ml of sulfuric acid (5.11). Add 20 ml of water (5.1) to the receiver (volumetric flask 250 ml) (Figure B.1, Key 7) and keep the end of backflow preventer (Figure B.1, Key 6) under the surface of water.

Heat the distillation flask (Figure B.1, Key 4) directly and pass steam after the temperature of the liquid in the distillation flask reach the temperature of about 140 °C.

Adjust the distillation temperature at 145 °C ± 5 °C and the distillate rate from 3 ml/min to 5 ml/min and continue the distillation until the fluid volume in the receiver (Figure B.1, Key 7) reaches 220 ml.

Remove the Liebig condenser (Figure B.1, Key 5) and the backflow preventer (Figure B.1, Key 6). Then, rinse the inner pipe of the condenser and the inside and outside of the backflow preventer (Figure B.1, Key 6) with water (5.1). Add the wash liquid to the receiver (Figure B.1, Key 7) and add water (5.1) to the marked line.

Other distillation apparatus and procedures are available.



Key

1 steam generation flask, 1 000 ml
2 inlet glass tube
3 trap
4 distillation flask, 500 ml
5 Liebig condenser, 300 ml
6 backflow preventer (about 50 ml)
7 receiver (volumetric flask, 250 ml)
8 interchangeable ground glass joint
9 interchangeable ball joint
10 holddown spring
11 thermometer, 200 °C
12 rubber tube
13 pinch cock
14 insert thermometer stopper
15 ball trap (Kildall type)

Figure B.1 — Example of a distillation apparatus

Annex C
(informative)

Results of the interlaboratory trial

Table C.1 — Results of the interlaboratory trial

No	Analyst	Sample	Results	Detection
1	USA, Analysis company	Synthetic sample	5,88 mg/l ± 0,017 mg/l, (*n* = 9), RSD 0,29 %	Ion selective electrode
2	Spain, University	Lake Water	0,82 mg/l ± 0,004 mg/l, (*n* = 5), RSD, 0,49 %	Colourimetric
3	Thailand, University	River water	0,91 mg/l ± 0,020 mg/l, (*n* = 3), RSD 2,0 %	Colourimetric
4	CANADA, Analysis company	Effluent of research institute	7,53 mg/l ± 0,026 mg/l, (*n* = 3), RSD 4,3 %	Ion selective electrode
5	Japan, University	Synthetic sample	4,62 mg/l ± 0,068 mg/l, (*n* = 10), RSD 1,5 %	Colourimetric
6	Japan, University	Effluent of research institute	7,49 mg/l ± 0,0002 mg/l, (*n* = 3), RSD 0,0031 %	Colourimetric
7	Japan, Analysis company	River water	1,02 mg/l ± 0,004 mg/l, (*n* = 9), RSD 0,39 %	Colourimetric
8	Japan, Analysis company	Lake Water	0,82 mg/l ± 0,003mg/l, (*n* = 9), RSD 0,29 %	Colourimetric
9	Japan, Research institute	River water	0,93 mg/l ± 0,017 mg/l, (*n* = 5), RSD 1,8 %	Colourimetric
10	Japan, Research institute	Synthetic sample	5,59 mg/l ± 0,025 mg/l, (*n* = 3), RSD 0,45 %	Colourimetric

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Bureau of Indian Standards

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Review of Indian Standards

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the website-www.bis.gov.in or www.standardsbis.in.

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