

इंटरनेट

मानक

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Whereas the Parliament of India has set out to provide a practical regime of right to information for citizens to secure access to information under the control of public authorities, in order to promote transparency and accountability in the working of every public authority, and whereas the attached publication of the Bureau of Indian Standards is of particular interest to the public, particularly disadvantaged communities and those engaged in the pursuit of education and knowledge, the attached public safety standard is made available to promote the timely dissemination of this information in an accurate manner to the public.

“जानने का अधिकार, जीने का अधिकार”

Mazdoor Kisan Shakti Sangathan

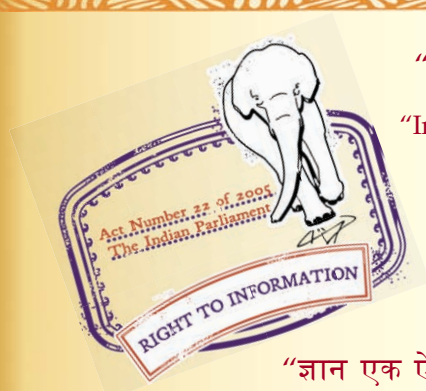
“The Right to Information, The Right to Live”

“पुराने को छोड़ नये के तरफ”

Jawaharlal Nehru

“Step Out From the Old to the New”

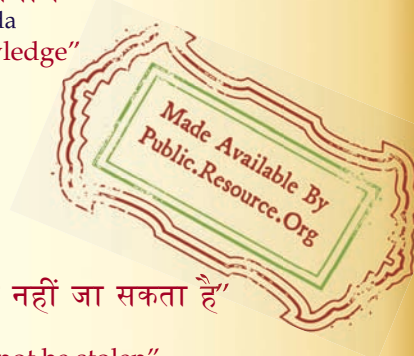
IS 3025 (Part 45) (1993, Reaffirmed 2003): Method of Sampling and Test (Physical and Chemical) for Water and Wastewater, Part 45: Sodium and Potassium (First Revision).
ICS 13.060.50



“ज्ञान से एक नये भारत का निर्माण”

Satyanarayan Gangaram Pitroda

“Invent a New India Using Knowledge”



“ज्ञान एक ऐसा खजाना है जो कभी चुराया नहीं जा सकता है”

Bhartrhari—Nitiśatakam

“Knowledge is such a treasure which cannot be stolen”

भारतीय मानक
जल और अपशिष्ट जल के नमूने लेने एवं परीक्षण
(भौतिक एवं रसायन) की पद्धतियां

भाग 45 सोडियम एवं पोटेशियम
(पहला पुनरीक्षण)

Indian Standard

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

PART 45 SODIUM AND POTASSIUM

(First Revision)

Third Reprint SEPTEMBER 2007
(Including Amendment No. 1)

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

FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Environmental Protection Sectional Committee had been approved by the Chemical Division Council.

Industrial wastes invariably contain significant quantities of sodium. Sodium salts being extremely soluble get leached early from soil and rocks and tend to remain in solution.

Sodium is not particularly significant in potable water except for those having an abnormal sodium metabolism. For high pressure boiler feed water even trace amounts of sodium are of concern.

Potassium rocks, such as silicates, are more soluble in water than sodium forms. Most natural waters contains less than 20 mg/l of potassium, but water containing several hundred milligrams per litre are occasionally found. Potassium is essential to animal nutrition.

In the preparation of this standard, considerable assistance has been derived from standard methods for the examination of water and waste water published by American Public Health Association, Washington, USA, 16th edition, 1985.

The composition of the technical committee responsible for this preparation of the standard is given in Annex A.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

AMENDMENT NO. 1 APRIL 2003
TO
IS 3025 (PART 45) : 1993 METHODS OF SAMPLING
AND TEST (PHYSICAL AND CHEMICAL) FOR WATER
AND WASTE WATER

PART 45 SODIUM AND POTASSIUM

(First Revision)

(Page 3, clause 5.6.2) — Substitute the following for the existing equation:

‘For bracketing approach,
sodium (or potassium), mg/l = $\left[\frac{(B-A)(s-a)}{(b-a)} + A \right] \times D$ ’

(CHD 32)

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Indian Standard

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

PART 45 SODIUM AND POTASSIUM

(First Revision)

1 SCOPE

This standard prescribes following three methods for determination of sodium and potassium:

- a) flame emission photometric method using either a flame photometer or an atomic absorption spectrophotometer in the flame emission mode;
- b) atomic absorption spectrometric method using an atomic absorption spectrophotometer in flame absorption mode; and
- c) gravimetric method for determination of sodium and colorimetric method for determination of potassium.

2 REFERENCES

The Indian Standards listed below are necessary adjuncts to this standard:

IS No.	Title
3025 (Part 1) : 1986	Methods of sampling and test (physical and chemical) for water and waste water : 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 TERMINOLOGY

For the purpose of this standard, definitions given in IS 7022 (Part 1) : 1973 and IS 7022 (Part 2) : 1979 shall apply.

4 SAMPLING AND STORAGE

The sampling and storage shall be done as prescribed in IS 3025 (Part 1) : 1986. Acidify the sample to pH 2 with concentrated nitric acid. Store all samples/stock solutions in polyethylene bottles.

5 FLAME PHOTOMETRY METHOD

5.1 Principle

A flame photometer measures photoelectrically the intensity of colour imparted to the flame of a Meker-type burner where the sample is introduced into the flame under carefully standardized conditions. The intensity of colour is proportional to the sodium/potassium content in the sample. Sodium and potassium are determined at a wavelength of 589 nm and 766.5 nm, respectively.

5.2 Interferences

5.2.1 Radiation interference caused by elements other than those being determined is the chief contributing factor for error in flame photometry. Of the elements encountered in these analyses, the major effect is due to interference of one alkali-metal on another. Some effects are positive and some are negative. Among the other common ions capable of causing interference are Cl^- , SO_4^{2-} and HCO_3^- in relatively higher concentration. The foreign element effects cannot be entirely compensated without employing calibration standards closely duplicating the composition of the sample or by applying an experimentally determined correction in those instances where the sample contains a single important interference. However, the effects may be minimized by operating at the lowest practical sodium or potassium concentration range or by removal of the interfering elements. For example, aluminium has a depressing effect on alkali-metal emission, which may be of serious consequence. The amount of aluminium normally present in water has been found to have a negligible effect on alkali-metal determination. However, aluminium in waste waters should be removed from the solution prior to flame photometry if its concentration has been found by preliminary tests to exceed that of the alkali metal being determined.

5.2.2 Self-absorption causes the curve of intensity versus concentration to decrease in slope at higher concentrations tending to reduce accuracy. Bracketing the unknown by known standard solution and inter-polating linearly between the two minimizes this interference so as to render it practically negligible.

5.2.3 Suspended matter which may interfere mechanically by clogging the burner shall be removed by filtration prior to analysis. Organic colouring matter does not cause interference and need not be removed.

5.2.4 Flame photometers operating on the internal standard principle may require adding a standard lithium solution to each working standard and sample. Follow the manufacturers instructions for the optimum lithium concentration.

5.2.4.1 Incorporate a non-ionic detergent in the standard lithium solution to assure proper aspirator function when using the internal standard type flame photometer.

5.2.5 In the internal standard technique, potassium interferes with the sodium determination if the potassium to sodium ratio is higher than 5 : 1 and sodium interferes with the potassium determination if the sodium to potassium ratio is higher than 5 : 1. Calcium interferes if the calcium to sodium/potassium ratio is higher than 10 : 1. In such cases an approximate concentration of the interfering ions may be added to the sodium/potassium calibration standards. Magnesium interference does not appear until magnesium to sodium/potassium ratio exceeds 100 : 1.

5.3 Apparatus

5.3.1 Flame Photometer

Either direct-reading or internal standard type or an atomic absorption spectrophotometer in the flame emission mode.

5.3.2 Glassware

Rinse all glassware with dilute nitric acid (1 : 15) followed by several portions of deionized distilled water.

5.4 Reagents

Use deionized distilled water to prepare all reagents, calibrations, standards and dilution water.

5.4.1 Stock Sodium Solution

Dissolve in deionised distilled water, 2.542 g of sodium chloride dried to constant mass at 140°C and make up to 1 000 ml with water, 1 ml = 1 mg of sodium.

5.4.2 Stock Potassium Solution

Dissolve in deionised distilled water 1.907 g of potassium chloride dried at 110°C, and make up to 1 000 ml with water, 1 ml = 1 mg of potassium.

5.4.3 Standard Lithium Solution

Weigh rapidly 6.109 g of lithium chloride (LiCl) or 9.93 g of lithium nitrate (LiNO₃) dried overnight in an oven at 105°C. Dissolve in water and make up to 1 000 ml, 1 ml = 1 mg of lithium.

NOTE — Prepare a new calibration curve whenever the standard lithium solution is changed.

5.5 Procedure

5.5.1 Pretreatment of Polluted Water and Waste-Water Samples

Take 100 ml of sample in a beaker and add 5 ml of concentrated nitric acid and evaporate to dryness. Repeat this operation once again by adding concentrated nitric acid. Dissolve the residue in minimum volume of concentrated hydrochloric acid. Boil to dissolve, dilute to 50 ml with water, filter and make up to 100 ml.

NOTE — No preliminary treatment of sample is needed except filtration through an 11 cm ashless filter paper of medium retentiveness if the sample contains only suspended matter as an impurity.

5.5.2 Precautions

Place the instrument away from direct sunlight or constant light emitted by an overhead fixture in an area free of drafts, dust and tobacco smoke. Avoid contamination from corks, filter paper, perspiration, soap, cleansers, cleaning mixtures and inadequately rinsed apparatus.

5.5.3 Instrument Operation

Follow manufacturer's instructions for selecting proper photocell and wavelength, adjusting slit width and sensitivity, appropriate fuel and air or oxygen pressures and the steps for warm-up, flame background, rinsing of burner, aspirating and igniting samples and measuring emission intensity.

5.5.4 Direct-Intensity Measurement

Prepare a blank and sodium/potassium standards in stepped amounts by diluting the stock solutions described in 5.4.1 and 5.4.2 for any of the following applicable ranges : 0 to 1.0 mg/l, 0 to 10 mg/l, or 0 to 100 mg/l, so that within each range there are equally spaced standards in tenths of the maximum. Starting with the highest calibration standard and working towards the most dilute standard, measure emission at 589 nm for sodium and 766.5 nm for

Add an appropriate volume of standard lithium solution to carefully measured volume of sample (or diluted portion), each sodium/potassium calibration standard and the blank, and then follow all the steps described in 5.5.4.

From the calibration curve, select and prepare sodium/potassium standards that immediately bracket the emission intensity of the sample. Determine emission intensities of the bracketing standards (one standard slightly less and the other slightly greater than the sample) and the sample as simultaneously as possible. Repeat the determination on bracketing standards and samples.

5.6.1 For direct intensity measurement (5.5.4) and internal standard measurements (5.5.5.) :

$$\frac{\text{Sodium (or potassium), mg/l}}{\text{Sodium (or potassium) in mg/l in portion} \times D}$$

approach sodium
(or potassium), $= \frac{(B-A)(s-a)}{(b-a)} + A \times D$
mg/l

B = sodium (or potassium) in upper
bracketing standard, mg/l;

A = sodium (or potassium) in lower bracketing standard, mg/l;

b = emission intensity of upper bracketing standard;

a = emission intensity of lower bracketing standard;

s = emission intensity of sample; and

$$D = \text{dilution ratio} = \frac{\text{sample in ml} + \text{distilled water in ml}}{\text{sample in ml}}$$

5.6.3 Repeat the calculation for the second set of data and average the two values to obtain the concentration of sodium (or potassium).

This method is based on the fact that when a solution containing salts of sodium and potassium is introduced in the flame, the intensity of the transmitted radiation will decrease in proportion to the extent of the concentration of these elements in solution. A hollow cathode lamp made of the element to be determined provides the source of radiation. The application range of this method is 0.20 to 4.0 mg/l when using the 766.5 nm resonance line for potassium and 0.20 to 3.0 mg/l when using the 589.6 nm resonance line for sodium.

6.2.1 No chemical interferences have been found. Sodium and potassium are partially ionized in air-acetylene flame (to suppress ionization add potassium nitrate or potassium chloride to give final concentration of 2 000 $\mu\text{g}/\text{ml}$ of potassium in the solution, in which sodium has to be measured and add any one of the cesium nitrate, cesium chloride, sodium nitrate and sodium chloride to give a final concentration of 1 000 $\mu\text{g}/\text{ml}$ of cesium or 2 000 $\mu\text{g}/\text{ml}$ of sodium as the case may be, in all solutions including blank where potassium has to be measured). Presence of appreciable concentrations of mineral acids in the solution, reduce the absorbance reading for both sodium and potassium. Hydrochloric acid, sulphuric acid, nitric acid and phosphoric acid cause decrease in the reading. Influence of nitric acid and sulphuric acid is mainly due to changes in physical properties and the aspiration rates of the solution. The influence of hydrochloric acid and phosphoric acid is due to the evaporation process in the flame.

6.2.2 Concentration of aluminium, phosphate, sulphate and silicate up to 160 mg/l do not interfere with low concentration of sodium, namely, to 2 to 10 mg/l. But it is found that slight decrease in absorption occurs in the presence of high concentration of phosphates. But the same amount of aluminium, phosphate, sulphate and silicate do not interfere with potassium, determination in the presence of aluminium chloride (1M).

Atomic absorption spectrophotometer in the absorption mode, set up and operated according to the manufacturer's instructions, equipped with an appropriate burner for air-acetylene flame and hollow cathode lamps for sodium and potassium determinations.

6.4 Reagents

6.4.1 Potassium Chloride Solution

Dissolve 40 g of potassium chloride in distilled water and make up to 1 litre.

6.4.2 Sodium Chloride Solution

Dissolve 50 g of sodium chloride in distilled water and make up to 1 litre.

6.4.3 Stock Sodium Solution — See 5.4.1.

6.4.4 Stock Potassium Solution — See 5.4.2.

6.4.5 Standard Sodium Solution

Take appropriate aliquots of the stock sodium solution (5.4.1) in a series of 100 ml volumetric flasks to give a range of standard solutions in the working range. Add 10 ml of potassium chloride solution (6.4.1) and dilute to 100 ml to have about 2 000 µg/l of potassium to suppress ionization while working in air-acetylene flame.

6.4.6 Standard Potassium Solution

Take appropriate aliquots of the stock potassium solution (4.5.2) in a series of 100 ml volumetric flasks to give a range of standard solutions in the working range. Add 10 ml of sodium chloride solution (6.4.2) and dilute to 100 ml to have about 2 000 µg/l of sodium in the working solution.

6.5 Procedure

6.5.1 Preparation of Sample Solution — See 5.5.1.

6.5.2 Preparation of Test Solutions

Take 10 ml of the sample in a 100 volumetric flask. Add 10 ml of potassium chloride solution (6.4.1) if sodium is to be determined and 10 ml of sodium chloride solution (6.4.2) if potassium is to be determined. Make up to the mark using distilled water.

6.5.3 Preparation of Blank Solution

Prepare appropriate blank solutions as in 6.4.5, 6.4.6 and 6.5.2 using the same reagents and same volume of distilled water instead of the sodium (or potassium) solutions or the sample.

6.5.4 Preparation of Calibration Curves

Aspirate the blank and calibration solutions and aspirate distilled water in between. Carry out the measurements using an air-acetylene (oxidizing) flame. The wavelength and working

range shall be as below:

Wavelength, nm		Working range, µg/ml	
Sodium	Potassium	Sodium	Potassium
589.0	766.5	0.15 to 0.60	0.5 to 2.0
589.6	769.9	0.5 to 2.0	1.5 to 6.0
330.2) 330.3)	404.4	100 to 400	200 to 800

NOTE — By rotation of burner on 589 nm and 589.6 nm for sodium and on 766.5 nm and 769.9 nm for potassium, measurements can be taken for 20 to 50 µg/ml. However, the working range and also the sensitivity may vary for different instrument specification.

6.5.5 Prepare calibration curve by plotting the absorbance reading against the sodium (or potassium) concentrations.

6.5.6 Aspirate the test solution and read the sodium (or potassium) concentration from the calibration curve.

6.7 Calculations

$$\text{Sodium (or Potassium), mg/l} = C \times \frac{V_1}{V}$$

where

C = sodium (or potassium) concentration from the calibration curve, mg/l,

V = volume of the original sample taken for analysis, ml, and

V_1 = volume of the volumetric flask (100 ml).

7 GRAVIMETRIC METHOD — FOR SODIUM

7.1 Principle

Sodium is precipitated as sodium zinc uranyl acetate hexahydrate, $[\text{NaC}_2\text{H}_3\text{O}_2 \cdot \text{Zn}(\text{C}_2\text{H}_3\text{O}_2)_2 \cdot 3\text{UO}_2(\text{C}_2\text{H}_3\text{O}_2)_2 \cdot 6\text{H}_2\text{O}]$ by adding a large volume of zinc uranyl acetate reagent previously saturated with the sodium salt to a small volume of the concentrated sample.

7.2 Interference

Lithium interferes by forming a slightly soluble salt with the reagent, potassium interferes if there are more than 25 mg in the 1 ml solution being tested. Organic acids, such as oxalic, citric and tartaric, interfere, as anions such as phosphate that give precipitates with the reagent. Sulphate must be absent when much potassium is present because potassium sulphate is only slightly soluble in the reagent. If the potassium and sulphate concentrations are known, the maximum possible error due to the precipitation of potassium sulphate can be calculated. Usually this error will be negligible because the potassium concentration in potable water is low and also because the calculation factor,

0.014 95, for converting mass of sodium is very small. Upon evaporation of the sample, silica may become partially dehydrated and precipitate out. Except in extreme cases, the error caused by silica will be negligible due to the following reasons:

- a) the precipitation of silica is not complete;
- b) some of the dehydrated silica adheres strongly to the glass surface of the beaker and is not transferred to the crucible to be weighed with the sodium salt;
- c) the high ratio of mass of the triple salt to that of sodium and the slight increase in the mass of precipitate due to precipitated silica has a relatively small effect on the calculated sodium concentration. A method for compensation of errors caused by precipitation of uranyl phosphate and silica is described in the procedure.

7.3 Apparatus

7.3.1 Beakers — 20-ml borosilicate.

7.3.2 Fritted Glass Crucible — 30-ml borosilicate of medium porosity or medium porosity; or porous porcelain crucibles.

7.3.3 Vacuum pump or aspirator, with manifold and individual petcocks.

7.4 Reagents

7.4.1 Zinc Uranyl Acetate Reagent

Mix 2.7 ml of glacial concentrated acetic acid with 100 ml distilled water. Add 10 g uranyl acetate dihydrate, $[\text{UO}_2(\text{C}_2\text{H}_3\text{O}_2)_2 \cdot 2\text{H}_2\text{O}]$ and 30 g of zinc acetate dihydrate $[\text{Zn}(\text{C}_2\text{H}_3\text{O}_2)_2 \cdot 2\text{H}_2\text{O}]$ and warm to dissolve. On cooling, add 2 to 3 mg of sodium chloride, let stand for 24 hours more, and filter off the precipitate of sodium zinc uranyl acetate, thus leaving the reagent saturated with the triple salt. Store in a pyrex bottle.

7.4.2 Ethyl Alcohol Wash Solution

Saturate 95 percent ethyl alcohol with pure sodium zinc uranyl acetate and decant or filter the solution just before use. Prepare the sodium zinc uranyl acetate by adding 25 ml zinc uranyl acetate reagent to 2 ml sodium chloride solution (10 mg NaCl), stir and collect the precipitate in a sintered glass crucible and wash three times with concentrated acetic acid and finally three times with diethyl ether.

7.4.3 Diethyl Ether

7.5 Procedure

If necessary, remove any suspended matter from the potable water sample by filtration. Select

a sample volume containing less than 8 mg of sodium and less than 25 mg of potassium. Pipette the clear sample into 120 or 50-ml pyrex beaker and evaporate to dryness on a steam or hot water bath. Cool the residue to room temperature, add 1.0 ml distilled water, rub with a stirring rod. If the residue fails to dissolve, add more 1.0-ml increments of distilled water to dissolve it. Ignore a feathery turbidity of calcium sulphate because of its subsequent solubility in the zinc uranyl acetate reagent.

Treat with zinc uranyl acetate reagent in the ratio of 10 ml reagent for each 10 ml increment of distilled water required to dissolve the residue. Mix, cover the beaker and let stand for 1 h. Stir periodically to prevent the formation of a supersaturated solution. Collect the precipitate under suction in a weighed medium-porosity sintered glass crucible. Substitute a porous-bottomed porcelain filtering crucible if desired. Drain the filter as dry as possible under suction. Wash the beaker, crucible, and precipitate five to eight times with 2 ml portions of zinc uranyl acetate reagent. Drain the crucible completely after the last wash to remove traces of the zinc uranyl acetate reagent. Wash five times with 2-ml portions of ethyl alcohol wash solution. Conclude the washing with three small portions of diethyl ether.

Continue suction for a few minutes until the diethyl ether is dry. Wipe the outside and inner bottom ring of the crucible with a cloth if salts have crystallized there. Transfer the crucible to the balance and weigh after 10 to 15 min and again 10 min later to check on the constancy of the mass. Return the crucible to the suction apparatus and dissolve the sodium zinc uranyl acetate by passing 100 ml warm distilled water in small portions through the filter. Dry the crucible with ethyl alcohol wash solution and diethyl ether, as previously directed, and reweigh. The difference in the mass before and after the distilled water treatment represents the mass of the sodium zinc uranyl acetate.

7.6 Calculation

$$\text{Sodium (Na), percent, mg/l} = \frac{A \times 14.95}{\text{ml of sample}}$$

where

A = triple-salt precipitate in mg.

7.7 Precision and Accuracy

A synthetic unknown sample containing sodium (19.9 mg/Na), calcium (108 mg/1 Ca), magnesium (82 mg/1 Mg), potassium (3.1 mg/1 K),

chloride (241 mg/l Cl), nitrite (0.25 mg/l N), nitrate (1.1 mg/l N), sulphate (259 mg/l SO_4) and 42.5 mg/l total alkalinity (contributed by NaHCO_3) was analyzed in four laboratories by the gravimetric method with a relative standard deviation of 11.3 percent and a relative error of 0.5 percent.

8 COLORIMETRIC METHOD — FOR POTASSIUM DETERMINATION

8.1 Principle

Potassium is determined colorimetrically by precipitating it with sodium cobalt nitrite, oxidizing the dipotassium sodium cobalt nitrate with standard potassium dichromate solution in the presence of sulphuric acid measuring the excess dichromate colorimetrically.

8.2 Interference

Ammonium ion interferes and should not be present. Silica causes no difficulty unless a silica gel is formed, either during evaporation or when the sample becomes acidic upon addition of the reagent. If this occurs, as evidenced by turbidity in the final coloured solution, filtration will be necessary. Other substances normally present in water do not interfere.

8.3 Minimum Detectable Concentration

This method can detect 0.5 mg potassium when a 15-cm light path is used. This represents a potassium concentration of 5 mg/l when a 100-ml sample is taken for analysis.

8.4 Apparatus

8.4.1 Colorimetric Equipment

One of the following is required:

- Spectrophotometer, for use at 425 nm, providing a light path of 1 cm or longer.
- Filter photometer, providing a light path of 1 cm or Nessler tubes, matches, 100-ml tall from.

8.4.2 Centrifuge

8.4.3 Centrifuge Tubes — 25 ml.

8.4.4 Small-diameter stirring rods, 2 to 3 mm, to stir the precipitate in the centrifuge tube.

8.5 Reagents

8.5.1 Nitric Acid — concentrated 1 N.

8.5.2 Trisodium Cobalt Nitrite Solution

8.5.3 Nitric Acid — 0.01 N.

Dilute 10 ml of 1 N nitric acid in 1 l with distilled water.

8.5.4 Standard Potassium Dichromate — 0.1000 N.

Dissolve 4.904 g anhydrous potassium dichromate in distilled water, and make up to 1 000 ml. This solution is stable for long period.

8.5.5 Sulphuric Acid — Concentrated.

8.5.6 Stock Potassium Solution

Dissolve 1.907 g potassium chloride dried at 110°C and dilute to 1 000 ml with deionized distilled water. 1 ml = 1.00 mg potassium.

8.5.7 Standard Potassium Solution

Dilute 1.00 ml intermediate potassium solution with deionized distilled water to 100 ml; 1.00 ml = 10.0 μg potassium. Use this solution for preparing the calibration curve in the potassium range of 0.1 to 1.0 mg/l.

8.6 Procedure

8.6.1 Sample Volume

If the sample contains from 100 to 700 mg/l potassium, use a 10-ml portion. If it contains less, concentrate a large portion by evaporation to about 5 ml, transfer to a 25 ml centrifuge tube, and make up to 10 ml.

8.6.2 Colour Production

At room temperature, add with mixing, 1 ml of 1 N nitric acid and 5 ml trisodium cobalt nitrite solution. Let stand for 2 h. In as much as the composition of the precipitate depends on the time allowed for precipitation as well as on the temperature; keep the time constants (± 15 min) for all samples. A variation of 10°C in either direction will give an error of 2 percent centrifuge for 10 minutes. Decant and wash the precipitate with 15 ml of 0.01 N nitric acid; using a small diameter stirring rod mix to ensure complete contact between precipitate and wash solution; centrifuge again, decant, and add with mixing 10 ml standard potassium dichromate solution and 5 ml concentrated sulphuric acid. Cool to room temperature. Make up to 100 ml with distilled water. Filter if the solution is turbid.

8.6.3 Preparation of Standards

Pipette 1, 2, 3, 4, 5, 6 and 7 ml standard potassium solution into 25-ml centrifuge tubes; dilute each to 10 ml and produce standards containing 1.0 to 7.0 mg potassium.

8.6.4 Colour Measurement

Measure the colour either photometrically or visually.

8.6.4.1 Photometric measurement

Use the longest light path available with the instrument to measure the absorbance or

transmittance of the sample and standards by a spectrophotometer with 2 cm light path. Prepare the calibration graph in the range of 2 to 7 mg potassium. For a filter photometer with a 15-ml light path, prepare the calibration curve in the range of 0.5 to 7 mg potassium.

8.6.4.2 Visual Colour Comparison

Compare the sample against the standards in the range from 2 to 7 mg potassium. Protect from dust and strong light to preserve stability

of the solutions for at least one week.

8.7 Calculation

$$\text{Potassium (K), mg/l, percent} = \left[\frac{\text{mg of potassium from calibration curve}}{\text{sample in ml}} \right] \times 1000$$

8.8 Precision and Accuracy

Both visual and spectrophotometric readings with a 2 cm light path can be made with an estimated accuracy of ± 0.5 mg potassium.

ANNEX A

(Foreword)

COMMITTEE COMPOSITION

Environmental Protection Sectional Committee, CHD 012

<i>Chairman</i>	<i>Representing</i>
SHRI N. S. TIWANA	Central Pollution Control Board, Delhi
<i>Members</i>	
DR K. R. RANGANATHAN (Alternate to Shri N. S. Tiwana)	
ADVISER (PHE)	Ministry of Rural Development
ADDL ADVISER (PHE) (Alternate)	
DR A. L. AGGARWAL	National Environmental Engineering Research Institute (CSIR), Nagpur
DR T. CHAKRABARTI (Alternate)	
DR A. ALAM	Indian Council of Agricultural Research, New Delhi
SHRI S. C. AHLUWALIA	National Council for Cement & Building Materials, New Delhi
SHRI A. D. AGNIHOTRI (Alternate)	
DR S. K. AWASTHI	Indian Petrochemicals Corporation Ltd, Vadodara
SHRI D. C. BHATT (Alternate)	
SHRI R. K. BANERJEE	Shriram Institute for Industrial Research, Delhi
SHRI P. K. MAIR (Alternate)	
SHRI B. BASU	National Thermal Power Corporation Ltd, New Delhi
DR S. MUKHERJEE (Alternate)	
SHRI V. S. BHATNAGAR	Central Scientific Instruments Organization (CSIR), Chandigarh
DR M. S. N. SRINIVAS (Alternate)	
SHRI S. CHAKRAVORTI	Directorate General Factory Advice Service & Labour Institutes, Bombay
DR M. H. FULEKAR (Alternate)	
SHRI M. GOPALKRISHNA	National Test House, Calcutta
DR S. RAHUT (Alternate)	
SHRI S. GOURISHANKAR	Bharat Heavy Electricals Ltd, Hyderabad
SHRI S. BALAGURUNATHAN (Alternate)	
DR HARISH CHANDRA	Industrial Toxicology Research Centre (CSIR), Lucknow
SHRI B. K. JAIN	The Fertilizer Association of India, New Delhi
DR (MS) B. SWAMINATHAN (Alternate)	
SHRI S. K. JAIN	Indian Oil Corporation Ltd (R&D Centre), Faridabad
SHRI R. K. MALHOTRA (Alternate)	
SHRI P. M. KULKARNI	Thermax Ltd, Pune
DR A. K. WAGLE (Alternate)	
SHRI A. LAHIRI	Hindustan Lever Ltd, Bombay
SHRI B. B. DAVE (Alternate)	
SHRI M. K. LODH	Projects & Development India Ltd, Sindri
SHRI D. N. P. SINGH (Alternate)	
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