भारतीय मानक
Indian Standard

IS 3025 (Part 37) : 2022

जल एवं अपशिष्ट जल के नमूने लेने तथा परीक्षण (भौतिक एवं रसायन) की पद्धतियाँ

भाग 37 आर्सेनिक

(दूसरा पुनरीक्षण)

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

Part 37 Arsenic

(Second Revision)

ICS 13.060.50

भारतीय मानक ब्यूरो
BUREAU OF INDIAN STANDARDS
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July 2022 **Price Group 4**

FOREWORD

'This Indian Standard (Part 37) (Second Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Water Quality Sectional Committee had been approved by the Chemical Division Council'.

Arsenic is a naturally occurring component of the earth crust and is distributed throughout the environment like air, water and land. Arsenic occurs in both organic and inorganic form in water but inorganic form of arsenic is comparatively more toxic. Arsenic may occur in water as a result of mineral dissolution, industrial discharges or application of insecticides.

The Technical Committee responsible for formulation of IS 3025: 1964 'Methods of sampling and test (physical and chemical) for water used in industry' and IS 2488 (Part 2): 1968 'Methods of sampling and test for industrial effluents' decided to revise the standard and publish it in separate parts. This standard superseded **7** of IS 2488 (Part 2): 1968 and **40** of IS 3025 : 1964 and was one among the different parts published under IS 3025 series of standards.

This standard was first revised in 1988. In the first revision of the standard, the following three methods for determination of arsenic, were given:

a) Atomic absorption method;

b) Mercuric bromide stain method; and

c) Silver diethyl dithiocarbamate method.

In this revision the following changes have been made:

a) Mercuric bromide method has been removed;

b) Inductively coupled plasma spectroscopy has been added; and

c) Safety precautions have been added.

In the preparation of this standard, considerable assistance has been derived from the method no 4500 P of — Standard Methods for the Examination of Water and Wastewater, published by the American Public Health Association, Washington, USA, 23rd Edition, 2017.

The composition of committee responsible for the formulation of this standard is given at Annex A.

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

Indian Standard

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

PART 37 ARSENIC

(Second Revision)

1 SCOPE

This standard (Part 37) prescribes three methods for determination of arsenic namely:

a) Atomic absorption method;

b) Silver diethyl dithiocarbamate method; and

c) Inductively coupled plasma spectroscopy method.

2 REFERENCES

The following standards contain provisions which through reference in this text constitute provisions of this standard. At the time of publications, 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 indicated below:

IS No.	*Title*
1070 : 1992	Reagent grade water — Specification (*third revision*)
3025 (Part 2) : 2019	Methods of sampling and test (physical and chemical) for water and wastewater: Part 2 Determination of selected elements by inductively coupled plasma optical emission spectrometry (ICP-OES)
3025 (Part 65) : 2022	Methods of sampling and test (physical and chemical) for water and wastewater: Part 65 Application of inductively coupled plasma mass spectrometry (ICP-MS) — Determination of selected elements including uranium isotopes (*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, the definitions given in IS 7022 (Part 1) and IS 7022 (Part 2) shall apply.

4 ATOMIC ABSORPTION METHOD

4.1 Scope and Application

Arsenic is converted into its volatile hydride by sodium borohydride reagent in acid solution. The hydride is purged continuously by argon or nitrogen into an appropriate atomizer of an atomic absorption spectrometer and converted to gas phase atoms. The sodium borohydride reducing agent, by rapid generation of elemental hydrides in an appropriate reaction cell, minimizes dilution of hydrides by the carrier gas and provides rapid, sensitive determination.

4.2 Interferences

Interferences are minimized because arsenic hydride is removed from solution containing most potential interfering substances. Slight response variations occur when acid matrices are varied. Treating the samples and standard in the same manner control this variations. Low concentrations of noble metals, copper, lead, nickel at or greater than 1.0 mg/l and hydride forming elements like bismuth, antimony, tin and telurium at concentrations between 0.1 and 1.0 mg/l may suppress the response of arsenic.

4.3 Apparatus

4.3.1 *Atomic Absorption Spectrometer*

Equipped with gas flow meters for argon (or nitrogen) and hydrogen, arsenic electrodeless discharge lamps with power supply background correction at measurement wavelengths and appropriate strip chart recorder.

4.3.2 *Atomizer*

Use one of the following:

a) Boiling-type burner head for argon (or nitrogen) air entrained-hydrogen flame;
b) Cylindrical quartz cell, 10 to 20 cm long electrically heated by external nlchrome wire to 800-900°C; and
c) Cylindrical quartz cell with internal fuel rich hydrogen-oxygen flame. The sensitivity of quartz cells deteriorates over several months of use. It may be restored by treatment with 40 percent hydrofluoric acid.

4.3.3 *Reaction Ceil for producing Arsenic Hydride*

Any commercially available system if it utilizes liquid sodium borohydride reagent, accepts samples digested in accordance with **4.5.3** to **4.5.5**, accepts 4.0 to 6.0 N hydrochloric acid, and is efficiently and precisely stirred by the purging gas and/or a magnetic stirrer.

4.3.3.1 *Eye dropper of syringe*

Capable of delivering 0.5 to 3.0 ml sodium borohydride reagent. Exact and reproducible addition is required so that production of hydrogen gas does not vary significantly between determinations.

4.4 Reagents

4.4.1 *Sodium Borohydride Reagent*

Dissolve 8 g sodium borohydride in 200 ml of 0.1 N sodium hydroxide solution. Prepare fresh daily.

4.4.2 *Sodium Iodide Pre-reductant Solution*

Dissolve 50 g of sodium iodide in 500 ml water. Prepare fresh daily.

4.4.3 *Sulphuric Acid,* 18N.

4.4.3.1 *Sulphuric Acid,* 2.5N.

4.4.4 *Potassium Persulphate*, 5.0 percent solution.

Dissolve 25.0 g of potassium persulphate in water and dilute to 500 ml. Store in glass and referigerate. Prepare weekly.

4.4.5 *Nitric Acid*, concentrated.

4.4.6 *Perchloric Acid*, concentrated.

4.4.7 *Hydrochloric Acid*, concentrated.

4.4.8 *Argon or Nitrogen,* commercial grade.

4.4.9 *Arsenic (III) Solutions*

4.4.9.1 *Stock arsenic (III) solution*

Dissolve 1.320 g arsenic trioxide in water containing 4 g of sodiumhydroxide. Dilute to 1 l [1.0 ml = 1.00 mg arsenic (III)].

4.4.9.2 *Intermediate arsenic (III) solution*

Dilute 10 ml arsenic stock solution lo 1 000 ml with water containing 5 ml concentrated hydrochloric acid. [1.00 ml = 10.0 µg arsenic (III)].

4.4.9.3 *Standard arsenic (III) solutions*

Dilute 10 ml of intermediate arsenic (III) solution to 1 000 ml with water containing the same concentration of acid used for sample preparation. [1.00 ml = 0·100 µg arsenic (III)], Prepare diluted solutions daily.

4.4.10 *Arsenic (V) Solutions*

4.4.10.1 *Stock arsenic (V) solution*

Dissolve 1.534 g arsenic pentaoxide in distilled water containing 4 g of sodium hydroxide. Dilute to 1 l [1.00 ml = 1.00 mg arsenic (V)].

4.4.10.2 *Intermediate arsenic (V) solution* *See* **4.4.9.2**. [1.0 ml = 10.0 µg arsenic (V)].

4.4.10.3 *Standard arsenic (V) solution* *See* **4.4.9.3**. [1.0 ml = 0.100 µg arsenic (V)].

4.4.11 *Organic Arsenic Solutions*

4.4.11.1 *Stock organic arsenic solution*

Dissolve 1.842 g dimethyl arsenic acid (cacodylic acid) in water containing 4 g of sodium hydroxide. Dilute to 1 l. (1.0 ml = 1.00 mg arsenic).

4.4.11.2 *Intermediate organic arsenic solution*

Prepare as given in **4.4.9.2**. (1.0 ml = 10.0 µg arsenic).

4.4.11.3 *Standard organic arsenic solution*

Prepare as given in **4.4.9.3**. (1.00 ml = 0.100 µg arsenic).

4.5 Procedure

4.5.1 *Setting up of Apparatus*

The setup is as given in Fig. 1 or according to manufacturer's instructions. Connect inlet of reaction cell with auxilary purging gas by flow meter, If a drying cell between the reaction cell and atomizer is necessary, use only anhydrous calcium chloride but not calcium sulphate. Before using the hydride generation/analysis, system, optimize operating parameters. Aspirate aqueous solutions of arsenic directly into the flame to facilitate atomizer alignment. Align quartz automizers

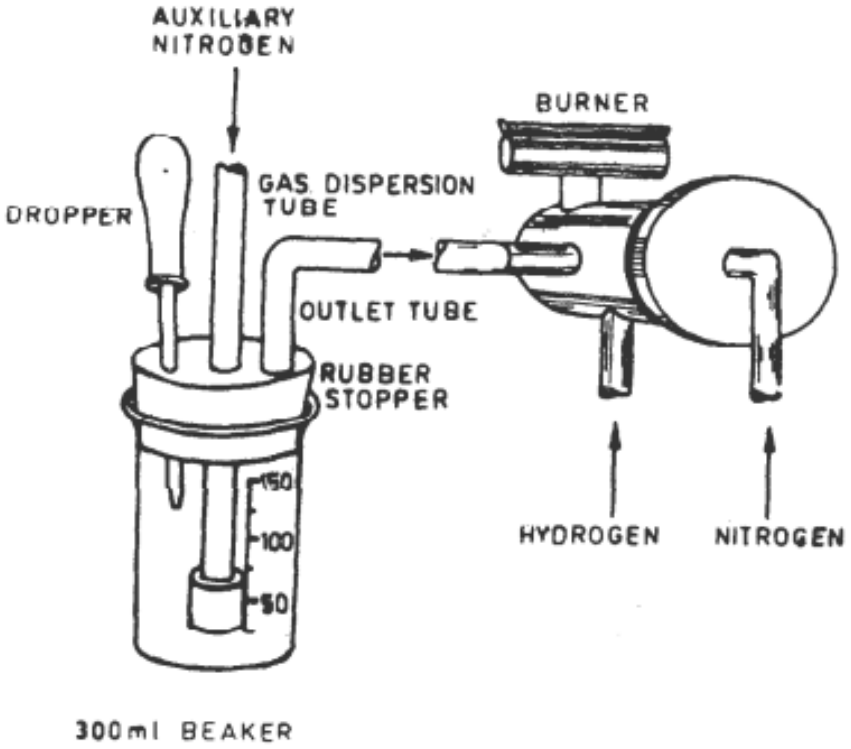

FIG. 1 REACTION CELL FOR PRODUCING ARSENIC ANHYDRIDE

for maximum absorbance. Establish purging gas flow concentration and rate of addition of sodium borohydride reagent solution volume and rate of the stirring for optimum instrument response. If quartz atomizer is used, optimize cell temperature. The recommended wavelength is 193.7 nm for arsenic.

4.5.2 *Calibration*

Transfer 0.00, 1.00, 2.00, 5.00, 10.00, 15.00 and 20.00 ml standard solutions of arsenic (III) to 100.0 ml volumetric flasks and make up to mark with water containing same acid concentration used for sample preservation. This yields standard solutions of 0.0, 1.0, 2.0, 5.0, 10.0 and 20.0 µg arsenic.

4.5.3 *Preparation ion of Samples and Standards for Total Recoverable Arsenic*

Add 50.0 ml sample or arsenic (III) standard to 200.0 ml beaker. Add 7.0 ml of 18.0 N sulphuric acid and 5.0 ml concentrated nitric acid. Add a small boiling chip of glass beads. Evaporate to sulphur trioxide fumes. Maintain oxidizing conditions at all times by adding small amounts of nitric acid. Maintain an excess of nitric acid until all organic matter is destroyed. Complete digestion usually as indicated by a light coloured solution. Cool slightly, add 25 ml water and 1 ml concentrated perchloric acid, and evaporate to fumes of sulphur trioxide to expel oxides of nitrogen. After final evaporation of sulphur trioxide fume, dilute to 50 ml.

4.5.4 *Preparation of Samples and Standards for Total Arsenic*

Add 50 ml sample or standard to 200 ml beaker. Add 1 ml of 2.50 N sulphuric acid and 5.0 ml of 5.0 percent potassium persulphate. Boil gently on a preheated hot plate for about 30 min or until the final volume is reduced to 10 ml. Do not let sample go to dryness. After manual digestion, dilute to 50 ml.

4.5.5 *Determination*

To 50 ml digested standard or sample in a 200 ml beaker, add 5 ml concentrated hydrochloric acid and mix. Add 5 ml of sodium iodide pre-reductant solution, mix and wait at least 30 min. Attach one beaker at a time to the rubber stopper containing the gas dispersion tube for the purging of gas, sodium borohydride reagent inlet and the outlet to the atomizer. Turn on strip chart recorder and wait until the base line is established by the purging gas and all is expelled from reaction cell. Add 0.5 ml of sodium borohydride reagent. After the instrument absorbance has reached a maximum and returned to the base line, remove beaker, rinse dispersion tube with water and proceed to next sample or standard. Periodically compare arsenic (III) and arsenic (V) curves for response consistency. Check for presence of chemical interferences that supress instrument response for arsenic by treating a digested sample with 10.0 µg/l arsenic (III) or arsenic (V) as appropriate, Average recoveries should be not less than 90 percent.

4.6 Calculation

Construct a standard curve by plotting peak heights of standards versus concentration of standards. Measure peak heights of samples and read concentrations from the curve. If sample was diluted before digestion, apply an appropriate factor.

5 SILVER DIETHYL DITHIOCARBAMATE METHOD

5.1 Scope and Application

Arsenite that contains trivalent arsenic is reduced by using aqueous sodium bronhydride solution to arsine in an aqueous medium of *p*H 6. However, arsenate, methylarsonic acid and dimethylarsonic acid are not reduced under these conditions and thus the generated arsine is swept by a stream of oxygen free nitrogen from a reduction vessel via a scrubber having a glass wool or cotton impregnated by using lead acetate solution into an absorber tube which contains silver dithiocarbamate and morpholine solubilized in chloroform. Intensity of colour developed take place at 520 nm. In order to determine the total inorganic arsenic in the absence of methylarsenic compounds, a portion of the sample is reduced to *p*H of 1. Alternatively, arsenate can be measured in the sample from where arsenite has been removed by reducing it to arsine gas at a *p*H of 6. Acidify the sample by using HCl and to this another portion of sodium borohydride is added. The arsine that is formed from arsenate is collected in the freshly prepared absorber solution.

5.2 Interference

Certain metals like chromium, cobalt, copper, mercury nickel, potassium and silver interfere in the generation of arsine. The concentration of these metals normally present in water and wastewaters do not interfere significantly. Antimony salt interferes with colour developments.

5.2.1 The minimum detectable quantity is 1 µg of arsenic.

5.3 Apparatus

5.3.1 *Arsine Generator and Absorption Tube* (*see* Fig. 2).

The setup is as given in Fig. 2. Three necked round bottom flask (200 ml) having a sidearm is used

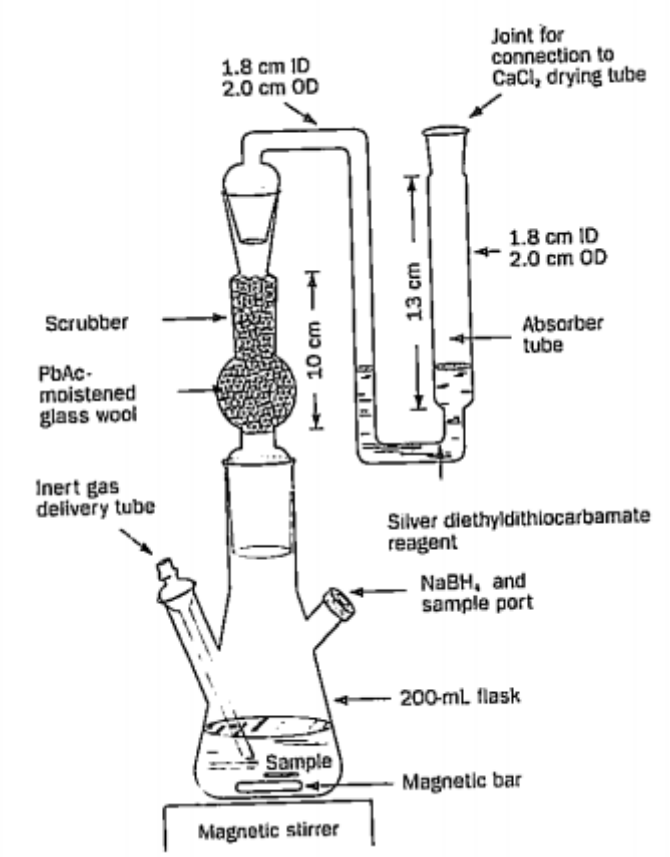

FIG. 2 ARSINE GENERATOR AND ARSINE ASSEMBLY

through which the inert gas delivery tube is inserted that reaches almost to the bottom of the flask. Use a 24/40 female ground glass joint to carry the scrubber and a second side arm is used which is closed by a rubber septum or preferably by a screw cap with a hole in its top so as to insert a TFE-faced silicone septum. Place a magnetic stirring bar in the flask and fit the absorber tube to the scrubber after filling it with silver diethyldithiocarbamate solution. Stoppers made of rubber or cork are not preferred as they may absorb arsine. The glass equipment is cleaned with nitric acid.

5.3.2 Photometric Equipment that consists of:

(a) *Spectrophotometer*, for use at 520 nm with 1 cm cells,

(b) Filter photometer with a green filter with a maximum transmittance in the range of 500-540 nm, and

(c) Cells for spectrophotometer or filter photometer having a path length of 1 cm. They should be clean, dry and should be equipped with tightly fitted cover.

5.4 Reagents

5.4.1 *Hydrochloric Acid*, 2 M.

Take 165 ml of concentrated hydrochloric acid and dissolve it in 1 000 ml of water.

5.4.2 *Lead Acetate Solution*

Dissolve 10 g of lead acetate [$Pb(C_2H_3O_2)_2.3H_2O$)] in 100 ml distilled water.

5.4.3 *Silver Diethyl Dithiocarbamate Solution*

Take 1 ml of morpholine (CAUTION-Corrosive-avoid contact with skin) and dissolve it in 70 ml of chloroform. Further add 0.30 g of silver diethyldithiocarbamate. Shake it in a stoppered flask till the maximum amount of it is dissolved. Dilute it to 100 ml with chloroform. Further, filter and store it in a tightly closed brown bottle in a refrigerator.

5.4.4 *Reagent Water,* (*see* IS 1070).

5.4.5 *Acetate Buffer of p*H 5.5

Mix about 428 ml of 0.2 M sodium acetate and 72 ml of acetic acid.

5.4.6 *Sodium Acetate of Concentration,* 0.2 M.

Take 16.46 g of anhydrous sodium acetate or take 27.36 g of sodium acetate trihydrate in water and dilute the same to 1 l.

5.4.7 *Acetic Acid,* 0.2 M.

Dissolve about 11.5 ml of glacial acetic acid in water and dilute the same to 1 000 ml.

5.4.8 *Sodium Borohydride Solution,* 1 percent.

Dissolve about 0.4 g of sodium hydroxide (4 pellets) in about 400 ml water. To this, add sodium borohydride and shake to dissolve and mix. Prepare the fresh solution after every few days.

5.4.9 *Standard Arsenite Solution*

Dissolve 0.173 4 g of sodium arsenite ($NaAsO_2$) in the water and then dilute the same in 1 l of water. Dilute 10.0 ml to 100.0 ml by using water. Dilute 10 ml of the intermediate solution to 100 ml by using water (1 ml = 1.00 µg of As).

CAUTION — $NaAsO_2$ is toxic in nature and thus its contact with skin should be avoided and it should not be ingested.

5.4.10 *Standard Arsenate Solution*

Take 0.416 g of $Na_2HAsO_4.7H_20$ and dissolve it in water. Further dilute the same upto 1 l of water. Dilute 10 ml of the intermediate solution to 100 ml (1 ml = 1.00 µg of As).

5.5 Procedure

5.5.1 *Arsenite*

5.5.1.1 *Preparation of scrubber and absorber*

Glass wool is dipped into the lead acetate solution and remove the excess of the solution by squeezing the glass wool. Further, press the glass wool in between the pieces of filter paper and fluff it afterwards. If cotton is used, treat it similarly and dry it in a dessicator. Fluff it thoroughly when dry. Place a plug of cotton or glass wool which is little loose in a scrubber tube (5 ml of it may be used to provide sufficient volume to rinse the spectrophotometer cell).

5.5.1.2 *Loading of arsine generator*

Pipette out not more than 70 ml sample containing not more than 20.0 µg As (arsinite) into the generator flask. Add about 10 ml of acetate buffer and if required, adjust the total volume of liquid up to 80 ml. Flush the flask with nitrogen at the rate of 60 ml/min.

5.5.1.3 *Generation of arsine and its measurement*

While passing the nitrogen from the system, use 30 ml syringe for the injection of 15 ml of 1 percent sodium borohydride within the time frame of 2 min. Vigorously stir the same by using a magnetic stirrer. Pass nitrogen from the system for 15 more min in order to flush the arsine into the absorber solution. Pour the absorber solution in a clean and dry spectroscopic cell and then

measure the absorbance at 520 nm. Concentration of arsenite is determined by using standard arsenite solutions. In order to determine the arsenate by using the same solution, the liquid is saved in the generator flask.

5.5.1.4 *Preparation of the standard curves*

Treat the solution of arsenite standard that contains 0.0, 1.0, 2.0, 5.0, 10.0, and 20.0 µg As as described above. Plot the curve of absorbance versus micrograms of arsenic in the standard.

5.5.2 *Arsenate*

After removal of arsenite as arsine, treat the sample so as to convert arsenate to arsine.

If the lead acetate which is impregnated with glass wool has become ineffective in removing hydrogen sulfide (in case it has become gray to black) replace glass wool. Pass the nitrogen through system at the rate of 60 ml/min and then carefully add 10 ml of 2.0 N HCl. Generate arsine as directed above and prepare the standard curves with standard solutions of arsenate in accordance with the procedure given above.

5.5.3 *Total Inorganic Arsenic*

Prepare the scrubber and absorber as directed above (**5.5.1.1**) and load the arsine generator as directed in (**5.5.1.2**) the above step by using 10 ml of 2.0 N HCl instead of the acetate buffer. Generate arsine and carry out the measurement as in **5.5.1.3**. Prepare the standard curves as per **5.5.1.4**. The curves that are obtained from the standard arsinite solution are almost identical to those that are obtained with arsenate standard solutions. Therefore, either use arsinite or arsenate standards.

5.6 Calculation

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

where

M = mass of arsenic calculated from calibration curve, in µg; and

V = volume of sample in the generator flask, in ml.

6 INDUCTIVELY COUPLED PLASMA SPECTROSCOPY

Arsenic can also be determined by inductively coupled plasma optical emission spectroscopy with reference to procedure given in IS 3025 (Part 2). Likewise, inductively coupled plasma mass spectroscopy with reference to procedure given in IS 3025 (Part 65) can also be used for the determination of arsenic.

7 SAFETY PRECAUTIONS

Avoid contact with skin, eyes, and clothing. Wash hands before breaks and immediately after handling the product. All operations involving arsenic should be carried out in a ventilated enclosure chemical fume hood to keep airborne concentrations below recommended exposure limits

Arsenic should be secured from unauthorized access. Arsenic is highly toxic, as such the container should be sealed tightly. Ground all equipment containing material. Keep away from all sources of ignition and heat.

7.1 In addition to arsenic specific symptoms, other compounds mixed with the arsenic will cause other symptoms which may make diagnosis difficult. The principle route of exposure of arsenic is ingestion and this occurs with poor hand cleaning hygiene in the lab. Arsenic is a ubiquitous and can be present in high levels in some drinking water and foods, which could contribute to the following symptoms:

List of symptoms typical of arsenic poisoning:

a) *Skin and Mucous Membranes* — Dermatitis, skin ulcers, hyperpigmentation, keratoses, skin cancer;

b) *Respiratory Tract* — Irritation of nose, throat and lungs, perforation of nasal septum, lung cancer; and

c) *Gastrointestinal* — irritation and nausea.

7.2 Short-Term (Acute) Arsenic Poisoning

Exposure to inorganic arsenic can cause nausea, vomiting, diarrhea, weakness, loss of appetite, cough, chest pain, giddiness, headache and breathing difficulty. Exposure to arsine gas can cause sudden death. Acute poisoning by arsenic compounds other than arsine gas rarely occurs in industry.

7.3 Long-Term (Chronic) Effects

Exposure to inorganic arsenic over time can cause weakness, nausea, vomiting, diarrhea, skin and eye irritation, hyperpigmentation, thickening of the palms and soles (hyperkeratosis), contact dermatitis, skin sensitisation, warts, ulceration and perforation of the nasal septum, and numbness and weakness in the legs and feet. Higher incidents of some types of skin, bladder and lung cancers can also result from chronic arsenic exposure.

ANNEX A

(*Foreword*)

COMMITTEE COMPOSITION

Water Quality Sectional Committee, CHD 36

Organization	*Representative(s)*
Chief Scientist, EPTRI, Hyderabad	Shri N. Raveendhar (***Chairman***)
Andhra Pradesh Pollution Control Board, Vijaywada	Shrimati M. Sreeranjan Shrimati A. Sri Samyuktha (*Alternate*)
Bhabha Atomic Research Centre, Mumbai	Dr S. K. Sahu Shri I. V. Saradhi (*Alternate*)
Central Institute of Mining and Fuel Research, Dhanbad	Dr (Mrs) Bably Prasad Dr Abhay Kumar Singh (*Alternate*)
Central Pollution Control Board, New Delhi	Shri N. C. Durgapal Dr J. C. Babu (*Alternate*)
Confederation of Indian Industry, New Delhi	Shri Shikhar Jain Ms Kamal Sharma (*Alternate*)
Delhi Jal Board, Delhi	Shri Ashutosh Kaushik Shri Sanjeev Kumar (YP) (*Alternate*)
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Environment Protection and Training Research Institute, Hyderabad	Dr J. S. Srinivas
FAD 14	Member Secretary
Gujarat Pollution Control Board, Gandhinagar	Dr D. N. Vansadia Shri K. B. Vaghela (*Alternate*)\
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National Institute of Oceanography, Vishakhapatnam	Dr V. V. S. S. Sarma
NTPC Ltd, New Delhi	Shri V. Ravi Babu Shri Sudhir Dahiya (*Alternate*)

Organization	*Representative(s)*
Sriram Institute for Industrial Research, New Delhi	Ms Shobha Kumar Shri Sumant Shekhar (*Alternate*)
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In personal capacity (*1221, Mahatma Gandhi Road, P. O., Haridevpur, Kolkata, 700 082*)	Shri Sanjib Kumar Goswami
In personal capacity, Hyderabad	Shri N. Murali Mohan
BIS Directorate General	Shri Ajay Kumar Lal, Scientist 'E' and Head (CHD) [Representing Director General (*Ex-officio*)]

Member Secretary

Ms Shubhanjali Umrao
Scientist 'B' (CHD), BIS

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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Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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