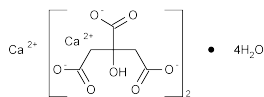


## Calcium Citrate



$C_{12}H_{10}Ca_3O_{14} \cdot 4H_2O$  570.49  
1,2,3-Propanetricarboxylic acid, 2-hydroxy-, calcium salt (2:3), tetrahydrate;  
Calcium citrate (3:2), tetrahydrate [5785-44-4].

### DEFINITION

Calcium Citrate contains four molecules of water of hydration. When dried at 150 ° to constant weight, it contains NLT 97.5% and NMT 100.5% of  $Ca_3(C_6H_5O_7)_2$ .

### IDENTIFICATION

- **A.**  
**Analysis:** Dissolve 0.5 g in a mixture of 10 mL of water and 2.5 mL of 2 N nitric acid. Add 1 mL of mercuric sulfate TS, heat to boiling, and add 1 mL of potassium permanganate TS.  
**Acceptance criteria:** A white precipitate is formed.
- **B.**  
**Sample:** 0.5 g of Calcium Citrate  
**Analysis:** Ignite completely the *Sample* at as low a temperature as possible, cool, and dissolve the residue in dilute glacial acetic acid (1:10). Filter, and add 10 mL of ammonium oxalate TS to the filtrate.  
**Acceptance criteria:** A voluminous white precipitate that is soluble in hydrochloric acid is formed.

### ASSAY

- **PROCEDURE**  
**Sample solution:** Dissolve 350 mg of Calcium Citrate, previously dried at 150 ° to constant weight, in 12 mL of 0.5 M hydrochloric acid, and dilute with water to about 100 mL.  
**Analysis:** While stirring the *Sample solution*, add 30 mL of 0.05 M edetate disodium VS from a 50-mL buret. Add 15 mL of 1 N sodium hydroxide and 300 mg of hydroxy naphthol blue, and continue the titration to a blue endpoint. Each mL of 0.05 M edetate disodium is equivalent to 8.307 mg of calcium citrate ( $Ca_3(C_6H_5O_7)_2$ ).  
**Acceptance criteria:** 97.5%–100.5% on the dried basis

### IMPURITIES

- **ARSENIC, Method I (211)**  
**Test preparation:** Dissolve 1 g of Calcium Citrate in 5 mL of 3 N hydrochloric acid, and dilute with water to 35 mL.  
**Acceptance criteria:** NMT 3 ppm
- **HEAVY METALS, Method I (231)**  
**Test preparation:** Dissolve 1 g of Calcium Citrate in a mixture of hydrochloric acid and water (2:20). Add 1.5 mL of ammonium hydroxide, and dilute with water to 25 mL.  
**Acceptance criteria:** NMT 20 ppm
- **LEAD (251)**  
**Test preparation:** Dissolve 0.5 g of Calcium Citrate in 20 mL of 3 N hydrochloric acid. Evaporate this solution on a steam bath to 10 mL, dilute with water to 20 mL, and cool. Use 5 mL of *Diluted Standard Lead Solution* (5 µg of Pb) for the test.  
**Acceptance criteria:** NMT 10 ppm
- **LIMIT OF FLUORIDE**  
[NOTE—Prepare and store all solutions in plastic containers.]  
**Standard stock solution:** 1000 µg/mL of fluoride ion from USP Sodium Fluoride RS in water  
**Standard solution:** 5 µg/mL of fluoride ion from *Standard stock solution*. [NOTE—Prepare on the day of use.]  
**Linearity solution A:** Transfer 1.0 mL of the *Standard solution* to a 250-mL plastic beaker. Add 50 mL of water, 5 mL of 1 N hydrochloric acid, 10 mL of 1.0 M sodium citrate,

and 10 mL of 0.2 M edetate disodium. If necessary, adjust with 1 N sodium hydroxide or 1 N hydrochloric acid to a pH of 5.5. Transfer to a 100-mL volumetric flask, and dilute with water to volume. This solution contains 0.05 µg/mL of fluoride.

**Linearity solution B:** Transfer 5.0 mL of the *Standard solution* to a 250-mL plastic beaker, and proceed as directed for *Linearity solution A* beginning with "Add 50 mL of water,". This solution contains 0.25 µg/mL of fluoride.

**Linearity solution C:** Transfer 10.0 mL of the *Standard solution* to a 250-mL plastic beaker, and proceed as directed for *Linearity solution A* beginning with "Add 50 mL of water,". This solution contains 0.50 µg/mL of fluoride.

**Sample solution:** Transfer 1.0 g of Calcium Citrate to a 100-mL beaker. Add 10 mL of water and, while stirring, 10 mL of 1 N hydrochloric acid. When dissolved, boil rapidly for 1 min, transfer the solution to a 250-mL plastic beaker, and cool in ice water. Add 15 mL of 1.0 M sodium citrate and 10 mL of 0.2 M edetate disodium, and adjust with 1 N sodium hydroxide or 1 N hydrochloric acid to a pH of 5.5. Transfer this solution to a 100-mL volumetric flask, and dilute with water to volume.

**Electrode system:** Use a fluoride-specific, ion-indicating electrode and a silver–silver chloride reference electrode connected to a pH meter capable of measuring potentials with a minimum reproducibility of ±0.2 mV (see *pH (791)*).

### Analysis

**Samples:** *Linearity solution A, Linearity solution B, Linearity solution C, and Sample solution*  
Transfer 50 mL of each *Linearity solution A, Linearity solution B, and Linearity solution C* to separate 250-mL plastic beakers, and measure the potential of each solution with the *Electrode system*. Between each reading wash the electrodes with water, and absorb any residual water by blotting the electrodes dry. Plot the logarithms of the fluoride concentrations (0.05, 0.25, and 0.50 µg/mL, respectively) versus potential to obtain a Standard response line.  
Transfer 50 mL of the *Sample solution* to a 250-mL plastic beaker, and measure the potential with the *Electrode system*. From the measured potential and the Standard response line determine the concentration, *C*, in µg/mL, of fluoride ion in the *Sample solution*. Calculate the percentage of fluoride in the specimen taken by multiplying *C* by 0.01.

**Acceptance criteria:** NMT 0.003%

### LIMIT OF ACID-INSOLUBLE SUBSTANCES

**Sample solution:** Dissolve 5 g of Calcium Citrate by heating with a mixture of hydrochloric acid and water (10:50) for 30 min.

**Analysis:** Filter, wash, and dry at 105 ° for 2 h the residue so obtained.

**Acceptance criteria:** The weight of the residue is NMT 10 mg (0.2%).

### SPECIFIC TESTS

- **LOSS ON DRYING (731):** Dry a sample at 150 ° for 4 h: it loses from 10.0% to 13.3% of its weight.

### ADDITIONAL REQUIREMENTS

- **PACKAGING AND STORAGE:** Preserve in well-closed containers.
- **USP REFERENCE STANDARDS (11)**  
USP Sodium Fluoride RS