भारतीय मानक
Indian Standard

IS 4505 : 2015

सोडियम फॉर्मेल्डीहाइड सलफोक्सीलेट — विशिष्टि

(पहला पुनरीक्षण)

Sodium Formaldehyde Sulphoxylate — Specification

(First Revision)

ICS 71.060.50

भारतीय मानक ब्यूरो
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Inorganic Chemicals Sectional Committee, CHD 01

FOREWORD

This Indian Standard (First 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.

The specification for sodium formaldehyde sulphoxylate was first published in 1968. Sodium formaldehyde sulphoxylate ($NaHSO_2.CH_2O.2H_2O$) a derivative of sodium hydrosulphite and formaldehyde, is a sodium salt of sulphoxylic acid formaldehyde, molecular weight 154. It typically contains sodium bisulphite formaldehyde ($NaHSO_3.CH_2O.H_2O$) and sodium sulphite as impurities.

Sodium formaldehyde sulphoxylate is a powerful reducing agent and exerts its full reducing action intensively only at high temperatures. Due to its specific characteristic in differing from its allied product sodium hydrosulphite, sodium formaldehyde sulphoxylate finds extensive application in textile industry for printing and stripping dyed textiles prior to redying and discharge printing. It is used in bleaching sugarcane juice for making jaggery, as a redox catalyst in emulsion of polymers and as a stabilizer/antioxidant in pharmaceutical drug formulations.

Sodium formaldehyde sulphoxylate is manufactured by reacting sulphur dioxide with zinc dust in aqueous suspension to form zinc hydrosulphite solution. Formaldehyde and more zinc dust are added to this solution to form basic zinc formaldehyde sulphoxylate which is subsequently decomposed with caustic soda to form sodium formaldehyde sulphoxylate and insoluble zinc hydroxide.

In this revision, the sodium formaldehyde sulphoxylate has been categorized into two grades based on the application and *p*H requirement has also been revised. Also, the requirements of iron, lead, copper and zinc have been made more stringent. For lead estimation, Atomic Absorption Spectrophotometric method has been adopted as a replacement of colorimetric method.

The composition of the Committee responsible for the formulation of this standard is given 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 : 1960, ‘Rules for rounding off numerical values (*revised*)’. 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 FORMALDEHYDE SULPHOXYLATE — SPECIFICATION
(First Revision)

1 SCOPE

This standard prescribes the requirements and the methods of sampling and tests for sodium formaldehyde sulphoxylate dihydrate.

2 REFERENCES

The standards listed 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 the standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent edition of these standards.

IS No.	*Title*
264 : 2005	Nitric acid — Specification (*third revision*)
265 : 1993	Hydrochloric acid (*fourth revision*)
266 : 1993	Sulphuric acid (*third revision*)
1070 : 1992	Reagent grade water — Specification (*third revision*)
2124 : 2000	Sodium bicarbonate — Specification (*second revision*)
2316 : 1990	Methods of preparation of standard solutions for colorimetric and volumetric analysis (*second revision*)
12074 : 1987	Method for determination of lead by atomic absorption spectrophotometer

3 GRADES

There shall be two grades of material, namely:

a) *Grade* 1 — Discharge agent for textile printing and stripping and bleaching of oils, fats, soaps and jaggery.
b) *Grade* 2 — For polymer and pharma applications.

4 REQUIREMENTS

4.1 Description

Sodium formaldehyde sulphoxylate shall be in the form of white chips. It shall be soluble in water at room temperature forming solution of up to 50 percent concentration giving a clear solution.

4.2 The material is hygroscopic in nature. It is stable for a long period if kept in dry condition and generally emits out a slight characteristic odour akin to formaldehyde. It is unaffected by alkalis but is unstable in acidic conditions.

NOTE — A foul smell emits out if the product deteriorates and the lumps of chips are formed, which later crumbles down to powder form.

4.3 The material shall comply with the requirements laid down in Table 1, when tested according to the methods prescribed in Annex A. Reference to the relevant clauses of Annex A is given in col 5 of Table 1.

5 KEEPING QUALITY

The material, when stored unopened in original containers, shall continue to satisfy the requirements given in Table 1 for at least six months from the date of packing.

6 PACKING AND MARKING

6.1 Packing

The material should be packed in sound, clean and dry air tight mild steel black plate containers of 50 kg capacity with polyethylene liner. The containers shall be stored in cool, dry place away from acids or acidic atmosphere.

6.2 Marking

6.2.1 The containers shall be securely closed and legibly and indelibly marked with the following information:

a) Name of the material;
b) Manufacturer’s name or recognized trade-mark, if any;
c) Mass of the material in the container;
d) Date and year of manufacture; and
e) Lot number in code or otherwise to enable the batch of manufacture to be traced from records.

6.2.2 *BIS Certification Marking*

The containers may also be marked with the Standard Mark.

Table 1 Requirement for Sodium Formaldehyde Sulphoxylate
(*Clauses* 4.3, 5, A-4.3.2, A-5.3.2 *and* A-6.3.1)

Sl No.	Characteristic	Requirement		Method of Test (Ref to IS or Clause No. of Annex A)
		Grade 1	Grade 2	
(1)	(2)	(3)	(4)	(5)
i)	Sodium formaldehyde sulphoxylate dihydrate ($NaHSO_2.CH_2O.2H_2O$), percent by mass, *Min*	97.0	99.0	**A-2**
ii)	Sodium formaldehyde bisulphate ($NaHSO_3.CH_2O.H_2O$), percent by mass, *Max*	4	4	**A-2**
iii)	Sodium sulphite (Na_2SO_3), percent by mass, *Max*	3	3	**A-2**
iv)	*p*H (of 1 percent solution)	9.0 to 11	9.0 to 11	**A-3**
v)	Iron (as Fe), percent by mass, *Max*	0.01	0.001	**A-4**
vi)	Lead (as Pb), percent by mass, *Max*	0.001	0.001	IS 12074
vii)	Copper (as Cu), percent by mass, *Max*	0.001	0.001	**A-5**
viii)	Zinc (as Zn), percent by mass, *Max*	0.01	0.001	**A-6**

The use of the Standard Mark is governed by the provisions of *Bureau of Indian Standards* Act, 1986 and the Rules and Regulations made thereunder. The details of conditions under which the license for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

7 SAMPLING

The method of drawing representative samples of the material, number of tests to be performed and the criteria for conformity of the material to the requirement of this standard shall be as prescribed in Annex B.

ANNEX A

(*Clauses* 4.3 and *Table* 1)

ANALYSIS OF SODIUM FORMALDEHYDE SULPHOXYLATE

A-1 QUALITY OF REAGENTS

A-1.1 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 contain impurities which affect the result of analysis.

A-2 DETERMINATION OF SODIUM FORMALDEHYDE SULPHOXYLATE, SODIUM FORMALDEHYDE BISULPHITE AND SODIUM SULPHITE

A-2.0 Principle

Sodium formaldehyde sulphoxylate reacts with iodine in neutral or acidic conditions, whereas sodium formaldehyde bisulphite does not react:

$$NaHSO_2.CH_2O.2H_2O + 2I_2 \rightarrow NaHSO_4 + HCHO + 4HI$$

Sodium sulphite present in the material is converted to sodium bisulphite formaldehyde by the addition of excess of formaldehyde solution. Sodium bisulphite formaldehyde reacts with iodine in alkaline solution:

$$NaHSO_3.CH_2O.H_2O + 3NaHCO_3 + I_2 \rightarrow Na_2SO_4 + 2NaI + 4H_2O + 3CO_2$$

A-2.1 Reagents

A-2.1.1 *Formaldehyde Solution,* 1: 9 (*v/v*).

A-2.1.2 *Starch Indicator* — Titrate 5 g of starch and 0.01 g of mercuric iodide with 30 ml of cold water and slowly pour it with stirring into one litre of boiling water. Boil for 3 min. Allow the solution to cool and decant off the supernatant clear liquid.

A-2.1.3 *Sodium Bicarbonate, see* IS 2124.

A-2.1.4 *Standard Iodine Solution*, 0.1 N (*see* **52.1** of IS 2316).

A-2.2 Procedure

A-2.2.1 Weigh accurately about 1 g of the material and dissolve it in water and make up the solution to 250 ml with water.

A-2.2.2 Take 25 ml of the solution as prepared in **A-2.2.1** in a conical flask, add 1 ml of starch indicator and titrate with iodine solution (V_1)

A-2.2.3 Take 25 ml of the solution as prepared in **A-2.2.1** in a conical flask. Add 5 ml of formaldehyde solution in water, shake well, add 1 ml of starch indicator solution and titrate with iodine solution (V_2).

A-2.2.4 Take 25 ml of the solution as prepared in **A-2.2.1** in a conical flask, add 1 g of sodium bicarbonate and stir well to dissolve it. Add 1 ml of starch indicator solution and titrate with iodine solution (V_3).

A-2.3 Calculation

$$\text{Sodium formaldehyde sulphoxylate, percent by mass} = \frac{V_2 N \times 38.52}{m}$$

$$\text{Sodium sulphite, percent by mass} = \frac{(V_1 - V_2) N \times 63.03}{m}$$

$$\text{Sodium bisulphite formaldehyde, percent by mass} = \frac{(V_3 - V_1) N \times 76.03}{m}$$

where

V_1 = volume in ml of standard iodine solution consumed in **A-2.2.2**;

V_2 = volume in ml of standard iodine solution consumed in **A-2.2.3**;

V_3 = volume in ml of standard iodine solution consumed in **A-2.2.4**;

N = normality of standard iodine solution used in the titration; 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*

A-3.2 Procedure — Weigh accurately 1 g of the material and dissolve in 20 ml of carbon dioxide free water and make up the volume to 100 ml in a volumetric flask. Determine the *p*H of the solution with the help of *p*H meter.

A-4 DETERMINATION OF IRON

A-4.0 Principle

Iron in ferric state gives a red colour with potassium thiocyanate. The colour produced by the material after oxidation is compared visually with the colour formed by a standard iron solution.

A-4.1 Apparatus

A-4.1.1 *Nessler Tubes,* 50 ml capacity.

A-4.2 Reagents

A-4.2.1 *Concentrated Sulphuric Acid, see* IS 266.

A-4.2.2 *Concentrated Hydrochloric Acid, see* IS 265.

A-4.2.3 *Dilute Hydrochloric Acid,* 5 N.

A-4.2.4 *Ammonium Persulphate*, solid

A-4.2.5 *Butanolic Potassium Thiocyanate Solution* — Dissolve 10 g of potassium thiocyanate in 10 ml of water. Add sufficient *n*-butanol to make up to 100 ml and shake vigorously until the solution is clear.

A-4.2.6 *Standard Iron Solution (A)* — Dissolve 0.702 0 g of ferrous ammonium sulphate [$FeSO_4 (NH_4)_2SO_4.6H_2O$] in 10 ml of dilute sulphuric acid (10 percent *v/v*) and dilute with water to 1 000 ml. Pipette out 10 ml of this solution and dilute with water to make up the volume to 100 ml. One ml of this solution contains 0.01mg of iron (as Fe).

A-4.2.7 *Standard Iron Solution (B)* — Dissolve 0.702 0 g of ferrous ammonium sulphate [$FeSO_4 (NH_4)_2SO_4.6H_2O$] in 10 ml of dilute sulphuric acid (10 percent *v/v*) and dilute with water to 1 000 ml. Pipette out 10 ml of this solution and dilute with water to make up the volume to 1 000 ml. One ml of this solution contains 0.001mg of iron (as Fe).

A-4.3 Procedure — Weigh accurately 1 g of the material and transfer it to an evaporating dish. Add 10 drops of concentrated sulphuric acid to wet the material with acid and ignite until no more fumes are produced and all carbon is oxidized. Heat the residue with 5 ml of concentrated hydrochloric acid and evaporate to dryness on a water bath. Dissolve the residue by warming with 50 ml of dilute hydrochloric acid. Cool and make the volume to 100 ml. Ten ml of this solution contains 0.1 g of the material.

A-4.3.1 Take 10 ml of the solution as prepared in **A-4.3** in a Nessler tube. Add 1 ml of concentrated hydrochloric acid, about 30 mg of ammonium persulphate and 15 ml of butanolic potassium thiocyanate solution. Shake vigorously for 30 s and allow the liquids to separate. Carry out a control test in another Nessler tube using 1 ml of standard iron solution (A) for Grade 1 and 1 ml of standard iron solution (B) for Grade 2.

A-4.3.2 The limit prescribed in Table 1 shall be taken as not having been exceeded if the intensity of red colour produced in the butanolic layer in the test with material is not greater that that produced in the control test.

A-5 DETERMINATION OF COPPER

A-5.0 Principle — Copper is determined colorimetrically by visual comparison in Nessler tubes using sodium diethyldithiocarbamate.

A-5.1 Apparatus

A-5.1.1 *Nessler Tubes*, 50- ml capacity.

A-5.2 Reagents

A-5.2.1 *Citric Acid Solution*, 10 percent (*w/v*).

A-5.2.2 *Gum Arabic Solution*, 1 percent (*w/v*).

A-5.2.3 *Dilute Ammonium Hydroxide*, approximately 5 N.

A-5.2.4 *Concentrated Hydrochloric Acid*, *see* IS 265.

A-5.2.5 *Dilute Hydrochloric Acid,* approximately 10 percent.

A-5.2.6 *Sodium Diethyldithiocarbamate Solution* — Dissolve 0.1 g of sodium diethyldithiocarbamate in 100 ml of water. Filter, if necessary. Preserve in an amber coloured glass bottle and store in dark place. This solution should not be used after two weeks.

A-5.2.7 *Standard Copper Solution* — Dissolve 0.196 4 g of copper sulphate pentahydrate ($CuSO_4.5H_2O$) in 1 000 ml water. Pipette out 10 ml of this solution and dilute to 1 000 ml with water. One ml of this diluted standard solution contains 0.000 5 mg of copper (as Cu). The dilute solution shall be freshly prepared.

A-5.3 Procedure

A-5.3.1 Take 25 ml of the solution of the material as prepared in **A-4.3**. Add 2.5 ml of citric acid solution. Neutralize the excess acid with ammonium hydroxide and add 10 ml in excess. Add 2.5 ml of gum arabic solution followed by 10 ml of sodium diethyldithiocarbamate solution. Make up the volume upto 50-ml mark and mix well. Carry out a control test using 5 ml of standard copper solution, proceeding exactly as for the material under test. Compare the intensity of the color produced in the two tubes.

A-5.3.2 The limit laid down in Table 1 shall be taken as not having been exceeded if the intensity of the colour produced with the material is not greater than that in the control test.

A-6 DETERMINATION OF ZINC

A-6.0 Principle

Zinc is determined colorimetrically by visual comparison in flat-bottom glass-stoppered tube using dithizone (diphenylthiocarbazone).

A-6.1 Apparatus

A-6.1.1 *Flat-Bottom Glass-Stoppered Tube for Colour Comparison,* 50-ml capacity.

A-6.1.2 *p*H *Meter*

A-6.2 Reagents

A-6.2.1 *Dithizone* (*Diphenyl Thiocarbazone*) *Solution*, 0.001 percent (*w/v*) in carbon tetrachloride.

A-6.2.2 *Acetate Buffer Solution, p*H approximately 4.75.

Mix equal volumes of 2 N sodium acetate and 2 N acetic acid and remove reacting heavy metals by shaking with dithizone solution. Filter through a small quantitative filter paper to remove droplets of carbon tetrachloride.

A-6.2.3 *Sodium Thiosulphate Solution*, 25 percent (*w/v*).

A-6.2.4 *Standard Zinc Solution (A)* — Weigh accurately 1 g of granulated zinc and dissolve in 7 ml of 5 N hydrochloric acid and dilute with water to 1 000 ml. Take 1 ml of this solution and dilute it further to 100 ml. One ml of this solution is equivalent to 0.01 mg of zinc.

A-6.2.5 *Standard Zinc Solution (B)* — Weigh accurately 1 g of granulated zinc and dissolve in 7 ml of 5 N hydrochloric acid and dilute with water to 1 000 ml. Take 1 ml of this solution and dilute it further to 1 000 ml. One ml of this solution is equivalent to 0.001 mg of zinc.

A-6.3 Procedure

Take 10 ml of the solution of the material as prepared in **A-4.3** in a flat–bottom glass–stoppered tube, add sufficient acetate buffer solution to adjust the *p*H to 4.5 to 4.75, add 1 ml of sodium thiosulphate solution and 2 ml of dithizone solution and shake vigorously for two minutes. Carry out a control test using 1 ml of standard zinc solution (A) for Grade 1 and 1 ml of standard zinc solution (B) for Grade 2, carried through the same steps as for the material and dilute it to the same volume. Compare the intensity of the colour produced in two tubes.

A-6.3.1 The limit prescribed in Table 1 shall be taken as not having been exceeded if the intensity of the colour produced in the test is not greater than that produced in the control test.

ANNEX B

(*Clause* 7)

SAMPLING OF SODIUM FORMALDEHYDE SULPHOXYLATE

B-1 GENERAL REQUIREMENTS OF SAMPLING

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

B-1.1.2 Precautions shall be taken to protect the sample, the material being sampled, the sampling instrument and the container for samples from adventitious contamination.

B-1.1.3 As the material is hygroscopic in nature and is unstable in acidic conditions, the sample and the material being sampled should not be exposed to acid or acidic atmosphere and should be exposed to the atmosphere only to the minimum possible extent which may be absolutely necessary for the purpose of sampling.

B-1.1.4 The samples shall be placed immediately after sampling in clean, dry and air-tight containers made of metal or fibre with polyethylene lining.

B-1.1.5 Each sample container shall be sealed air-tight immediately after filling.

B-1.1.6 On each sample container full details of sampling, such as sample number, the lot number, date of sampling, etc shall be marked so that the identity and source of sample can easily be established.

B-1.1.7 Sampling shall be done in the presence of the buyer and seller or their representatives. Each sample container after being properly filled and closed shall carry the seals and signature of both the parties or their representatives.

B-2 SCALE OF SAMPLING

B-2.1 Lot

In any consignment all the containers of the same size, drawn from the same batch of manufacture shall be grouped together to constitute a lot.

B-2.2 For ascertaining the conformity of the material in the lot to the requirements of this standard, test shall be carried out for each lot separately. The number of containers to be selected for this purpose shall depend on the size of the lot and shall be in accordance with Table 2.

B-2.3 These containers shall be selected at random from the lot. To ensure randomness of selection, a random number table may be used. In case such a table is not available, the following procedure may be adopted:

Starting from any container in the in the lot, count them as 1, 2, 3,….. up to r and so on in one order. Every rth container thus counted shall be withdrawn to constitute the sample, where r is the integral part of N/n, N being the number of containers in the lot and n being the number of containers to be selected.

Table 2 Number of Containers to be Selected for Sampling

(*Clause* B-2.2)

Number of Containers in the Lot N (1)	Number of Containers to be Selected n (2)
Up to 25	2
26 to 50	3
51 to 100	4
101 to 300	5
301 and over	6

B-3 PREPARATION OF SAMPLES

B-3.1 From each of the selected containers, draw with an appropriate sampling instrument a representative portion of the material not less than 300 g. Divide it into three equal parts of about 100 g and transfer each to a clean and moisture-proof sample container which shall be closed air-tight, ensuring that during this whole procedure the material and the sample are exposed to the atmosphere as little as possible and are not exposed at all to acids or acidic atmosphere. Each of these three samples shall be an individual sample representing that particular container.

B-3.2 There shall be three sets of individual samples. Each set shall contain a sample representing each selected container. One of these sets shall be marked for the purchaser, another for the supplier and the third shall be as a referee set.

B-3.3 The referee set consisting of individual samples representing the selected containers shall bear the seals of both the purchaser and the supplier or their representatives. They shall be kept at a place and under conditions agreed to between the parties. The referee set shall be used only in case of dispute.

B-4 NUMBER OF TESTS

B-4.1 Tests for the determination of the following shall be done on each of the individual samples in a set:

a) Sodium formaldehyde sulphoxylate dihydrate,
b) Sodium formaldehyde bisulphite,
c) Sodium sulphite, and

d) *p*H value.

B-4.2 Tests for all other requirements of this standard shall be conducted on the composite sample which shall be formed by mixing together equal quantities of the material from each of the individual samples in a test.

B-5 CRITERIA FOR CONFORMITY

B-5.1 For Individual Samples

The lot shall be declared as conforming to the requirements of sodium formaldehyde sulphoxylate dihydrate, sodium formaldehyde bisulphite, sodium sulphite and *p*H value, if each of the individual samples separately satisfies the relevant requirements.

B-5.2 For Composite Samples

For declaring the conformity of the lot to the requirements of the characteristics tested on the composite sample, the test result for each characteristic shall satisfy the corresponding requirements of this standard.

ANNEX C

(*Foreword*)

COMMITTEE COMPOSITION

Inorganic Chemical Sectional Committee, CHD 01

Organization	*Representative(s)*
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Scientist 'B' (CHD), BIS

Bureau of Indian Standards

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Review of Indian Standards

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Catalogue' and 'Standards : Monthly Additions'.

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Amendments Issued Since Publication

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