

COEI-1-CITACI Citric acid, monohydrate**Monohydrated 3-Carboxy-3-hydroxypentanedioic acid****Acidum citricum****SIN NO. 330****1. Objective, Origin and Scope of Application**

Citric acid can be used to chemically acidify wines or as a stabilizing agent to limit, in particular, the risks of iron breakdown, or again, for prewashing filter plates. Its maximum proportions in wine may be subject to statutory limits.

2. Labelling

The label should indicate product concentration, even when included in mixtures, as well as its safety and storage conditions.

3. Properties

Citric acid is found in the form of colorless, translucent crystals which are rather friable and slightly efflorescent, or in crystalline powder form.

$$D_{4^{\circ}C}^{20^{\circ}C} = 1,542$$

4. Solubility

| | |
|----------------------|--------------|
| Water at 20 °C | very soluble |
| Alcohol, 95% by vol. | very soluble |
| Glycerol | very soluble |
| Ethyl ether | 31.5 g/l |

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Aqueous citric acid is inert in polarized light.

5. Identifying Characteristics

- 5.1. Verify total solubility in water. A 1 pp 100 solution (m/v) shows an acid reaction to methyl orange (R).
- 5.2. Place 2 ml of an aqueous 1 g/l citric acid solution and 0.5 ml of mercury (II) sulfate solution (R) in a test tube. Bring to a boil and add several drops of 2 pp 100 potassium permanganate solution (R). A white precipitate should form.
- 5.3. Add 1 drop of bromine water (R), 3 drops of concentrated sulfuric acid (R) and 1 drop of saturated potassium permanganate solution to 0.1 ml of 10 pp 100 (m/v) aqueous citric acid solution. Bring to a boil.

Add 2 ml of concentrated sulfuric acid (R). Heat again until completely dissolved. Let cool, then add 0.1 ml of beta-naphthol (R). A green coloring should appear. A pink coloring is obtained under the same conditions if sulforesorcin reagent (R) is used under the same conditions.

- 5.4. Place 5 ml of chloroform or dichloromethane in a test tube. Add 100-200 mg of citric acid. Shake. The crystals or crystalline powder should collect together at the surface of the liquid. Under these same conditions, tartaric acid collects at the very bottom of the tube.

6. Tests

6.1. Foreign Substances

Citric acid should be soluble without residue in its weight of water and in twice its weight of 95% alcohol (by volume).

6.2. Sulfur Ash

After calcination at $600\text{ }^{\circ}\text{C} \pm 25\text{ }^{\circ}\text{C}$, the concentration of sulfur ash should not be greater than 0.5 g/kg.

6.3. Tartaric Acid Determination

Add 2 drops of sulforesorcinic reagent (R) and 2 drops of 10 pp 100 (m/v) citric acid solution to 2 ml of concentrated sulfuric acid (R). Heat to $150\text{ }^{\circ}\text{C}$. The solution should not develop a violet coloring.

6.4. Preparing the Solution for Tests

Prepare a 10 parts per 100 (m/v) solution.

6.5. Chlorides

Add 14.5 ml of water, 5 ml of nitric acid diluted to 10 pp 100 (R) and 0.5 ml of 5 pp 100 silver nitrate solution (R) to 0.5 ml of the solution prepared for tests under paragraph

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6.4. After sitting for 15 minutes in the dark, there should be no clouding. If clouding does occur, it should be less intense than that observed in a control prepared as indicated in the Annex. (Chloride content expressed in terms of hydrochloric acid should be less than 1 g/kg).

6.6. Sulfates

Add 18 ml of water, 1 ml of diluted hydrochloric acid (R) and 2 ml of 10 pp 100 barium chloride solution diluted to 10 pp 100 (R) to 1 ml of the solution prepared for tests under paragraph 6.4. After 15 minutes, there should be no clouding. If clouding does occur, it should be less intense than that observed in a control prepared by replacing the test solution with 1 ml of 0.1 g/l sulfuric acid solution. (Sulfate content expressed in terms of sulfuric acid should be less than 1 g/kg).

6.7. Oxalic Acid and Barium

Neutralize 5 ml of the solution prepared for tests under paragraph 6.4 by adding concentrated ammonium hydroxide (R). Add 2 drops of acetic acid (R) and 5 ml of saturated calcium sulfate solution (R). There should be no clouding. (Oxalate content expressed in terms of oxalic acid should be less than 0.1g/kg).

6.8. Iron

Add 1 ml of concentrated hydrochloric acid (R) and 2 ml of 5 pp 100 potassium thiocyanate solution (R) to 10 ml of the solution prepared for tests under paragraph 6.4. The resulting red coloration should be less intense than that observed in a control using 1 ml of iron (III) salt solution in a concentration of 0.010 g of iron per liter, 9 ml of water and the same quantities of the same reagents. (Iron content should be less than 10 mg/kg).

Iron may also be analytically quantified by atomic absorption spectrometry in accordance with the technique detailed in the Compendium.

6.9. Cadmium

Using the method described in the Annex, quantify cadmium analytically in the test solution prepared according to Par. 6.4. (Cadmium content should be less than 1 mg/kg).

6.10. Lead

Using the method described in the Compendium, determine lead content analytically in the test solution prepared according to Par. 6.4. (Lead content should be less than 1 mg/kg).

6.11. Mercury

Using the method described in the Annex, determine the mercury content analytically in the test solution prepared according to Par. 6.4. (Mercury content should be less

than 1 mg/kg).

6.12. Arsenic

Using the method described in the Annex, determine the arsenic content analytically in the test solution prepared according to Par. 6.4. (Arsenic content should be less than 1 mg/kg).

7. Storage

Citric acid should be stored in a dry place in air-tight bags.