भारतीय मानक

Indian Standard

IS 5464 : 2023

सिट्रिक एसिड, मोनोहाइड्रेट — विशिष्टि

(दूसरा पुनरीक्षण)

Citric Acid, Monohydrate — Specification

(Second Revision)

ICS 71.080.40

भारतीय मानक ब्यूरो
BUREAU OF INDIAN STANDARDS
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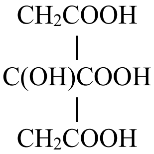
March 2023 **Price Group 7**

Organic Chemicals, Alcohols and Allied Products Sectional Committee, PCD 09

FOREWORD

This Indian Standard (Second Revision) was adopted by the Bureau of Indian Standards after the draft finalized by the Organic Chemicals, Alcohols and Allied Products Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

Citric acid also known as 2-hydroxypropane-1, 2, 3-tricarboxylic acid ($C_6H_8O_7$) occurs in many fruits, especially unripe fruits of the citrus family such as lemons and oranges. It is commercially manufactured by mould fermentation of carbohydrates in which a dilute solution of molasses is fermented by using certain moulds (citromycetes). This method is particularly suitable for India on account of huge quantities of molasses available from sugar mills. It is represented by the following structural formula:



$$\begin{array}{c} CH_2COOH \\ | \\ C(OH)COOH \\ | \\ CH_2COOH \end{array}$$

Citric acid is extensively used in soft drinks and other food industries. The functional use in food is that of a sequestering agent, antioxidant, dispersing agent and acidifier. It is also put to pharmaceutical uses besides being used as analytical reagent and general industrial purposes such as mordant in dyeing or cleaning agent for boiler water in thermal power plants. Ferric ammonium citrate on exposure to sunlight changes to ferrous salt and is therefore used in making blue-print paper. Esters of citric acid (tributyl citrate) are good plasticizers for lacquers and varnishes.

This standard was first issued in 1970 and subsequently revised in 1995. In 1970 version, the requirements, the methods of sampling and test for two grades of citric acid monohydrate namely, grade 1 suitable for use as analytical reagent and grade 2 intended for use in food additives were prescribed.

The first revision was taken up to delete grade 2 intended for use in food additives and to retain the requirements of the material for general purpose, as a separate standard was formulated by the relevant committee under Agricultural and Food Division of the Bureau wherein requirements of the material used as food additives was stipulated (*see* IS 13186).

In this revision, ICP-AES method has been incorporated to replace 2, 2'-bipyridyl method as prescribed in **7** of IS 4542 for determination of iron (*as* Fe), as IS 4542 is withdrawn. ICP-AES method is also incorporated as an alternate method for determination of heavy metals (*as* Pb). The cross-referred standards and BIS certification marking clause have also been updated.

The composition of the committee, responsible for the formulation of this standard is listed in Annex L.

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

CITRIC ACID, MONOHYDRATE — SPECIFICATION

(Second Revision)

1 SCOPE

1.1 This standard prescribes the requirements, the methods of sampling and testing for citric acid, monohydrate.

1.2 This standard does not include the requirements of citric acid used for pharmaceutical purposes or as food additives.

2 REFERENCES

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

IS No.	*Title*
IS 265 : 2021	Hydrochloric acid — Specification (*fifth revision*)
IS 266 : 1993	Sulphuric acid — Specification (*third revision*)
IS 915 : 2012/ISO 1042 : 1998	Laboratory glassware — One-mark volumetric flasks (*third revision*)
IS 1070 : 1992	Reagent grade water — Specification (*third revision*)
IS 1448 (Part 172) : 2020	Methods of test for petroleum and its products: Part 172 ICP-AES method for determination of trace elements in petroleum products
IS 2088 : 1983	Methods for determination of arsenic (*second revision*)
IS 2362 : 1993	Determination of water by the karl fischer method (*second revision*)
IS 4161 : 1967	Specification for nessler cylinders
IS 4905 : 2015/ISO 24153 : 2009	Random sampling and randomization procedures (*first revision*)

3 REQUIREMENTS

3.1 Description

The material shall be colourless translucent crystals or a white granular to line crystalline powder. It shall be odourless with a strong acid taste.

3.1.1 It shall be freely soluble in water, ethyl alcohol and sparingly soluble in ether. It shall form a clear solution in water.

3.2 The material shall also comply with the requirements prescribed in Table 1 when tested according to the method given in col (4) of Table 1.

3.2.1 *Quality of Reagents*

Unless specified otherwise, 'pure chemicals' and distilled water (*see* IS 1070) shall be used in tests.

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

4 PACKING AND MARKING

4.1 Packing

The material shall be packed in well-closed containers made of glass or in polyethylene liners within containers made of cardboard, fiberboard, plywood or tinplate.

Table 1 Requirements for Citric Acid, Monohydrate

(*Clause* 3.2)

Sl No. (1)	Characteristic (2)	Requirements (3)	Method of Test, Ref to Annex (4)
i)	Citric acid, percent by mass (as monohydrate), *Min*	98.5	A
ii)	Sulphate (as SO_4), ppm, *Max*	100	B
iii)	Halides (as Cl), ppm, *Max*	100	C
iv)	Sulphated ash, percent by mass, *Max*	0.10	D
v)	Heavy metals (as Pb), ppm, *Max*	10	E/ IS 1448 (Part 172)$^{1)}$
vi)	Iron (as Fe), ppm, *Max*	10	IS 1448 (Part 172)
vii)	Oxalates (as $C_2H_2O_4$)	To pass the test	F
viii)	Water Content, percent by mass, *Max*	8.8	G
ix)	Readily carbonizable substance	To pass the test	H
x)	Arsenic, ppm, *Max*	1	J

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1) In case of disputes, IS 1448 (Part 172) shall be referee method for determination of heavy metals (*as* Pb)

4.2 Marking

4.2.1 Each container shall bear legibly and indelibly the following information:

a) Name of the material;
b) Name of the manufacturer and his recognized trademark, if any;
c) Batch number or lot number, in code or otherwise;
d) Month and year of manufacture;
e) Net and gross weight; and
f) Any other statutory requirement.

4.2.2 *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.

5 SAMPLING

5.1 The method of drawing representative samples of the material and the criteria for conformity of the material shall be as prescribed in Annex K.

ANNEX A

[*Table* 1, *Sl No*. (i)]

DETERMINATION OF CITRIC ACID, MONOHYDRATE CONTENT

A-1 OUTLINE OF THE METHOD

A known quantity of the material is dissolved in water and limited with sodium hydroxide solution. Citric acid monohydrate content is calculated from the volume of sodium hydroxide solution used up.

A-2 REAGENTS

A-2.1 Standard Sodium Hydroxide Solution, 1 N.

A-2.2 Thymol Blue Indicator

Dissolve 0.04 g of thymol blue in 100 ml of 50 percent rectified spirit.

A-3 PROCEDURE

Weigh accurately about 3 g of the material in a tared flask. Dissolve in 100 ml of water and titrate with standard sodium hydroxide solution using thymol blue indicator.

A-4 CALCULATION

Citric acid, monohydrate, percent by mass =

$$\frac{0.070\,05 \times V \times N \times 100}{M}$$

where

V = volume of standard sodium hydroxide solution required for neutralization, in ml;

N = normality of standard sodium hydroxide solution; and

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

ANNEX B

[*Table* 1, *Sl No*. (ii)]

DETERMINATION OF SULPHATES (*as* SO_4)

B-1 OUTLINE OF THE METHOD

The material is dissolved in water, denatured spirit and dilute hydrochloric acid added and mixed. Barium chloride solution is then added and turbidity compared with that produced by a known quantity of sulphate.

B-2 APPARATUS

B-2.1 One-Mark Graduated Flasks, 100 ml and 1 000 ml (*see* IS 915).

B-2.2 Nessler Cylinders, 50 ml (*see* IS 4161).

B-3 REAGENTS

B-3.1 Denatured Spirit

B-3.2 Dilute Hydrochloric Acid, approximately 5 N.

B-3.3 Barium Chloride ($BaCl_2 . 2H_2O$), 10 percent (*m/v*).

B-3.4 Standard Sulphate Solution

Dissolve 1.48 g of the ignited sodium sulphate (Na_2SO_4) in water. Transfer quantitatively to the 1 000 ml one-mark graduated flask and make it up to the volume with water up to the mark. Pipette 10 ml of this solution into the 100 ml one-mark graduated flask and make up the volume with water up to the mark. 1 ml of this solution contains 0.1 mg of sulphate (*as* SO_4).

B-4 PROCEDURE

B-4.1 Dissolve 5.0 g of the material in 40 ml of water and transfer quantitatively into one of the nessler cylinders. Add 10 ml of denatured spirit and 1 ml of dilute hydrochloric acid; mix and add 1 ml of barium chloride solution. Allow to stand for 1 h. In another nessler cylinder carry out control test under same conditions using 5 ml of standard sulphate solution, 37 ml of water, 10 ml of denatured spirit, 1 ml of dilute hydrochloric acid, and 1 ml of barium chloride solution.

B-4.2 The material shall be taken to have not exceeded the limit prescribed in Table 1, if the turbidity produced with the material is not greater than that obtained in the control test.

ANNEX C

[*Table* 1, *Sl No.* (iii)]

DETERMINATION OF HALIDES (*as* Cl)

C-1 OUTLINE OF THE METHOD

The material is dissolved in water and acidified with nitric acid. Silver nitrate is then added and turbidity compared with that produced by a known quantity of chloride.

C-2 APPARATUS

C-2.1 One-Mark Graduated Flasks, 1 000 ml (*see* IS 915)

C-2.2 Nessler Cylinders, 50 ml (*see* IS 4161).

C-3 REAGENTS

C-3.1 Dilute Nitric Acid, approximately 5 N

C-3.2 Silver Nitrate Solution, 10 percent *(m/v)*

C-3.3 Standard Sodium Chloride Solution

Dissolve 1.65 g of sodium chloride in water and transfer quantitatively to one of the 1 000 ml one-mark graduated flasks. Make up the volume with water up to the mark. Pipette out 10 ml of this solution into the second one-mark graduated flask and again dilute with water to 1 000 ml mark. One ml of this solution contains 0.01 mg of chloride (*as* Cl).

C-4 PROCEDURE

C-4.1 Dissolve 5.00 g of the material in 30 ml of water and transfer this solution quantitatively to one of the nessler cylinders. Add 5 ml of dilute nitric acid and 1 ml of silver nitrate solution. Dilute with water to 50 ml mark and mix well. In the second nessler cylinder carry out a control test under the same conditions using 5 ml of standard chloride solution in place of the material. Dilute the contents with water to 50 ml mark and mix well.

C-4.2 The material shall be taken to have not exceeded the limits prescribed in Table 1 if the opalescence or turbidity produced with the material is not greater than that obtained in the control test.

ANNEX D

[*Table* 1, *Sl No.* (iv)]

DETERMINATION OF SULPHATED ASH

D-1 OUTLINE OF THE METHOD

A known quantity of the material is ignited and burnt until only ash and carbon remain. After cooling the residue is moistened with sulphuric acid, ignited and finally the mass of the sulphated ash is determined.

D-2 APPARATUS

D-2.1 Crucible, of suitable capacity, squat form and made of silica on platinum.

D-3 REAGENT

D-3.1 Sulphuric Acid, relative density 1.836 (*see* IS 266).

D-4 PROCEDURE

Weigh accurately about 5 g of the material in the crucible and dry at (105 ± 2) °C for four hours. Ignite completely and to the ash add 2 drop to 3 drops of sulphuric acid. Re-ignite. Conduct the ignition in a place protected from air currents, and use as low temperature as possible to effect the combustion of the carbon. When the carbon has completely disappeared; continue heating till no more sulphuric acid fumes are evolved; cool the crucible in a desiccator and weigh.

D-5 CALCULATION

$$\text{Sulphated ash, percent by mass} = \frac{100 \times (M_1 - M_2)}{(M_3 - M_2)}$$

where

M_1 = mass of the crucible with the ignited residue, in g;

M_2 = mass of the crucible, in g; and

M_3 = mass of the crucible with the material, in g.

ANNEX E

[*Table* 1, *Sl No*. (v)]

DETERMINATION OF HEAVY METALS (*as* Pb)

E-1 OUTLINE OF THE METHOD

The material is dissolved in ammonium hydroxide and then heated with sodium sulphide. The colour produced by the heavy metals present in the material is compared with that produced by a known quantity of lead in a freshly prepared standard lead solution.

E-2 APPARATUS

E-2.1 One-Mark Graduated Flasks, 1 000 ml (*see* IS 915).

E-2.2 Nessler Cylinders, 50 ml (*see* IS 4161).

E-3 REAGENTS

E-3.1 Ammonium Hydroxide, approximately 6 N, free from lead.

E-3.2 Potassium Cyanide Solution, approximately 10 percent (*m/v*).

E-3.3 Sodium Sulphide Solution, approximately 10 percent (*m/v*).

E-3.4 Standard Lead Solution

Dissolve 1.600 g of lead nitrate in water and transfer to one of the 1 000 ml one-mark graduated flasks. Make up the solution to 1 000 ml mark. Pipette 10 ml of the solution into the second 1 000 m1 one-mark graduated flask and dilute it again with water to 1 000 ml mark. 1 ml of this solution contains 0.01 mg of lead (*as* Pb). This solution shall be freshly prepared before use.

E-4 PROCEDURE

Weigh 2.000 g of the material and transfer to a Nessler cylinder. Dissolve it in 10 ml of ammonium hydroxide and then dilute with water to 50 ml. Add two drops of sodium sulphide solution. Carry out a control test in another nessler cylinder, using 2 ml of standard lead solution and the same quantities of the other reagents to make the volume up to 50 ml. Allow both the cylinders to stand for 10 min on a white surface and then compare the colour viewing downwards.

The limits for heavy metals shall be taken as not having been exceeded if the intensity of the colour produced in the test with the material is not greater than that produced in the control test.

ANNEX F

[*Table* 1, *Sl No*. (vii)]

DETERMINATION OF OXALATES (*as* $C_2H_2O_4$)

F-1 OUTLINE OF THE METHOD

The material is dissolved in water and neutralized with dilute ammonium hydroxide; dilute acetic acid and calcium chloride solution are then added. Turbidity produced, if any, is noted.

F-2 REAGENTS

F-2.1 Dilute Ammonium Hydroxide, approximately 5 N.

F-2.2 Dilute Acetic Acid, approximately 5 N.

F-2.3 Calcium Chloride Solution, 10 percent *(m/v).*

F-3 PROCEDURE

F-3.1 Neutralize 10 ml of a 5 percent *(m/v)* solution of the material with dilute ammonium hydroxide. Add 5 drops of dilute acetic acid, cool and add 2 ml of calcium chloride solution.

F-3.2 The material shall be regarded to have passed the test if no turbidity is produced.

ANNEX G

[*Table* 1, *Sl No*. (viii)]

DETERMINATION OF WATER CONTENT

G-1 PROCEDURE

Weigh 0.5 g of the material and determine the moisture content using karl fischer method as described in IS 2362.

ANNEX H

[*Table* 1, *Sl No*. (ix)]

TEST FOR READILY CARBONIZABLE SUBSTANCES

H-1 OUTLINE OF THE METHOD

The colour produced by the material with sulphuric acid is compared with that of the matching fluid.

H-2 APPARATUS

H-2.1 Nessler Cylinders, 50 ml (*see* IS 4161)

H-3 REAGENTS

H-3.1 Hydrochloric Acid, [*see* IS 265 percent (*m/v*)].

H-3.2 Hydrogen Peroxide Solution, 30 percent (*m/v*).

H-3.3 Sodium Hydroxide Solution, 1 : 5 (*m/v*).

H-3.4 Potassium Iodide, solid.

H-3.5 Dilute Sulphuric Acid, 1 : 4 (*v/v*).

H-3.6 Standard Sodium Thiosulphate Solution, 0.1 N.

H-3.7 Starch Solution

Saturate 5 g of starch and 0.01 g of mercuric iodide with 30 ml of cold water and slowly pour it with stirring into 1 litre of boiling water. Boil for 3 min. Allow to cool and decant off the supernatant clear liquid.

H-3.8 Sulphuric Acid, relative density 1.836 (*see* IS 266).

H-3.9 Cobaltous 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 1000 ml. Pipette 5 ml of this solution into a 250 ml iodine flask, add 5 ml of hydrogen peroxide solution and 15 ml of sodium hydroxide solution. Boil for 10 min, cool and add 2 g of potassium iodide and 20 ml of dilute sulphuric acid.

When the precipitate has dissolved, titrate the liberated iodine with standard sodium thiosulphate solution, adding starch so1ution as the indicator. Perform a blank determination with the same quantities of the same reagents and make the necessary correction. Taking each ml of standard sodium thiosulphate solution as equivalent to 23.79 mg of cobaltous chloride, adjust the final solution by the addition of enough of the mixture of hydrochloric acid and water to make each ml contain 59.5 mg of cobaltous chloride ($CoCl_2.\ 6H_2O$).

H-3.10 Dilute Ferric Chloride Solution

Dissolve about 55 g of ferric chloride ($FeCl_3.\ 6H_2O$) 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, 3 g of potassium iodide and 5 ml of hydrochloric acid, and allow the mixture to stand for 15 min. Dilute with 100 ml of water, and titrate the liberated iodine with standard sodium thiosulphate solution adding starch solution as the indicator. Perform a blank determination with the same quantities of the same reagents, and make any necessary correction. Taking each ml of standard sodium thiosulphate solution as equivalent to 27.03 mg of ferric chloride, 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 45.0 mg of ferric chloride ($FeCl_3.\ 6H_2O$).

H-3.11 Matching Fluid

Prepare the matching fluid by mixing together 1.0 ml of cobaltous chloride solution and 9 ml of dilute ferric chloride solution.

H-4 PROCEDURE

H-4.1 Heat 1.00 g of the material with 10 ml of sulphuric acid in a water-bath at (90 ± 1) °C in the dark; maintaining the level of the water in the bath above the level of the sulphuric acid. Shake to mix after 1 min intervals and continue heating for a total of 60 min. Cool to the room temperature. Transfer quantitatively to one of the nessler cylinders and dilute with water to the mark. In the second nessler cylinder transfer the matching fluid and dilute with water to the mark. Compare the intensity of colour of the solutions in the two nessler cylinders.

H-4.2 The material shall be regarded to have passed the test if the intensity of the colour of the test solution is not deeper than the colour of the matching fluid.

ANNEX J

[*Table* 1, *Sl No*. (x)]

DETERMINATION OF ARSENIC CONTENT (*as* As)

J-1 PROCEDURE

Dissolve 1.000 g of the material in 15 ml of water. Carry out the test for arsenic as prescribed in IS 2088 except that the following standard arsenic solution shall be used in place of the standard arsenic trioxide solution defined in IS 2088.

J-1.1 Standard Arsenic Solution

Dissolve 1.32 g of resublimed arsenic trioxide (As_2O_3) in 25 ml of sodium hydroxide solution and neutralize with dilute sulphuric acid. Dilute with freshly distilled water containing 10 ml of concentrated sulphuric acid per litre and make up the volume to 1 000 ml. Again dilute 10 ml of this solution to 1 litre with water containing sulphuric acid and finally dilute 100 ml of this solution to 1 litre with water containing sulphuric acid. 1 ml of this solution contains 0.001 mg of arsenic. The dilute solution shall be prepared freshly when required.

ANNEX K

(*Clause* 5.1)

SAMPLING OF CITRIC ACID, MONOIIYDRATE

K-1 GENERAL REQUIREMENTS FOR SAMPLING

K-1.1 Samples shall be taken in a protected place not exposed to damp air, dust or soot.

K-1.2 The sampling instrument shall be clean and dry.

K-1.3 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the sample containers from adventitious contamination.

K-1.4 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by suitable means.

K-1.5 The samples shall be placed in suitable, clean, dry and air-tight glass bottles or other suitable containers on which the material has no action.

K-1.6 Sample containers shall be of such a size that they are almost completely filled by the sample.

K-1.7 The sample containers shall be sealed airtight after filling and marked with full identification particulars of the lot and consignment and the date of sampling.

K-2 SCALE OF SAMPLING

K-2.1 Lot

In a single consignment, all the containers of the same size, containing material from the same batch of manufacture, shall constitute a lot.

K-2.2 For ascertaining type conformity of the material to the requirements of this specification, samples shall be tested for each lot separately. The number of containers to be selected from a lot shall depend on the size of the lot and shall be in accordance with col (2) and col (3) of Table 2.

Table 2 Number of Containers to be Selected for Sampling

(*Clause* 2.2)

Sl No. (1)	Number of Containers in the Lot (2)	Number of Containers to be Selected (3)
i)	Up to 25	3
ii)	26 to 50	4
iii)	51 to 100	5
iv)	101 to 300	6
v)	301 to 500	7
vi)	501 to 800	8
vii)	801 to 1 300	9
viii)	1 301 and above	10

K-2.3 These containers shall he chosen at random from the lot and in order to ensure randomness of selection, procedure given in IS 4905 may be followed.

K-3 TEST SAMPLES AND REFEREE SAMPLE

K-3.1 From each of the containers selected according to **K-2.2**, a representative portion of the material, about 200 g, shall be drawn. These samples shall constitute individual samples.

K-3.2 From each of these individual samples (**K-3.1**), an equal quantity of the material shall be drawn and thoroughly mixed to constitute a composite sample not less than 600 g. The composite sample shall be transferred to clean bottles and labelled with full identification particulars of the sample. This composite sample shall be divided into three equal parts, one for the purchaser, another for the supplier and the third to be used as referee sample.

K-4 TESTS

K-4.1 Tests for the determination of all the characteristics of this specification shall be carried out on the composite samples.

K-5 CRITERIA FOR CONFORMITY

K-5.1 For determining the conformity of the lot to this specification, the test results on the composite samples shall meet the corresponding requirements specified in this standard.

ANNEX L

(*Foreword*)

COMMITTEE COMPOSITION

Organic Chemicals, Alcohols and Allied Products Sectional Committee, PCD 09

Organization	*Representative(s)*
National Chemical Laboratory (NCL), Pune	Dr C. V. Rode **(*Chairperson*)**
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All India Alcohol-Based Industries Development Association (AABIDA), Mumbai	Shri K. L. Raphael Shri Kirti Gajjar (*Alternate*)
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Member Secretary
KUMARI ADITI CHOUDHARY
SCIENTIST ‘B’/ASSISTANT DIRECTOR
(PETROLEUM, COAL AND RELATED PRODUCTS), BIS

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Review of Indian Standards

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Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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