*भारतीय मानक* **IS 248 : 2023**

***Indian Standard***

# सोडियम बाईसल्फाईट, तकनीकी (सोडियम मेटाबाईसल्फाईट) — विशिष्टि

*( पांचवा पुनरीक्षण )*

# Sodium Bisulphite, Technical (Sodium Metabisulphite) — Specification

*( Fifth Revision )*

ICS 71.060

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Inorganic Chemicals Sectional Committee, CHD 01

## FOREWORD

This Indian Standard (Fifth 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.

Sodium bisulphite (sodium metabisulphite) is used in the preparation of dyes, intermediates, organic chemicals, perfumery; in the digestion of wood pulp, in leather tanning, as an antichlor and mordant in textile industry. It is also used as food preservative, in photography as reducing agent, in copper and brass plating, as colour preservative for pale crepe rubber, general antiseptic, as pesticides and a source of sulphur dioxide.

This standard was first published in 1954 and subsequently revised in 1961, 1971, 1978 and 1987. In this revision, reference, packing and marking clauses have been updated. In addition to this, instrumental test method for the analysis of Iron and Lead has been incorporated.

This standard, however, does not cover its application as food preservative and pesticides.

The composition of the committee responsible for formulation of this standard is listed in Annex C.

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*

# SODIUM BISULPHITE, TECHNICAL (SODIUM METABISULPHITE) — SPECIFICATION

*( Fifth Revision )*

## 1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for sodium bisulphite (sodium metabisulphite).

## 2 REFERENCES

The Indian Standards given 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.

| *IS No.* | *Title* |
|---|---|
| IS 265 : 2021 | Hydrochloric acid — Specification (*fifth revision*) |
| IS 1070 : 1992 | Reagent grade water (*third revision*) |
| IS 3025 (Part 2) : 2019/ISO 11885 : 2007 | 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) (*first revision*) |

## 3 REQUIREMENTS

### 3.1 Description

The material shall be dry, white or cream colored powder, free from extraneous matter and having a faint smell of Sulphur dioxide gas.

**3.2** The material shall also conform to the requirements laid down in Table 1, when tested in accordance with the methods prescribed in Annex A.

## 4 PACKING AND MARKING

### 4.1 Packing

The material shall be suitably packed taking care that it does not come in direct contact with iron container either during storage or in transit.

### 4.2 Marking

The packages shall be securely closed and bear legibly and indelibly the following information:

a) Name and grade of the material;
b) Name of the manufacturer and his recognized trade mark, if any;
c) Gross and net mass;
d) Date of manufacture; and
e) Batch number.

**4.2.1** *BIS Certification Marking*

The containers may also be marked with the Standard Mark.

**4.2.1.1** The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act*, 2016 and the Rules and Regulations framed thereunder, and the products may be marked with the Standard Mark.

## 5 SCALE OF SAMPLING AND CRITERIA FOR CONFORMITY

**5.1** The scale of sampling and criteria for conformity of the material to the standard shall be as prescribed in Annex B.

**Table 1 Requirements for Sodium Bisulphite**
(*Clause* 3.2)

| Sl No. (1) | Characteristic (2) | Requirement (3) | Method of Test, (Ref to Cl No. in Annex A) (4) |
|---|---|---|---|
| i) | Purity (as $SO_2$ content), percent by mass, *Min* | 60.0 | **A-2** |
| ii) | *p*H of 5 percent solution | 4.5 to 5.5 | **A-3** |
| iii) | Matter insoluble in water, percent by mass, *Max* | 0.1 | **A-4** |
| iv) | Iron (*as* Fe), percent by mass, *Max* | 0.02 | **A-5** or **A-8** |
| v) | Heavy metals (as Pb), percent by mass, *Max* | 0.1 | **A-6** or **A-8** |
| vi) | Appearance of solution | To pass test | **A-7** |

## ANNEX A

(*Clause* 3.2 *and Table* 1)

## METHODS OF TEST FOR SODIUM BISULPHITE

### A-l QUALITY OF REAGENTS

**A-1.1** Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be used in tests.

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

### A-2 DETERMINATION OF PURITY OF SODIUM BISULPHITE

### A-2.1 Reagents

**A-2.1.1** *Starch Solution*

Take 3 g of starch and make a paste with cold water. Pour the paste into 1 litre of boiling water and add 10 ml of one percent salicylic acid.

**A-2.1.2** *Potassium Iodide* — Crystals.

**A-2.1.3** *Iodine Solution*

Approximately 0.1 N. Dissolve in a 1 000 ml volumetric flask about 12.5 g of resublimed iodine in a concentrated solution of 35 g of potassium iodide. Make up the solution to 1 000 ml.

**A-2.1.4** *Standard Sodium Thiosulphate Solution*

0.1 N. Dissolve about 25 g of crystallized sodium thiosulphate in 1 000 ml of recently boiled water in a volumetric flask. Standardize the solution against standard potassium dichromate or freshly standardized iodine solution, using towards the end of the reaction, starch solution as indicator. The solution shall be prepared freshly.

### A-2.2 Procedure

Weigh accurately about 0.2 g of the material and add it to exactly 50 ml of standard iodine solution. Allow to stand for 5 min, add 1 ml of hydrochloric acid and titrate the excess of iodine with standard sodium thiosulphate solution, using starch solution as indicator. The end point of the reaction being indicated by the disappearance of blue colour. Carry out a blank titration with same amounts of reagents but without sample.

### A-2.3 Calculation

Calculate the percentage of sulphur dioxide ($SO_2$) on the basis that 1 ml of 0.1 N iodine solution is equivalent to 0.003203 g of sulphur dioxide ($SO_2$).

Purity (as $SO_2$ content),

$$\text{percent by mass} = 3.203 \times \frac{(V_1 - V)\,\mathrm{N}}{M}$$

where

$V_1$ = volume in ml of standard thiosulphate required for blank,
$V$ = volume in ml of standard thiosulphate required for test,
$\mathrm{N}$ = normality of standard thiosulphate solution, and
$M$ = mass in g of the material taken for the test.

### A-3 DETERMINATION OF *p*H

### A-3.1 Apparatus

**A-3.1.1** *p*H *Meter* — Equipped with a standard calomel electrode and a glass electrode.

### A-3.2 Procedure

Dissolve 5 g of the material in 100 ml of carbon-dioxide-free water. Determine the *p*H by *p*H meter.

### A-4 DETERMINATION OF MATTER INSOLUBLE IN WATER

### A-4.1 Apparatus

**A-4.1.1** *Gooch Crucible or Sintered Glass Crucible* — Porosity No. 4.

### A-4.2 Procedure

Weigh accurately about 50 g of the material and dissolved in 300 ml of water. Filter through the tared Gooch crucible or tared sintered glass crucible and wash well with water. Dry at 105 °C ± 2 °C to constant mass.

### A-4.3 Calculation

Matter insoluble in water,

$$\text{percent by mass} = \frac{100 \times m}{M}$$

where

$m$ = Mass in g of the residue obtained, and
$M$ = Mass in g of the sample taken for the test.

### A-5 TEST FOR IRON

**A-5.1** The following two methods are prescribed:

Colorimetric method, and spectrophotometric method.

### A-5.1 Colorimetric Method

**A-5.1.1** *Principle*

Iron is determined colorimetrically by visual comparison of the colour in Nessler cylinders using potassium thiocyanate.

**A-5.1.2** *Reagents*

**A-5.1.2.1** *Ammonium persulphate* — Solid.

**A-5.1.2.2** *Concentrated hydrochloric acid* — *see* IS 265.

**A-5.1.2.3** *Butanolic potassium thiocyanate solution*

Dissolve 10 g of potassium thiocyanate in 10 ml of water. Add sufficient *n*-butanol to make 100 ml and shake vigorously until solution is clear.

**A-5.1.2.4** *Standard iron solution*

Dissolve 0.702 of ferrous ammonium sulphates [$FeSO_4.(NH_4)_2SO_4.6H_2O$] in water containing 10 ml of dilute sulphuric acid (10 percent) and dilute to one litre in a volumetric flask. One millilitre of the solution contains 0.1 mg of iron (as Fe). Further dilute 100 ml of the solution to 1 000 ml. One millilitre of the diluted solution contains 0.01 mg of iron (as Fe).

**A-5.1.3** *Apparatus* — Nessler cylinder with 50 ml capacity.

**A-5.1.4** *Procedure*

Dissolve 1.00 g of the sample in 30 ml of hot water, add 5 ml of hydrochloric acid and evaporate to dryness on a steam bath. Add 15 ml of hot water and 2 ml of hydrochloric acid and evaporate again to complete dryness. Dissolve the residue in 10 ml of water and transfer to a beaker. Add 1 ml of hydrochloric acid, about 30 mg of ammonium persulphate, heat to boiling, cool and transfer to a Nessler cylinder. Add 15 ml of butanolic potassium thiocyanate. Shake vigorously for 30 s and allow the two layers to separate. For control test, take 2 ml of standard iron solution and repeat the test as above.

**A-5.1.3.1** The limit prescribed in Table 1 shall be taken as not having been exceeded, if the red colour produced in the butanolic layer of the sample solution is not more intense than that produced in the control test.

### A-5.2 Spectrophotometric Method

**A-5.2.1** *Outline of the Method*

Iron is reduced to ferrous state by thioglycollic acid and then treated with excess of ammonium hydroxide. The intensity of the resultant pink coloured complex is measured in a spectrophotometer at 535 nm at *p*H 10.

**A-5.2.2** *Apparatus*

**A-5.2.2.1** *Any suitable spectrophotometer*

**A-5.2.3 Reagents**

**A-5.2.3.1** *Dilute sulphuric acid* — 10 percent (*v*/*v*).

**A-5.2.3.2** *Standard iron solution*

Weigh 0.702 g of ferrous ammonium sulphates [$FeSO_4.(NH_4\ )_2SO_4.\ 6H_2O$] and dissolve in 10 ml of dilute sulphuric acid. Dilute with water to make up the volume to 1 000 ml. Transfer 10 ml of this solution and again dilute with water to make up the volume to 100 ml. One millilitre of this solution is equivalent to 0.01 mg of iron (as Fe).

**A-5.2.3.3** *Concentrated Hydrochloric acid* — *see* IS 265.

**A-5.2.3.4** *Dilute ammonium hydroxide* — Approx 6 N.

**A-5.2.3.5** *Thioglycollic acid AR (*$SH.CH_2COOH$) - 10 percent (*v*/*v*).

### A-5.3 Procedure

Weigh about 10 g of the sample, dissolve in about 75 ml of water. Add 1 ml of concentrated hydrochloric acid and boil for two min. Cool to 15 °C and add 10 ml of 10 percent thioglycollic acid solution. Add dilute ammonium hydroxide solution drop by drop till pale pink color appears keeping the temperature of the solution below 15 °C. Add one ml more of dilute ammonium hydroxide solution to bring to *p*H about 10. Dilute to 100 ml in a standard volumetric flask. Read the intensity of the color in a spectrophotometer using 535 nm wavelength. Note down the reading. Run a blank on all the reagents following the above procedure, substract this reading from the reading obtained with the sample. Let this be *A.*

**A-5.3.1** Prepare a series of standard iron solution by taking 2 ml, 5 ml, 7 ml, 10 ml, 15 ml and 20 ml of standard iron solution. Subject them to above procedure and note down the readings. Plot a standard curve for readings obtained against concentration of iron (as Fe).

### A-5.4 Calculation

Iron (as Fe), percent by mass = $\frac{A}{10\ M}$

where

$A$ = mg of Fe from the calibration curve, and

$M$ = mass in g of prepared sample taken for test.

**A-5.5** Alternatively, Iron may also be determined by an instrumental method given in **A-8**.

## A-6 TEST FOR HEAVY METALS

### A-6.1 Method A

**A-6.1.1** *Apparatus*

**A-6.1.1.1** *Nessler cylinders* — 50 ml capacity.

**A-6.1.2** *Reagents*

**A-6.1.2.1** *Acetic Acid* — Approximately 33 percent (*m*/*v*).

**A-6.1.2.2** *Dilute Ammonium Hydroxide Solution* — Approximately 10 percent (*v*/*v*).

**A-6.1.2.3** *Concentrated Hydrochloric Acid* — *see* IS 265.

**A-6.1.2.4** *Standard Lead Solution*

Dissolve 1.60 g of lead nitrate in water and make up the solution to 2 l in a volumetric flask. 1 ml of the solution contains 0.5 mg of lead (as Pb). Further dilute 100 ml of the solution to 1 000 ml. 1 ml of the diluted solution contains 0.05 mg of lead (as Pb).

**A-6.1.2.5** *Sodium Sulphide Solution* — 10 percent (*m*/*v*), freshly prepared.

**A-6.1.3** *Procedure*

Dissolve 4 g of the sample in 20 ml of hot water. Add 6 ml of hydrochloric acid and evaporate the contents nearly to dryness on a water bath. Add 15 ml of hot water, 3 ml of hydrochloric acid and evaporate again on the water bath. Finally heat for l h at 150 °C. Dissolve the residue in water, filter and make up the filtrate to 50 ml in a volumetric flask.

**A-6.1.3.1** Pipette into a Nessler cylinder 12.5 ml of the sample solution, 4 ml of standard lead solution and 1 ml of acetic acid. Add 2 drops of sodium sulphide solution and make up to the mark with water. Mix well.

**A-6.1.3.1.1** To another Nessler cylinder add the remaining 37.5 ml of the sample solution and 1 ml of acetic acid. Add 2 drops of sodium sulphide solution and make up to the mark with water. Mix well.

**A-6.1.3.2** The limits prescribed in Table 1 shall be taken as not having been exceeded if the colour produced in the second cylinder is not more intense than that produced in the first.

### A-6.2 Spectroscopic Determination of Lead (Method B)

**A-6.2.1** *Principle*

A sample containing microgram quantities of lead is extracted with dithizone solution in chloroform. The extraction is carried out in the presence of strong ammoniacal citrate-cyanide reducing agent (*p*H 10 to 11.5). The quantity of lead present in the sample is determined spectrophotometrically by measuring the absorbance at 510 nm in chloroform extract containing the lead dithizonate complex.

**A-6.2.2** *Minimum Detection Limit*

One µgPb/10 ml dithizone solution (extract).

**A-6.2.3** *Apparatus*

**A-6.2.3.1** Spectrophotometer for use at 510 nm with a path length of 1 cm or longer.

**A-6.2.3.2** *p*H *meter*

**A-6.2.3.3** *Standard volumetric glasswares.*

**A-6.2.3.4** *TEF beaker* — 100 ml for acid digestion.

**A-6.2.3.5** *Separatory funnels* — 250 ml and 500 ml.

All glasswares are to be cleaned with 1 : 1 $HNO_3$, and rinsed thoroughly with distilled water.

**A-6.2.4** *Reagents*

**A-6.2.4.1** *Quality of reagents*

Only analytical or equivalent grade reagents, unless specified otherwise, are to be used. All reagents are to be prepared in lead-free distilled water.

**A-6.2.4.2** *Stock lead solution*

Dissolve 0.159 9 g lead nitrate [$(Pb(NO_3)_2$, minimum purity, 99.5 percent (*w/w*)] in about 200 ml of water. Add 10 ml concentrated $HNO_3$ and dilute to 1 000 ml with water, 1.0 ml of this solution will contain 100 µg of Pb.

**A-6.2.4.3** *Standard lead solution*

Dilute 2.0 ml of stock lead solution to 100 ml with water, 1.0 ml of this solution will contain 2 µg of Pb.

**A-6.2.4.3.1** *Nitric acid* — Concentrated (18 N).

**A6.2.4.3.2** *Nitric acid — Dilute,* 20 percent, *v/v*.

**A-6.2.4.3.3** *Ammonium hydroxide* — Concentrated (14 N).

**A-6.2.4.3.4** *Ammonium hydroxide* — Dilute 10 percent, *v/v* and 1 percent, *v/v*.

**A.6.2.4.3.5** *Citrate-cyanide reducing solution*

Dissolve 200 g anhydrous ammonium citrate $[(NH_4)_2\ HC_6H_5O_7]$ 10 g anhydrous sodium sulphite $(Na_2SO_3)$, 5 g hydroxylamine hydrochloride, 20 g potassium cyanide (KCN) in water and dilute to 500 ml, and mix with one litre of concentrated $NH_4OH$.

CAUTION — KCN is a poisonous solution. Handle with extreme care and do not pipette by mouth.

**A-6.2.4.4** *Stock dithizone solution*

Dissolve 25 mg dithizone in about 50 ml chloroform $(CHCl_3)$ taken in a 200 ml beaker and filter through Whatman No. 42 (or equivalent) filter paper. Collect the filtrate and two washings (10 ml each) in a 250 ml conical flask. Transfer the combined filtrate to a

Five hundred millilitre separatory funnel. Add about 100 ml 1 percent (*v/v*) $NH_4OH$ solution, shake moderately for about 1 min. Transfer the $CHCl_3$ layer to another 250 ml separatory funnel retaining the orange-red aqueous layer in the 500 ml separatory funnel. Repeat the extraction (of the $CHCl_3$ layer) with 100 ml of 1 percent (*v/v*) $NH_4OH$ solution, transfer the $CHCl_3$ layer to another 250 ml separatory funnel and the aqueous layer to the original 500 ml separatory funnel containing the first extract. One more repetition, of extraction and transferring to the main aqueous layer is carried out. To the combined aqueous extract in the 500 ml separatory funnel add 1 : 1 HCl in 2 ml portions, mixing after each addition, until dithizone precipitation is complete and the solution is no longer orange-red. Extract the precipitated dithizone with three 25 ml portions of $CHCl_3$. Dilute the combined extract to 250 ml with $CHCl_3$, 1 ml of this solution will contain 100 µg of dithizone.

**A-6.2.4.5** *Working dithizone solution*

Dilute 100 ml stock dithizone solution to 250 ml in a standard volumetric flask with $CHCl_3$, 1 ml of this solution will contain 40 µg of dithizone.

**A-6.2.5** *Procedure*

**A-6.2.5.1** *Sample digestion*

Digest all samples for lead as per standard digestion procedure using minimum amount of $HNO_3$-$H_2SO_4$ and $HNO_3$-$HClO_4$. Dilute the acidified sample (*p*H = 2) to 100 ml and add 20 ml of dilute (20 percent, *v/v*) $HNO_3$, filter if required through a filter paper (Whatman No. 41 or equivalent), and transfer it to a 250 ml separatory funnel. Add 60 ml ammoniacal citrate-cyanide solution, mix and cool to room temperature. Add 10 ml of dithizone working solution. Shake the stoppered funnel vigorously for about 30 s, allow to stand (to get two separate layers). Discard 1 ml to 2 ml $CHCl_3$ layer and then fill the absorption cell. Measure the absorbance at 510 nm using working dithizone solution as reagent blank.

**A-6.2.5.2** *Calibration curve*

Plot a calibration curve using at least five standard lead solutions, after adding 50 ml ammoniacal citrate-cyanide solution to the individual lead standard solutions and extracting the same with 10 ml of dithizone working solution.

**A-6.2.6** *Calculation*

mg Pb/l = µg (in 10 ml extract obtained from calibration curve)/Volume of sample (ml)

**A-6.3** Alternatively, Lead may also be determined by an instrumental method given in **A-8**.

## A-7 APPEARANCE OF SOLUTION

**A-7.1** Dissolve 20 g of the material in a little quantity of water and make up the volume to 100 ml. The solution shall not have more than a pale yellow color and shall be free from extraneous impurities other than slight flocculence.

## A-8 DETERMINATION OF LEAD AND IRON BY INDUCTIVELY COUPLED PLASMA OPTICAL EMISSION SPECTROMETER (ICP-OES) METHOD

### A-8.1 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 are 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 2 Recommended Wavelengths, Achievable Limits of Quantification for Different Configuration of Instruments and Important Spectral Interferences

(*Clauses* 8.2 *and* 8.4)

| Sl No. | Element | Wavelength (nm) | Approx Achievable Limits | | Interfering Elements |
|---|---|---|---|---|---|
| | | | Radial viewing (µg) | Axial viewing (µg) | |
| (1) | (2) | (3) | (4) | (5) | (6) |
| i) | Pb | 220.353 | 14 | 5 | Al, Co, Fe, Ti |
| | | 283.305 | (70) | (20) | Cr, Fe |
| ii) | Fe | 238.204 | 14 | (3) | Co |
| | | 259.940 | 6 | 2 | Co |
| | | 271.441 | - | - | - |

**A-8.2 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 2. 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.

Additionally, Table 2 lists the most important spectral interferences at the recommended wavelengths for analysis.

**A-8.3 Reagents and Solutions**

**A-8.3.1** *Nitric Acid (65 percent) Suprapure*

**A-8.3.2** *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 or Iron in 2 percent to 5 percent nitric acid. It is preferable to prepare single stock solution of multi elemental standards for analysis.

**A-8.3.3** *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.

**A-8.3.4** *Sample Preparation*

Weigh about 2.5 g sample in a 50.0 ml volumetric flask and add 1.0 ml nitric acid and make up the volume with water.

**A-8.3.5** *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.

**A-8.4 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 of standard solution of element as well as data selected carefully from Table 2.

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.

**A-8.5 Procedure**

**A-8.5.1** *Calibration*

Profile and calibrate the instrument according to the instrument manufacturer's recommended procedures, using the intermediate mixed standard solutions (**A-8.3.3**). 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.

**A-8.5.2** 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 (**A-8.3.5**) 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 (and/or iron) in the sample solution.

NOTE — It is recommended that IS 3025 (Part 2)/ISO 11885 may be referred and practiced for ensuring precise and reproducible analysis.

### A-8.6 Calculation

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

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

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 B

(*Clause* 5.1)

## SAMPLING OF SODIUM BISULPHITE

### B-l GENERAL REQUIREMENTS OF SAMPLING

**B-1.1** In drawing, preparing, storing and handling test samples, the precautions given in **B-l.2** to **B-l.8** shall be observed.

**B-1.2** Samples shall not be taken at a place exposed to weather.

**B-1.3** Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

**B-1.4** To draw a representative sample, the contents of each container selected for sampling shall be mixed thoroughly by suitable means.

**B-1.5** The samples shall be placed in suitable, clean, dry and air-tight opaque glass or plastics containers.

**B-1.6** The sample containers shah be of such a size that they are almost completely filled by the sample.

**B-1.7** Each sample container shall be sealed air-tight after filling and marked with full details of sampling, the date of sampling and the year of manufacture.

**B-1.8** Samples shall be stored in such a manner that the temperature of the material does not vary unduly from the normal temperature.

### B-2 SCALE OF SAMPLING

### B-2.1 Lot

All the containers in a single consignment of the material drawn from a single batch of manufacture shall constitute a lot.

**B-2.1.1** Samples shall be tested from each lot for ascertaining the conformity of the material to the requirements of the specification

**B-2.2** The number (*n*) of containers to be chosen from a lot depends on the size of the lot (*N*) and shall be in accordance with co1 (1) and co1 (2)of Table 3.

**B-2.3** The containers to be selected for sampling shall be drawn at random from the lot. For random sampling procedures, guidance may be obtained from IS 4905.

### B-3 PREPARATION OF TEST SAMPLES

**B-3.1** Draw, with an appropriate sampling instrument, 50 g of sodium bisulphite from different parts of each container selected. This portion shall be transferred to suitable sample container. From each of the sample containers approximately equal quantities of the material shall be taken and mixed together to give a composite sample weighing about 100 g. The remaining material in each of the sample container is termed as individual sample.

### B-4 CRITERIA FOR CONFORMITY

**B-4.1** Test for purity of the material shall be conducted on individual samples. The lot shall be considered as conforming to the specification if all the individual samples pass the test.

**B-4.2** Tests for all other characteristics shall be carried out on composite sample. The lot shall be declared as conforming to the specification if the test results on the composite sample satisfy the corresponding requirements

**Table 3 Scale of Sampling of Containers**

(*Clause* B-2.2)

| Sl No.<br><br>(1) | Lot Size<br>(*N*)<br>(2) | Number Of Containers To Be Selected In The Sample<br>(*n*)<br>(3) |
|---|---|---|
| i) | up to 50 | 3 |
| ii) | 51 to 100 | 4 |
| iii) | 101 to 300 | 5 |
| iv) | 301and above | 7 |

## ANNEX C
(*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<br>Shri H. S. Das (*Alternate*) |
| Bhabha Atomic Research Centre, Mumbai | Dr A. V. R. Reddy<br>Dr S. N. Achary (*Alternate*) |
| Central Drugs Standard Control Organization | Dr Raman Mohan Singh |
| Consumer Voice, Delhi | Shri M. A. U. Khan<br>Shri K. C. Chaudhary (*Alternate*) |
| Delhi Jal Board, New Delhi | Shri Ashutosh Kaushik |
| DGQA, New Delhi | Dr Gurbachan Singh<br>Shri B. S. Tomar (*Alternate*) |
| Geological Survey of India, Kolkata | Shri P. V. V. R. Sarma |
| Global Adsorbents Pvt Ltd, Kolkata | Shri Sanjay Dhanuka |
| Grasim Industries Ltd, Nagda | Shri Alok Singh<br>Shri Pankaj Gupta (*Alternate*) |
| Gujarat Alkalies and Chemicals Ltd, Vadodara | Shri V. K. Mahida<br>Shri Shailesh Patel (*Alternate*) |
| Hindalco, Mumbai | Shri Nageswar Kapuri<br>Shri Ajith Ramachandra (*Alternate*) |
| Hindustan Lever Ltd, Mumbai | Ms Vrinda Rajwade<br>Shri Sojan Varghese (*Alternate*) |
| Indian Chemical Council (ICC) | Dr U. Shetkar<br>Dr Rakesh Kumar (*Alternate*) |
| Indian Institute of Chemical Technology, Hyderabad | Dr Praveen R. Likhar<br>Dr Rajender Reddy (*Alternate*) |
| Industrial Carbon Pvt Ltd, Ankleshwar | Shri Satyan Rohit Kumar |
| Ministry of Chemicals & Fertilizers | Dr Rohit Misra<br>Dr O. P. Sharma (*Alternate*) |
| Ministry of Defence (DGQA), Kanpur | Shri R. N. Aparajit |
| National Chemical Laboratory, Pune | Dr Darbha Srinivas<br>Dr Paresh Dhepe (*Alternate*) |

| *Organization* | *Representative(s)* |
|---|---|
| National Metallurgical Laboratory, Jamshedpur | Dr Trilochan Mishra<br>Shri Devbrata Mishra (*Alternate*) |
| National Mineral Development Corporation Ltd, Hyderabad | Shri Rajan Kumar<br>Dr Prashant Sharma (*Alternate*) |
| National Peroxide Ltd | Dr Joy Anthony |
| National Physical Laboratory, New Delhi | Dr Nahar Singh<br>Dr S. P. Singh (*Alternate*) |
| National Test House (NR) | Ms Richa Kundra |
| Office of the Development Commissioner (MSME), New Delhi | Dr Karthikeyan<br>Ms Anna Backiam (*Alternate*) |
| Shriram Institute for Industrial Research, Delhi | Dr (Ms) Laxmirawat<br>Shri B. Govindan (*Alternate*) |
| Tamil Nadu Petroproducts Ltd, Chennai Tata | Shri Ravi Muthukrishnan |
| Chemicals Ltd, Gujarat | Shri Najmul Hasan Khan |
| The Dharamsi Morarji Chemicals Co Ltd Vaibhav | Shri Mandar Gaikwad |
| Analytical Services, Ahmedabad | Shri Gaurang Oza |
| In Personal Capacity (*Hari Nagar Co-Op-Society, Gotri Road, Vadodara, Gujrat-390007*) | Shri R. S. Baghel |
| In Personal Capacity (*514 Veer Apt, Sector B, Rohini,New Delhi-110085*) | Shri D. K. Jain |
| BIS Directorate General | Shri A. K. Lal, Scientist 'E'/Director and Head (Chemical Department) [Representing |

*Member Secretary*
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Scientist 'C'/Deputy Director
(Chemical Department), BIS

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