

Citric Acid Solution 46% Food Grade

Specifications and Tests

1. Appearance: clear and practically colourless solution

2. Identity: corresponds

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

3. 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

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

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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.

- 4. Arsenic: max. 1 ppm**
Proceed as described under position 3)
- 5. Lead: max. 0.5 ppm**
Proceed as described under position 3)
- 6. Mercury: max. 1 ppm**
Proceed as described under position 3)
- 7. Copper: max. 1 ppm**
Proceed as described under position 3)
- 8. Zinc: max. 1 ppm**
Proceed as described under position 3)
- 9. Iron: max. 5 ppm**
Proceed as described under position 3)
- 10. Barium: max. 1 ppm**
Proceed as described under position 3)
- 11. Calcium: max. 20 ppm**
Proceed as described under position 3)
- 12. Magnesium: max. 1 ppm**
Proceed as described under position 3)
- 13. Aluminium: max. 0.2 ppm**
Proceed as described under position 3)

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14. Chlorides: **max. 5 ppm**
Proceed according to the test described in the monograph "Citric Acid Monohydrate" of Ph. Eur.

15. Sulphates: **max. 50 ppm**
Proceed as described under position 3)

16. Oxalates: **max. 10 ppm**
High Performance Liquid Chromatography

a) Apparatus

| | |
|------------------|--------------------------------------------|
| Pump | Waters 626 inert |
| Injection system | Waters 600 S |
| Column | Ion Pac AS11 (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 μ l |

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 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

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

17. Related substances: **passes test**

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Proceed according to the test described in the monograph "Citric Acid" of JP.

- 18. Sulphated ash: meets USP requirements**
Proceed according to the test "Residue on ignition" described in the monograph "Citric Acid" of USP (weight = 20g Citric Acid).
- 19. Organic Volatile Impurities: meets USP requirements**
Proceed according to the test described in the monograph "Citric Acid" of USP.
- 20. Tridodecylamine: meets FCC requirements**
Proceed according to the test described in the monograph "Citric Acid" of FCC.
- 21. Polycyclic aromatic hydrocarbons: meets JP requirements**
Proceed according to the test described in the monograph "Citric Acid" of JP.
- 22. Colour (500 g/l, T at 405 nm in a 1 cm cell): min 97 %**
- 23. Assay: 46%, 47%, 50%, 69 % (anhydrous)**
(04 3300 4, 04 8046 0, 04 3302 0, 04 3301 2)

Dilute the equivalent of 0.550 g 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.

Note: ppm = mg/kg Citric Acid