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भारतीय मानक मसौदा

सैकरीन, खाद्य ग्रेड – विशिष्टि

(आइ एस 6385 का तीसरा पुनरीक्षण)

Draft Indian Standard

SACCHARIN, FOOD GRADE - SPECIFICATION

(Third Revision of IS 6385)

ICS No. 67.220.20

Food Additives Committee, FAD 08

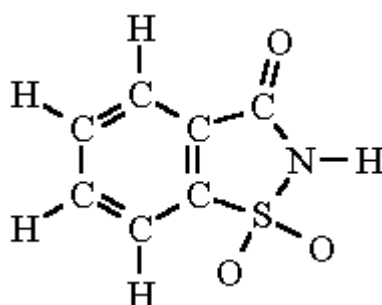
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FOREWORD

(Formal clauses would be added later)

Saccharin is a non-nutritive sweetener. It is normally used 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 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 Formula -The recognized chemical names are 0-benzosulfimide; 2,3 dihydro-3-oxobenzisoxazolone; and 1,2 benzisothiazole-3-one-1,1-dioxide. Its empirical formula is $C_7H_5NO_3S$. Molecular weight is 183.18 and structural formula is as under:



This standard was first published in 1971 and was first revised in 1978 to make the following changes/additions:

- To provide a separate clause for description incorporating the solubility properties to keep the standard in line with the Food Chemical Codex, NRC.

- b) To reduce the limit for toluene sulphonamides.
- c) To substitute the requirement of lead by heavy metals and the corresponding changes in test method.
- d) To include directions for storage and expiry date under the marking clause.

In preparation of this standard, considerable assistance has been derived from the following publications:

- a) Compendium of Food Additive Specifications, Volume 2, Joint FAO/WHO Expert Committee on Food Additives (JECFA), 1992.
- b) Specifications for identity and purity of some food additives, 1975, FAO/WHO, Rome.
- c) Food Chemical Codex. Third Edition.
- d) National Academy of Sciences, National Research Council, Washington DC, USA.

In this revision, the following major changes have been made:

- a) The requirement for heavy metals has been removed as the limit of lead (contaminant in food colours) is already covered through the standard.
- b) The marking requirements have been updated.
- c) One amendment issued to the previous version of the standard has been incorporated.

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 : 2002 '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.

1 SCOPE

This standard prescribes the requirements and methods of tests for saccharin, food grade.

2 REFERENCES

The following Indian Standards 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 editions of the standards indicated below:

<i>IS No.</i>	<i>Title</i>
IS 1070 : 2023	Reagent grade water – Specification (<i>fourth revision</i>)
IS 1699 : 1995	Methods of sampling and test for food colours (<i>second revision</i>)
IS 5345 : 1978	Sodium saccharin, food grade – Specification (<i>first revision</i>)

3 DESCRIPTION

It shall be in the form of white crystals or white crystalline powder. It shall be odourless or having a faint aromatic odour. It has intensely sweet taste. The material shall be slightly soluble in water, sparingly soluble in ethanol, slightly soluble in chloroform and ether and is readily absorbed by dilute solution of ammonia, solutions of alkali hydroxides or solutions of alkali carbonates with the evolution of carbon dioxide.

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 mean of identification or determination of purity and dependence must be placed on other specifications.

4 REQUIREMENTS

4.1 Identification

4.1.1 A saturated aqueous solution of saccharin shall be acidic to litmus.

4.1.2 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 evolves ammonia. 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 100ml) to the filtrate shall produce a violet colour.

4.1.3 Mix 20 mg of the material with 40 mg of the resorcinol, add 10drops of concentrated sulphuric acid and heat the mixture in a liquid bath at 200°C for 3 minutes. After cooling add 10ml of water and an excess of 10 percent sodium hydroxide solution. A fluorescent green liquid shall be produced.

4.1.4 Melting range of the material shall be between 226 and 230°C.

NOTE - Melting point is the temperature at which liquefaction of the substance occurs, which is indicated by the formation of a definite meniscus. The melting point of the substance should fall within the range specified.

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

Table 1 Requirements for Saccharin, Food Grade

(Clause 4.2)

Sl. No.	Characteristic	Requirements	Method of Test, Ref to
(1)	(2)	(3)	(4)
i)	Purity as C ₇ H ₅ NO ₃ S, percent by mass, on dry basis, <i>Min</i>	99.0	Annex A (A-1)
ii)	Moisture, percent by mass on drying at 105 °C for 2 hrs, <i>Max</i>	1.0	IS 2362
iii)	Benzoic and salicylic acids	To pass the test	Annex A (A-2)
iv)	Readily carbonizable substances, <i>Max</i>	To pass the test	IS 5345
v)	Sulphated ash, percent by mass, <i>Max</i>	0.2	Annex A (A-3)
vi)	Toluenesulfonamides, percent by mass, <i>Max</i>	25.0	Annex A (A-4)
vii)	Arsenic (as As), percent by mass, <i>Max</i>	3.0	IS 1699
viii)	Selenium (as Se), percent by mass, <i>Max</i>	30.0	IS 5345
ix)	Lead (as Pb), percent by mass, <i>Max</i>	2.0	IS 1699

5 PACKING AND STORAGE

5.1 Packing

The material shall be securely packed in well-filled containers so as to preclude contamination of the contents.

5.2 Storage

The material shall be stored in a cool and dry place so as to avoid 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) Best beforemonths from manufacture; and
- g) Any other requirements as specified under the *Legal Metrology (Packaged Commodities) Rules, 2011* and *Food Safety and Standards (Packaging) Regulations, 2018* 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

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

8 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.

ANNEX A
[Table 1, Sl No. (i)]
METHODS OF TEST FOR SACCHARIN, FOOD GRADE

A-1 PURITY

A-1.1 Two methods have been specified for determination of purity. In case of dispute, Method II shall be regarded as the reference method.

A-1.2 Method I

A-1.2.1 Reagents

A-1.2.1.1 *Sodium hydroxide solution* - 0.1 N.

A-1.2.1.2 *Phenolphthalein indicator*.

A-1.2.2 Procedure

A-1.2.2.1 Dissolve about 0.5 g of previously dried (105°C for 2 hours) sample, accurately weighed in 75 ml hot water. Cool quickly, add phenolphthalein indicator. Titrate with sodium hydroxide solution. Each millilitre of 0.1 N sodium hydroxide is equivalent to 18.32 mg of saccharin (C₇H₅NO₃S).

A-1.3 Method II

A-1.3.1 Reagents

A-1.3.1.1 *Sodium hydroxide solution* - 30 percent (m/v) in water, and N/10.

A-1.3.1.2 *Hydrochloric acid* - concentrated.

A-1.3.1.3 *Sulphuric acid* - N/10.

A-1.3.1.4 *Methyl red indicator* - Dissolve 1 g of methyl red in 100 ml of 95 percent alcohol.

A-1.3.2 Procedure

Transfer about 540 mg of previously dried at 105° for 2 hours and accurately weighed sample, to long necked flask, having a capacity of 200 ml and add to it 10 ml of 30 percent (m/v) solution of sodium hydroxide. Boil over a small flame for 2 minutes, avoiding loss by evaporation; cool, add 15 ml of hydrochloric acid and boil again for 50 minutes under a reflux condenser. Cool, rinse the condenser with 50 ml of water, and pass a current of air through the flask to remove acid vapour. Connect with-an ammonia distillation apparatus, add 20 ml of 30 percent (m/v) solution of sodium hydroxide, and distil the ammonia into 40 ml of sulphuric acid; titrate the excess of acid with N/10 sodium hydroxide using solution of methyl red as indicator. Each millilitre of N/10 sulphuric acid, neutralized by, the ammonia in the distillate, is equivalent to 18.32 mg of saccharin (C₇H₅NO₃S).

A-2 TEST FOR BENZOIC AND SALICYLIC ACID

Dissolve 0.5 g of the sample in 10 ml of boiling water and 3 drops of 9 percent ferric chloride. No precipitate or violet colour shall appear.

A-3 SULPHURED ASH

A.3.1 Apparatus

A-3.1.1 Flat-Bottom Dish – of silica or platinum.

A-3.1.2 Muffle Furnace - maintained at $550 \pm 20^\circ\text{C}$.

A-3.1.3 Desiccator

A-3.2 Procedure

Weigh accurately about 3 g of the material in the dish, previously dried in an air-oven and weighed. Heat the dish gently on a flame at first and then strongly in a muffle furnace at $550 \pm 200^\circ\text{C}$ till grey ash results. Cool the dish in a desiccator. Moisten the residue with one millilitre of sulphuric acid and cautiously heat the dish again at $550 \pm 200^\circ\text{C}$ for 30 minutes. Cool the dish in a desiccator and weigh. Repeat this process of heating for 30 minutes, cooling and weighing until the difference between two successive weighing is less than 1 mg. Record the lowest mass.

A-3.3 Calculation

$$\text{Sulphated ash, percent by mass} = \frac{100 (M_2 - M)}{M_1 - M}$$

where,

M = mass in g of the empty dish;

M_1 = mass in g of the dish with the material taken for the test; and

M_2 = mass in g of the dish with the ash;

A-4 DETERMINATION OF TOLUENE SULFONAMIDES

A-4.1 Any of the two methods given under IS 5345 may be used except that 15 percent sodium bicarbonate solution should be used in place of 5 percent solution in test preparation of IS 5345 in Method 1.

A-5 HEAVY METALS

Prepare and test a 2 g sample as per the method given in IS 1699 using 20 μg of lead ion (Pb) in the solution A.