भारतीय मानक **IS 5345 : 2024**

Indian Standard

सोडियम सैकरीन, खाद्य ग्रेड — विशिष्टि

(तीसरा पुनरीक्षण)

Sodium Saccharin, Food Grade — Specification

(Third Revision)

ICS 67.220.20

भारतीय मानक ब्यूरो
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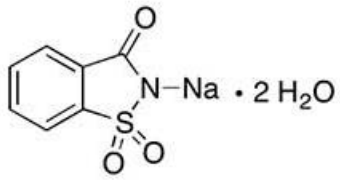
Food Additives Sectional Committee, FAD 08

FOREWORD

This Indian Standard (Third Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Food Additives Sectional Committee had been approved by the Food and Agriculture Division Council.

Sodium saccharin is a non-nutritive sweetener. It is taken by diabetics and those who need low calorie diet as a substitute for cane sugar. It is about 500 times sweeter than sugar. Use of sodium saccharin, food grade as artificial sweetener has been permitted in selected food items under the *Food Safety and Standards* (*Food Products Standards and Food Additives*) *Regulation*, 2011.

Chemical names and formulae



The recognized chemical names are sodium *o*-benzosulflmide; sodium salt of 2, 3-dihydro-3-oxobenzisosulfonazole, 1, 2-benzisothiazolin-3-one-l, l-dioxide sodium salt dihydrate. Its common name is soluble saccharin. Empirical formula is $C_7H_4NNa_3O_3S.2H_2O$. Its molecular weight is 241.19 and structural formula is as under:

This standard was first published in 1969 and subsequently revised in 1978 and 1997. In the second revision, the limit for toluenesulfonamide was reduced; and directions for storage and expiry date were incorporated.

In this revision the following major changes have been done:

a) The requirement for heavy metals has been removed as the limit of lead (contaminant in food colours) is already covered through the standard; and

b) The marking requirements have been updated.

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 SACCHARIN, FOOD GRADE — SPECIFICATION

(Third Revision)

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for sodium saccharin, food grade.

2 REFERENCES

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

IS No.	*Title*
IS 1070 : 2023	Reagent grade water — Specification (*fourth revision*)
IS 1699 : 2024	Food colours — Methods of sampling and test (*third revision*)
IS 2362 : 1993	Determination of water by Karl Fischer method — Test method (*second revision*)

3 DESCRIPTION

It shall be in the form of white crystals or white crystalline powder. It shall be odourless or having a faint odour and intensely sweet to taste even in dilute solution 1 g is soluble in 1.5 ml of water and in about 50 ml of alcohol.

NOTE — The solubility is intended only as information regarding approximate solubility and is not to be considered as a quality requirement and is of minor significance as a means of identification or determination of purity.

4 REQUIREMENTS

4.1 Identification

4.1.1 Dissolve about 100 mg of the material in 5 ml of 5 percent sodium hydroxide solution. Evaporate to dryness and gently fuse the residue over a small flame until it no longer ammonia evolves. After the residue has cooled, dissolve it in 20 ml of water, neutralize the solution with dilute hydrochloric acid and filter. The addition of a drop of ferric chloride solution (9 g of ferric chloride and sufficient water to make 100 ml) to the filtrate shall produce a violet colour.

4.1.2 Mix 20 mg of the material with 40 mg of the resorcinol, add 10 drops of sulphuric acid and heat the mixture in a liquid bath at 200 °C for 3 min. After cooling add 10 ml of water and an excess of sodium hydroxide solution (dissolve 4.3 g sodium hydroxide in water and make it to 100 ml). A fluorescent green liquid shall result.

4.1.3 The residue obtained by igniting a 2 g sample shall give positive test for sodium, when tested by the method given in 4.1.3.1.

4.1.3.1 *Test for sodium*

A solution of a sodium compound, previously converted to chloride or nitrate, when mixed with 5 times its volume of cobalt-uranyl acetate, a golden yellow precipitate is formed on shaking. Sodium compounds impart an intense yellow colour to a non-luminous flame.

4.1.4 To 10 ml of a 10 percent solution, add 1 ml of hydrochloric acid. Wash the crystalline precipitate of saccharin formed with cold water and dry at 105 °C for 2 h. The residue shall melt between 226 °C and 230 °C.

4.2 The material shall also conform to the requirements given in Table 1.

5 PACKING AND STORAGE

5.1 Packing

The material shall be filled in amber colour glass containers or any other containers with as little air space as possible. The container shall be such as to preclude contamination of the contents with metals or other impurities.

5.2 Storage

The material shall be stored in a cool and dry place so as to avoid excessive exposure to heat.

6 MARKING

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

a) Name of the material, including the words 'Food Grade';

b) Name of the manufacturer or his registered trade-mark, if any;

c) Net quantity when packed;

d) Lot/batch No.;

e) Month and year of manufacture;

f) Expiry date; and

g) Any other requirements as specified under the *Legal Metrology* (*Packaged Commodities*) *Rules*, 2011 and *Food Safety and Standards* (*Labelling and Display*) *Regulations*, 2020.

6.2 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.

7 SAMPLING

7.1 The representative samples of the material shall be drawn according to the method prescribed in IS 1699.

7.2 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.

Table 1 Requirements for Sodium Saccharin, Food Grade

(*Clause* 4.2)

Sl No.	Characteristic	Requirement	Method of Test, Ref to
(1)	(2)	(3)	(4)
i)	Purity as $C_7H_4NNaO_3S$, after drying at 120 °C for 4 h, percent by mass	99 to 101	A-1
ii)	Loss on drying, percent by mass, *Max*	15	A-2
iii)	Acidity and alkalinity	Pass the test	A-3
iv)	Benzoate and salicylate	Pass the test	A-4
v)	Readily carbonizable substances	Pass the test	A-5
vi)	Toluenesulfonamides, mg/kg, *Max*	25	A-6
vii)	Arsenic (as As), mg/kg, *Max*	2	IS 1699
viii)	Selenium (as Se), mg/kg, *Max*	30	A-7
ix)	Lead (as Pb), mg/kg, *Max*	2	IS 1699

ANNEX A

(*Table 1*)

METHODS OF TEST FOR SODIUM SACCHARIN

A-1 PURITY

A-1.1 Two methods have been specified. Any methods may be used depending on the facilities available.

A-1.2 Method I

A-1.2.1 *Procedure*

Weigh accurately about 500 mg of the material and transfer it quantitatively to a separator with the aid of 10 ml of water. Add 2 ml of diluted hydrochloric acid, and extract the precipitated saccharin first with 30 ml, then with four 20 ml portions of a solvent composed of 9 volumes of chloroform and 1 volume of alcohol. Filter each extract through a small filter paper moistened with the solvent mixture, and evaporate the combined filtrates on a steam-bath to dryness. Dissolve the residue in 75 ml of hot water, cool, add phenolphthalein and titrate with 0.1 N sodium hydroxide. Each millilitre of 0.1 N sodium hydroxide is equivalent to 20.52 mg of sodium saccharin ($C_7H_4NNaO_3S$).

A-1.3 Method II

A-1.3.1 *Reagents*

A-1.3.1.1 *Acetic acid* — glacial

A-1.3.1.2 *Crystal violet* — glacial acetic acid indicator solution

A-1.3.1.3 *Perchloric acid* — 0.1 N

A-1.3.2 *Procedure*

Dissolve about 0.3 g of previously dried sample, accurately weighed, in 20 ml of glacial acetic acid. Add 2 drops of crystal violet-glacial acetic acid indicator, and titrate with 0.1 N perchloric acid. End-point shall be when violet colour of solution change to green *via* blue. Perform a blank determination, and make any necessary correction. Each millilitre of 0.1 N perchloric acid is equivalent to 20.52 mg of sodium saccharin ($C_7H_4NNaO_3S$).

A-2 LOSS ON DRYING

For routine purposes, loss on drying shall be determined by drying the sample at 120 °C for 4 h. In case of dispute, it shall be determined as moisture by the Karl Fischer method described in IS 2362.

A-3 TEST FOR ACIDITY AND ALKALINITY

Dissolve 1 g of the sample in 10 ml of freshly boiled and cooled water. Add one drop of phenolphthalein indicator. No pink colour shall appear. Add one drop of 0.1 N sodium hydroxide. A pink colour shall appear.

A-4 TEST FOR BENZOATE AND SALICYLATE

To 10 ml of a 5 percent solution, previously acidified with 5 drops of dilute acetic acid, add 3 drops of 9 percent ferric chloride. No precipitate or violet colour shall appear.

A-5 READILY CARBONIZABLE SUBSTANCES

A-5.1 Reagents

A-5.1.1 *Sulphuric Acid*

A-5.1.2 *Matching Fluid*

Composed of 0.1 ml of cobalt chloride solution (*see* A-5.1.2.1), 0.4 ml of ferric chloride solution (*see* A-5.1.2.2), 0.1 ml cupric sulphate solution (*see* A-5.1.2.3) and 4.4 ml of water.

A-5.1.2.1 *Cobalt chloride solution*

Dissolve about 65 g of cobaltous chloride ($CoCl_2.6H_2O$) in enough of a mixture of 25 ml of hydrochloric acid and 975 ml of water to make 1 000 ml. Place exactly 5 ml of this solution in a 250 ml iodine flask, add 5 ml of 3 percent hydrogen peroxide and 15 ml of 20 percent sodium hydroxide solution. Boil for 10 min, cool and add 2 g of potassium iodide and 20 ml of 25 percent sulphuric acid. When the precipitate has dissolved, titrate the liberated iodine with 0.1 N sodium thiosulphate solution, adding starch. Each millilitre of 0.1 N sodium thiosulphate is equivalent to 23.8 mg of cobaltous chloride. Adjust the final volume of the solution by adding enough of the mixture of hydrochloric acid and water to make each millilitre contain 59.5 mg of cobaltous chloride.

A-5.1.2.2 *Ferric chloride solution*

Dissolve about 55 g of ferric chloride ($FeC1_3.6H_2O$) in enough of a mixture of 25 ml hydrochloric acid and 975 ml of water to make 1 000 ml. Place 10 ml

of this solution in a 250 ml iodine flask, add 15 ml of water and 3 g of potassium iodide and allow the mixture to stand for 15 min. Dilute with 100 ml of water and titrate the liberated iodine with 0.1 N sodium thiosulphate solution, adding starch as indicator. Each milliliter of 0.1 N sodium thiosulphate is equivalent to 27.03 mg of ferric chloride. Adjust the final volume of the solution by adding enough of the mixture of hydrochloric acid and water to make each millilitre contain 45.0 mg of ferric chloride.

A-5.1.2.3 *Cupric sulphate solution*

Dissolve about 65 g of cupric sulphate ($CuSO_4.5H_2O$) in enough of a mixture of 25 ml of hydrochloric acid and 975 ml of water to make 1 000 ml. Place 10.0 ml of this solution in a 250 ml iodine flask, add 40 ml of water, 4 ml of acetic acid and 3 g of potassium iodide. Titrate the liberated iodine with 0.1 N sodium thiosulphate solution, adding starch. Each millilitre of 0.1 N sodium thiosulphate is equivalent to 24.97 mg of cupric sulphate. Adjust the final volume of the solution by adding enough of the mixture of hydrochloric acid and water to make each millilitre contain 62.4 mg of cupric sulphate.

A-5.2 Procedure

Dissolve 200 mg of sodium saccharin in 5 ml of sulphuric acid. Keep at 48 °C to 50 °C for 10 min. The solution shall not have deeper colour than the matching fluid (*see* A-5.1.2).

A-6 DETERMINATION OF TOLUENESULFONAMIDES

Two methods have been specified. Any method may be used depending upon the facilities available.

A-6.1 Method I — Gas Chromatography

A-6.1.1 *Reagents*

A-6.1.1.1 *Methylene chloride*

Use a suitable pure grade, equivalent to the product obtained by distillation in all-glass apparatus.

A-6.1.1.2 *Internal standard stock solution*

Transfer 100 mg of 95 percent n-tricosane into a l0 ml volumetric flask. Dissolve in n-heptane, dilute to volume with the same solvent and mix.

A-6.1.1.3 *Stock standard preparation*

Transfer 20 mg each of reagent grade o-toluenesulfonamide and p-toluenesulfonamide into a 10 ml volumetric flask. Dissolve in methylene chloride, dilute to volume with the same solvent, and mix.

A-6.1.1.4 *Diluted standard preparation*

Pipette into five 10 ml volumetric flasks, 0.1 ml, 0.25 ml, 1.0 ml, 2.5 ml and 5.0 ml respectively, of the ‘stock standard preparation’. Pipette 0.25 ml of the ‘internal standard stock solution’ into each flask, dilute each to volume with methylene chloride and mix. These solutions contain 250 µg of *n*-tricosane, plus respectively, 20 µg, 50 µg, 200 µg, 500 µg and 1 000 µg per ml of each toluenesulfonamide, plus 250 mg of *n*-tricosane.

A-6.1.1.5 *Test preparation*

Dissolve 2.00 g of the sample in 8.0 ml of 5 percent sodium bicarbonate solution. Mix the solution thoroughly with 10.0 g of chromatographic siliceous earth (celite 545 or equivalent). Transfer the mix into a 25 mm × 250 mm chromatographic tube having a fritted glass disk and a Teflon stopcock at the bottom, and a reservoir at the top. Pack the contents of the tube by tapping the column on a padded surface, and then by tamping firmly from the top. Place 100 ml of methylene chloride in the reservoir and adjust the stopcock so that 50 ml of eluate is collected in 20 min to 30 min. To the eluate add 25 µl of ‘internal standard stock solution’. Mix and then concentrate the solution to a volume of 1.0 ml in a suitable concentrator tube fitted with a modified Snyder column, by using Kontes tube heater maintained at 90 °C.

A-6.1.2 *Procedure*

A-6.1.2.1 Inject 2.5 µl of the test preparation (*see* A-6.1.1.5) into a suitable gas chromatograph equipped with a flame-ionization detector. The column should be of glass, approximately 3 m in length and 2 mm in inside diameter and packed with 3 percent phenylmethyl silicone on 100 to 120 mesh equivalent to 150 micron to 125 micron IS test sieve [*see* IS 460 (Parts 1) (Part 2) and (Part 3)] silanized calcined diatomaceous silica.

CAUTION — The glass column should extend into the injector for on-column injection and into the detector base to avoid contact with metal.

The carrier is helium flowing at a rate of 30 ml per minute. The injection port, column, and detector are maintained at 225 °C, 180 °C and 250 °C respectively. The instrument attenuation setting should be such that 2.5 µl of the ‘diluted standard preparation’ containing 200 µg per ml of each toluenesulfonamide gives a response of 40 percent to 80 percent of full-scale deflection. Record the

chromatogram, note the peaks for o-toluenesulfonamide, p-toluenesulfonamide and the n-tricosane internal standard and calculate the areas for each peak by suitable means. The retention times for *o*-toluenesulfonamide, *p*-toluenesulfonamide, and *n*-tricosane are about 5 min, 6 min and 15 min, respectively.

A-6.1.2.2 In a similar manner, obtain the chromatogram for 2.5 µl portions of each of the five 'diluted standard preparations' and for each solution determine the areas of the *o*-toluenesulfonamide, *p*-toluenesulfonamide and *n*-tricosane peaks. From the values thus obtained, prepare standard curves by plotting concentration of each toluenesulfonamide, in µg per ml versus the ratio of the respective toluenesulfonamide peak area to that of n-tricosane. From the standard curve determine the concentration, in µg per ml, of each toluenesulfonamide in the 'test preparation'. Divide each value by 2 to convert the result to parts per million of toluenesulfonamide in the 2 g sample taken for analysis.

NOTE — If the toluenesulfonamide content of the sample is greater than about 500 parts per million, the impurity may crystallize out of the methylene chloride concentrate (*see* A-6.1.1.5). Although this level of impurity exceeds that permitted by the specification, the analysis may be completed by diluting the concentrate (usually 1: 10 is satisfactory) with methylene chloride containing 250 µg of n-tricosane per ml, and by applying appropriate dilution factors in the calculation. Care shall be taken to redissolve completely any crystalline toluenesulfonamide to give a homogeneous solution.

A-6.2 Method II — Thin Layer Chromatography

A-6.2.1 *Apparatus*

A-6.2.1.1 *Flat glass plates* — 200 mm × 100 mm

A-6.2.1.2 *Micro-pipette*

A-6.2.1.3 *Developing chamber* — lined with filter paper

A-6.2.2 *Reagents*

A-6.2.2.1 *Silica gel G*

A-6.2.2.2 *Chloroform*

A-6.2.2.3 *Methyl alcohol*

A-6.2.2.4 *Ammonia solution* — strong

A-6.2.2.5 4-*sulphamoylbenzoic acid* — reference material

A-6.2.2.6 *Toluene-2-sulfonamide acid* — reference material

A-6.2.2.7 *Sodium hypochloride solution*

Diluted with water to contain 0.5 percent (*m/v*) of available chlorine.

A-6.2.2.8 *Potassium iodide*

A-6.2.2.9 *Starch mucilage*

A-6.2.2.10 *Glacial acetic acid*

A-6.2.2.11 *Solution A*

4 volumes of methyl alcohol plus 1 volume of acetone plus 0.5 percent (*m/v*) of the sample.

A-6.2.2.12 *Solution B*

Four volumes of methyl alcohol plus 1 volume of acetone plus 0.005 percent (*m/v*) of the 4-sulphamoylbenzoic acid.

A-6.2.2.13 *Solution C*

Four volumes of methyl alcohol plus 1 volume of acetone plus 0.005 percent (*m/v*) of the toluene-2-sulfonamide.

A-6.2.3 *Procedure*

A-6.2.3.1 Prepare suspension of silica1 gel G. Spread the suspension on the plates about 0.25 mm thick. Allow to stand until the coating sets and then dry the plates at 105 °C to 110 °C for 1 h. Protect the plates from moisture. Pour into the developing chamber sufficient quantity of mobile phase (100 volumes of chloroform + 50 volumes of methyl alcohol + 11.5 volume of strong ammonia solution) to form a layer about 15 mm deep. Close the tank for one hour at 20 °C to 27 °C. Remove the narrow strips of the coating, about 5 mm inside from the margins of the chromatoplate. Using micro-pipette apply separately to the chromatoplates 2 ml each of solutions A, B and C. These spots should be about 25 mm from the bottom of the plates and not less than 25 mm from the sides of the plates. The diameter of the spots should not be more than 6 mm. Dry the spots and place the chromatoplates in the developing chamber at 20 °C to 27 °C until the mobile phase has ascended to the 150 mm line. Remove the plates and dry them in current of warm air. Then heat at 105 °C for 5 min. Spray the hot plates with the sodium hypochlorite solution. Dry in a current of cold air until sprayed area of the plate below the line of application give at most a faint blue colour with a drop of a mixture, prepared by dissolving 0.5 percent (*m/v*) of potassium iodide in starch mucilage containing 1 percent

(*m/v*) of glacial acetic acid. Avoid prolongedexposure to cold air. Spray the plates with the same mixture. The spots in the chromatograms obtained with solution (B) and (C) should be more intense than any corresponding spots in the chromatogram obtained with solution (A).

A-7 DETERMINATION OF SELENIUM

A-7.1 Reagents

A-7.1.1 *Selenium Stock Solution*

Transfer 120 mg of metallic selenium (Se) into a 1 000 ml volumetric flask, and 100 ml of dilute nitric acid (1 in 2), warm gently on a steam-bath to effect solution, and dilute to volume with water. Transfer 5 ml of this solution into a 200 ml olumetric flask at A-7.1.1 , dilute to volume with water, and mix. Each millilitre of this solution contain 3 µg of selenium ion (Se).

A-7.1.2 *Standard Selenium Solution*

Just prior to use, transfer 20.0 ml of selenium stock solution (60 µg Se) into a 200 mm × 25 mm test tube, add 20 ml of hydrochloric acid, and mix.

A-7.1.3 *Sample Solution*

Transfer 2 g of the sample to a 250 ml Erlenmeyer flask and cautiously add 10 ml of 30 percent hydrogen peroxide. After the initial reaction has subsided, add 6 ml of 70 percent perchloric acid, heat slowly until white fumes of perchloric acid are copiously evolved, and continue heating gently for a few minutes to ensure decomposition of any excess peroxide. If the solution is brownish in colour due to non-decomposed organic matter, add a small portion of the peroxide solution and heat again to white perchloric acid fumes, repeating if necessary until decomposition of the organic matter shall be complete and a colourless solution is obtained. Cool, add 10 ml of water and filter into a 200 mm × 25 mm test tube. Wash the filter paper with hot water until the filtrate measures 20 ml, add 20 ml of hydrochloric acid and mix.

A-7.2 Procedure

Place the test tubes containing the standard selenium solution and the sample solution in a water-bath and heat until the temperature of the solution reaches 40 °C. To each tube, add 400 mg of ascorbic acid stir until dissolved and maintain at 40 °C for 30 min. Cool the solution, dilute with water to 50 ml and mix. Any pink colour produced by the sample shall not exceed that produced by the standard selenium solution (A-7.1.2).

Bureau of Indian Standards

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Review of Indian Standards

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