भारतीय मानक **IS 297 : 2024**

Indian Standard

सोडियम सल्फाइड, तकनीकी — विशिष्टि

(तीसरा पुनरीक्षण)

Sodium Sulphide, Technical — Specification

(Third Revision)

ICS 71.060.50

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

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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 originally published in 1951 and subsequently revised in 1970 and 2001. In the first revision, the limits for the percentage of iron and aluminum compounds, sodium sulphate, sodium chloride and reducing compounds (other than sodium sulphide and sodium hydro sulphide) were reduced. Additional requirements of sodium hydrosulphide, excess alkali and water insoluble (other than iron and aluminum compounds) were incorporated and changes were also made in some of the methods of test and sampling procedure. In the second revision, the testing procedure for determination of reducing compounds (other than sodium sulphide and sodium hydrosulphide), sodium chloride, alternative method for determination of excess alkali and the calculation given for determination of total sulphides, hydro sulphides and excess alkali were modified.

In this revision, instrumental test method for the determination of aluminium and iron has been incorporated. Also, packing and marking clause and reference clause have been updated.

Sodium sulphide is widely used in tanneries, dyestuff and textile industries, and ore beneficiation. The solution being caustic, it needs proper care in handling.

The composition of the Committee responsible for formulation of this standard is given in Annex K.

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 SULPHIDE, TECHNICAL — SPECIFICATION

(Third Revision)

1 SCOPE

This standard prescribes the requirements and methods of sampling and test for sodium sulphide, technical.

2 REFERENCES

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

IS No.	*Title*
IS 265 : 2021	Hydrochloric acid — Specification (*fifth revision*)
IS 323 : 2009	Rectified spirit for industrial use — Specification (*second revision*)
IS 1070 : 2023	Reagent grade water — Specification (*fourth revision*)
IS 2316 : 1990	Methods of preparation of standard solutions for colorimetric and volumetric analysis (*second revision*)
IS 3025	Methods of sampling and test (physical and chemical) for water and wastewater:
(Part 2) : 2019/ ISO 11885 : 2007	Determination of selected elements by inductively coupled plasma optical emission spectrometry (ICP-OES) (*first revision*)
(Part 65) : 2022/ISO 17294-2 : 2016	Application of inductively coupled plasma mass spectrometry (ICP-MS) — Determination of selected elements including uranium isotopes (*first revision*)

3 REQUIREMENTS

3.1 Description

Sodium sulphide, technical, shall be in the form of flesh-coloured or dark-brown mass or flakes. It shall be soluble in water.

3.2 The material shall also comply with the requirements laid down in Table 1, when tested according to the methods prescribed in Annex A to Annex G. Reference to the relevant Annex is given in col (4) of the Table 1.

4 PACKING AND MARKING

4.1 Packaging

The material shall be packed in closed containers as agreed to between the purchaser and the supplier.

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 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 there under, and the products may be marked with the Standard Mark.

5 SAMPLING

The method of drawing representative samples of the material, number of tests to be performed and the criteria of conformity of the material to the requirements of this specification shall be as prescribed in Annex J.

access Indian Standards click on the link below:
https://www.services.bis.gov.in/php/BIS_2.0/bisconnect/knowyourstandards/Indian_standards/isdetails/

6 QUALITY OF REAGENTS

Unless specified otherwise, pure chemicals and reagent grade water (*see* IS 1070) shall be employed in tests.

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

Table 1 Requirements for Sodium Sulphide, Technical

(*Clauses* 3.2, J-4.2 *and* J-5.2)

Sl No. (1)	Characteristic (2)	Requirement (3)	Method of Test, Ref to Annex (4)
i)	Total sulphides (as Na_2S) percent by mass, *Min*	60.0	A
ii)	Sodium hydrosulphide (as NaHS), percent by mass, *Max*	2.5	A
iii)	Excess alkali (as Na_2CO_3), percent by mass, *Max*	2.0	A or G[1)]
iv)	Reducing compounds (other than sodium sulphide and sodium hydrosulphide) (as $Na_2S_2O_3$) percent by mass, *Max*	2.0	B
v)	Iron and aluminum compounds (as Fe_2O_3), percent by mass, *Max*	0.5	C or H
vi)	Sodium sulphate (as Na_2SO_4) percent by mass, *Max*	1.0	D
vii)	Sodium chloride (as NaCl), percent by mass, *Max*	1.0	E
viii)	Water insolubles (other than iron and aluminum compounds), percent by mass, *Max*	0.25	F

[1)] In case of dispute volumetric method prescribed in A shall be the referee method.

ANNEX A

[*Table* 1, *Sl No.* (i), (ii) *and* (iii)]

DETERMINATION OF TOTAL SULPHIDES, HYDROSULPHIDE AND EXCESS ALKALI

A-1 OUTLINE OF THE METHOD

To a known volume of excess of iodine solution and standard hydrochloric acid, a known mass of the sample is added. Excess of iodine is titrated against standard sodium thiosulphate solution. The amount of iodine consumed gives the total reducing compounds, from which the amount of iodine equivalent to the amount of sodium hydrosulphide and other reducing compounds (in terms of NaS_2O_3), when present, is deducted to give the iodine equivalent to total sulphides. After the completion of titration against sodium thiosulphate, titration is continued against standard sodium hydroxide solution using phenolphthalein indicator for the estimation of either sodium hydrosulphide or excess alkali, whichever is present. If the amount of standard 0.1 N sodium hydroxide solution required is more than the known amount of standard 0.1 N hydrochloric acid added, the excess acid produced is accounted for sodium hydrosulphide; if less, the amount of standard 0.1 N hydrochloric acid consumed is accounted for excess alkali.

A-2 REAGENTS

A-2.1 Glycerine Water — approximately 5 percent (*m*/*v*)

A-2.2 Standard Iodine Solution — approximately 0.1 N

A-2.3 Standard Hydrochloric Acid — 0.1 N

A-2.4 Standard Sodium Hydroxide Solution — 0.1 N

A-2.5 Standard Sodium Thiosulphate Solution — 0.1 N

A-2.6 Starch Indicator Solution

Triturate 5 g of pure starch and 0.01 g of mercuric iodide with 30 ml of water in a mortar. Pour the resulting paste into 1 litre of boiling water, boil for 3 min, allow the solution to cool, and decant off the clear liquid.

A-2.7 Phenolphthalein Indicator Solution

Dissolve 0.1 g of phenolphthalein in 100 ml of rectified spirit (*see* IS 323).

A-3 PROCEDURE

A-3.1 Preparation of Sample Solution

Weigh accurately 10 g of the material, transfer to a stoppered flask and dissolve in about 150 ml of glycerine water. Filter off insoluble, if any, collecting the filtrate in a 500 ml volumetric flask. Make up the filtrate and washings to 500 ml with glycerine water. Preserve this solution for tests purposes.

A-3.2 Pipette out 50 ml of standard iodine solution and 50 ml of standard hydrochloric acid into a conical flask and dilute to about 150 ml. Add to this 10 ml of prepared sample solution from **A-3.1**. Titrate the excess of iodine against standard thiosulphate solution using 1 ml of starch solution as indicator. Let this be B ml. Add four drops of phenolphthalein indicator and citrate the contents against standard sodium hydroxide solution. Let it be C ml.

A-3.3 Carry out a separate titration of 50 ml of iodine solution in the presence of 50 ml of standard hydrochloric acid against standard sodium thiosulphate solution using 1 ml of starch solution as indicator. Let this be A ml.

A-3.4 Titrate 50 ml of standard hydrochloric acid against standard sodium hydroxide solution using phenolphthalein indicator. Let this be D ml.

If C is greater than D:

The excess acid due to sodium hydrosulphide = $(C - D) = E$

If D is greater than C which is due to the excess alkali,

the amount of acid consumed by excess alkali = $(D - C) = F$

A-4 CALCULATION

A-4.1 Total sulphides (as Na_2S), percent by mass =

$$\frac{(X - Y - E) \times N \times 3.9}{M}$$

where

X = volume in ml of sodium thiosulphate, equivalent to ml of iodine consumed by total reducing compounds, as determined in **A-3.3**;

Y = volume, in ml, of standard sodium thiosulphate solution equivalent to reducing compounds [other than sodium sulphide and sodium hydrosulphide ($Na_2S_2O_3$)], when present, as determined in **B-2**;

E = volume, in ml, of standard sodium thiosulphate solution equivalent to sodium hydrosulphide (NaHS), when present, as determined in A-3.4;

N = normality of standard sodium thiosulphate solution; and

M = mass, in g, of the sample in 10 ml of the prepared sample solution from A-3.1 taken for the test.

A-4.2 Sodium Hydrosulphide (when C is Greater than D)

Sodium hydrosulphide (as NaHS), percent by mass

$$= \frac{E \times N_1 \times 5.6}{M}$$

where

E = The excess acid due to sodium hydrosulphide;

N_1 = normality of standard sodium hydroxide solution; and

M = mass, in g, of the sample in 10 ml of the prepared sample solution from A-3.1 taken for the test.

A-4.3 Excess Alkali

Either sodium hydroxide or sodium carbonate (when D is greater than C)

Excess alkali (as Na_2CO_3), percent my mass

$$= \frac{F \times N_1 \times 5.3}{M}$$

where

F = the amount of acid consumed by excess alkali;

N_1 = normality of standard sodium hydroxide solution; and

M = mass, in g, of the sample in 10 ml of the prepared sample solution from A-3.1 taken for the test.

ANNEX B

[*Table 1, S1 No.* (iv)]

DETERMINATION OF REDUCING COMPOUNDS (OTHER THAN SODIUM SULPHIDE AND SODIUM HYDROSULPHIDE)

B-1 REAGENTS

B-1.1 Glycerine Water — approximately 5 percent (*m*/*v*)

B-1.2 Sodium Carbonate Solution — 10 percent (*m*/*v*)

B-1.3 Zinc Sulphate Heptahydrate ($ZnSO_4.7H_2O$) Solution — 40 percent (*m*/*v*)

B-1.4 Zinc Carbonate Suspension

Add 200 ml of sodium carbonate solution to 100 ml of zinc sulphate heptahydrate solution and dilute with previously boiled and cooled water to 400 ml. The suspension shall be freshly prepared at a week's interval.

B-1.5 Standard Hydrochloric Acid — approximately 1 N

B-1.6 Standard Iodine Solution — approximately 0.1 N

B-1.7 Standard Sodium Thiosulphate Solution — 0.1 N

B-2 PROCEDURE

Transfer accurately 10 ml of prepared sample solution from A-3.1 and add 100 ml of glycerine water and 40 ml of zinc carbonate suspension to precipitate the sulphide. Allow to stand for half an hour. Filter and wash the precipitates; collect the filtrate and washing in a 250 ml iodine flask, and to this add 10 ml of standard iodine solution and 50 ml diluted hydrochloric acid solution. Titrate the excess of iodine with standard thiosulphate solution. Similarly carry out a blank with same quantity of reagent and iodine solution.

B-3 CALCULATION

Reducing compound (as $Na_2S_2O_3$), percent by mass

$$= \frac{(V_1 - V_2) \times N \times 158.1 \times 5}{M}$$

where

V_1 = volume, in ml, of standard thiosulphate solution consumed for blank titration;

V_2 = volume, in ml, of standard thiosulphate solution required for titration of sample;

N = normality of standard sodium thiosulphate solution; and

M = mass, in g, of sample in 10 ml of the prepared sample solution from A-3.1 taken for the test.

ANNEX C

[*Table* 1, *Sl No.* (v)]

DETERMINATION OF IRON AND ALUMINUM COMPOUNDS

C-1 REAGENTS

C-1.1 Concentrated Hydrochloric Acid — *see* IS 265

C-1.2 Dilute Nitric Acid — 1 : 1 (*v*/*v*)

C-1.3 Bromine — potassium bromide solution

Dissolve 160 g of potassium bromide in the minimum quantity of water. Add 200 ml of bromine to it, mix the solution thoroughly and dilute to 1 000 ml.

C-1.4 Ammonium Hydroxide — 20 percent (*m*/*m*)

C-2 PROCEDURE

Weigh accurately about 10 g of the material into a 250 ml flask. Add 50 ml of concentrated hydrochloric acid and boil off the hydrogen sulphide. Filter, if necessary. Add a few milliliters of dilute nitric acid and then add bromine-potassium bromide solution to clear the solution from sulphur an doxidize the iron. Boil off bromine, make alkaline with ammonia and boil till vapours do not smell of ammonia and keep for two minutes. Filter, wash the precipitate and ignite at about 1 200 °C. Cool and weigh. Repeat the operation till constant mass is obtained.

C-3 CALCULATION

Express the mass of the residue as percentage of the material taken for the best.

C-4 Alternative Method

Iron and aluminium may alternatively be determined by instrumental test method as prescribed at Annex H or ICP-MS method as prescribed in IS 3025 (Part 65).

Instrumental test method using ICP-OES or ICP-MS shall be used as referee method in case of any dispute.

ANNEX D

[*Table* 1, *Sl No.* (vi)]

DETERMINATION OF SODIUM SULPHATE

D-1 REAGENTS

D-1.1 Concentrated Hydrochloric Acid — *see* IS 265

D-1.2 Barium Chloride Solution — approximately 10 percent (*m*/*v*)

D-2 PROCEDURE

Weigh accurately about 5 g of the material into a 400 ml flask. Add 25 ml of concentrated hydrochloric acid and boil off the hydrogen sulphide. Filter, and to the boiling filtrate and washings add drop by drop about 20 ml of hot barium chloride solution. Boil for 15 min and then allow to stand for 4 h. Filter through a tared sintered glass crucible No. G-4. Wash the precipitate thoroughly with hot water till it is free from chlorides, and dry to constant mass at 105 °C to 110 °C.

NOTE — Excess of barium chloride is necessary to reduce the volubility of barium sulphate. Precipitation in hot solution by addition of barium chloride solution in a slow stream with stirring minimizes mechanical occlusion of barium chloride and gives a coarse precipitate which is less soluble in acids.

D-3 CALCULATION

Sodium sulphate (as Na_2SO_4), percent by mass

$$= 60.86 \times \frac{M_1}{M_2}$$

where

M_1= mass, in g, of barium sulphate precipitate; and;

M_2 = mass, in g, of the material taken for the test.

ANNEX E

[*Table* 1, *S1 No.* (vii)]

DETERMINATION OF SODIUM CHLORIDE

E-1 REAGENTS

E-1.1 Dilute Nitric Acid — approximately 4 N

E-1.2 Chromic Acid

E-1.3 Silver Nitrate Solution — approximately 5 percent (*m*/*v*)

E-2 PROCEDURE

Weigh accurately about 5 g of the material and dissolve in 25 ml of water in a beaker. Add to the solution 30 ml of dilute nitric acid and heat to expel the hydrogen sulphide and other gases. Filter, if necessary. Add few drops of chromic acid to the filtrate. Heat the solution to 50 °C and add to the hot solution sufficient volume of silver nitrate solution to precipitate the chloride completely. Protect the silver chloride precipitate from light by wrapping black paper around the container. Cool the solution, filter through a tared Gooch crucible or a sintered glass crucible no. G-4 and wash the precipitate with cold water. Dry the crucible and its contents to constant mass at (130 ± 2) °C.

E-3 CALCULATION

Sodium chloride (as NaCl), percent by mass

$$= 40.78 \times \frac{A}{M}$$

where

A = mass, in g, of silver chloride precipitate; and

M = mass, in g, of the material taken for the test.

ANNEX F

[*Table* 1, *Sl No.* (viii)]

DETERMINATION OF WATER INSOLUBLES (OTHER THAN IRON AND ALUMINUM COMPOUNDS)

F-1 PROCEDURE

Weigh accurately about 5 g of the sample and dissolve in about 150 ml of water. Warm the solution and filter through a weighed sintered glass crucible no. G-4. Wash till the filtrate is colourless. Dry the insoluble at a temperature of 105 °C to 110 °C till constant mass is obtained. Deduct the amount of iron and aluminum as determined in **C-2**.

F-2 CALCULATION

Water insoluble (other than iron and aluminum compounds) percent by mass $= 100 \times \frac{M_1 A}{M}$

where

M_1 = mass, in g, of the residue obtained;

A = percent of iron and aluminum compounds as determined in **C-2**; and

M = mass, in g, of the material taken for the test.

ANNEX G

[*Table* 1, *Sl No.* (iii)]

DETERMINATION OF EXCESS ALKALI (ALTERNATIVE METHOD)

G-1 APPARATUS

The apparatus, as assembled, shall be as shown in Fig. 1.

G-2 REAGENTS

G-2.1 Barium Hydroxide Solution — saturated

G-2.2 Standard Hydrochloric Acid — 0.1 N

G-2.3 Standard Sodium Hydroxide Solution — 0.1 N

G-2.4 Methyl Orange Indicator Solution

Dissolve 0.05 g of methyl orange in 100 ml of water.

G-2.5 Chromic Acid — 25 percent

G-3 PROCEDURE

Take about 5 g of the material, accurately weighed in the decomposition flask *C*. The flask is closed by inserting the funnel tube *B*, fitted with tube connecting to soda lime bottle *A* and connected by means of a condenser *D* to the train for removing impurities from carbon dioxide, leading to the absorption bottle *E* which is of 125 ml capacity and is filled with 50 ml of clear saturated barium hydroxide solution.

Sweep out the apparatus with a current of dry purified air before attaching the absorption bottle. This is accomplished by applying gentle suction at the end of the purifying train. Fill the acid funnel with 50 ml of 25 percent chromic acid, the stop-cock B_1, being closed. Allow the acid in *B* to run slowly on the sample at a rate that evolves gas not too rapidly to be absorbed. Close the stopcock B_1 retaining 1 to 2 ml of acid in *B* to act as a seal. When the action has ceased, heat the solution in C to boiling. Then apply gentle suction to the absorption end of the apparatus and open the stopcock B_1, allowing the remainder of the acid to flow into the flask Canal admitting a current of airpurified by passing through the soda lime in *A*. The suction should be gentle at first and then the speed of the flow increased to the fill capacity of the absorption bottle.

From the absorption bottle, transfer the contents completely to a beaker. Filter the precipitate and wash with warm water till the filtrate is free from alkali. Dissolve the precipitate in 50 ml of standard 0.1 N hydrochloric acid, wash with water and titrate the excess acid against standard 0.1 N sodium hydroxide solution using methyl orange as indicator. Conduct a blank titration of 50 ml of standard 0.1 N hydrochloric acid against standard 0.1 N sodium hydroxide solution using the same indicator.

G-4 CALCULATION

Calculate on the basis that 1 ml of 0.1 N hydrochloric acid is equivalent to 0.005 3 g of sodium carbonate.

Excess alkali (as Na_2CO_3), percent by mass

$$= \frac{(V - V_1) \times N \times 0.053 \times 100}{M}$$

where,

V = volume, in ml, of standard hydrochloric acid consumed by barium carbonate;

V_1 = volume, in ml, of hydrochloric acid consumed by in blank titration;

N = normality of standard hydrochloric acid; and

M = mass, in g, of the material taken for the test.

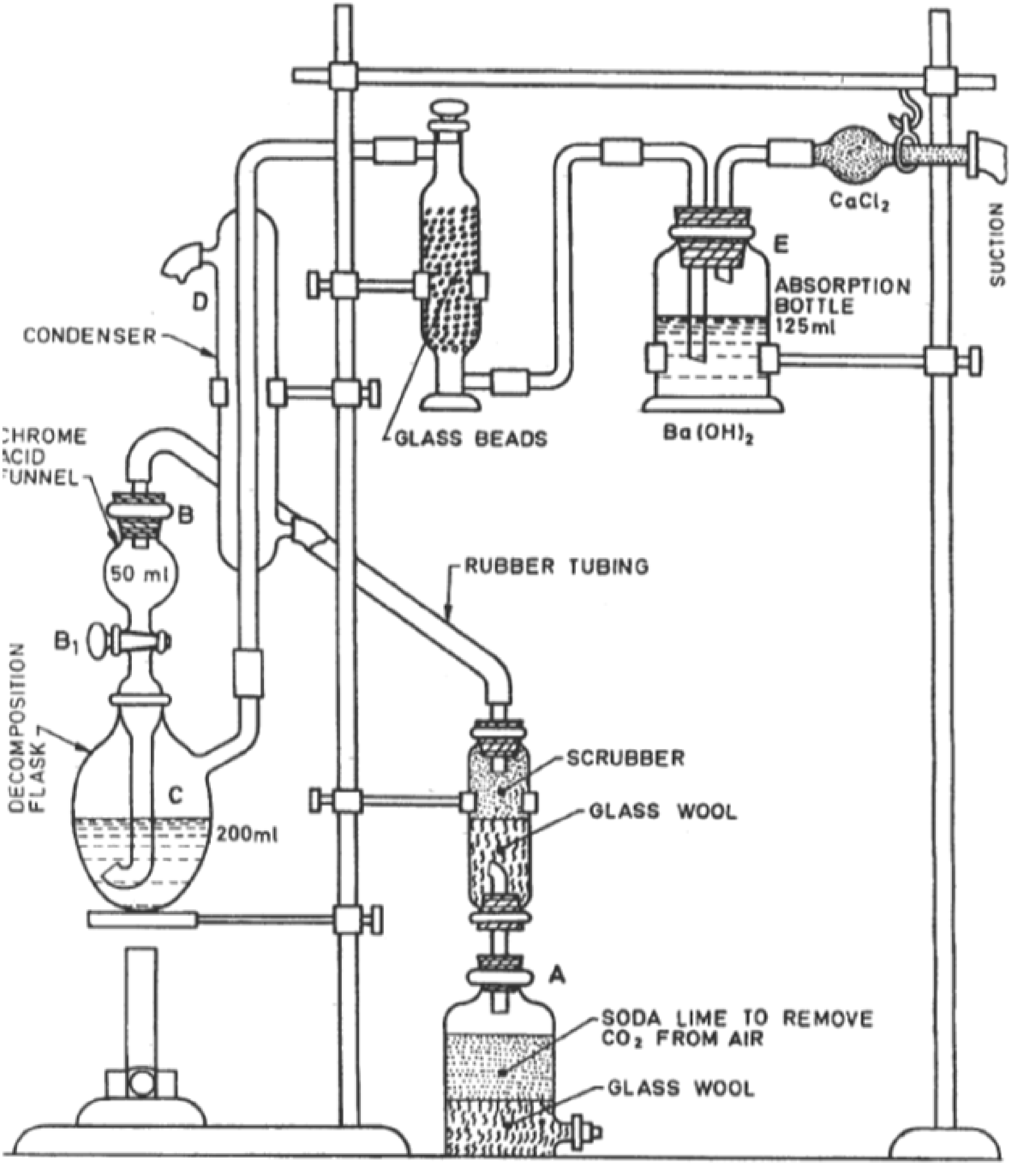

FIG. 1 APPARATUS FOR DETERMINATION OF EXCESS ALKALI

ANNEX H

[*Table 1, Sl No.* (v)]

DETERMINATION OF ALUMINIUM AND IRON BY INDUCTIVELY COUPLED PLASMA OPTICAL EMISSION SPECTROMETER (ICP-OES) METHOD

H-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.

H-1.1 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.

H-1.2 Reagents and Solutions

H-1.2.1 *Nitric Acid* (*65 Percent*) *Suprapure*

H-1.2.2 *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, calcium, magnesium, manganese, arsenic, molybdenum, aluminium and mercury in 2 percent to 5 percent nitric acid. It is preferable to prepare single stock solution of multi elemental standards for analysis.

H-1.2.3 *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.

H-1.2.4 *Sample Preparation*

Weigh about 2.5 g of the sample in a 50 ml volumetric flask and add 1 ml nitric acid and make up the volume with water.

NOTE — Sample should be clear before injecting to the instrument.

H-1.2.5 *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, polytetrafluoroethylene (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.

H-1.3 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.

For analysis of mercury and arsenic, a gas (hydride)/vapour generating system is coupled with ICP, instead of using of nebulizer. The mercury vapour/arsine generated through the system is carried by the carrier gas (Ar) to 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.

H-1.4 Procedure

H-1.4.1 *Calibration*

Profile and calibrate the instrument according to the instrument manufacturer’s recommended procedures, using the intermediate mixed standard solutions (**H-1.2.3**). 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.

H-1.4.2 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 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 aluminium (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.

H-1.4.3 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).

Table 2 Recommended Wavelengths, Achievable Limits of Quantification for Different Configuration of Instruments and Important Spectral Interferences

(*Clauses* H-1.1 *and* H-1.3)

Sl No.	Element	Wavelength (nm)	Approximately Achievable Limits		Interfering Elements
			Radial Viewing (µg)	Axial Viewing (µg)	
(1)	(2)	(3)	(4)	(5)	(6)
i)	Fe	238.204	14	(3)	Co
		259.940	6	2	Co
		271.441	–	–	–
ii)	Al	167.079	1	2	Fe, Pb
		308.215	100	17	Fe, Mn, OH, V
		396.152	10	6	Cu, Fe, Mo, Zr

ANNEX J

(*Clause* 5)

SAMPLING OF SODIUM SULPHIDE, TECHNICAL

J-1 GENERAL REQUIREMENTS OF SAMPLING

J-1.1 In drawing, preparing, storing and handling the samples, the following precautions shall be observed.

J-1.2 Samples shall not be taken at a place exposed to the adverse effects of weather.

J-1.3 The sampling instrument and sample containers shall be clean and dry.

J-1.4 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination particularly from absorption of or contact with water and contact with, oxygen and oxidizing agents.

J-1.5 Before taking the sample, the contents of the selected drums shall be thoroughly mixed.

J-1.6 After filling, each sample container shall be sealed air-tight with a suitable stopper and marked with the relevant particulars.

J-1.7 The material being caustic, particular care shall be taken in its handling.

J-2 SCALE OF SAMPLING

J-2.1 Lot

All the drums in a consignment of the material drawn from a single batch of manufacture shall constitute a lot. Each lot shall be tested separately for all the requirements of this specification.

J-2.2 The number of drums to be selected at random from a lot depends on the size of the lot and shall be as given in Table 3.

J-3 PREPARATION OF TEST SAMPLES

J-3.1 Draw with an appropriate sampling instrument about 100 g of sodium sulphide from different parts of each of the selected drums. These samples are termed as individual samples representing the selected drums. These individual samples shall be kept indifferent sample containers which shall be marked with full details of sampling.

J-3.2 Equal quantities from each of the individual samples shall be taken and mixed together to form a composite sample weighing approximately 200 g.

J-4 NUMBER OF TESTS

J-4.1 Tests for the determination of total sulphides, sodium hydrosulphide and excess alkali shall be conducted on each of the individual samples.

J-4.2 Tests for the remaining characteristics given in Table 1 shall be conducted on the composite sample.

J-5 CRITERIA FOR CONFORMITY

J-5.1 For Individual Samples

From the test results for total sulphides, sodium hydrosulphide and excess alkali as percent by mass, the arithmetic mean ($\bar{X}$) and range (R) shall be computed (range is defined as the difference between the maximum and the minimum values of the test results).

The lot shall be declared as conforming to the requirements of total sulphides if the value of the expression ($\bar{X} - 0.6\ R$) is greater than or equal to 60.0. In the case of sodium hydrosulphide and excess alkali, the lot shall be declared as conforming to the requirements if ($\bar{X} + 0.6\ R$) as calculated from the relevant test results is less than or equal to 2.5 and 2.0 respectively.

J-5.2 For Composite Sample

For declaring the conformity of the lot to the requirements of all other characteristics, the composite sample shall satisfy the relevant requirements specified in col (3) of Table 1.

Table 3 Scale of Sampling

(*Clause* J -2.2)

Sl No. (1)	Lot Size (2)	Number of Drums to be Selected (3)
i)	Up to 25	3
ii)	26 to 50	4
iii)	51 to 100	5
iv)	101 to 200	6
v)	201 and above	8

ANNEX K

(*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 Manufacturers Association of India, Delhi	Shri K. Srinivasan Shri H. S. Das (*Alternate*)
Bhabha Atomic Research Centre, Mumbai	Dr A. V. R. Reddy Dr S. N. Achary (*Alternate*)
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Grasim Industries Ltd, Nagda	Shri Alok Singh Shri Pankaj Gupta (*Alternate*)
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Hindalco, Mumbai	Shri Nageswar Kapuri Shri Ajith Ramachandra (*Alternate*)
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Indian Institute of Chemical Technology, Hyderabad	Dr Praveen R. Likhar Dr Rajender Reddy (*Alternate*)
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Ministry of Chemicals & Fertilizers, New Delhi	Dr Rohit Misra Dr O. P. Sharma (*Alternate*)
Ministry of Defence (DGQA), Kanpur	Shri R. N. Aparajit
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Headquarters:

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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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