

भारतीय मानक ब्यूरो /BUREAU OF INDIAN STANDARDS  
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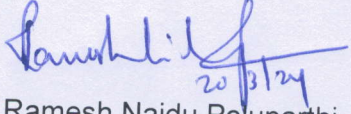
हमारा संदर्भ/Our Ref: ERL(C)/ IS 10116 : 2015

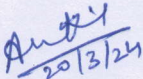
20/03/2024


विषय / **Subject:** Comments on method of Chemical Analysis of Arsenic (as As) in Boric Acid:

इस संबंध में प्रस्तावित बदलाव(ओं) पर विचार करने के लिए सी ई डी तथा संबंधित तकनीकी समिति द्वारा निम्नलिखित टिप्पणियों पर ध्यान दिया जाए:

क्र. सं. Sl.No	Clause no of IS	प्रस्तावित बदलाव/ <b>Proposed change</b>	वजह/ <b>Justification</b>
1	Annex-8	<i>Add Clause A-8.1.2 after Clause A-8.1.1 "Alternatively Arsenic may be estimated by Inductively Coupled Plasma Optical emission Spectrometry (ICP-OES) method as per Clause 7 of IS 2088:2023". Or "Atomic Absorption Spectrophotometer (AAS) method as per IS 11124:1984" Or "Inductively Coupled plasma- Mass Spectrometry (ICP-MS) method as per IS 3025(Part 65):2022".</i>	<i>Clause 8 of IS 10116:2015 refers a classical method of analysis for the estimation of Arsenic in Boric Acid wherein the method involved the handling and exposure of critical chemicals like Mercuric Bromide and Lead acetate and the method is also very laborious and tedious. In view of this, there is a need for incorporation of instrumental methods as specified in IS 2088:2023 (ICP-OES) or AAS - Vapor generation methods as specified in IS 11124:1984 or ICP-MS method as specified in IS 3025 (Part 65):2022"</i>

  
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out a control test using 2 ml of standard lead solution and the same quantity of reagents in the same total volume of the reaction mixture. Compare the colour of the solutions to 10 min after the addition of hydrogen sulphide solution.

**A-5.3.1** The prescribed limit shall be taken as not having been exceeded if the intensity of the colour produced in the test with the material is not greater than that produced in the control test.

## A-6 DETERMINATION OF IRON

### A-6.0 Outline of the Method

Iron is determined colorimetrically by visual comparison using potassium thiocyanate.

#### A-6.1 Apparatus

**A-6.1.1** *Nessler Cylinders* — 100 ml capacity (see IS 4161).

#### A-6.2 Reagents

**A-6.2.1** *Concentrated Hydrochloric Acid* — (see IS 265).

**A-6.2.2** *Ammonium Persulphate* — solid.

**A-6.2.3** *Butanolic Potassium Thiocyanate* — Dissolve 10 g of potassium thiocyanate in 10 ml of water. Add sufficient *n*-butanol to make up the volume to 100 ml and shake vigorously until the solution is clear.

**A-6.2.4** *Standard Iron Solution* — Dissolve 0.702 g of ferrous ammonium sulphate [ $\text{FeSO}_4(\text{NH}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O}$ ] in water and add 10 ml of dilute sulphuric acid (10 percent *v/v*). Dilute the solution to one litre. Take 10 ml of this solution and dilute to 100 ml before use. One millilitre of this solution contains 0.01 mg of iron (as Fe).

#### A-6.3 Procedure

Dissolve 5 g of the material for SQ grade and 0.02 g in case of explosive grade in 25 ml of water and transfer to a Nessler cylinder. Add 2 ml of hydrochloric acid, 30 mg of ammonium persulphate and 15 ml of butanolic potassium thiocyanate solution. Make up to 100 ml, shake vigorously for 30 s and allow the liquids to separate. Carry out a control test in another Nessler cylinder with 1 ml of standard iron solution and the same quantities of other reagents in the same total volume of the reaction mixture. Compare the colour of the butanol layer in the two sets.

**A-6.3.1** The limits prescribed in Table 1 for SQ grade and 4.4 for explosive grade shall be taken as not having been exceeded, if the colour of the butanol layer in the test with the material is not darker than the colour produced in the control test.

## A-7 CALCIUM

### A-7.1 Apparatus

**A-7.1.1** *Platinum Basin*

### A-7.2 Reagents

**A-7.2.1** *Methanol*

**A-7.2.2** *Hydrochloric Acid* — see IS 265.

**A-7.2.3** *Sulphuric Acid* — see IS 266.

**A-7.2.4** *Strong Ammonia Solution*

**A-7.2.5** *Ammonia – Ammonium Chloride Buffer Solution*

**A-7.2.6** *Sodium Sulphide Solution*

**A-7.2.7** *EDTA Solution* — 0.01 M.

**A-7.2.8** *Methyl Thymol Blue Indicator Solution*

**A-7.3** Dissolve 2.5 g of the powdered sample in 25 ml of methanol in a platinum basin, add 5 drops of hydrochloric acid and evaporate to dryness. To the residue add 15 ml of methanol and 3 drops of hydrochloric acid and again evaporate to dryness. Moisten the residue with sulphuric acid and ignite. Dissolve the residue in 50 ml of water; add 10 ml of ammonia-ammonium chloride buffer solution, 25 ml of strong ammonia solution and 5 drops of sodium sulphate solution. Titrate with EDTA solution using methyl thymol blue as indicator, until the blue solution becomes colourless or grey. Not more than 0.15 ml of EDTA is required to pass the test.

## A-8 ARSENIC

### A-8.1 Procedure

Dissolve 2.5 g of the material in 10 ml of the water. Carry out the test for arsenic as prescribed in IS 2088 using for comparison a stain obtained with 0.002 5 mg of arsenic trioxide.

**A-8.1.1** The material shall be taken to have satisfied the requirement of the test, if the length and intensity of the stain is not greater than that produced in the control test.

## A-9 SODIUM

Determine the sodium by flame photometer at 589 mm according to the directions of the manufacturer of apparatus.

## A-10 CHLORIDE

### A-10.0 Outline of the Method

Excess silver nitrate in nitric acid solution produces a white suspension of silver chloride on addition to a solution containing chloride ion.



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 (nephelometric) 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



*Indian Standard***METHOD FOR ATOMIC  
ABSORPTION SPECTROPHOTOMETRIC  
DETERMINATION OF ARSENIC****0. FOREWORD**

**0.1** This Indian Standard was adopted by the Indian Standards Institution on 31 October 1984, after the draft finalized by the Chemical Standards Sectional Committee had been approved by the Chemical Division Council.

**0.2** The atomic absorption spectrophotometric method is dependent on the fact that atoms in the ground state will absorb light of the same wavelength they emit when excited. When vapour containing arsenic ( $\text{AsH}_3$ ) is introduced in the flame, the intensity of the transmitted radiation will decrease in proportion to the amount of ground state atoms present in the flame, that is, the extent of concentration of arsenic. A hollow cathode lamp made of the element to be determined provides the source of radiation. Vapour generation kit is used to provide  $\text{AsH}_3$  vapour which is passed into the flame and the resultant arsenic absorption is seen as a peak signal. A monochromator eliminates the unwanted radiations and a photosensitive device measures the intensity of light. The absorbance is taken as the measure of concentration of the analyzed element.

**0.3** 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\*.

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**1. SCOPE**

**1.1** This standard prescribes the atomic absorption spectrophotometric method for the determination of arsenic by hydride generation technique.

\*Rules for rounding off numerical values (*revised*).

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