भारतीय मानक
Indian Standard

IS 13186 : 2024

सिट्रिक अम्ल, खाद्य ग्रेड — विशिष्टि

(पहला पुनरीक्षण)

Citric Acid, Food Grade — Specification

(First Revision)

ICS 67.220.20

भारतीय मानक ब्यूरो
BUREAU OF INDIAN STANDARDS
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April 2024 **Price Group 6**

Food Additives Sectional Committee, FAD 08

FOREWORD

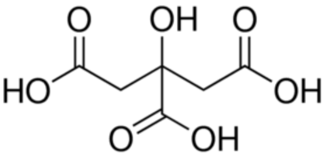
This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Food Additives Sectional Committee had been approved by the Food and Agriculture Division Council.

With the increased production of processed foods, manufacturers have started adding a large number of substances, generally in small quantities, to improve appearance, flavour, texture or storage properties 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 by Bureau of Indian Standards to cover purity and identification of these substances. It is hoped that these standards would help in checking purity which requires to be checked at the stage of manufacture, for it is extremely difficult (and in many cases impossible) 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 specification that are accepted by scientists, health authorities and international bodies.

Citric acid is used as an antioxidant, synergist, sequestrant acidulant and flavouring agent. Its use has been permitted in the *Food Safety and Standards* (*Food Products Standards and Food Additives*) *Regulation*, 2011.

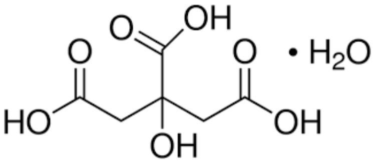
Chemical names and formula — Citric acid is also known as 2-hydroxy-l,2,3-propanetricarboxylic acid; beta-hydroxytricarboxylic acid. Its empirical formulae, molecular weight and structural formulae are given below:

Structural formula of citric acid (anhydrous) is:



Anhydrous form
Empirical formula – $C_6H_8O_7$
Molecular weight – 192.13

Structural formula of citric acid (monohydrate) is:



Monohydrate form
Empirical formula – $C_6H_8O_7.H_2O$
Molecular weight – 210.15

In the preparation of this standard considerable assistance was derived from the FAO/WHO food and nutrition papers No. 4 — ‘Specification for identity and purity of thickening agents, anticaking agents, antimicrobial, antioxidants and emulsifiers’ published by FAO, Rome 1984.

(Continued on third cover)

Indian Standard

CITRIC ACID, FOOD GRADE — SPECIFICATION

(First Revision)

1 SCOPE

This standard prescribes the requirements and methods of sampling and test for citric acid anhydrous and monohydrate, 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 is encouraged to investigate the possibility of applying the most recent editions of these standards:

IS No.	*Title*
IS 1070 : 2023	Reagent grade water — Specification (*fourth revision*)
IS 1699 : 1995	Methods of sampling and test for food colours (*second revision*)
IS 2362 : 1993	Determination of water by Karl Fisher method — Test method (*second revision*)
IS 3025 (Part 32) : 1988	Methods of sampling and test (physical and chemical) for water and wastewater : Part 32 chloride (*first revision*)
IS 3025 (Part 40) : 1991	Water and wastewater — Methods of sampling and test (physical and chemical) : Part 40 calcium (*first revision*)

3 REQUIREMENTS

3.1 Description

The material shall be in the form of white or colourless, odourless, crystalline solid. The monohydrate effloresces substance in dry air.

3.2 Identification Tests

3.2.1 *Solubility*

The material is very soluble in water, freely soluble in ethanol and slightly soluble in ether.

3.2.2 Boil a neutralized solution of the sample with dilute sulphuric acid test solution and a few drops of one percent potassium permanganate. Warm until the violet colour disappears and then add an excess of bromine test solution A; white precipitate forms either immediately or on cooling.

3.3 The material shall also comply with the requirement given in Table 1.

3.4 Additional Requirements for ECO-Mark

3.4.1 *General Requirements*

3.4.1.1 The product shall conform to the requirements prescribed under 3.1 to 3.3.

3.4.1.2 The product manufacturer shall produce the consent clearance as per the provisions of the *Water* (*Prevention & Control of Pollution*) *Act*, 1974, the *Water* (*Prevention & Control of Pollution*) *Cess Act*, 1977 and the *Air* (*Prevention & Control of Pollution*) *Act*, 1981 along with the authorization, if required, under the *Environment* (*Protection*) *Act*, 1986 and the rules made thereunder to Bureau of Indian Standards while applying for the Eco-Mark and the product shall also be in accordance with the *Prevention of Food Adulteration Act*, 1954 and the rules made thereunder.

3.4.1.3 The product/packing shall display in brief the criteria based on which the product has been labelled as environment friendly.

3.4.1.4 The material used for product packaging shall be recyclable or biodegradable.

3.4.1.5 The date of manufacture and best before date shall be declared on the product package by the manufacturer.

3.4.1.6 The product package or leaflet accompanying it may display instructions of proper use and storage so as to maximize the product performance, safety and minimize wastage.

3.4.2 *Specific Requirements*

The product shall also conform to the requirements given in Table 2.

Table 1 Requirement for Citric Acid, Food Grade

(*Clause* 3.3 *and* 7.1)

Sl No.	Characteristic	Requirement	Method of Test, Reference to
(1)	(2)	(3)	(4)
i)	Purity, as $C_6H_8O_7$, percent by mass, (calculated on the anhydrous basis), *Min*	99.5	Annex A-1
ii)	Moisture, percent by mass:		
	a) anhydrous, *Max*	0.5	
	b) monohydrate	7.5 to 8.8	Annex A-2
iii)	Sulphated ash, percent by mass, *Max*	0.01	Annex A-3
iv)	Lead, mg/kg, *Max*	0.5	IS 1699
v)	Arsenic, mg/kg, *Max*	3.0	IS 1699
vi)	Oxalates, mg/kg, *Max*	100.0	Annex A-4
vii)	Readily carbonizable substance	To pass the test	Annex A-5
viii)	Sulphates (as Na_2SO_4), mg/kg, *Max*	150.0	IS 1699

Table 2 Additional Requirements for Citric Acid, Food Grade

(*Clause* 3.4.2)

Sl No.	Characteristic	Requirement	Method of Test, Reference to
(1)	(2)	(3)	(4)
i)	Water insoluble matter, ppm, *Max*	30.0	Annex B
ii)	Chloride (as Cl), ppm, *Max*	5.0	IS 3025 (Part 32)
iii)	Phosphate (as P_2O_5), ppm, *Max*	5.0	Annex C
iv)	Calcium, ppm, *Max*	25.0	IS 3025 (Part 40)
v)	Tridodecylamine, ppm, *Max*	0.1	Annex D

4 PACKING, STORAGE AND MARKING

4.1 Packing

The material shall be packed in air tight containers. The containers shall be such as to preclude contamination of the contents with metals or other impurities.

4.2 Storage

The material shall be stored in a cool place protected from light.

5 MARKING

5.1 Each container shall be legibly and indelibly marked with 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;
f) Instruction for storage;

g) Best before date (month and year to be given by the manufacturer); and

h) Any other requirements as specified under the *Legal Metrology* (*Packaged Commodities*) *Rules*, 2011 and *Food Safety and Standards* (*Packaging and Labelling*) *Regulation*, 2011 and the rules framed thereunder.

5.2 BIS Certification Marking

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provision of *Bureau of Indian Standards Act*, 2016 and Rules and Regulation framed there under and the product(s) may be marked with the Standard Mark.

5.3 ECO-Mark

The product may also be marked with the Eco-Mark, the details of which may be obtained from Bureau of Indian Standards.

6 SAMPLING

The representative samples of the material shall be drawn and conformity of the materials to the requirements of this specification shall be determined according to the procedure prescribed in IS 1699.

7 TESTS

7.1 Tests shall be carried out by the methods specified in col (4) of Table 1 and Table 2.

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 result of analysis.

ANNEX A

[*Table 1*]

METHOD OF TEST FOR CITRIC ACID, FOOD GRADE

A-1 PURITY (TITRIMETRIC METHOD)

A-1.1 Procedure

Dissolve about 3 g of the accurately weighed sample in 40 ml of water. Add phenolphthalein (1 g in 100 ml alcohol) and titrate with 1 N sodium hydroxide. Each ml of 1 N sodium hydroxide is equivalent to 64.04 mg of $C_6H_8O_7$.

A-2 MOISTURE

Moisture content shall be determined by the Karl Fisher method (*see* IS 2362).

A-3 SULPHATED ASH

A-3.1 Reagent — concentrated sulphuric acid

A-3.2 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 oxidized. Cool the crucible in a desiccator and weigh.

A-3.3 Calculation

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

where

M_1 = mass, in g, of the residue; and
M_2 = mass, in g, of the material taken for the test.

A-4 OXALATES

A-4.1 Method I

A-4.1.1 *Reagents*

A-4.1.1.1 *Ammonia solution* — 10 percent

A-4.1.1.2 *Dilute acetic acid*

A-4.1.1.3 *Calcium chloride*

A-4.1.2 *Procedure*

Neutralize 10 ml of a 10 percent solution of the sample with 10 percent ammonia solution. Add 5 drops of dilute acetic acid test solution, cool and add 2 ml of calcium chloride test solution. No turbidity shall be produced (indicates not more than about 1.0 percent of oxalic acid).

A-4.2 Method II

A-4.2.1 *Procedure*

Dissolve 1.0 g of sample in 4 ml of deionized water, add 3 ml concentrated hydrochloric acid and then 1 g of granulated zinc. Heat for 1 min in a boiling water bath. Let stand for 2 min at room temperature; decant the supernatant solution into a test tube containing 0.25 ml of a 1 percent solution of phenylhydrazine hydrochloride. Mix, heat to boiling and cool immediately. Transfer the solution into a glass cylinder with a ground glass stopper and add an equal volume of concentrated hydrochloric acid. Add 0.25 ml of a 5 percent solution of potassium hexacyanoferrate (III), mix well and let stand for 30 min. The absorbance of the solution, read at 520 nm, is not more than that of a standard solution. Prepare the standard solution by dissolving 100 mg of oxalic acid (140 mg oxalic acid dehydrate) in 1 000 ml of deionized water and dilute 1 ml with 3 ml of deionized water.

A-5 READILY CARBONIZABLE SUBSTANCES

A-5.1 Reagents

A-5.1.1 *Sulphuric Acid* — 95 ± 0.5

A-5.1.2 Mix 1 g of suitable starch with 10 mg of red mercuric oxide and sufficient cold water to make a thin paste. Add 20 ml of boiling water to this and boil for 1 min with continuous stirring and cool. Only clear solution shall be used.

A-5.1.3 *Cobaltous Chloride*

Dissolve about 65 g of cobaltous chloride ($CoCl_2.6H_2O$) is enough of a mixture of 25 ml of hydrochloric acid and 975 ml of water to make 1 000 ml. Pipette 5 ml of this solution into a 250 ml iodine flask, add 5 ml hydrogen peroxide solution (containing between 2.5 g and 3.5 g of H_2O_2 in each 100 ml) and 15 ml of sodium hydroxide solution (1 in 5). Boil for 10 min, cool and add 2 g of potassium iodide and 20 ml of dilute sulphuric acid (1 in 4). When the precipitate had dissolved, titrate the liberated iodine with 0.1 N sodium thiosulfate. The titration is sensitive to air oxidation and should be blanketed with carbon dioxide. Each ml of 0.1 N sodium thiosulfate is equivalent to 23.79 mg of $CoCl_2.6H_2O$. Adjust the final volume of the solution by the addition of enough of the mixture of hydrochloric acid and water to make each ml contain 59.5 mg of $CoCl_2.6H_2O$.

A-5.1.4 *Ferric Chloride*

Dissolve about 55 g of ferric chloride ($FeCl_2.6H_2O$) in enough of a mixture of 25 ml of hydrochloric acid and 975 ml of water to make 1 000 ml. Pipette 10 ml of this solution into a 250 ml iodine flask, add 15 ml of water, 5 ml of hydrochloric acid, 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 thiosulfate, adding starch solution (*see* **A-5.1.2**) as the indicator. Perform a blank determination with the same quantities of the same reagent and in the same manner, and make any necessary correction. Each ml of 0.1 N sodium thiosulphate is equivalent to 27.03 mg of $FeCl_2.6H_2O$. Adjust the final volume of the solution by the addition of the mixture of hydrochloric acid and water to make each ml contain 45.0 mg of $FeCl_2.6H_2O$.

5.2 Procedure

Transfer 1.0 g, finely powdered material, to a 22 mm × 175 mm test tube, previously rinsed with 10 ml of sulphuric acid solution and allowed to drain for 10 min. Add 10 ml of sulphuric acid solution, agitate the tube until solution is complete, and immerse the tube in water bath at 90 °C ± 1 °C for 60 min ± 0.5 min, keeping the level of the acid below the level of the water during the heating period. Cool the tube in a stream of water, and transfer the acid solution to a color comparison tube. The color of the acid solution shall not be darker than that of the same volume of a mixture of 0.5 parts cobaltous chloride solution (**A-5.1.3**) and 4.5 parts ferric chloride solution (**A-5.1.4**) (*v/v*).

ANNEX B

[*Table* 2, *Sl No.* (i)]

DETERMINATION OF WATER INSOLUBLE MATTER

B-1 PROCEDURE

Treat 10 g of sample, accurately weighed, with 100 ml of hot water and filter through a tared filtering crucible. Wash the insoluble residue with hot water, dry at 105 °C for 2 h, cool and weigh.

ANNEX C

[*Table* 2, *Sl No.* (iii)]

DETERMINATION OF PHOSPHATE

C-1 PROCEDURE

Weigh accurately about 200 mg to 300 mg of sample, dissolve in 25 ml of water and 10 ml of diluted nitric acid test solution and boil for 30 min. Filter if necessary, and wash any precipitate, then dissolve the precipitate by the addition of 1 ml diluted nitric acid test solution. Adjust the temperature to about 50 °C, add 75 ml of ammonium molybdate test solution, and maintain the temperature at about 50 °C for 30 min, stirring occasionally. Allow to stand for 16 h or overnight at room temperature. Decant off the supernatant, through a filter paper, wash the precipitate once or twice with water by decantation using 30 ml to 40 ml each time, and pour the washings through the same filter. Transfer the precipitate to the same filter, and wash with potassium nitrate solution (1 in 100) until the filtrate is no longer acid to litmus paper.

Transfer the precipitate with filter paper to the original precipitation vessel, add 50.0 ml of 1 N sodium hydroxide, agitate and stir until the precipitate is dissolved, add 3 drops of phenolphthalein test solution and titrate the excess alkali with 1 N sulphuric acid. Each ml of 1 N sodium hydroxide consumed is equivalent to 3.088 mg of P_2O_5.

ANNEX D

[*Table 2, Sl No.* (v)]

DETERMINATION OF TRIDODECYLAMINE

D-1 REAGENTS

D-1.1 Buffered Indicator Solution — Prepare a mixture consisting of 700 ml of 0.01 M citric acid (anhydrous, reagent grade), 200 ml of 0.2 M disodium phosphate and 50 ml each of 0.2 percent bromophenol blue and of 0.2 percent bromocresol green in spectro-grade methanol.

D-1.2 No Indicator Buffer Solution — Prepare a mixture consisting of 700 ml of 0.1 M of citric acid (anhydrous, reagent grade), 200 ml of 0.2 M disodium phosphate and 100 ml of spectro-grade methanol.

D-1.3 Amine Stock Solution — Transfer between 40 mg and 45 mg of tridodecyl (trilauryl) amine, accurately weighed into a 500 ml volumetric flask, dilute to volume with isopropyl alcohol and mix. Discard after three weeks.

D-1.3 Standard Amine Solution — Using a graduated 5 ml pipette, transfer into a 100 ml volumetric flask an amount of Amine stock solution equivalent to 400 µg of tridodecylamine, dilute to volume with isopropyl alcohol and mix. Prepare this solution fresh on the day of use.

D-2 PROCEDURE

Dissolve 160 g of anhydrous reagent grade citric acid (not the sample to be tested) in 320 ml of water and divide the solution equally between two 250 ml separators, S_1 and S_2. To S_1 add 5 ml of no indicator buffer solution. To S_2, add 2.0 ml of standard amine solution and 5 ml of buffered indicator solution. To prepare solutions of the sample being tested, dissolve 160 g of anhydrous citric acid monohydrate sample in 306 ml of water. Divide the test solution equally between two 250 ml separators, S_3 and S_4. Add 5 ml of no indicator buffer solution to S_3 and 5 ml of buffered indicator solution to S_4. To each of the four separators, add 20 ml of 1 : 1 mixture (*v/v*) prepared from spectrograde chloroform and n-heptane. Shake for 15 min on a mechanical shaker and allow the phases to separate for 45 min. Drain all except the last few drops of the lower (aqueous) phases and discard. Hand-shake the organic phases with 25 ml each of 0.05 N sulphuric acid for 30 s and allow the phase to separate for 10 min. Drain all except the last few drops of the lower (organic) phase through a Whatman No. 40 (or equivalent) paper and collect the aqueous filtrates in separate small glass-stoppered containers. Determine the absorbance of each solution in a 5 cm cell at 400 nm, with a suitable spectrophotometer standardized prior to analysis, against chloroform heptane (1 : 1 *v/v*). The net absorbance of the sample (S_4 - S_3) is not greater than that of the standard (S_2 - S_1).

ANNEX E

(*Foreword*)

COMMITTEE COMPOSITION

Food Additives Sectional Committee, FAD 08

Organization	*Representative(s)*
CSIR - Indian Institute of Toxicology Research, Lucknow	Dr Yogeshwar Shukla **(*Chairperson*)**
All India Food Processors Association, New Delhi	Ms Shreya Pandey Shri Krishna Kumar Joshi (*Alternate*)
Association of Food Scientists and Technologists India, Mumbai	Dr Vikas Singh Chauhan Dr Nandini P. Shetty (*Alternate*)
Bose Institute, Kolkata	Prof Gaourishnkar
Confederation of Indian Food Trade and Industry, New Delhi	Dr Jasvir Singh Ms Priyanka Sharma (*Alternate*)
Confederation of Indian Industry, New Delhi	Ms Neha Aggarawal Ms Mamta Arora Budhiraja (*Alternate*)
Consumer Education and Research Centre, Ahmedabad	Ms Anindita Mehta Ms Dolly A. Jani (*Alternate*)
Consumer Guidance Society of India, Mumbai	Dr Sitaram Dixit Dr M. S. Kamath (*Alternate*)
CSIR - Central Food Technological Research Institute, Mysuru	Dr Ngaseppam Iboyiama Shri Aruna Kumar (*Alternate*)
Defence Food Research Laboratory, Mysuru	Dr G. K. Sharma Dr D. D. Wadikar (*Alternate*)
Department of Chemical Technology, University of Calcutta, Calcutta	Prof Arup Mukherjeee Dr Farooque Abdullah (*Alternate*)
Envirocare Labs Private Limited, Thane	Dr Priti Amritkar Dr Nilesh Amritkar (*Alternate*)
Food Ingredients Manufacturers & Suppliers of India Association	Shri Firoz H. Naqvi
Grasim Industry, Mumbai	Shri Pankaj Kumar Gupta
ICMR - National Institute of Nutrition, Hyderabad	Dr J. Padmaja
Indian Institute of Chemical Technology, Hyderabad	Dr Ashok Kumar Tiwari Dr T. Kumaraguru (*Alternate*)
Indian Pharmacopoeia Commission, Ghaziabad	Ms Shruti Rastogi Shri Jai Shiv Chauhan (*Alternate*)
Indian Salt Manufacturers Association, Ahmedabad	Shri B. C. Rawal Shri P. R. Dhruve (*Alternate*)

Organization	*Representative(s)*
Office of the Salt Commissioner, Jaipur	SHRI M. A. ANSARI SHRI B. S. NAGAR (*Alternate*)
Protein Foods and Nutrition Development Association of India, Mumbai	SHRI BHUPINDER SINGH DR JAGADISH PAI (*Alternate*)
Roha Dyechem, Mumbai	SHRI ZAINULABIDIN DHANSE
VR Food Tech Pvt Ltd, Mumbai	DR ASHLESH PARCHURE DR N. RAM (*Alternate*)
BIS Directorate General	MS SUNEETI TOTEJA, SCIENTIST ‘E’/DIRECTOR AND HEAD (FOOD AND AGRICULTURE) [REPRESENTING DIRECTOR GENERAL (*Ex-officio*)]

Member Secretary
SHRI KULDEEP MITTAL
SCIENTIST ‘B’/ASSISTANT DIRECTOR
(FOOD AND AGRICULTURE), BIS

(*Continued from second cover*)

This standard was originally published in 1991. This revision of the standard has been undertaken to incorporate the amendments issued to the standard and also to align the requirements of the product with (*Food Safety and Standards (Food Product Standards and Food Additives*) *Regulations*, 2011. Also, the following changes have been incorporated:

a) The description clause has been modified;
b) The solubility clause has been modified;
c) The limit of the sulphates has been changed to bring in line with Eco-Mark;
d) Alternate method for determination of oxalates have been incorporated to bring in line with JECFA; and
e) Requirement of lead has been incorporated deleting the requirement of 'heavy metals'.

A scheme for labelling environment friendly products known as Eco-Mark has been introduced at the instance of the Ministry of Environment and Forests (MoEF), Government of India which is based on the gazette notification No. 215(E) dated 17 May 1996 for labelling food additives as environment friendly products, published by the Ministry of Environment and Forests. The Eco-Mark would be administered by the Bureau of Indian Standards (BIS) under the *Bureau of Indian Standards Act*, 2016 as per the resolution No. 71 dated 20 February 1991 as published in the Gazette of the Government of India vide GSR No. 85(E) dated 21 February 1991. For a product to be eligible for Eco-Mark it shall also carry the Standard Mark of BIS for quality besides meeting additional environmental friendly (EF) requirements.

The composition of the Committee responsible for the formulation of this standard is given in Annex E.

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.

Bureau of Indian Standards

BIS is a statutory institution established under the *Bureau of Indian Standards Act*, 2016 to promote harmonious development of the activities of standardization, marking and quality certification of goods and attending to connected matters in the country.

Copyright

Review of Indian Standards

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the website- www.bis.gov.in or www.standardsbis.in.

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Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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