

PFP7 Flame Photometer

Protocol: P05-005A

Determination of calcium in milk

■ Introduction

To measure the calcium in milk, a sample is ashed and then dissolved in dilute hydrochloric acid to prevent low calcium readings. Lanthanum chloride is added to mask phosphate, sulphate and aluminium interferences. The standards and blank solution are prepared and treated in the same way.

■ Materials required

Equipment

Jenway flame photometer with calcium filter fitted
Accurate balance weighing to 0.0005g
Silica crucible
High temperature oven
Volumetric flasks
Filter paper

Reagents

Concentrated hydrochloric acid
10% Lanthanum chloride solution
Deionised water
Calcium carbonate

■ Method

Reagent preparation

1. Dilute concentrated hydrochloric acid 1:4 with deionised water.

CAUTION: ALWAYS ADD ACID TO WATER WITH GREAT CARE. DO NOT ADD WATER TO CONCENTRATED ACID.

Blank Preparation

1. Measure 2.5ml of diluted hydrochloric acid into a 100ml volumetric flask.
2. Add 2.0ml of 10% lanthanum chloride solution
3. Make up to 100ml with deionised water. This is the blank solution.

Standard Preparation

1. Place 1.249gm A.R. calcium carbonate in approximately 50ml of deionised water.
2. Add drop-wise concentrated hydrochloric acid until the calcium carbonate is dissolved (it should take approx. 10ml).
3. Make up to 100ml with deionised water.
4. This will give 500ppm stock calcium standard.
5. The stock solution should be diluted to give standards of 2.5, 5.0, 7.5 and 10ppm calcium.

Sample preparation

1. Place 4g (accurately weighed) of milk in a dry silica crucible.
2. Ash sample in an oven at 500-525°C.
3. When cool, dissolve the ash in 5ml of diluted hydrochloric acid.
4. Transfer to 100ml volumetric flask and make up to mark with deionised water.
5. Filter through Whatman filter paper.
6. Pipette 50ml of the filtrate into 100ml volumetric flask.
7. Add 2ml of 10% lanthanum chloride solution and make up to mark with deionised water.

Method

1. Aspirate the blank solution and set the zero.
2. Aspirate the 10ppm calcium standard and set the full scale.
3. Reset the zero.
4. Aspirate the intermediate standards, note the readings and construct a calibration curve.
5. Aspirate the test samples and note readings.
6. The result is in ppm of calcium for milk.
7. If percent calcium required, multiply calcium ppm reading by 0.025 and this gives the percentage calcium in milk. The normal level for milk is approximately 0.11%.