

Flame Photometer PFP7

Protocol: P05-011A

## The Simple Flame Photometric Determination of Calcium

### ■ Introduction

A PFP7 flame photometer offers a quick and easy method for the determination of a samples' calcium content. The following method is suitable for the determination of calcium in aqueous samples that have no interfering ions present.

### ■ Materials Required

#### Equipment

Jenway flame photometer  
Accurate balance weighing to 0.0005g  
Volumetric flasks

#### Reagents

Calcium Standard – 1000ppm (Part number 025 009)  
Deionised Water

### ■ Method

#### Sample preparation

If there are interfering ions present, calcium should be precipitated as calcium oxalate and dissolved in perchloric acid.

Dissolve 1.0g of sample in deionised water in a 100.0ml volumetric flask. Dilute to volume with deionised water. If required, perform serial dilutions to produce a sample with a calcium concentration between 10 and 100ppm.

#### Standard preparation

From the 1000ppm calcium standard, prepare standards of 100, 75, 50 and 25ppm using deionised water as diluent.

For lower concentration samples, a set of calcium standards of 50, 40, 20 and 10ppm should be prepared. Do not set a top standard lower than 50 due to the insensitivity of the technique to low calcium concentrations.

#### Method

1. Set up the flame photometer as detailed in the instruction manual for calcium analysis.
2. Aspirate the blank solution and set the zero.
3. Aspirate the standards into the flame photometer.
4. Plot a standard curve of calcium concentration against intensity.
5. Aspirate the sample solution into the flame and record the reading.

### ■ Calculation

From the calibration graph, determine the samples' concentration from the recorded reading. If required, multiply the determined concentration by the dilution factor.

## ■ Interfering Ions

If there are interfering ions present, the calcium should be precipitated as calcium oxalate by adding 1.0ml of ammonium oxalate/oxalic acid mixture

### **Ammonium oxalate/oxalic acid mixture**

- a. 5ml of 0.1M oxalic acid
- b. 95ml of 0.1M ammonium oxalate.

The precipitate should be filtered from the sample, washed with deionised water and dissolved using 25ml 0.1M perchloric acid solution. Dilute to 100.0ml with deionised water

Ensure that the standard and blank solutions contain the same concentration of perchloric acid as the aspirated sample solution.