

Citric Acid Anhydrous Fine Granular

Specifications and Tests

1. **Appearance:** **colourless crystals or white, granular to fine powder**

2. **Odour:** **odourless**

3. **Identity:** **corresponds**

Proceed according to the Identification Tests A, C and E described in the monograph "Anhydrous Citric Acid" of Ph. Eur.

4. **Water:** **max. 0.2 %**

(K. Fischer)

Proceed according to the test described in the monograph "Anhydrous Citric Acid" of Ph. Eur.

5. **Extraneous matter:** **passes CBT test**

Dissolve 250 g Citric Acid in 500 ml pure demineralized water. Filter on a 0.8 µm membrane filter. The surface of the filter has to remain practically white without obnoxious particles or detrimental particles and has essentially no physical matter.

6. **Heavy metals:** **max. 1 ppm**

1. Principle

Inductively Coupled Plasma (ICP)

2. Reagents

a) Standard Solutions: Titrisol Merck (1.0 g/l)

b) Citric Acid, free of cations, sulphate and other acids

c) Intermediate Solution 1

Pipet 1.0 ml each of the following standard solutions: Al, As, Ba, Ca, Cd, Cr, Cu, Fe, Mg, Ni, Pb, Si and Zn and dilute to 100.0 ml with twice deionized water

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- d) Intermediate Solution 2
Pipet 10.0 ml of the Hg standard solution and dilute to 100.0 ml with twice deionized water

3. Procedure

- a) Sample preparation
Dissolve 10.0 g of Citric Acid in 100.0 ml twice deionized water
- b) Standard preparation
To 10.0 g of Citric Acid (2.b) add 5.0 ml Intermediate Solution 1, 0.1 ml standard solution of K, 0.3 ml standard solution of SO₄, 50 µl Intermediate Solution 2 and dilute to 100.0 ml with twice deionized water.
- c) Blank
Dissolve 10.0 g of Citric Acid (2.b) in 100.0 ml of twice deionized water.

7. **Arsenic:** **max. 1 ppm**
Proceed as described under position 6)

8. **Lead:** **max. 0.5 ppm**
Proceed as described under position 6)

9. **Mercury:** **max. 1 ppm**
Proceed as described under position 6)

10. **Copper:** **max. 1 ppm**
Proceed as described under position 6)

11. **Zinc:** **max. 1 ppm**
Proceed as described under position 6)

12. **Iron:** **max. 1 ppm**
Proceed as described under position 6)

13. **Barium:** **max. 1 ppm**
Proceed as described under position 6)

14. **Calcium:** **max. 10 ppm**
Proceed as described under position 6)

15. **Magnesium:** **max. 1 ppm**

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Proceed as described under position 6)

- 16. Aluminium:** **max. 0,2 ppm**

Proceed as described under position 6)

- 17. Chlorides:** **max. 5 ppm**

Proceed according to the test described in the monograph "Anhydrous Citric Acid" of Ph. Eur.

- 18. Sulphates:** **max. 30 ppm**

Proceed as described under position 6)

- 19. Oxalates:** **max. 10 ppm**

High Performance Liquid Chromatography

a) Apparatus

Pump	Waters 626 inert
Injection system	Waters 600 S
Column	Ion Pac AS 11 (DIONEX) 250 x 4.0 mm i.d.
Detector	PED (DIONEX)

b) Working conditions

Mobile phase 1	twice deionized water
Mobile phase 2	30 mMol NaOH in twice deionized water
Flow rate	1.8 ml / min
Injected volume	25 µl

c) Standards

Gradient from 15 % to 80 % of eluent 2 in 40 min.

Oxalic acid standard: dissolve 140 mg of oxalic acid in 1000.0 ml of twice deionized water.

Citric Acid standard: dissolve 10.0 g of Citric Acid free of cations, sulphates and other acids in 100.0 ml of twice deionized water.

Mix oxalic acid standard with Citric Acid standard to obtain concentrations of 0, 5, 10, 20 and 50 mg of oxalic acid per kg of citric acid.

d) Sample Solution

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Dilute 1.0 g of sample in 100.0 ml of twice deionized water

- 20. Readily carbonisable substances: passes test**
Proceed according to the test described in the monograph "Anhydrous Citric Acid" of Ph. Eur.
- 21. Related substances: passes test**
Proceed according to the test described in the monograph "Citric Acid" of JP.
- 22. Sulphated ash: meets USP requirements**
Proceed according to the test "Residue on ignition" described in the monograph "Citric Acid" of USP (weight = 20 g).
- 23. Organic Volatile Impurities: meets USP requirements**
Proceed according to the test described in the monograph "Citric Acid" of USP.
- 24. Tridodecylamine: meets FCC requirements**
Proceed according to the test described in the monograph "Citric Acid" of FCC.
- 25. Polycyclic aromatic hydrocarbons: meets JP requirements**
Proceed according to the test described in the monograph "Citric Acid" of JP.
- 26. Colour (500 g/l, T at 405 nm in a 1 cm cell): min 98 %**
- 27. Assay: 99.8 - 100.2 % (on anhydrous substance)**

Dissolve 0.550 g of Citric Acid in 50 ml of water. Using 0.5 ml of phenolphthalein solution as indicator, titrate with 1N sodium hydroxide until a pink colour is obtained.

1 ml of 1N sodium hydroxide is equivalent to 64.03 mg of Citric Acid.
- 28. Fineness:**
(US Standard sieves)
- | | | |
|---------------|--------------------|----------|
| On sieve | No. 30 (0.595 mm) | max. 5 % |
| Through sieve | No. 100 (0.149 mm) | max. 5 % |