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भारतीय मानक मसौदा

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(आइ एस 6793 का दूसरा पुनरीक्षण)

Draft Indian Standard

FUMARIC ACID, FOOD GRADE — SPECIFICATION

(Second Revision of IS 6793)

ICS No. 67.220.20

Food Additives Sectional Committee, FAD 08 **Last Date of Comments:** 10 October 2024

FOREWORD

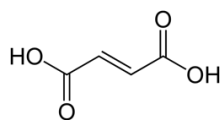
(Formal clauses would be added later)

Food additives are added to improve the appearance, flavour, texture or storage properties, etc of the processed foods. As certain impurities in these substances have been found to be harmful, it is necessary to have a strict quality control of these food additives. A series of standards have, therefore, been prepared to cover purity and identification of these substances. These standards would help in checking purity, which requires to be checked at the stage of manufacture, for it is extremely difficult to detect the impurity once these substances have been added to the processed foods. Besides, these standards are intended to guide the indigenous manufacturers in making their product conform to specifications that are accepted by scientists, health authorities and national/ international bodies.

Use of fumaric acid as a food additive is permitted under the *Food Safety and Standards (Food Products Standards and Food Additives) Regulations, 2011*.

Chemical Description

Its chemical names are *trans*-butenedioic acid and *trans*-1,2 ethylenedicarboxylic acid. Its empirical formula is C₄H₄O₄. Its molecular weight is 116.07. Its structural formula is given below:



This standard was first published in 1972. It was first revised in 1996 to incorporate the requirement of solubility keep in line with Food Chemical Codex (FCC); and the requirement of lead was replaced by heavy metals with its corresponding test method.

In this revision, one amendment issued to the previous version of the standard has been incorporated and 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.

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*)' This 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 sampling and test for fumaric acid, 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 : 202X	Methods of sampling and test for food colours (<i>third revision</i>) [<i>Under preparation Doc: FAD 08 (23204)F</i>]
IS 2362 : 1993	Determination of water by Karl Fischer method — Test method (<i>second revision</i>)
IS 5762 : 1970	Methods for determination of melting point and melting range

3 DESCRIPTION

The material may be in the form of white, odour-less granules or as a crystalline powder with a characteristic acid taste. Fumaric acid is soluble in alcohol, slightly soluble in water and ether and very slightly soluble in chloroform.

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 Tests

Place 50 mg of fumaric acid in a test tube, add 2 to 3 mg of resorcinol and 1 ml of sulphuric acid, stir, heat at 120 °C to 130 °C for 5 min and cool. Dilute with water to 5 ml, and add sodium hydroxide solution (2 : 5) dropwise while cooling to make the solution alkaline. Dilute with water to 10 ml. The solution shall have a greenish blue fluorescence under ultraviolet light.

4.1.1 Melting Range

The melting range of the product when tested as per the method given in IS 5762 shall be from 286 °C to 302 °C.

4.2 The material shall also comply with the requirements given in Table 1.

5 PACKING

The material shall be securely packed in well-filled containers with minimum access to light and moisture. The containers shall be such as to preclude contamination of the contents with metals or other impurities.

Table 1 Requirements of Fumaric Acid, Food Grade
(Clause 4.2)

Sl. No.	Characteristic	Requirements	Method of Test, Ref to
(1)	(2)	(3)	(4)
i)	Purity (as C ₄ H ₄ O ₄) on anhydrous basis, percent by mass, <i>Min</i>	99.5	Annex A (A-1)
ii)	Moisture, percent by mass, <i>Max</i>	0.5	IS 2362
iii)	Sulphated ash, percent by mass, <i>Max</i>	0.1	Annex A (A-2)
iv)	Maleic acid, percent by mass, <i>Max</i>	0.1	Annex A (A-3)
v)	Arsenic (as As), mg/kg, <i>Max</i>	3	IS 1699
vi)	Lead (as Pb), mg/kg, <i>Max</i>	2	IS 1699

6 STORAGE

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

7 MARKING

7.1 The containers shall be securely closed and shall bear legibly and indelibly the following information:

- a) Name of the material including the words 'Food Grade';
- b) Source of manufacture;
- c) Net quantity when packed;
- d) Batch or code number;
- e) Date of manufacture; and
- f) Expiry/ Best before date;
- g) Any other requirements as specified under the *Legal Metrology (Packaged Commodities) Rules, 2011* and *Food Safety and Standards (Labelling and Display) Regulations, 2020*.

7.2 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.

8 SAMPLING

Representative samples of the materials shall be drawn according to the method prescribed in IS 1699.

9 TESTS

Tests shall be carried out by the methods specified in col (4) of Table 1.

10 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), (ii) and (iii)]

METHOD OF TEST FOR FUMARIC ACID, FOOD GRADE

A-1 PURITY

A-1.1 Procedure

Transfer about 2 g, accurately weighed previously dried material into a 250 ml conical flask, add 50 ml of methanol and dissolve the sample by warming gently on a steam-bath. Cool, add phenolphthalein indicator and titrate with 1 N sodium hydroxide solution. Perform a blank determination and make any necessary correction. Each ml of 1 N sodium hydroxide is equivalent to 58.04 mg of fumaric acid.

A-2 SULPHATED ASH

A-2.1 Reagent

A-2.1.1 Concentrated Sulphuric Acid

A-2.2 Procedure

Weigh accurately about 2 g of the material in a tared crucible. Ignite, gently at first until the material is thoroughly charred. Cool, moisten the residue with 1 ml sulphuric acid and ignite gently till the carbon is completely consumed. Cool the crucible in a desiccator and weigh.

NOTE – Carry out the ignition in a place protected from air currents and use as low temperature as possible to effect the combustion of carbon.

A-2.3 Calculation

$$\text{Sulphated ash, percent by mass} = \frac{M_1}{M_2} \times 100$$

where

$$\begin{aligned} M_1 &= \text{mass, in g, of the residue; and} \\ M_2 &= \text{mass, in g, of the material taken for the test.} \end{aligned}$$

A-3 MALEIC ACID

A-3.1 Reagents

A-3.1.1 Buffer Solution

Dissolve 53.5 g of ammonium chloride in about 900 ml of water, adjust the pH to 8.2 with approximately 0.3 N ammonium hydroxide, and dilute with water to 1000 ml.

A-3.1.2 Standard Solution

Transfer to a 100 ml volumetric flask about 100 mg, accurately weighed, maleic acid of the highest purity available, dissolve in about 10 ml of water, then dilute to volume with water and mix.

A-3.1.3 Sample Solution

Transfer about 50 g of the sample, accurately weighed, into a 250 ml beaker, add 80 ml of water, and stir for 10 min with a mechanical stirrer. Filter, using suction, and wash with about 40 ml of water. Transfer the combined filtrate and washings to a 250 ml beaker, add an additional 50 g sample, accurately weighed, to the beaker and repeat the stirring, filtration, and washing procedure. Transfer the combined filtrate and washings to a 250 ml volumetric flask, add 2 drops of phenolphthalein indicator, then add sodium hydroxide solution with stirring, until a light pink colour persists for at least 30 seconds, and dilute to volume with water.

A-3.2 Procedure

Transfer 10.0 ml of the sample solution into a 100 ml volumetric flask, add 20 ml of buffer solution, dilute to volume with water, and mix (solution *A*). Rinse a polarographic cell with a portion of the solution, then add a suitable volume of the solution to the cell, immerse it in a water-bath regulated at 24.5 °C to 25.5 °C, and deaerate by bubbling purified nitrogen through the solution for at least 6 min. Insert the dropping mercury electrode of a suitable polarograph, and record the polarogram from –1 volt to –2 volts, using a saturated calomel electrode as the reference electrode. Determine the height of the wave occurring at the half-wave potential near –1.36 volts. In the same manner polarograph a solution prepared by adding 10 ml of the sample solution, 20 ml of the buffer solution and 2.0 ml of the standard solution to a 100 ml volumetric flask and diluting to volume with water (solution *B*).

A-3.3 Calculation

Calculate the mass, in mg, of maleic acid in the total mass of sample taken by the formula:

$$\frac{2500 \times C \times A}{(B-A)}$$

where

- A* = wave height of solution *A*;
- B* = wave height of solution *B*; and
- C* = concentration, in mg/ml, of added maleic acid in solution *B*.