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SPECIFICATION FOR SODIUM FORMALDEHYDE SULPHOXYLATE

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Gr 4 *March* 1968

Indian Standard

SPECIFICATION FOR
SODIUM FORMALDEHYDE SULPHOXYLATE

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Indian Standard

SPECIFICATION FOR SODIUM FORMALDEHYDE SULPHOXYLATE

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 29 January 1968, after the draft finalized by the Inorganic Chemicals (Miscellaneous) Sectional Committee had been approved by the Chemical Division Council.

0.2 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 as impurity sodium bisulphite formaldehyde ($NaHSO_3 . CH_2O.H_2O$) as well as sodium sulphite.

0.3 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. It is used in bleaching oils, fats and soaps and also employed as a delayed action coagulant for rubber latex.

0.4 The production of the sodium formaldehyde sulphoxylate has been recently started in the country. It 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.

0.5 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*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

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

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and tests for sodium formaldehyde sulphoxylate dihydrate which is also known commercially as sodium sulphoxylate formaldehyde.

2. REQUIREMENTS

2.1 Description — Sodium formaldehyde sulphoxylate shall be in the form of small white lumps or white granules. It shall be soluble in water at room temperature forming solution of up to 50 percent concentration giving a clear solution.

2.1.1 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 crumble down to powder form.

2.2 The material shall comply with the requirements laid down in Table 1, when tested according to the methods prescribed in Appendix A. Reference to the relevant clauses of Appendix A is given in col 4 of the table.

TABLE 1 REQUIREMENTS FOR SODIUM FORMALDEHYDE SULPHOXYLATE

SL No.	CHARACTERISTIC	REQUIREMENT	METHOD OF TEST (REF TO CL NO. of APPENDIX A)
(1)	(2)	(3)	(4)
i)	Sodium formaldehyde sulphoxylate, dihydrate ($NaHSO_2.CH_2O.2H_2O$), percent by weight	97.102	A-2
ii)	Sodium formaldehyde bisulphite ($NaHSO_2.CH_2O.H_2O$), percent by weight, *Max*	4	
iii)	Sodium sulphite (Na_2SO_2), percent by weight, *Max*	3	
iv)	*p*H (of 1 percent solution)	7.5 to 9.0	A-3
v)	Iron (as Fe), percent by weight, *Max*	0.02	A-4
vi)	Lead (as Pb), percent by weight, *Max*	0.01	A-5
vii)	Copper (as Cu), percent by weight, *Max*	0.01	A-6
viii)	Zinc (as Zn), percent by weight, *Max*	0.02	A-7

3. KEEPING QUALITY

3.1 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.

4. PACKING AND MARKING

4.1 Packing — The material shall 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.

4.2 Marking — 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) Weight 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.

4.4.1 The containers may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark it governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

5. SAMPLING

5.1 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 requirements of this specification shall be as prescribed in Appendix B.

APPENDIX A

(*Clause* 2.2)

ANALYSIS OF SODIUM FORMALDEHYDE SULPHOXYLATE

A-1. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1960*) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results 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 acid conditions, while sodium bisulphite formaldehyde does not react:

$$NaHSO_2.\ CH_2O.2H_2O + 4I = NaHSO_4 + HCHO + 4\ HI$$

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:

$$NaHSO_2.\ HCHO.H_2O+3NaHCO_2+2\ I=Na_2SO_4+2NaI+4H_2O+3CO_2$$

A-2.1 Reagents

A-2.1.1 *Fomaldehyde Solution* — 1 : 9 (*v/v*).

A-2.1.2 *Starch Indicator* — Triturate 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 minutes. Allow the solution to cool and decant off the supernatant clear liquid.

A-2.1.3 *Sodium Bicarbonate* — *see* IS : 2124-1962†.

A-2.1.4 *Standard Iodine Solution* — 0.1 N (*see* 24 of IS : 2316-1963‡).

A-2.2 Procedure — Weigh accurately about 1 g of the material and dissolve it in water and make up the solution to 250 ml with water.

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*Specification for water, distilled quality (*revised*).

†Specification for sodium bicarbonate.

‡Methods of preparation of standard solutions for colorimetric and volumetric analysis.

A-2.2.1 Take 25 ml of the solution as prepared in **A-2.2** in a conical flask, add 1 ml of starch indicator and titrate with iodine solution *(V_1).*

A-2.2.2 Take 25 ml of the solution as prepared in **A-2.2** 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-2.2.3 Take 25 ml of the solution, as prepared in **A-2.2** in a conical flask, add 1 g of sodium bicarbonate and stir well to dissolve it. Add one millilitre of starch indicator solution and titrate with iodine solution (V_3).

A-2.3 Calculation

Sodium formaldehyde sulphoxylate, percent by weight $= \dfrac{V_2 N \times 38{\cdot}52}{W}$

Sodium sulphite, percent by weight $= \dfrac{(V_1 - V_2)\, N \times 63{\cdot}03}{W}$

Sodium bisulphite formaldehyde, percent by weight $= \dfrac{(V_3 - V_1)\, N \times 76{\cdot}03}{W}$

where

V_1 = volume in ml of standard iodine solution consumed in **A-2.2.1;**
V_2 = volume in ml of standard iodine solution consumed in **A-2.2.2;**
V_3 = volume in ml of standard iodine solution consumed in **A-2.2.3;**
N = normality of standard iodine solution used in the titration; and
W = weight in g of the material taken for the test.

A-3. DETERMINATION OF pH

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 : 262-1963*.

A-4.2.2 *Concentrated Hydrochloric Acid* — *see* IS : 265 - 1962†.

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 thiocyanatc 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* — 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 1000 ml. Pipette out 10 ml of this solution and dilute with water to make up the volume to 100 ml. One millilitre of this solution contains 0.01 mg of iron (as Fe).

A-4.3 Procedure — Weigh accurately one gram 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 any 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 wanning with 50 ml of dilute hydrochloric acid. Cool and make the volume to 100 ml. Ten millilitres 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 seconds and allow the liquids to separate. Carry out a control test in another Nessler tube using 2 ml of standard iron solution.

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 than that produced in the control test.

A-5. DETERMINATION OF LEAD

A-5.0 Principle — Lead is determined colorimetrically by visual comparison of the colour produced with sodium sulphide against that produced by a standard lead solution.

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*Specification for sulphuric acid (*revised*).

†Specification for hydrochloric acid (*revised*).

A-5.1 Reagents

A-5.1.1 *Dilute Ammonium Hydroxide* — approximately 5N.

A-5.1.2 *Potassium Cyanide Solution* — 10 percent (*w/v*).

A-5.1.3 *Sodium Sulphide Solution* — 12 percent (*w/v*).

A-5.1.4 *Concentrated Nitric Acid* — *see* IS : 264-1950*.

A-5.1.5 *Standard Lead Solution* — Weigh accurately 1.60 g of lead nitrate, dissolve in 50 ml of nitric acid and make up to 1 000 ml with water. Dilute one millilitre of this solution to 100 ml with water immediately before use. One millilitre of this diluted standard solution is equivalent to 0.01 mg of lead (as Pb).

A-5.2 Procedure — Take 10 ml of the solution as prepared in **A-4.3.** Neutralize the excess of hydrochloric acid with ammonium hydroxide, add 15 ml in excess and add 1 ml of potassium cyanide solution. Dilute with water to 50 ml and add 2 drops of sodium sulphide solution. Carry out a control test using one millilitre of standard lead solution carried through the same steps as for the material and dilute it to the same volume. Compare the intensity of the colour produced in the two tubes.

A-5.2.1 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.

A-6. DETERMINATION OF COPPER

A-6.0 Principle — Copper is determined colorimetrically by visual comparison in Nessler tubes using sodium diethyldithiocarbamate.

A-6.1 Apparatus

A-6.1.1 *Nessler Tubes* — 50 ml capacity.

A-6.2 Reagents

A-6.2.1 *Citric Acid Solution* — 10 percent (*w/v).*

A-6.2.2 *Gum Arabic Solution* — 1 percent (*w/v*).

A-6.2.3 *Dilute Ammonium Hydroxide* — approximately 5 N.

A-6.2.4 *Concentrated Hydrochloric Acid* — *see* IS : 265-1962†.

A-6.2.5 *Dilute Hydrochloric Acid* — approximately 10 percent.

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*Specification for nitric acid.

†Specification for hydrochloric acid (*revised*).

A-6.2.6 *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 shall not be used after two weeks.

A-6.2.7 *Standard Copper Solution* — Dissolve 0.196 4 g of copper sulphate pentahydrate ($CuSO_4.5H_2O$) in 1 000 ml of water. Pipette out 10 ml of this solution and dilute to 1 000 ml with water. One millilitre of this diluted standard solution contains 0.000 5 mg of copper (as Cu). The dilute solution shall be freshly prepared.

A-6.3 Procedure

A-6.3.1 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 to 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 colour produced in the two tubes.

A-6.3.2 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-7. DETERMINATION OF ZINC

A-7.0 Principle — Zinc is determined colorimetrically by visual comparison in flat-bottom glass-stoppered tube using dithizone (diphenylthiocarbazone).

A-7.1 Apparatus

A-7.1.1 *Flat-Bottom Glass-Stoppered Tube for Colour Comparison* — 50 ml capacity.

A-7.1.2 *p*H *Meter*

A-7.2 Reagents

A-7.2.1 *Dithizone (Diphenyl Thiocarbazone) Solution* — 0.001 percent (*w/v*) in carbon tetrachloride.

A-7.2.2 *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-7.2.3 *Sodium Thiosulphate Solution* — 25 percent (*w/v*).

A-7.2.4 *Standard Zinc Solution* — Weigh accurately one gram of granulated zinc and dissolve in 7 ml of 5 N hydrochloric acid and dilute with water to 1 000 ml. Take one millilitre of this solution and dilute it further to 100 ml. One millilitre of this solution is equivalent to 0.01 mg of zinc.

A-7.3 Procedure — Take 10 ml of the solution of the material as prepared in **A-4.3.1** 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 2 ml of standard zinc solution 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-7.3.1 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.

APPENDIX B

(*Clause* 5.1)

SAMPLING OF SODIUM FORMALDEHYDE SULPHOXYLATE

B-1. GENERAL REQUIREMENTS OF SAMPLING

B-1.0 In drawing, preparing, storing and handling samples the following precautions and directions shall be observed.

B-1.1 Precautions shall be taken to protect the sample, the material being sampled, the sampling instrument and the container for samples from adventitious contamination.

B-1.2 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-1.3 The samples shall be placed immediately after sampling in clean, dry and air-tight containers made of metal or fibre with polyethylene lining.

B-1.4 Each sample container shall be sealed air-tight immediately after filling.

B-1.5 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-1.6 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 signatures 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 specification, 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

TABLE 2 NUMBER OF CONTAINERS TO BE SELECTED FOR SAMPLING

NUMBER OF CONTAINERS IN THE LOT N (1)	NUMBER OF CONTAINERS TO BE SELECTED n (2)
Up to 25	2
26 „ 50	4
51 „ 100	4
101 „ 300	5
301 and over	6

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 lot, count them as 1, 2, 3,… …up to *r* and so on in one order. Every *r*th 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.

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 specification 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 Sample — 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 specification.

BUREAU OF INDIAN STANDARDS

Headquarters :

Manak Bhavan, 9 Bahadur Shah Zafar Marg, NEW DELHI 110002

Telephones : 3 31 01 31, 3 31 13 75 Telegrams : Manaksanstha
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