भारतीय मानक
Indian Standard

IS 2088 : 2023

आर्सेनिक के निर्धारण के तरीके

(तीसरा पुनरीक्षण)

Methods for Determination of Arsenic

(Third Revision)

ICS 71.060

भारतीय मानक ब्यूरो
BUREAU OF INDIAN STANDARDS
मानक भवन, 9 बहादुर शाह ज़फर मार्ग, नई दिल्ली - 110002
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI - 110002
www.bis.gov.in www.standardsbis.in

June 2023 **Price Group 6**

Inorganic Chemicals Sectional Committee, CHD 01

FOREWORD

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

This standard was first published in 1962 covering only modified Gutzeit method. Newer techniques for determination of arsenic were increasingly applied, and one such method was based on silver diethyldithiocarbamate. The standard was revised in 1971 to incorporate this method. This method assisted in determining the actual arsenic content as against the limit test provided by modified Gutzeit method. Selection of either silver diethyldithiocarbamate or modified Gutzeit method were to be judged from the end use.

The second revision of the standard was undertaken in 1983 to align it with ISO 2590 'General method for the determination of arsenic-silver diethyldithiocarbamate photometric method'. Before the test could be applied to the material, any arsenic present had to be quantitatively converted into its simple derivatives like arsenic trichloride or pentachloride which could completely be reduced to arsine by action of zinc in presence of dilute acid. These derivatives had to be free from interfering substances like nitrate, copper, silver, mercury, cobalt, nickel, molybdenum and palladium which are to be removed suitably and taken in the desired volume containing the desired amount of free acid. Though the preparation of test solution had to be dealt by individual material specifications, the possible treatments either alone or in combination, was considered suitable for most samples. Such treatments are prescribed in Annex A for guidance.

Further, in the third revision, alternative test method, ICP-OES has been incorporated in the standard. In addition to this, reference clause has also been updated.

The composition of the committee responsible for formulation of this Standard is listed in Annex B.

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 : 2022 'Rules for rounding off numerical values (*second revision*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

METHODS FOR DETERMINATION OF ARSENIC

(Third Revision)

1 SCOPE

1.1 This standard prescribes methods for determination of arsenic.

1.1.1 Modified Gutzeit method of test for arsenic shall be employed in cases where the actual arsenic content is not needed and only a knowledge of limit is desired. In cases where the actual arsenic content is to be determined, silver diethyldithiocarbamate method or ICP-OES method may be followed. The silver diethyldithiocarbamate method is applicable to quantities of arsenic (as As) in the range of 1 µg to 20 µg.

2 REFERENCES

The Indian Standards listed below contain provisions which through reference in this text, constitute provision 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 listed below:

IS No.	*Title*
IS 265 : 2021	Hydrochloric acid — Specification (*fifth revision*)
IS 323 : 2009	Rectified spirit for industrial use — Specification (*second revision*)
IS 336 : 1973	Specification for ether (*second revision*)
IS 695 : 2020	Acetic acid — Specification (*fourth revision*)
IS 1070 : 1992	Reagent grade water — Specification (*third revision*)

3 APPARATUS

3.1 The apparatus for modified Gutzeit method shall be as given in Fig. 1.

3.2 For silver diethyldithiocarbamate method the apparatus shall consist of the following:

3.2.1 *Evolution and Absorption Apparatus*

It shall consist of a conical flask A of 100 ml capacity for evolution of arsine, a connecting tube B to trap hydrogen sulphide, and absorption tube C with a spherical or conical groundglass joint. A spring clip may be used to ensure firm joint between the connecting tube B and absorption tube C when a spherical joint is used. Suitable forms of apparatus using spherical joint with fritted glass are shown in Fig. 2, 3 and 4.

3.2.2 *Spectrophotometer or Photoelectric Absorptiometer* — with filters in the range 520 nm to 560 nm and 10 mm cells

NOTE — The glassware should not be rinsed with organic solvents to facilitate drying. Traces of organic matter, especially, acetone may adversely affect the reaction between zinc and acid.

4 QUALITY OF REAGENTS

4.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be used in the tests.

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

4.2 All the reagents used shall be free from arsenic.

5 REAGENTS

5.1 Lead Acetate Solution — 10 percent (*m/v*). Add sufficient acetic acid (*see* IS 695) to obtain a clear solution.

5.2 Lead Acetate Paper Strips

Soak filter paper strips of 70 mm × 50 mm size in lead acetate solution contained in a glass bottle. Before use, take out the strips and dry them in an atmosphere free from hydrogen sulphide.

5.2.1 *Absorbent Cotton Wool Impregnated with Lead Acetate*

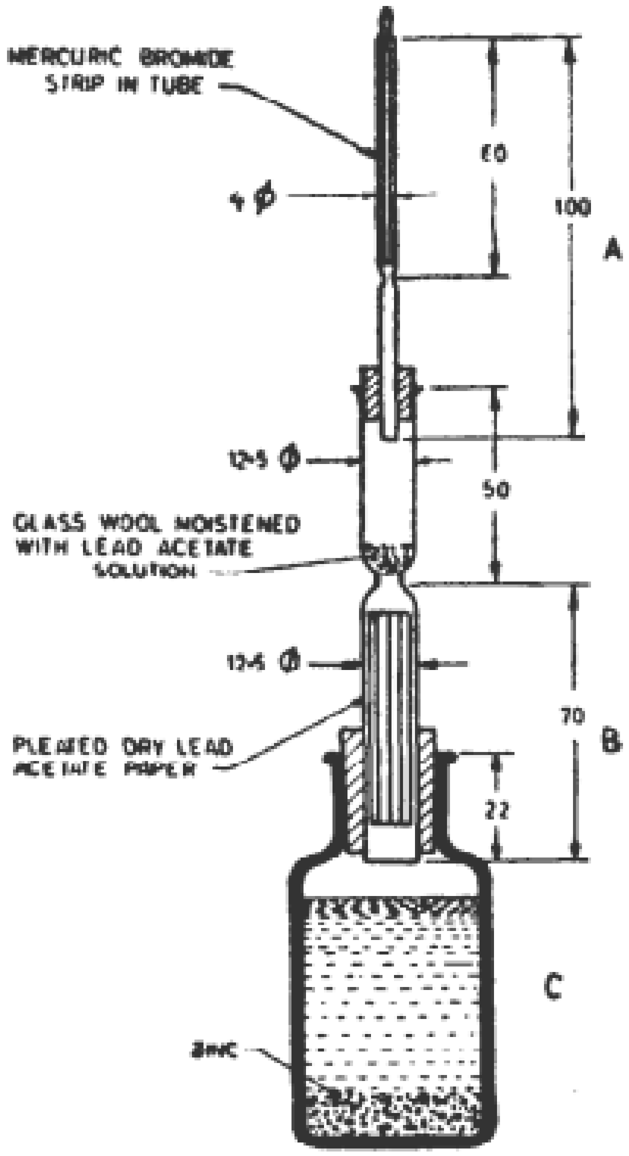
Saturate cotton wool with lead acetate solution, drain pass and dry under vacuum at room temperature.

5.3 Mercuric Bromide Solution

Dissolve 5 g of mercuric bromide in 100 ml of rectified spirit (*see* IS 323).

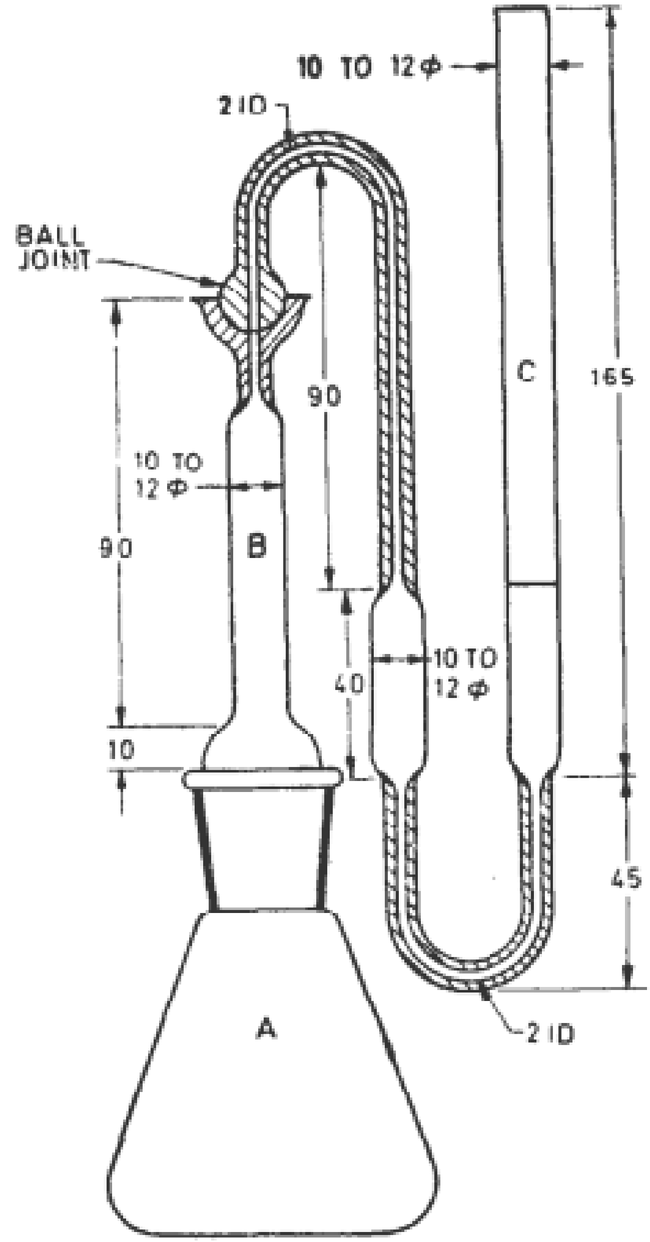
5.4 Sensitized Mercuric Bromide Paper Strips

Soak filter paper strips cut to a size of 70 mm × 50 mm in mercuric bromide solution contained in an amber-coloured glass bottle in dark. Before use, remove the strip, press it between sheets of filter paper, and dry it in an atmosphere free from hydrogen sulphide.



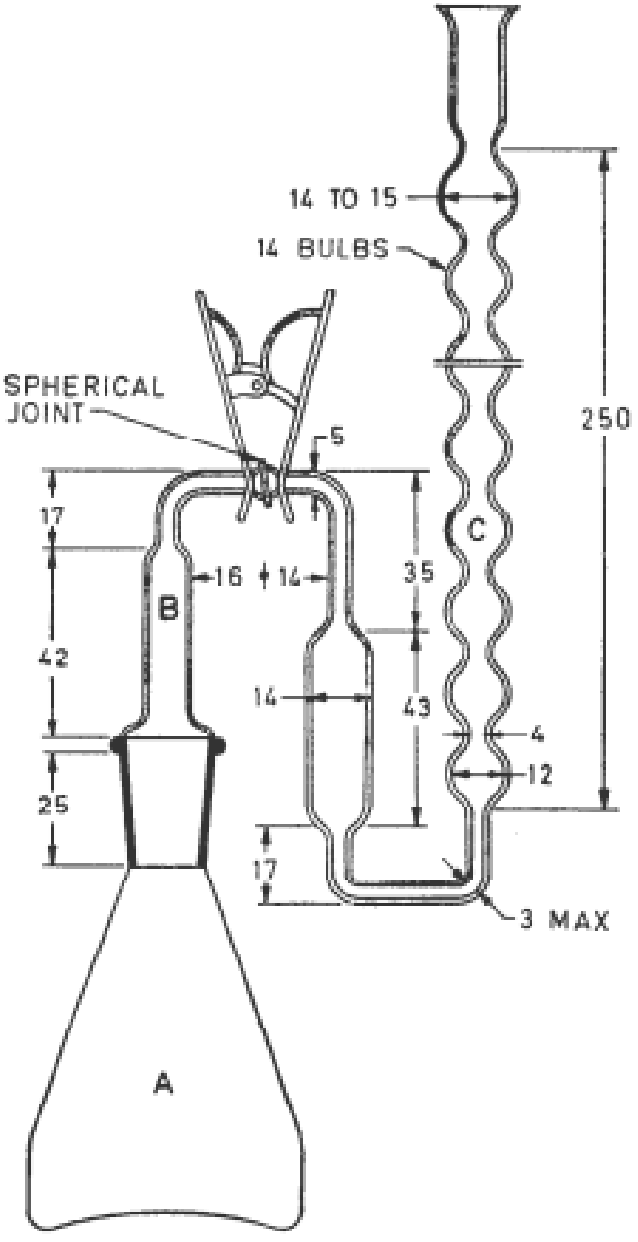
All dimensions in millimetres.

FIG. 1 APPARATUS FOR TEST OF ARSENIC (MODIFIED GUTZEIT METHOD)



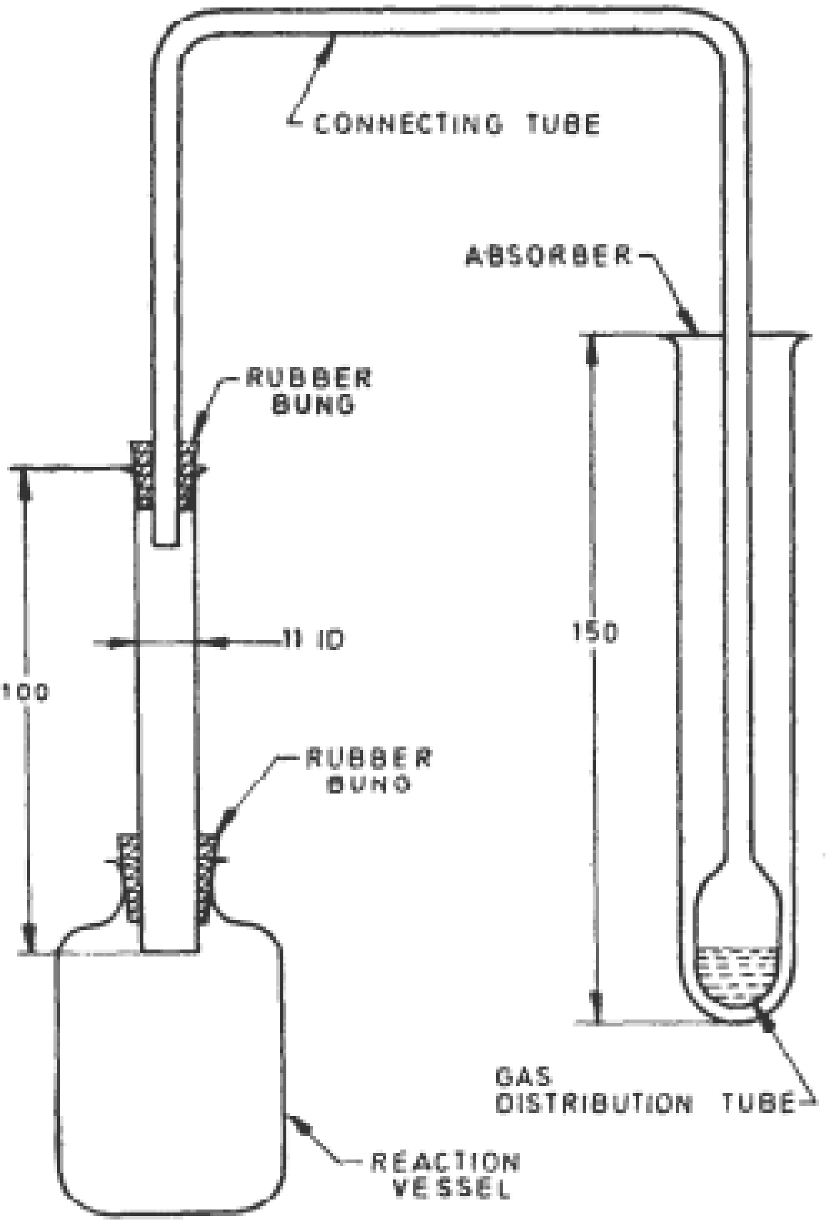
All dimensions in millimetres.

Fig. 2 Apparatus for Arsenic Determination



All dimensions in millimetres.

FIG. 3 APPARATUS FOR DETERMINATION OF ARSENIC (SILVER DIETHYLDITHIOCARBAMATE METHOD)



All dimensions in millimetres.

FIG. 4 APPARATUS WITH FRITTED GLASS FOR DETERMINATION OF ARSENIC (SILVER DIETHYLDITHIOCARBAMATE METHOD)

5.5 Dilute Sulphuric Acid — Approximately 5 N.

5.6 Concentrated Hydrochloric Acid — *see* IS 265.

5.7 Potassium Iodide Solution — 15 percent (*m*/*v*).

5.8 Stannous Chloride Solution

Dissolve 40 g of stannous chloride in 100 ml of hydrochloric acid. Discard this solution if an appreciable deposit forms.

5.9 Zinc — Granules, of size 0.5 to 1.0 mm.

5.10 Standard Arsenic Stock Solution

Weigh, to the nearest 0.1 mg, 0.132 0 g of arsenic trioxide and transfer it to a beaker of suitable capacity (for example 100 ml). Dissolve the arsenic trioxide with 2 ml of sodium hydroxide solution containing 50 g/l. Transfer the solution to a 1 000 ml volumetric flask. Wash the beaker several times. Collect the washings in the volumetric flask and make up the volume. One millilitre of this standard solution contains 100 µg of arsenic (As).

5.10.1 *Diluted Standard Arsenic Solution*

Containing 2.5 mg of arsenic per litre. Take 25 ml of the standard arsenic solution, transfer it to a 1 000 ml volumetric flask and make up the volume. Prepare this solution at the time of use. One millilitre of this standard solution contains 2.5 µg of arsenic (As).

5.11 Silver Diethyldithiocarbamate Solution

Dissolve 1 g of silver diethyldithiocarbamate in water-white pyridine and dilute to 200 ml with pyridine. Store the solution in stoppered-glass bottles away from light.

5.11.1 Silver diethyldithiocarbamate may be commercially available or it may be prepared from sodium diethyldithiocarbamate as follows:

Dissolve 10 g of sodium diethyldithiocarbamate $[(C_2H_5)_2N.CS.SNa.3H_2O]$ in 35 ml of rectified spirit (IS 323) and filter. Add to the solution with stirring, 100 ml of ether (*see* IS 336). Filter it by using suction, wash the precipitate with ether and dry them in air. Dissolve separately 22 g of the material in 100 ml of water, and 1.7 g of silver nitrate in 100 ml of water. Slowly mix these two solutions with constant stirring. Keep the temperature of the mixture below 10 °C. Decant the supernatant liquid, wash the precipitate 3 or 4 times with water, at a temperature below 10 °C. Filter and dry the product in vacuum at room temperature. Preserve the material protected from light in a cool place. This silver diethyidithiocarbamate solution in water white pyridine is stable for about 2 months.

NOTE — Alternatively, silver diethyldithiocarbamate may be prepared as follows:

Dissolve 2.2 g of freshly purified sodium diethyldithiocarbamate (as described above) in 250 ml of water-white pyridine.

Dissolve 1.95 g silver nitrate in 250 ml of water-white pyridine. With continuous stirring pour the former solution into the latter. Allow to stand over-night and filter through a fast filter paper. Store the solution in a well-stoppered dark coloured bottle protected from light in a cool plate. The solution is stable for at least 3 months.

6 PROCEDURE

6.1 Modified Gutzeit Method

Prepare the solution of the material as specified in the individual material specification (*see* also Annex A). Place the dry lead acetate paper in the lower portion of the tube B and cotton wool moistened with lead acetate in its upper portion. Place the sensitized strip of mercuric bromide paper in tube A and connect the tubes together with a rubber stopper. Introduce the solution of the material into the bottle C and add 10 ml of dilute sulphuric acid. Add 0.5 ml of stannous chloride solution, 5 ml of potassium iodide solution and make up the volume with water to about 50 ml. Mix the contents and drop in about 10 g of zinc. Fit in position the rubber stopper carrying the tube B immediately. Place the bottle in a warm place at about 40 °C. Remove the test strips after 40 min with the help of tweezers. Conduct the test in exactly similar manner by using a volume of standard arsenic solution as specified in the material specification, in place of the solution of the material and compare the stain produced with the material with that produced with the arsenic solution.

6.1.1 The limit prescribed in the material specification shall be taken as not having been exceeded if the length of the stain as well as the intensity of its colour produced in the test with the material is not greater than those produced with the corresponding standard arsenic solution.

6.2 Silver Diethyldithiocarbamate Method

6.2.1 *Principle*

Absorption of arsine in a solution of silver diethyldithiocarbamate involves the reaction:

The colloidally dispersed silver, purplish-red colour, is measured by photometry (nephelometry) at 540 nm.

6.2.2 *Preparation of Calibration Curve*

The curve shall be confirmed every time when a new solution of silver diethyldithiocarbamate is prepared.

6.2.3 *Evolution of Arsine*

Transfer to a series of 100 ml conical flasks, aliquots of standard arsenic solution corresponding to 0 µg, 5 µg, 10 µg, 15 µg, 20 µg and 25 µg of arsenic and proceed as given in **6.2.3.1**.

6.2.3.1 Add 10 ml of concentrated hydrochloric acid and dilute to 50 ml ± 5 ml with 1 litre water. Add 2 ml of potassium iodide and stannous chloride solution respectively. Mix well and let it stand for

15 min to 20 min. Pack lightly the top third of the connecting tube with impregnated absorbent cotton wool and assemble the absorption train. Transfer 5.0 ml of silver diethyldithiocarbamate solution to absorption tube C. After 15 min to 20 min, introduce 5 g of zinc granules into the conical flask A and quickly reassemble the apparatus. Allow the reaction to proceed for 45 min to 60 min at room temperature.

6.2.3.2 *Spectrophotometric measurements*

Disconnect the absorption tube and tilt the absorber so that the reagent solution flows back and forth between the absorber and bulb to disperse the solid contents, if any, and to mix in the solution well. Transfer the solution to a photometric cell and measure its absorbance at the wavelength of maximum absorption, 540 nm using water as reference liquid.

NOTE — The colour of the dispersion is not very stable for long time and hence absorptiometric (nephplometric) measurement should be made within 2 h of the development of colour. Care should also be taken to prevent the evaporation of solution as its volume is small.

In the case of fritted glass absorber raise and lower the connecting tube into the absorber several times, to allow the solution to pass through the frit back and forth effecting the dispersal of the red deposit. Let the connecting tube finally drain into the absorber.

10 µg standard shall have an absorption of the order of 0.4.

6.2.3.3 *Plotting of the calibration curve*

Calculate corrected absorbance by subtracting the reading obtained for the solution containing no standard arsenic solution from the observed reading. Plot a graph of corrected absorbance of solution against their arsenic contents.

6.2.3.4 The test solutions shall be prepared as prescribed in relevant individual material specifications so as to contain 1 µg to 10 µg of arsenic in a solution of 5.0 ml ± 0.5 ml volume. Transfer the solution to the conical flask, cool to room temperature if necessary and proceed as prescribed in **6.2.3**, **6.2.3.1** and **6.2.3.2**.

6.2.4 *Blank Test*

Carry out a blank test, as prescribed in **6.2.3**, **6.2.3.1** and **6.2.3.2** omitting the sample.

6.2.5 *Calculation*

Calculate the corrected absorbance by subtracting the value obtained for the blank solution from that obtained for the test solution and read from the calibration curve the corresponding mass of arsenic.

$$\text{Arsenic content, parts per million by mass} = \frac{M_1}{M_2}$$

where

M_1 = mass in µg, of arsenic found; and
M_2 = mass in g, of sample in the solution tested.

7 DETERMINATION OF ARSENIC BY INDUCTIVELY COUPLED PLASMA OPTICAL EMISSION SPECTROMETER (ICP-OES) METHOD

7.1 Principle

The sample solution under analysis is nebulized through a nebulizer inside a spray chamber. The aerosol formed is aspirated to argon plasma torch produced by a radio-frequency inductively coupled plasma (ICP), where the molecules break into constituent atoms and/or molecular species and atoms get excited. These excited atoms then return back to the lower energy state by emitting radiation of specific wavelength. These emitted radiations are characteristic of an element and are measured by the Photomultiplier tube detector and intensity of such emitted radiation is directly proportional to the concentration of respective constituent element in the sample.

Table 1 Recommended Wavelengths, Achievable Limits of Quantification for Different Configuration of Instruments and Important Spectral Interferences

(*Clauses* 7.2 and 7.4)

Sl No.	Element	Wavelength (nm)	Approx. Achievable Limits		Interfering Elements
			Radial viewing (µg)	Axial viewing (µg)	
(1)	(2)	(3)	(4)	(5)	(6)
i)	As	188.979	18	14	Al, Cr, Fe, Ti
		193.696	5	14	Al, Co, Fe, W, V
		197.197	(100)	31	Al, Co, Fe, Pb, Ti

7.2 Recommended Wavelength, Limit of Quantification and Important Spectral Interferences

Elements along with the recommended wavelengths and typical estimated limits of quantification are listed in Table 1. Actual working detection limits are dependent on the type of instrumentation, detection device and sample introduction system used and on the sample matrix. Therefore, these concentrations can vary between different instruments.

7.3 Reagents and Solutions

7.3.1 *Nitric Acid (65 Percent) Suprapure*

7.3.2 *Standard Stock Solution*

Either prepare by dissolving proportionate amount of soluble compounds of elements (preferably spectroscopic grade), or use commercially available certified stock solution of 10 µg/ml, 100 µg/ml or 1 000 µg/ml of lead, iron or arsenic in 2 percent to 5 percent nitric acid. It is preferable to prepare single stock solution of multi elemental standards for analysis.

7.3.3 *Standard Solution*

Pipette out 5 ml from 100 µg/ml standard stock solution into a 100 ml volumetric flask and make up volume with 2 percent nitric acid to prepare 5 µg/ml solution. From this 5 µg/ml solution, an aliquot of 1.0 ml, 3.0 ml and 5.0 ml taken in 50 ml volumetric flasks (separate) and make up volume with 2 percent nitric acid to prepare 0.1 µg/ml, 0.3 µg/ml and 0.5 µg/ml solution of respective elements under reference.

7.3.4 *Sample Preparation*

Weigh about 2.5 g sample in a 50 ml volumetric flask and add 1.0 ml nitric acid and make up the volume with water.

7.3.5 *Reagent Blank Solution*

Place 50 ml of nitric acid and 1 000 ml of water into an HDPE or PP container. For ultra-trace analysis, polytetrafluorethylene (PTFE) containers should be used. Prior to analysis, make sure that the acid matrix and concentration of the reagent blank solution is the same as in the standard and sample solutions.

7.4 Instrument

Set up the instrument as per the manufacturer's instructions manual for recommended operating parameters, based on the manufacturers operating manual and evaluated by internal check analysis using standard solution of element as well as data selected carefully from Table 1.

For analysis of arsenic, a gas (hydride)/vapour generating system is coupled with ICP, instead of using nebulizer. The arsine generated through the system is carried by the carrier gas (Ar) plasma torch, and other instrumental conditions shall be the same as above.

NOTE — Sensitivity, instrumental detection limit, precision, linear dynamic range and interference effects will be investigated and established for each individual analyte line on that particular instrument.

7.5 Procedure

7.5.1 *Calibration*

Profile and calibrate the instrument according to the instrument manufacturer's recommended procedures, using the intermediate mixed standard solutions (**7.3.3**). The relationship between concentration and intensity is linear up to six orders of magnitude. Examine the spectra of the element and make necessary adjustments (if required) for the exact peak positions and baselines to ensure proper measurements of the respective peak intensities.

Flush the system with the reagent blank solution between each standard.

7.5.2 Before beginning the sample run, re-analyse the reference standard with the highest concentration as if it were a sample. Ensure that the concentration values do not deviate from the actual values by more than ± 5 percent (or the established control limits, whichever is lower). If they do, follow the recommendations of the instrument manufacturer to correct for this condition. Begin the sample run by flushing the system with the reagent blank solution (**7.3.5**) between each sample. It is recommended to analyse a calibration check solution and the calibration blank solution every 10 samples. Analyze the sample solution and calculate the concentration in µg/ml of the lead (arsenic or iron) in the sample solution.

7.6 Calculation

The mass concentrations for each element are determined with the aid of the instrument software by following steps:

a) Relate emission signals from calibration blank and calibration solutions with the signals from reference elements and establish a calibration plot; and

b) Determine the mass concentrations of samples with the aid of the emissions and the calibration graphs and calculate the quantity in mg/kg of the constituent elemental impurities in the sample, by multiplying the value by 20 (dilution factor).

ANNEX A

(*Foreword and Clause* 6.1)

PREPARATION OF TEST SOLUTIONS OF ARSENIC

A-1 PREPARATION OF SOLUTION

A-1.1 The preparation of test solutions may be considered under the three possibilities described in **A-1.2** to **A-1.4**.

A-1.2 Sample Solution

A large portion of samples may be simply dissolved in water, with or without concentration, bromine treatment or addition of acid.

A-1.3 Wet Oxidation

The organic matter is destroyed and arsenic compounds not directly reducible under conditions of test are broken down. Care shall be taken that fuming of the acid does not start until all the peroxide is used up for destruction of organic compounds. Generally, 50 percent (*v*/*v*) hydrogen peroxide is used for destruction of organic compounds.

A-1.4 Solution of Difficult-to-Dissolve Substances

A-1.4.1 For opening up of siliceous materials; a mixture of hydrofluoric and sulphuric acid may be used but some hydrogen peroxide should be added before fuming of sulphuric acid starts.

A-1.4.2 Fusion with a mixture of sodium peroxide and sodium carbonate is useful in some cases where direct treatment with acids is not effective. If the sample is highly ferrogenous as in case of iron, ore, borax may be added in equal quantity.

ANNEX B

(*Foreword*)

COMMITTEE COMPOSITION

Inorganic Chemicals Sectional Committee, CHD 01

Organization	*Representative(s)*
Central Salt and Marine Chemicals Research Institute, Bhavnagar	Dr Kannan Srinivasan (***Chairperson***)
Alkali Mfrs Association of India, Delhi	Shri K. Srinivasan Shri H. S. Das (*Alternate*)
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Consumer Voice, Delhi	Shri M. A. U. Khan Shri K. C. Chaudhary (*Alternate*)
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Molecular Products Group India Pvt Ltd	Shri Vikrant Patil
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Shriram Institute for Industrial Research, Delhi	Dr LaxmiRawat Shri B. Govindan (*Alternate*)

Organization	*Representative(s)*
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In Personal Capacity (*Hari Nagar Co-Op-Society, Gotri Road, Vadodara, Gujarat-390007*)	DR R. S. BAGHEL
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Member Secretary
SAGAR SINGH
SCIENTIST 'C' (CHEMICAL), BIS

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Review of Indian Standards

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Amendments Issued Since Publication

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BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110002
Telephones: 2323 0131, 2323 3375, 2323 9402 *Website*: www.bis.gov.in

Regional Offices:		*Telephones*
Central	: 601/A, Konnectus Tower-1, 6th Floor, DMRC Building, Bhavbhuti Marg, New Delhi 110002	2323 7617
Eastern	: 8th Floor, Plot No 7/7 & 7/8, CP Block, Sector V, Salt Lake, Kolkata, West Bengal 700091	2367 0012 2320 9474
Northern	: Plot No. 4-A, Sector 27-B, Madhya Marg Chandigarh 160019	265 9930
Southern	: C.I.T. Campus, IV Cross Road, Taramani, Chennai 600113	2254 1442 2254 1216
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