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

भाग 52 क्रोमियम

(पहला पुनरीक्षण)

Indian Standard

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

PART 52 CHROMIUM

(First Revision)

ICS 13.060.50

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

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Environment Protection and Waste Management Sectional Committee, CHD 32

FOREWORD

This Indian Standard (Part 52) (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.

The wide use of chromium (Cr) by modern industries has resulted in a large quantity of this element being discharged into the environment via air emission, waste water or land disposal. One of the important sources of the Cr in the waste water is due to the use of chromates in cooling water for corrosion control. Cr may exist in water in both hexavalent and trivalent states. Trivalent Cr has a lower toxicity than that in the hexavalent state.

Important sources which add chromium (Cr) to the environment are : ferro-chromium production units, refining of ores, chemical industries and the combustion fossil fuels. In rural areas chromium content in ambient air is usually less than 0.1 µg/cubic metre while in industrial areas it may range from 0.01-0.03 µg/cubic metres. Particulates ejected from coal fired power generation plants may contain 2.3-32 ppm of chromium. Waste waters from tanneries may contain 10-50 ppm, textile wastes up to 32 ppm while spent chrome liquors may contain up to 4 500 mg/litre of Cr. Only trivalent and hexavalent forms of Cr are of biological significance. Trivalent Cr is the most common form of the metal in nature and it is in this state the metal almost always occurs in a living system. Hexavalent Cr is capable of crossing the cell membrane and is rapidly reduced to trivalent state inside a living cell.

Small quantities of trivalent Cr are essential to carbohydrate metabolism in mammals while it is also a co-factor for action of insulin. A little amount of Cr is helpful in improving glucose tolerance in diabetic patients, weak and old individuals. Most of the mammals can tolerate almost a hundred times more Cr in trivalent state than their usual body burden without any toxic effects. Acute Cr toxicity causes serious renal tubular necrosis. Exposure to hexavalent Cr has been found to cause dermatities, allergic skin reactions, chronic ulceration and injury to nasal septum, gastrointestinal ulcers etc. Chronic Cr toxicity has been associated with incidences of cancers of respiratory track in occupationally involved workers. Both forms of Cr (hexavalent and trivalent) are considered equally potent carcinogenic agent.

In view of the paradoxical role of the Cr, as an essential micronutrient in human and animal nutrition at low concentrations, and a known carcinogen at the elevated levels, there is a growing concern about the fate and effects of Cr in the environment.

As per IS 10500 : 1991 'Drinking water—Specification (*first revision*)' the permissible limit for hexavalent Cr in drinking water is 0.05 mg/l , *Max*. Above this limit, Cr can be carcinogenic.

The technical committee responsible for the formulation of IS 3025 : 1964 had decided to revise the standard and publish it as separate parts. This standard supersedes **38** 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.

The Atomic Absorption Method (Direct) specified in this standard is technically equivalent to the Method B of ISO 9174 : 1990 'Water quality — Determination of total chromium atomic absorption spectrophotometric method'. The diphenylcarbazide method specified in this standard is technically equivalent to the method given in ISO 11083 : 1994 'Water quality — Determination of chromium (vi) — Spectrometric method using 1,5 diphenylcarbazide'.

The composition of the Committee responsible for formulating 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*)'.

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Indian Standard

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

PART 52 CHROMIUM

(First Revision)

1 SCOPE

1.1 This standard prescribes two methods for the determination of chromium:

a) Diphenylcarbazide method, and
b) Atomic absorption method.

1.2 Depending upon the concentration range and interference levels, choice of the method is made.

2 REFERENCES

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

IS No.	*Title*
3025 (Part 1) : 1987	Methods of sampling and test (physical and chemical) for water and wastewater : Part 1 Sampling (*first revision*)
7022 (Part 1) : 1973	Glossary of terms relating to water, sewage and industrial effluents, Part 1
7022 (Part 2) : 1979	Glossary of terms relating to water, sewage and industrial effluents, Part 2

3 TERMINOLOGY

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

4 SAMPLING AND STORAGE

The sampling shall be done as prescribed in IS 3025 (Part 1). The sampling bottles shall be cleaned thoroughly with dilute nitric acid (6N), 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 the determination of dissolved chromium content filtration through 0.45 mm membrane filter, at the time of sampling, is required. The analysis of such samples is to be carried out within 24 hours of sampling. For preservation, the samples should be acidified with concentrated nitric acid (2 ml of conc nitric acid in 1 litre sample, just to bring down the *p*H below 2). The acidified samples can be stored for a few days (up to 5 days) in a refrigerator. In case, when dissolved hexavalent chromium or total hexavalent chromium is to be estimated adjust the *p*H of the sample to 8 or above with 1N NaOH and refrigerate

5 PURITY OF THE REAGENTS

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

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

6 DIPHENYLCARBAZIDE METHOD

6.1 Principle

This procedure measures only hexavalent chromium(Cr^{6+}). Therefore, to determine the total chromium, convert all the chromium to the hexavalent state by oxidation with potassium permanganate. The hexavalent chromium is determined spectrophotometrically by reaction with diphenylcarbazide in acid solution. A red-violet colour of unknown composition is produced. The coloured complex obeys Beer's law and is suitable for spectrophotometric measurements at 540 nm. This method is applicable in the range of 30 to 20 000 µg/l of chromium.

6.2 Interferences

The reaction with diphenylcarbazide is nearly specific for chromium. Hexavalent molybdenum and mercury salts will react to form colour with the reagent, but the intensities are much lower than that for chromium at the specified *p*H. Concentrations as high as 200 mg/l of Mo or Hg can be tolerated. Pentavalent vanadium interferes, strongly but concentrations up to 10 times that of chromium will not cause trouble. Potential interference from permanganate is eliminated by prior reduction with sodium azide. Iron in concentrations greater than 1 mg/l may produce a yellow colour, but the colour is not strong and no difficulty is encountered normally if the absorbance is measured spectrophotometrically at 540 nm.

NOTE — Interfering amounts of V and Fe can be removed by extraction of cufferrates of these metals into chloroform. Do not use it unless necessary because residual cufferron and chloroform in aqueous solution complicate later oxidation. Follow extractions by addition and reaction with acid fuming to decompose these compounds.

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6.3 Apparatus

6.3.1 Spectrophotometer, for use at 540 nm, with a light path of 1 cm.

6.3.2 p*H Meter*

6.3.3 *Standard Volumetric Glasswares*

NOTE — Thoroughly cleaned glassware with nitric acid or hydrochloric acid to remove chromium traces. Do not use glassware previously treated with chromic acid. New and unscratched glassware will minimize chromium absorption on glassware during oxidation procedure.

6.4 Reagents

6.4.1 *Stock Chromium Solution*

Dissolve 141.4 mg of $K_2Cr_2O_7$ in water and dilute to 100 ml (1.0 ml = 500 µg of Cr).

6.4.2 *Standard Chromium Solution*

Dilute 1.0 ml of stock chromium solution to 100 ml; (1.0 ml = 5.0 µg of Cr).

6.4.3 *Nitric Acid* — concentrated (16 N).

6.4.4 *Sulphuric Acid* — concentrated (36 N); 1:1; 6 N and 0.2 N.

6.4.5 *Phosphoric Acid* — concentrated (41 N).

6.4.6 *Methyl Orange Indicator Solution*

Dissolve 50 mg methyl orange in 100 ml water.

6.4.7 *Ammonium Hydroxide* — concentrated (14 N).

6.4.8 *Potassium Permanganate Solution*

Dissolve 4 g of $KMnO_4$ in 100 ml water.

6.4.9 *Sodium Azide Solution*

Dissolve 0.5 g of sodium azide (NaN_3) in 100 ml water.

6.4.10 *Diphenylcarbazide Solution*

Dissolve 250 mg of 1,5-diphenylcarbazide (1,5-diphenylcarbohydrazide) in 50 ml acetone. Store in an amber coloured bottle. Discard when the solution becomes discoloured.

6.4.11 *Acetone*

6.5 Procedure

6.5.1 *Preparation of Calibration Curve*

Pipette out measured volumes of standard chromium solution ranging from 2 to 20 ml (to give standards for 10-100 µg of Cr), into 100 ml beakers. Make up the volume to about 50 ml with water. Use 0.2 N H_2SO_4 and a *p*H meter to adjust the *p*H of each solution to 1.0 ± 0.3. Transfer quantitatively each of these solutions into 100 ml volumetric flasks and add 2.0 ml of diphenyl carbazide solution. Dilute to 100 ml with water, mix and let these stand for 5 to 10 min for full colour development. Meanwhile, prepare a reagent blank in an identical manner using 10 ml of water. Measure the absorbance of the standard solutions at 540 nm, using reagent blank as reference solution. Construct a calibration curve by plotting absorbance values against micrograms of chromium in 100 ml of the final volume.

6.5.2 *Determination of Hexavalent Chromium (Cr^{6+})*

Pipette out a portion of filtered sample (filtered through 0.45 µm membrane filter), containing 10 to 100 µg of Cr into a 100 ml beaker. Make up the volume to about 50 ml with water. Adjust *p*H of this solution to 1.0 ± 0.3 using 0.2N H_2SO_4, and a *p*H meter. Transfer quantitatively in to a 100 ml volumetric flask, add 2.0 ml of diphenylcarbazide solution. Dilute to 100 ml with water, mix well and allow to stand for 5 to 10 min. Measure absorbance at 540 nm, using reagent blank as reference solution (*see* **5.5.1**). From the absorbance data, determine the micrograms of chromium present in 100 ml of the final solution using the calibration curve. From the absorbance data, determine the micrograms of Cr present in 100 ml of the final solution using the calibration curve.

6.5.3 *Determination of Total Chromium*

If the sample contains organic matter and considerable suspended or colloidal particles are present, digest the sample with HNO_3 and H_2SO_4 and then take it for analysis. (In the absence of organic matter or suspended matter, filter the sample and determine the total chromium as described in **6.5.5**.)

6.5.4 *Digestion Process*

Transfer a suitable volume of homogenized sample to a beaker (Sample volume depends upon the expected Cr content. If the expected Cr concentration is of the order of 1 mg/l, the sample volume will be about 800 ml. If it is between 10 to 100 mg/l, about 100 ml of the sample will be sufficient). Add 5 ml of conc HNO_3 and a few porcelain pieces or glass beads. Heat to boil the solution, and concentrate carefully on a hot plate to a lowest possible volume. If required, cool the solution and transfer quantitatively, into a beaker of smaller size (say 150 ml beaker). Add 5 ml of conc HNO_3 and 10 ml of conc H_2SO_4. Heat up to the evolution of dense white fumes of SO_3. If the solution is not clear, add 5 ml of HNO_3 and re-heat until a clear solution (no evolution of brown fumes) results. Cool and transfer quantitatively into a 100 ml volumetric flask. Dilute up to the mark with water, and mix well. Use this solution for the determination of total chromium, as described in **6.5.5**.

6.5.5 *Oxidation and Determination of Chromium*

Pipette out a portion of filtered sample or solution obtained from acid digestion, containing 10 to

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100 µg of Cr, into a 150 ml beaker. Add water to make up the volume to about 50 ml. Add a few drops of methyl orange indicator, then add concentrated NH_4OH, until the solution just begins to turn yellow. Now add 1:1 H_2SO_4 dropwise, until the solution just begins to turn pink, then 1 ml in excess. Add a few porcelain pieces or glass beads, and heat to boiling. Add $KMnO_4$ dropwise till a permanent pink colour persists. Boil for a few more minutes. Add 1 ml of NaN_3 solution and continue to boil gently. If the red colour does not fade completely, after boiling for about 30 s, add one more ml of NaN_3 solution. Continue boiling, for 1 min after, the colour has faded completely, then cool. Add 5 drops of conc H_3PO_4. Adjust the *p*H to 1.0 ± 0.3 using 0.2N H_2SO_4, and transfer quantitatively in to a 100 ml volumetric flask. Add 2.0 ml of diphenylcarbazide solution and dilute to mark with water. Mix well and allow to stand for 5 to 10 min. Meanwhile, prepare a reagent blank exactly in an identical manner (using water instead of the sample solution). Measure the absorption of the sample at 540 nm, using reagent blank as reference. From the absorbance data, determine the micrograms of chromium present in 100 ml of the final solution using the calibration curve.

6.6 Calculation

6.6.1 *Soluble Hexavalent or Total Chromium (Direct Determination Without Digestion Step)*

$$\text{mg Cr/l} = \frac{\mu\text{g of Cr (in 100 ml of the final solution)}}{V}$$

where

V = volume in ml, of the sample used.

6.6.2 Total chromium (when digestion is carried out)

$$\text{mg Cr/l} = \frac{\mu\text{g of Cr (in 100 ml of the final solution)}}{V_1 \times V_2} \times 100$$

where

V_1 = volume in ml of the sample used, and
V_2 = total volume in ml of the digested solution used for the Cr determination.

6.6.3 *Precision and Accuracy*

The relative standard deviation reported in the literature for Cr in the 100 µg/l concentration range is 16.3 percent.

7 ATOMIC ABSORPTION METHOD (DIRECT)

7.1 Principle

The chromium content of the sample is determined by atomic absorption spectrophotometry. For dissolved chromium the filtered sample is directly aspirated to the atomizer. For total recoverable chromium, HNO_3-H_2SO_4 digestion is to be carried out prior to aspiration of the sample. This method is applicable in the range from 0.2 to 10 mg/l of chromium. However, the concentration range will vary with the sensitivity of the instrument used.

7.2 Interferences

Chromium absorption is suppressed in the air acetylene flame by the presence of iron and nickel. If the analysis is performed in lean flame the interference can be lessened. The addition of 2 percent NH_4Cl to sample and standard solution controls the interference caused by iron. An excess of phosphate will depress the chromium response and can be overcome by the addition of calcium.

7.3 Apparatus

Atomic absorption spectrophotometer with air-acetylene flame. Hollow-cathode lamp or electrodeless discharge lamp for use at 357.9 nm.

CAUTION — Acetylene gas is an explosive hazard in the laboratory. Follow manufacturer's instructions. Do not allow gas contact with copper, brass with more than 65 percent copper, silver or liquid mercury.

7.4 Reagents

7.4.1 *Hydrochloric Acid* — concentrated (11 N) and 1:1.

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

7.4.3 *Sulphuric Acid* — concentrated (36 N).

7.4.4 *Hydrogen Peroxide* — (30 percent *m/m*).

7.4.5 *Stock Chromium Solution*

Dissolve 0.192 3 g of CrO_3 in water. When solution is complete, acidify with 10 ml conc HNO_3 and dilute to 1 000 ml with water in a volumetric flask (1.0 ml = 100 µg of Cr).

7.5 Procedure

7.5.1 *Calibration*

Prepare a reagent blank and sufficient standards containing 0 to 60 mg/l of chromium by diluting suitable volumes of standard chromium solution with nitric acid (1 : 499) to 100 ml in volumetric flasks. Add 1 ml of 30 percent H_2O_2 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 357.9 nm.

7.5.2 *Determination of Chromium*

For the determination of dissolved chromium content, filtration through 0.45 µm membrane filter, at the time of sampling, is required. For total recoverable chromium,

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HNO_3-H_2SO_4 digestion is to be carried out (*see* **6.5.4**). Pipette out a suitable volume of the sample (or solution obtained after digestion) containing 50 to 60 µg of chromium in a 100 ml volumetric flask. Add 0.5 ml of conc nitric acid, and make up the volume upto the mark with water. Add 1ml 30 percent H_2O_2 solution to this. Prepare a reagent blank with 100 ml water, 0.5 ml conc nitric acid and 1ml H_2O_2. Rinse the nebulizer by aspirating water containing 1.5 ml conc HNO_3/l. Aspirate the reagent blank and carry out zero adjustment. Aspirate the sample solution and measure the absorbance at 357.9 nm. From the absorbance data, determine the micrograms of Cr present in 100 ml of the final solution using the calibration data.

7.6 Calculation

7.6.1 *Soluble Chromium (Direct Determination Without Digestion Step)*

$$\text{mg Cr/l} = \frac{\mu\text{g of Cr(in 100 ml of the final solution)}}{V}$$

where

V = volume in ml, of the sample used.

7.6.2 Total chromium (when the digestion is carried out)

$$\text{mg Cr/l} = \frac{\mu\text{g of Cr(in 100 ml of the final solution)}}{V_1 \times V_2} \times 100$$

where

V_1 = volume in ml of the sample used, and

V_2 = total volume in ml of the digested solution used for Cr determination.

7.6.3 *Precision and Accuracy*

The relative standard deviation reported in the literature for Cr in the 3.0 mg/l concentration range is 10.0 percent.

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ANNEX A

(Foreword)

COMMITTEE COMPOSITION

Environment Protection and Waste Management Sectional Committee, CHD 32

Organization	*Representative(s)*
Central Pollution Control Board, Delhi	Shri Dilip Biswas (***Chairman***)
Automotive Research Association of India, Pune	Representative
Bhabha Atomic Research Centre, Mumbai	Dr S. Sadashivan Dr T. N. Mahadeven (*Alternate*)
Bharat Heavy Electricals Limited, Haridwar	Dr N. G. Shrivastava
Cement Manufacturers' Association, New Delhi	Dr K. C. Narang
Central Fuel Research Institute, Dhanbad	Dr Gulab Singh Dr L. C. Rane (*Alternate*)
Central Leather Research Institute, Chennai	Dr S. Rajamani
Central Mechanical Engineering Research Institute, Durgapur	Representative
Central Mining Research Institute, Dhanbad	Dr B. K. Tewary
Central Pollution Control Board, New Delhi	Dr S. D. Makhijani Dr C. S. Sharma (*Alternate*)
Confederation of Indian Industries (CII), New Delhi	Shri A. K. Ghose Shri R. P. Sharma (*Alternate*)
Department of Industrial Policy & Promotion, New Delhi	Representative
Department of Science & Technology (TIFAC), New Delhi	Representative
Directorate General Factory Advice Service and Labour Institute, Mumbai	Shri S. S. Gautam Shri M. R. Rajput (*Alternate*)
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Industrial Toxicology Research Centre, Lucknow	Representative
Ministry of Defencce (R&D), DRDO, New Delhi	Representative
Ministry of Environment and Forests, New Delhi	Representative
Ministry of Non-Conventional Energy Research, New Delhi	Shri Anil Kumar Varshney
Ministry of Urban Development, New Delhi	Representative
Municipal Corporation of Delhi, New Delhi	Representative
Municipal Corporation of Greater Mumbai, Mumbai	Representative
National Institute of Occupational Health (ICMR), Ahmedabad	Dr D. J. Parikh Dr T. S. Patel (*Alternate*)

(*Continued on page* 6)

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(*Continued from page* 5)

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National Organic Chemical Industries Limited, Mumbai	**SHRI B. K. CHOPRA** **SHRI B. V. BAPAT (*Alternate*)**
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