

भारतीय मानक मसौदा
सोडियम धातु— विशिष्टि
(IS 8814 का पहला पुनरीक्षण)

Draft Indian Standard
Sodium Metal — Specification
(*First Revision of IS 8814*)

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ICS 71.060.50

Inorganic Chemicals Sectional Committee, CHD 01

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Inorganic Chemicals Sectional Committee, CHD 01

FOREWORD

(Formal clauses will be added later)

This standard was first published in 1978. In this first revision, instrumental test methods for the determination of iron and calcium have been added as alternate test methods. In addition to this, editorial corrections have been made wherever required. Also, Reference clause has been incorporated and Packing & Marking clause has been updated.

Sodium metal is used in the production of tetraethyl lead, sodium methoxide, sodium ethoxide, fatty alcohols, ethyl acetoacetic acid, in dye intermediates and pharmaceutical preparations.

Sodium metal is also used in fast breeding reactor plants. The requirements of this material are quite stringent. The commercial grade has to be specially purified before feeding the same in the circulating system of the reactor. The reactor grade of the material used in fast breeding atomic reactors is not covered in this standard.

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.

Draft Indian Standard
SODIUM METAL — SPECIFICATION
(First Revision)

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for sodium metal for commercial use.

2 REFERENCES

The standards listed in Annex A 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.

3 REQUIREMENTS**3.1 Description**

The material shall be yellowish coloured lumps having inside silver white matter, free from visible foreign matter.

3.2 The material shall comply with the requirements prescribed in Table 1, when tested according to the methods prescribed in Annex B. Reference to the relevant clauses of Annex B is given in col (4) of the table 1.

Table 1 Requirement for Sodium Metal

(Clause 3.2)

SI No.	Characteristic	Requirement	Method of Test. Ref to Cl No. In Annex B
(1)	(2)	(3)	(4)
i)	Total alkali metal content (as Na), percent by mass, <i>Min</i>	99.3	B-2
ii)	Potassium, percent by mass, <i>Max</i>	0.25	B-3
iii)	Iron, percent by mass, <i>Max</i>	0.005	B-4
iv)	Calcium, percent by mass, <i>Max</i>	0.2	B-5

4 PACKING AND MARKING**4.1 Packing**

The sodium bricks are completely coated with high flash point liquid paraffin oil. The material shall then be placed in air-tight steel containers. The containers shall contain sodium up to 80 percent of their volume in a stream of inert gas (nitrogen or argon) free from oxygen and moisture.

4.2 Marking

The container shall be legibly and indelibly marked with the following information:

- a) Name and description (technical grade) of the material;
- b) Name of the manufacturer and his recognized trade-mark, if any;
- c) Net mass of the material;
- d) Date of manufacture; and
- e) Batch number

4.2.1 The following words shall also appear on the container:

‘CAUTION — INFLAMMABLE. EXPLODES ON CONTACT WITH WATER’

4.2.2 *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 thereunder, 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 method of finding out the criteria of conformity of the material to the requirements of this specification shall be as prescribed in Annex C.

ANNEX A

(Clause 2)

LIST OF REFERRED STANDARDS

<i>IS No.</i>	<i>Title</i>
IS 264 : 2005	Nitric acid — Specification (<i>third revision</i>)
IS 265 : 2021	Hydrochloric acid — Specification (<i>fifth revision</i>)
IS 266 : 2024	Sulphuric acid — Specification (<i>fourth revision</i>)
IS 323 : 2009	Rectified spirit for industrial use — Specification (<i>second revision</i>)
IS 695 : 2020	Acetic acid — Specification (<i>fourth revision</i>)
IS 1070 : 2023	Reagent grade water — Specification (<i>fourth revision</i>)
IS 4905 : 2015/ISO 24153 : 2009	Random sampling and randomization procedures (<i>first revision</i>)

ANNEX B

(Clause 3.2, and Table1)

METHODS OF TEST

B-1 QUALITY OF REAGENTS

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

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

B-2 TOTAL ALKALI METAL CONTENT**B-2.1 Reagents**

B-2.1.1 *Rectified Spirit* — *see* IS 323

B-2.1.2 *Standard Hydrochloric Acid* — 0.1 N

B-2.1.3 *Bromophenol Indicator Solution*

Dissolve 0.01 g of bromophenol blue in 100 ml of rectified spirit.

B-2.2 Procedure

B-2.2.1 Remove a piece of the sodium metal from the jar with a pair of forceps, remove the oil from its surface, quickly cut about 2.5 g of sodium out of the middle of the piece, place it in dry tarred weighing bottle and weigh to the nearest 0.000 2 g.

B-2.2.2 Transfer the weighed piece of metallic sodium with a pair of forceps into a 200 ml to 300 ml porcelain cup or beaker, containing 60 ml of rectified spirit. Cover the cup or beaker with a watch glass.

B-2.2.3 After the metallic sodium has dissolved, add 30 ml to 40 ml of water. Transfer the resulting solution quantitatively into a 1 litre volumetric flask and make up to the mark with water. Use the solution for determining the alkalinity in **B-2.2.4** and iron content in **B-4**.

B-2.2.4 Pipette 25 ml aliquot of the solution prepared in **B-2.2.3** into a conical flask and titrates with standard hydrochloric acid using bromophenol blue indicator solution. One milliliter of hydrochloric acid is equivalent to 0.002 3 g of metallic sodium.

B-2.3 Calculation

$$\text{Total alkali metal content (as Na), percent by mass} = \frac{V \times 0.0023 \times 1000 \times 100}{25 \times M}$$

where

V = volume in ml, of standard hydrochloric acid used in the titration; and
 M = mass in g, of the material taken for the test.

B-3 POTASSIUM CONTENT**B-3.1 Reagents**

B-3.1.1 Rectified Spirit — see IS 323

B-3.1.2 Perchloric Acid — 40 to 60 percent

B-3.1.3 Concentrated Hydrochloric Acid — see IS 265

B-3.1.4 Phenolphthalein Indicator Solution — dissolve 0.1 g of the material in 100 ml of 60 percent rectified spirit.

B-3.1.5 Acetic Acid — glacial (see IS 695) and 20 percent solution.

B-3.1.6 Sodium Cobaltinitrite Solution

Dissolve 50 g of sodium cobaltinitrite and 25 g of sodium acetate in 250 ml of water. Allow the solution to stand for 24 h and then filter.

B-3.2 Procedure

B-3.2.1 Dissolve about 2 g of the material in 60 ml of rectified spirit. Filter the solution, transfer it to a cylinder and add 2 ml of perchloric acid. This should not cause the appearance of a white precipitate. If there is a precipitate, determine potassium as follows.

B-3.2.1.1 Place about 2 g of the material weighed to the nearest 0.000 2 g into a 100 ml evaporating dish and dissolve it in 60 ml of rectified spirit. Neutralize the solution with glacial acetic acid or hydrochloric acid in the presence of phenolphthalein indicator solution and evaporate on a water-bath almost to dryness, stirring periodically to keep the deposit from adhering to the bottom of the dish. Dissolve the deposit in 10 ml of water, add 0.5 g of 20 percent acetic acid and drop by drop (in the course of 5 min), while stirring, 10 ml of the sodium cobaltinitrite solution, and allow to stand for 3 hours. Then filter through filter crucible previously washed, dried at a temperature of 100 °C to 110 °C to constant mass and weighed to the nearest 0.000 2 g

B-3.2.1.2 Wash the residue on the filter three times with 20 percent acetic acid using 5 ml each time, and twice with rectified spirit, using 5 ml each time, dry at a temperature of 100 °C to 110 °C to constant mass, cool in a desiccator and weigh to the nearest 0.0002 g. One gram of a precipitate of the composition $K_2NaCo(NO_2)_6 \cdot H_2O$ contains 0.172 1 g of potassium.

B-3.3 Calculation

$$\text{Potassium, percent by mass} = \frac{M_1 \times 0.1721 \times 100}{M}$$

where

M_1 = mass in g, of the dried residue; and
 M = mass in g, of the material taken for the test.

B-4 IRON**B-4.1 General**

Three methods are prescribed for determining iron, namely, Method A, ICP-OES method as prescribed at **B-6** and ICP-MS method as prescribed in IS 3025 (Part 65). In case of dispute, ICP-MS shall be used as referee method.

B-4.2 Method A**B-4.2.1 Reagents****B-4.2.1.1** *Rectified spirit* — see IS 323**B-4.2.1.2** *Concentrated hydrochloric acid* — see IS 265**B-4.2.1.3** *Phenolphthalein indicator solution* — dissolve 0.1 g of phenolphthalein in 100 ml of 60 percent rectified spirit.**B-4.2.1.4** *Concentrated nitric acid* — see IS 264**B-4.2.1.5** *Concentrated sulphuric acid* — see IS 266**B-4.2.1.6** *Dilute sulphuric acid* — 4 N**B-4.2.1.7** *Ammonium thiocyanate solution* — 10 percent**B-4.2.1.8** *Isoamyl alcohol***B-4.2.1.9** *Standard iron solution* — 1 ml of the solution containing 0.01 mg of iron (as Fe).**B-4.2.2 Procedure**

Measure 25 ml aliquot of the solution, prepared as in **B-2.2.3** into a beaker. Neutralize the solution with concentrated hydrochloric acid to phenolphthalein, add 0.5 ml of nitric acid and heat to boiling, cool and transfer quantitatively into a cylinder with a ground-glass stopper.

B-4.2.2.1 Add to the solution 0.5 ml of dilute sulphuric acid, 5 ml of ammonium thiocyanate solution and 10 ml of isoamyl alcohol. Dilute the solution in the cylinder with water to make up 50 ml. Measure 33 ml of water, 0.5 ml of dilute sulphuric acid, 5 ml of ammonium thiocyanate solution and 10 ml of isoamyl alcohol into a similar cylinder. Then add standard iron solution to the contents of the second cylinder, drop by drop, from a microburette until the colour of the alcoholic layer is the same as the colour of the alcoholic layer of the solution under test contained in the first cylinder.

B-4.2.3 Calculation

$$\text{Iron, percent by mass} = \frac{V \times 1\,000 \times 0.000\,01 \times 100}{25 \times M}$$

where

V = volume in ml, of standard iron solution required to equalize the colour; and
 M = mass in g, of the material taken for the test.

B-5 CALCIUM**B-5.1 General**

Three methods are prescribed for determining calcium, namely, Method A, ICP-OES method as prescribed at **B-6** and ICP-MS method as prescribed in IS 3025 (Part 65). In case of dispute, ICP-MS shall be used as referee method.

B-5.2 Method A**B-5.2.1 Reagents****B-5.2.1.1** *Rectified spirit* — see IS 323**B-5.2.1.2** *Dilute hydrochloric acid* — 1: 1 (v/v)**B-5.2.1.3** *Ammonium chloride solution* — 20 percent (m/v)**B-5.2.1.4** *Ammonium hydroxide* — 20 percent (m/m)**B-5.2.1.5** *Magnesium chloride* — solid

B-5.2.1.6 *Sodium salt of ethylene diaminetetraacetic acid (EDTA) — 0.02 N solution*

Dissolve 4 g of EDTA in 500 ml of water, add 0.1 g of magnesium chloride, mix and add water to the mark in a 1 000 ml volumetric flask.

B-5.2.1.7 *Buffer solution*

Mix 100 ml of the ammonium chloride solution and 100 ml of ammonium hydroxide in a 1 000 ml volumetric flask and make up to the mark with water.

B-5.2.1.8 *α -naphtholphthalein indicator solution — 0.1 g in 100 ml of percent rectified spirit.***B-5.2.1.9** *Hydroxylamine Hydrochloride Solution — 1 percent***B-5.2.1.10** *Acid chrome blue-black indicator*

Dissolve 0.5 g of acid chrome blue-black in alcohol in a 100 ml volumetric flask, then add 2.5 g of hydroxylamine hydrochloride, 0.06 g of α -naphtholphthalein and make up to the mark with alcohol.

B-5.2.1.11 *Magnesium sulphate solution — 0.01 N***B-5.2.1.12** *Congo paper***B-5.2.1.13** *Standardization of EDTA solution*

Measure 10 ml of the prepared magnesium sulphate solution into a conical flask, add 50 ml of water, 10 ml of the buffer solution, 7 to 15 drops of the indicator (depending on the diameter of the pipette) and titrate with the EDTA solution until the colour of the solution changes from cherry-red to blue.

Calculate the correction factor (K) from the formula:

$$K = \frac{10}{V}$$

where

V = volume in ml, of EDTA solution used.

B-5.2.2 *Procedure*

B-5.2.2.1 Dissolve 5 g of the metallic sodium, weighed to the nearest 0.01 g in 100 ml of rectified spirit in a porcelain beaker, adding the spirit, drop by drop.

B-5.2.2.2 When the sodium is fully dissolved dilute the solution with water and neutralize it in the presence of a piece of Congo paper with hydrochloric acid, then evaporates to dryness on a water-bath. Dissolve the residue in water, transfer to a 250 ml volumetric flask and make up to the mark with water while mixing thoroughly.

B-5.2.2.3 Pipette 100 ml of the resulting solution into a 200 ml conical flask, add 50 ml of water, 7 ml of the buffer solution, 7 to 15 drops of acid chrome blue-black indicator and titrate with the EDTA solution until the wine-red colour changes to blue-violet, and the latter colour does not change on adding one or two drops of the EDTA solution. Towards the end of titration add the EDTA solution slowly, drop by drop, stirring intensively. One ml of 0.02 N EDTA solution is equivalent to 0.000 4 g of calcium.

B-5.2.3 *Calculation*

$$\text{Calcium, percent by mass} = \frac{V \times 0.000\ 4 \times 250 \times 100}{M \times 100}$$

where

V = volume in ml, of EDTA solution used in the titration; and

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

B-6 DETERMINATION OF CALCIUM AND IRON BY INDUCTIVELY COUPLED PLASMA OPTICAL EMISSION SPECTROMETER (ICP-OES) METHOD**B-6.1 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.

B-6.2 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.

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

(Clause B-6.2 and B-6.4)

SI 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)	Ca	315.887	100	13	Co, Mo
		317.933	26	4	Fe, V
		393.366	0.4	25	V, Zr
		422.673	-	-	V, Mo, Zr

B-6.3 Reagents and Solutions**B-6.3.1. Nitric Acid (65 percent) Suprapure****B-6.3.2 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, 100 or 1000 $\mu\text{g}/\text{ml}$ of Lead, Iron, calcium, magnesium, manganese, arsenic, molybdenum, aluminium and mercury in 2 to 5 percent nitric acid. It is preferable to prepare single stock solution of multi elemental standards for analysis.

B-6.3.3 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, 3.0 and 5.0 ml taken in 50 ml volumetric flasks (separate) and make up volume with 2 percent nitric acid to prepare 0.1, 0.3 and 0.5 µg/ml solution of respective elements under reference.

B-6.3.4 Sample Preparation

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

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

B-6.3.5 Reagent Blank Solution

Place 50 ml of nitric acid and 1000 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.

B-6.4 Instrument

Set up the instrument as per the manufacturer's instructions manual for recommended operating parameters, based on the manufacturer's 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.

B-6.5 Procedure**B-6.5.1 Calibration**

Profile and calibrate the instrument according to the instrument manufacturer's recommended procedures, using the intermediate mixed standard solutions (B-6.3.5). 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.

B-6.5.2 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 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 manganese, arsenic) 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.

B-6.6 Calculation

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

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

- ii) 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 C

(Clause 5)

SAMPLING

C-1 GENERAL REQUIREMENTS OF SAMPLING

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

C-1.2 Samples shall not be drawn in an exposed place,

C-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.

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

C-1.5 The samples shall be placed in clean, dry, air-tight glass or other suitable containers which have no action on the material.

C-1.6 The sample containers shall be of such a size that they are almost completely filled by the sample.

C-1.7 Each sample container shall be sealed air-tight with a stopper after filling, and marked with all particulars of the material and the date of sampling.

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

C-2 SCALE OF SAMPLING

C-2.1 Lot

All containers, in a single consignment, of the same size and belonging to the same batch of manufacture shall be grouped together to constitute a lot.

C-2.2 For ascertaining the conformity of the lot to the requirements of this specification, samples shall be tested from each lot separately.

C-2.3 The number of containers to be selected for this purpose, shall depend on the size of the lot and shall be according to Table 3.

Table 3 Scale of Sampling

(Clause C-2.3)

SI No.	Lot Size	No. of Containers To Be Selected
	N	n
(1)	(2)	(3)
i)	Up to 50	5

ii)	51 to 100	6
iii)	101 to 300	7
iv)	301 to 500	8
v)	501 and above	9

C-2.3.1 These containers shall be selected at random from the lot. In order to ensure the randomness of selection, a random number table shall be used. For guidance and use of random number tables IS 4905 may be referred. In the absence of a random number table, following procedure may be adopted:

Starting from any container in the lot, count them as 1, 2, 3,..... and so on up to r where r is the integral part of N/n (N being lot size and n the sample size). Every r^{th} container so counted shall be withdrawn so as to constitute the required sample size.

C-3 NUMBER OF TESTS AND CRITERIA FOR CONFORMITY

C-3.1 Each of the containers selected according to **C-1.4** shall be tested for all the requirements given in **3** of the specification. For this purpose sufficient quantity of material shall be taken from each selected container so as to make triplicate determinations for all the characteristics given in the specification. The quantity of material so selected from each container shall be divided into three equal parts, one for the purchaser, another for the supplier and the third for the referee. Each of these parts of material shall constitute an individual test sample corresponding to the container selected.

C-3.2 The referee samples, bearing the seals of both the purchaser and the supplier shall be kept at a place agreed to between the two. These shall be used in case of any dispute between the two.

C-3.3 The lot shall be declared as conforming to the requirements of this specification if each of the test results on the individual samples satisfies the corresponding requirement given in the specification, otherwise not.