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भारतीय मानक मसौदा
लैक्टिक एसिड, खाद्य ग्रेड — विशिष्टि
(आई एस 9971 का पहला पुनरीक्षण)

Draft Indian Standard
LACTIC ACID, FOOD GRADE — SPECIFICATION
(First Revision of IS 9971)

ICS 67.220.20

Food Additives Sectional Committee, FAD 08 **Last date of comments: 22 February 2025**

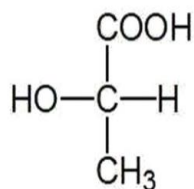
FOREWORD

(Adoption 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 is required 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.

Lactic acid is used as acidulent in beverages like soft drinks, mineral water, carbonated fruit juice, etc, to acid pickles, relishes, salad dressings and variety of fruit products such as jams, jellies, gelatins, marmalades, frozen fruit desserts, etc. It is also used in the manufacture of beer and wine, and dairy products like processed cheese, unsalted butter and in processing of meat, egg whites and sauces for canned fish. It is permitted under the *Food Safety and Standards (Food Products Standards and Food Additives) Regulations, 2011*.

Chemical Name — Lactic acid is 2-hydroxy propionic acid or 1-hydroxyethane-1-carboxylic acid. Its empirical formula is C₃H₆O₃. Its molecular mass is 90.08 and structural formula is:



This standard was first published in 1981. In the preparation of this standard, considerable assistance was derived from specification for identity and purity of some food additives, FAO Nutrition Meeting Report Series No. 57, FAO/WHO, Rome 1977; and Food Chemical Codex, 1966. Ed 1, National Academy of Sciences and National Research Council, Washington, D.C.

In this revision, two amendments issued to the previous version of the standard have 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; 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.

1 SCOPE

This standard prescribes the requirements and methods of sampling and test for lactic acid, food grade.

2 REFERENCES

The standards given 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 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:

<i>IS No.</i>	<i>Title</i>
IS 1070 : 2023	Reagent grade water — Specification (<i>fourth revision</i>)
IS 1699 : 2024	Food colours – Methods of sampling and test (<i>third revision</i>)

3 REQUIREMENTS

3.1 Description

Lactic acid shall be yellowish to colorless syrupy liquid with an acidic taste and no odor. It consists of a mixture of lactic acid ($C_3H_6O_3$) and lactic acid lactate ($C_3H_{10}O_5$). It is obtained by lactic fermentation of sugars or is prepared synthetically.

NOTE — Lactic acid is hygroscopic and when concentrated by boiling, it condenses to form lactic acid lactate, which on dilution and heating hydrolyzes to lactic acid.

3.2 Identification

3.2.1 Solubility — Lactic acid shall be miscible with water and ethanol.

3.2.2 Test for Acid — One in ten solution of the sample shall be acidic to litmus paper.

3.3 Test for Lactate – The material shall give positive test for lactate.

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

Table 1 Requirements for Lactic acid
(*Clause 3.4*)

Sl. No.	Characteristics	Requirements	Method of Test, Ref to
(1)	(2)	(3)	(4)
i)	Purity ($C_3H_6O_3$), percent by mass, <i>Max</i>	95.0 to 105.0	Annex A (A-1)
ii)	Sulphated ash, percent by mass, <i>Max</i>	0.1	Annex A (A-2)
iii)	Chlorides, percent by mass, <i>Max</i>	0.2	Annex A (A-3)

iv)	Sulphates (as SO ₄), percent by mass, <i>Max</i>	0.25	Annex A (A-4)
v)	Citric, oxalic, phosphoric and tartaric acids	Conform to test	Annex A (A-5)
vi)	Sugar	Conform to test	Annex A (A-6)
vii)	Readily carbonizable substances	Conform to test	Annex A (A-7)
viii)	Volatile fatty acid	Conform to test	Annex A (A-8)
ix)	Cyanide	Conform to test	Annex A (A-9)
x)	Methanol, percent by mass, <i>Max</i>	0.2	Annex A (A-10)
xi)	Iron (as Fe), mg/kg, <i>Max</i>	10	Annex A (A-11)
xii)	Arsenic (as As), mg/kg, <i>Max</i>	3	IS 1699
xiii)	Lead, mg/kg, <i>Max</i>	2	IS 1699

4 PACKING

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

5 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 marked legibly and indelibly to give the following information:

- a) Name of the material including the words 'Food Grade';
- b) Source of manufacture;
- c) Net content 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*.

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 TESTS

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

8.2 Quality of Reagents

Unless specified otherwise pure chemicals and distilled water (*see* IS 1070) shall be employed 10 tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

ANNEX A
[Table 1]
METHODS OF TEST FOR LACTIC ACID

A-1 PURITY

A-1.1 Reagent

A-1.1.1 Sodium Hydroxide — 1 N.

A-1.1.2 Phenolphthalein Indicator — Dissolve 0.2 g of phenolphthalein in 60 ml of 90 percent ethanol and add water to make 100 ml.

A-1.1.3 Sulphuric Acid — 1 N.

A-1.2 Procedure

Weigh accurately a portion of the sample equivalent to 5 g of lactic acid, transfer to a 250 ml flask, add 50 ml of sodium hydroxide, mix, and boil for 20 min. Add phenolphthalein, and titrate the excess alkali in the hot solution with 1 N sulphuric acid, and perform a blank determination. Each ml of 1 N sodium hydroxide is equivalent to 90.08 mg of lactic acid.

A-2 SULPHATED ASH

A-2.1 Reagent

A-2.1.1 Sulphuric Acid — concentrated.

A-2.2 Procedure

Weigh accurately 5 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 of 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 a temperature as possible to effect complete combustion of carbon.

A-2.3 Calculation

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

Where,

M_1 = mass, in g, of the residue; and

M_2 = mass, in g, of the material taken for the test.

A-3 CHLORIDES

A-3.1 Reagents

A-3.1.1 *Sodium Hydroxide Solution* — 1: 4.

A-3.1.2 *Potassium Chromate Solution* — 10 percent (m/v) in water.

A-3.1.3 *Silver Nitrate* — 0.1 N.

A-3.2 Procedure

Weigh accurately a portion of the sample equivalent to about 5 g of lactic acid, dissolve in 50 ml of water and neutralize to litmus with sodium hydroxide solution (1: 4). Add 2 ml of potassium chromate and titrate with 0.1 N silver nitrate to the first appearance of a red tinge. Each ml of 0.1 N silver nitrate is equivalent to 3.545 mg of Cl.

A-4 SULPHATES

A-4.1 Reagents

A-4.1.1 *Sodium Hydroxide Solution* — 1: 2.

A-4.1.2 *Barium Chloride Solution* — 12 percent (m/v) in water.

A-4.2 Procedure

Weigh accurately a portion of the sample equivalent to 50 g of lactic acid, transfer into a 600 ml beaker, dissolve in 200 ml of water, and adjust the pH to between 4.5 and 6.5 by addition of sodium hydroxide solution (1: 2), making the final adjustment with a more dilute alkali solution. Filter, if necessary, and heat the filtrate or clear solution to just below the boiling point. Add 10 ml of barium chloride solution stirring vigorously, boil the mixture gently for 5 min, and allow to stand for at least 2 h, or preferably overnight. Collect the precipitate of barium sulphate in a tared gooch crucible, wash until free from chloride, dry, and ignite at 600 °C to constant mass. The mass of barium sulphate so obtained, multiplied by 0.412, represents the mass of SO₄ in the sample taken.

A-5 CITRIC, OXALIC, PHOSPHORIC AND TARTARIC ACIDS

A-5.1 Procedure

Dilute 1 g of the sample to 10 ml with water. Add 40 ml of calcium hydroxide solution [0.14 g of Ca(OH)₂ in 100 ml of water] and boil for 2 min. No turbidity shall be produced.

A-6 SUGARS

A-6.1 Procedure

Add 5 drops of the sample to 10 ml of hot alkaline cupric tartrate solution (Fehling's solution). No red precipitate shall be formed.

NOTE — Fehling's solution is a mixture of equal volumes of solution 1 and 2 prepared immediately before use. Solution 1 consists of 34.64 g of copper sulphate in a mixture of 0.5 ml of sulphuric acid

and sufficient water to produce 500 ml and Solution 2 is 176 g of sodium potassium tartrate and 77 g of sodium hydroxide in sufficient water to produce 500 ml.

A-7 READILY CARBONIZABLE SUBSTANCES

A-7.1 Procedure

Superimpose carefully 5 ml of the sample kept at 15 °C on 5 ml of concentrated sulphuric acid kept at 15 °C. No deep grey color shall be produced within 15 min at the contact zone of the two liquids.

A-8 VOLATILE FATTY ACID

A-8.1 Heat 5 ml of the sample on a water bath. No strong fatty acid like odor shall be evolved.

A-9 CYANIDE

A-9.1 Reagent

A-9.1.1 *p*-Phenylenediamine-Pyridine Mixed Reagent — Dissolve 200 mg of *p*-phenylenediamine hydrochloride in 100 ml of water, warming to effect solution. Cool, allow the solids to settle, and use the supernatant liquid to make the mixed reagent. Dissolve 128 ml of pyridine in 365 ml of water, add 10 ml of hydrochloric acid, and mix. To prepare the mixed reagent, mix 30 ml of the *p*-phenylenediamine solution with all of the pyridine solution, and allow to stand for 24 h before using. The mixed reagent is stable for about three weeks when stored in an amber bottle.

A-9.1.2 *Sample Solution* — Transfer an accurately weighed quantity of the sample, equivalent to 20.0 g of 100 percent lactic acid into a 100 ml volumetric flask, dilute to volume with water, and mix.

A-9.1.3 *Cyanide Standard Solution* — Dissolve 2.5 g of potassium cyanide in 1000 ml of 0.1 N sodium hydroxide. Transfer a 10 ml aliquot into a 100 ml volumetric flask, dilute to volume with 0.1 N sodium hydroxide, and mix. Each ml of this solution contains 10 µg of CN.

A-9.2 Procedure

Pipette a 10 ml aliquot of the sample solution into a 50 ml beaker. Into a second 50 ml beaker pipette 10 ml of the cyanide standard solution, and add 10 ml of water. Place the beakers in an ice bath, and adjust the *pH* to between 9 and 10 with 20 percent sodium hydroxide, stirring slowly and adding the reagent slowly to avoid overheating.

Allow the solutions to stand for 3 min and then slowly add 10 percent phosphoric acid to a *pH* between 5 and 6. Transfer the solution into 100 ml separators containing 25 ml of cold water, and rinse the beakers and *pH* meter electrodes with a few ml of cold water, collecting the washings in the respective separator. Add 2 ml of bromine, stopper and mix. Add 2 ml of 2 percent of sodium arsenite solution, stopper, and mix. To the clear solutions, add 10 ml of *n*-butanol, stopper, and mix. Finally, add 5 ml of *p*-phenylenediamine-pyridine mixed reagent, mix, and allow to stand for 15 min. Remove and discard the aqueous phases, and filter the alcohol phases into 10 mm cells.

The absorbance of the sample, determined at 480 nm with a suitable spectrophotometer, is no greater than that of the standard.

A-10 METHANOL

A-10.1 Reagents

A-10.1.1 Potassium Permanganate Solution — Dissolve 30 g of potassium permanganate and 15 ml of phosphoric acid in 100 ml water. This solution may be retained for a month.

A-10.1.2 Sodium Salt of Chromotropic Acid Solution — 5 percent (m/v).

A-10.1.3 Calcium Carbonate

A-10.2 Preparation of Sample

To 5 ml of the sample add 8 ml of water and 5 g of calcium carbonate and distil. Dilute 5 ml of the initial distillate with water to 100 ml.

A-10.3 Determination

Pipette 2 ml potassium permanganate solution into a 50 ml volumetric flask. Chill in the ice-bath. Add 1 ml of sample solution (*see A-10.2*) and let it stand in ice-bath for 30 min. Decolorize with a little dry sodium bisulphate and add 1 ml of chromotropic acid solution. Add 15 ml of sulphuric acid slowly with swirling and place in hot (60-75 °C) water bath for 15 min. Cool, and change water to bring to 50 ml mark, mix and dilute to volume with water at room temperature.

A-10.4 The color of sample solution treated as in **A-10.3** shall not be darker than that of control prepared by 1 ml of methyl alcohol diluted to 10000 times and subjected to same treatment as given in **A-10.3**.

NOTE — Temperature of standard and sample solution should be within 1 °C.

A-11 IRON

A-11.1 Reagents

A-11.1.1 Dilute Hydrochloric Acid — 1: 2.

A-11.1.2 Ammonium Persulphate — Crystals.

A-11.1.3 Ammonium Thiocyanate Solution — 7.6 percent (m/v) in water (approximately 1 N).

A-11.1.4 Standard Iron Solution — Containing 20 µg of Fe.

A-11.2 Procedure

To the ash obtained in the test for sulphated ash (*see A-2.2*) and 2 ml of dilute hydrochloric acid and evaporate to dryness on a steam bath. Dissolve the residue in 1 ml of hydrochloric acid, dilute to 40 ml with water, and add about 40 mg of ammonium persulphate crystals and 10 ml of the ammonium thiocyanate solution. Any red or pink color shall not exceed that produced by 2.5 ml

of the iron standard solution in an equal volume of solution containing the quantities of reagents used in the test.