

भारतीय मानक

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

भाग 55 एल्यूमिनियम

( पहला पुनरीक्षण )

*Indian Standard*

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

PART 55 ALUMINIUM

( *First Revision* )

ICS 13.060.50

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**BUREAU OF INDIAN STANDARDS**  
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NEW DELHI 110002

## FOREWORD

This Indian Standard (Part 55) (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.

Pollution caused by substances, on which biotic and abiotic agencies of decomposition are ineffective, is a unique type of pollution. Toxic trace elements and heavy metals come under the category of non-degradable pollutants. The problem caused by these elements is in fact due to their concentration in the environment in the bio-available state and above a certain concentration become harmful to the living organism.

Aluminium is the third most abundant element of the earth's crust (estimated range from 8.1-8.8 percent by weight). This wide distribution accounts for the presence of aluminium in nearly all natural waters. In natural waters, the aluminium concentrations ranges from 3 µg/l to 20 µg/l. Soluble, colloidal and insoluble aluminium may also appear in treated water or waste water as residual from the alum coagulation. The aqueous solubility of aluminium is lowest in approximately neutral pH environment, but it increases in high and in low pH environments.

Plenty of aluminium (Al) is present in foodstuff and water. It has been estimated that a normal individual ingests about 80.5 mg of this element per day. Aluminium levels as high as 50-60 times the average daily intake do not appear to cause any interference in the metabolism of the human body. Higher levels of aluminium, however, may interfere with phosphate metabolism, inhibit absorption of fluorides, calcium and iron compounds. Pulmonary effects of aluminium follow inhalation of bauxite aerosols. Both restrictive and obstructive pulmonary diseases develop as a sequel to fibrosis in tissues of lung. Aluminium compounds dissolved in water are much more injurious than as particulates in suspended state. An aluminium content higher than 1.5 µg/ml may cause anorexia, decreased activity and gill hyperplasia in fishes. To plants the injuring action of aluminium is enhanced under acidic conditions (pH 5 or below). Roots become thick, lack finer branches and are unable to absorb water and nutrients efficiently. Plants become stunted, stubby and brittle. Usually young seedlings are more susceptible to the toxic effects of aluminium than older plants.

However, in view of the environmentally prevalent nature of Aluminium compounds in levels should be closely monitored to avoid any likelihood of toxicity caused by this element. As per IS 10500 : 1991 'Drinking water — Specification (*first revision*)' the permissible limit for aluminium in drinking water is 0.03 µg/l, maximum. Beyond this limit, a cumulative effect is reported to cause dementia.

The committee responsible for formulation of IS 3025 : 1964 had decided to revise the standard and publish it as separate parts. This standard (Part 55) supersedes 31 of IS 3025 : 1964.

In the preparation of this standard considerable assistance has been derived from Standard Methods for the Examination of Water and Waste Water, 19th Edition, 1995, published by the American Public Health Association, Washington, USA.

Atomic absorption method specified in this standard is technically equivalent to the Flame AAS method specified in ISO 12020 : 1997, 'Water quality — Determination of aluminium — Atomic absorption spectrometric methods'. ISO 10566 : 1994, 'Water quality — Determination of aluminium — Spectrometric method using pyrocatechol violet' specifies Spectrometric method using the indicator 'Pyrocatechol violet' against the use of 'Eriochrome cyanine R' as indicator in Eriochrome cyanine R method of this standard.

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

In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

*Indian Standard***METHODS OF SAMPLING AND TEST (PHYSICAL AND CHEMICAL) FOR WATER AND WASTEWATER****PART 55 ALUMINIUM***( First Revision )***1 SCOPE**

1.1 This standard (Part 55) prescribes two methods for the determination of aluminium:

- a) Eriochrome cyanine R method, and
- b) Atomic absorption method.

1.2 Depending upon the concentration range and interference levels, choice of the method is made. For dissolved aluminium content, filtration through 0.45 mm membrane filter is required. Do not use filter paper, absorbent cotton, or glass wool for filtering any solution that is to be tested for aluminium, because these will remove most of the soluble aluminium.

**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.

<i>IS No.</i>	<i>Title</i>
3025 (Part 1): 1987	Methods of sampling and test (physical and chemical) for water and wastewater : Part 1 Sampling ( <i>first revision</i> )
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 SAMPLING AND STORAGE**

The sampling bottles shall be cleaned thoroughly with dilute nitric acid (6 N), prior to the final rinsing with water. The water samples should be collected and stored preferably in polypropylene bottles or chemically resistant glass containers. For preservation, the samples should be acidified with concentrated nitric acid (2 ml of concentrated nitric acid in 1 litre sample, just to bring down the pH below 2). Unacidified samples should be analysed on the same day, while

the acidified samples can be stored for a few days (up to 5 days) in a refrigerator.

**4 PURITY OF THE REAGENTS**

Unless specified otherwise, only pure chemicals and Aluminium free distilled water shall be used in tests.

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

**5 ERIOCHROME CYANINE R METHOD****5.1 Principle**

Aluminium forms a red to pink complex with 'Eriochrome cyanine R' at pH 6.0. The colored complex obeys Beer's law and is suitable for spectrophotometric measurements between 525 nm and 535 nm. This method is applicable in the range of 20 to 300 µg/l of aluminium.

**5.2 Interferences**

Interference of heavy metals like iron and manganese, often found in water, is eliminated by adding ascorbic acid. Fluoride and polyphosphates, give rise to negative errors. Interference from fluoride can be overcome by adding known amounts (depending upon amount of fluoride present in the sample) of fluoride to the set of standards. Similarly, interference due to complex phosphate can be eliminated during sample treatment (*see 5.5.3.2*). The interference by alkalinity can be removed by acidifying samples just beyond neutralization point of methyl orange. Sulphate do not interfere up to concentration of 2 µg/l.

**5.3 Apparatus**

5.3.1 *Spectrophotometer* — for use at 535 nm with 1 cm cells.

5.3.2 *pH Meter*

5.3.3 *Standard Volumetric Glasswares*

**5.4 Reagents**

5.4.1 *Sulphuric Acid* — 0.02 N and 6 N

5.4.2 *Ascorbic Acid Solution* — 0.1 percent w/v in water. Prepare fresh reagent every day.

### 5.4.3 Buffer Solution

Dissolve 136g of sodium acetate  $\text{NaC}_2\text{H}_3\text{O}_2 \cdot 3\text{H}_2\text{O}$  in water, add 40 ml of 1 N acetic acid, and dilute to 1 litre with water.

### 5.4.4 Acetic Acid Solution — 1 : 1 and 1 N.

### 5.4.5 Sodium Hydroxide Solution — 0.1 N and 1 N.

### 5.4.6 Stock Eriochrome Cyanine R Dye Solution

Dissolve 300 mg of dye in about 50 ml of water. Adjust pH to about 2.9, with the help of 1 : 1 acetic acid. Dilute with water to 100 ml in a volumetric flask.

### 5.4.7 Working Eriochrome Cyanine R Dye Solution

Dilute 10 ml of stock dye solution to 100 ml in a volumetric flask.

NOTE — Working dye solution is stable for at least 6 months.

### 5.4.8 Stock Aluminium Solution

Dissolve 8.791 g of aluminium potassium sulphate,  $\text{AlK}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$  in water and dilute to 1 000 ml with water in a volumetric flask (1 ml = 500 µg of aluminium).

### 5.4.9 Standard Aluminium Solution

Dilute 10 ml of the stock aluminium solution to 1 000 ml with water in a volumetric flask (1 ml = 5.0 mg of aluminium).

NOTE — The standard aluminium solution is stable for at least one week.

### 5.4.10 Methyl Orange Indicator Solution

Dissolve 50 mg of methyl orange in 100 ml of water.

## 5.5 Procedure

### 5.5.1 Preparation of Calibration Curve

Prepare a series of aluminium standards from 0 to 7 µg (0 ml for the reagent blank), by accurately measuring calculated volumes of standard aluminium solution into 50 ml volumetric flasks (see 5.5.3.1). Add distilled water to make a total volume of approximately 25 ml. Add 1 ml of 0.02 N of  $\text{H}_2\text{SO}_4$  and 1 ml of ascorbic acid solution to each of the volumetric flasks and mix. Add 10 ml of buffer solution and mix. Pipette out 5.0 ml of working dye reagent into it and mix. Immediately make up to 50 ml with water. Mix and let stand for 5 to 10 min. The colour generally begins to fade after 15 min. Measure the absorbance of the aluminium complex at 535 nm using reagent blank as reference solution. Construct a calibration curve by plotting absorbance values against micrograms of aluminium in 50 ml of the final solution.

### 5.5.2 Determination of Aluminium in the Absence of Fluoride and Phosphates

Place 25.0 ml of the sample, or a portion diluted to 25 ml, in a conical flask, add a few drops of methyl

orange indicator, and titrate with 0.02N  $\text{H}_2\text{SO}_4$  to a faint pink colour. Record the reading and discard the solution. To two similar samples taken in 50 ml volumetric flasks add the same amount of  $\text{H}_2\text{SO}_4$  used in the titration and 1 ml in excess. To one sample add 1 ml of EDTA solution. This will serve as a reagent blank by complexing any aluminium present, and compensating for colour and turbidity. To both samples add 1 ml of ascorbic acid, 10 ml of buffer solution, and 5.0 ml of working dye solution. Dilute with water up to the mark. Set instrument to zero absorbance using the reagent blank containing EDTA. After 5 to 10 min of contact time, read the absorbance at 535 nm and determine the aluminium concentration from the calibration curve.

### 5.5.3 Determination of Aluminium in the Presence of Phosphate and Fluoride

#### 5.5.3.1 Removal of fluoride interference

Interference from fluoride is overcome by adding known amounts (depending upon the amount of fluoride present in the sample) of fluoride to the set of standards used during the calibration stage.

#### 5.5.3.2 Removal of phosphate interference

Add 1.7 ml of 6 N  $\text{H}_2\text{SO}_4$  to 100 ml of the sample in a 200 ml conical flask. Heat on a hot plate for at least 90 min, keeping solution temperature just below the boiling point (At the end of the heating period the volume of the solution should be about 25 ml. Add water, if necessary, to keep it at around that volume). After cooling, neutralize to a pH of 4.3 to 4.5 with NaOH, using 1N NaOH at the start and 0.1N for the final fine adjustment. Monitor with a pH meter. Make up to 100 ml in volumetric flask with water, mix, and use a 25 ml portion for the aluminium determination as described in 5.5.2. Run a blank in the same manner, using water and 1.7 ml of 6 N  $\text{H}_2\text{SO}_4$ . Subtract the blank reading from the sample reading or use the latter to set the instrument to zero absorbance before, taking the reading with the sample.

## 5.6 Calculation

$$\text{Aluminium, mg/l} = \frac{M}{V}$$

where

$M$  = mass of aluminium present in µg in 50 ml of the final solution, and

$V$  = volume of the sample in ml.

## 5.7 Precision and Accuracy

The relative standard deviation reported in the literature for aluminium in the 500 µg/l concentration range is 34.4 percent.

## 6 ATOMIC ABSORPTION METHOD (DIRECT)

### 6.1 Principle

The aluminium content of the sample is determined by atomic absorption spectrophotometry. For dissolved aluminium the filtered sample is directly aspirated to the atomizer. For total recoverable aluminium, a pretreatment with conc HCl is carried out, prior to aspiration of the sample. This method is applicable in the range from 5 to 100 mg/l of aluminium. However, the concentration range will vary with the sensitivity of the instrument used.

### 6.2 Apparatus

**6.2.1 Atomic Absorption Spectrophotometer** — with nitrous oxide-acetylene flame, hollow-cathode lamp or electrodeless discharge lamp for use at 309.3 nm.

NOTE — Use nitrous oxide with strict adherence to manufacturer's directions. Improper sequencing of gas flows at start up and shut down of instrument can produce explosion from flash back.

### 6.2.2 Standard Volumetric Glasswares

### 6.3 Reagents

**6.3.1 Hydrochloric Acid** — concentrated (11 N).

**6.3.2 Nitric Acid** — concentrated (16 N) and dilute (1 : 499).

**6.3.3 Potassium Chloride Solution**

Dissolve 250 g of KCl in water and dilute to 1 000 ml in a volumetric flask.

**6.3.4 Stock Aluminium Solution**

Dissolve 8.791 g of aluminium potassium sulphate,  $\text{Al K}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$  in water and dilute to 1 000 ml with water in a volumetric flask (1 ml = 500  $\mu\text{g}$  of Al).

**6.3.5 Standard Aluminium Solution**

Dilute 10 ml of stock aluminium solution to 1 000 ml with water in a volumetric flask (1 ml = 5.0  $\mu\text{g}$  of Al).

NOTE — Fresh standard aluminium solution to be prepared after a week.

### 6.4 Procedure

#### 6.4.1 Calibration

Prepare a reagent blank and sufficient standards containing 0 to 60 mg/l of aluminium by diluting suitable volumes of standard aluminium solution with nitric acid (1 : 499) to 100 ml in volumetric flasks. Add 2 ml of KCl solution to each of the volumetric flasks. Aspirate the reagent blank and carry out zero adjustment. Aspirate sequentially the standard solutions and measure the absorbance at 309.3 nm.

#### 6.4.2 Determination of Aluminium

Add 0.5 ml of concentrated nitric acid to 100 ml of the sample taken in a 250-ml beaker. (If total recoverable aluminium is to be determined, use 100 ml of the sample obtained after a pretreatment as given below. Add 5 ml of concentrated hydrochloric acid to 100 ml of the sample taken in a 250-ml beaker. Heat on a hot plate to reduce the volume to about 50 ml and filter the sample through 0.45  $\mu\text{m}$  membrane. Transfer quantitatively the contents of the beaker to a 100 ml volumetric flask. Make up to the mark in a volumetric flask.) Add 2ml of KCl solution to this. Prepare a reagent blank with 100 ml of water. Rinse the atomizer by aspirating water containing 1.5 ml conc  $\text{HNO}_3$ /l. Aspirate the reagent blank and carry out zero adjustment. Aspirate the sample solution and measure the absorbance at 309.3 nm. From the absorbance data, determine the micrograms of aluminium present in 100 ml of the final solution.

### 6.5 Calculation

$$\text{Aluminium, } \mu\text{g/l} = \frac{M}{V}$$

where

$M$  = mass of aluminium present in  $\mu\text{g}$  in 100 ml of the final solution, and

$V$  = volume of the sample in ml.

### 6.6 Precision and Accuracy

The relative standard deviation reported in the literature for aluminium in the 4.5 mg/l concentration range is 4.2 percent.

## ANNEX A

*(Foreword)*

## COMMITTEE COMPOSITION

## Environment Protection and Waste Management Sectional Committee, CHD 32

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### Amendments Issued Since Publication

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