

## The Flame Photometric Determination of Sodium and Potassium in Silicates, Minerals and Ores

### ■ Introduction

A PFP7 flame photometer offers a method for the determination of the sodium and potassium content of a wide variety of silicates, minerals and ore samples.

**Warning: - This method uses Hydrofluoric Acid. Extreme care should be taken as this chemical can cause severe burns. Please read the relevant MSDS before use.**

### ■ Materials Required

#### Equipment

Jenway flame photometer fitted with sodium and calcium filters  
Accurate balance weighing to  $\pm 0.0005\text{g}$   
Volumetric flasks  
Plastic pipette or measuring cylinder  
Platinum crucibles

#### Reagents

Sodium Standard Solution – 1000ppm (Jenway Part Number 025 021)  
Potassium Standard Solution – 1000ppm (Jenway Part Number 025 023)  
Hydrofluoric Acid 40% w/w  
Hydrochloric Acid 36% w/w  
Sulphuric Acid 98% w/w  
Deionised Water

### ■ Method

#### Diluent Preparation

Prepare a 1 in 10 solution of concentrated hydrochloric acid by diluting 100ml of acid with 900ml of deionised water.

#### Standard preparation

1. Accurately pipette 10.0ml of the 1000ppm sodium standard and 10.0ml of the 1000ppm potassium standard into a 1.0L volumetric flask containing 400ml of deionised water.
2. Add 100ml of concentrated hydrochloric acid.
3. Make up to volume with deionised water. This is a 10ppm sodium and potassium standard solution.
4. From the 10ppm standard solution, pipette 6.0, 4.0 and 2.0ml into separate 100.0ml volumetric flasks and dilute to volume with diluent. These standard solutions have sodium and potassium concentrations of 6, 4 and 2ppm respectively.

#### Sample preparation

1. Accurately weigh 1.0g of a finely powdered sample of the substance under test into a platinum crucible.
2. Moisten the sample with 5 drops of deionised water.

3. Add 5 drops of sulphuric acid and using a **plastic** pipette or measuring cylinder, 5ml of hydrofluoric acid (40%).

**(Take all necessary safety precautions when handling Hydrofluoric Acid)**

4. Evaporate and fume to dryness on a hotplate.
5. Repeat the addition and evaporation of the sulphuric and hydrofluoric acid until the sample is completely decomposed.
6. Extract the residue with diluent and quantitatively transfer the solution to a 200.0ml volumetric flask. Dilute the solution to volume with diluent.

## Method

1. Set up the flame photometer as detailed in the instruction manual for the element under test.
2. Aspirate the diluent solution and set the zero reading.
3. Aspirate the 10, 6, 4 and 2ppm standards into the flame photometer.
4. Plot a standard curve of sodium (or potassium) concentration against intensity.
5. Aspirate the sample solution into the flame and record the reading. If the reading is above the value recorded for the 10ppm standard, dilute the sample until the value is within the range of the calibration curve

## ■ Calculation

From the results obtained from the calibration graphs, the percentage of sodium or potassium in the sample can be determined. The results should be multiplied by any dilution factor used in the samples preparation.

$$\% \text{ Sodium or Potassium} = \frac{\text{Na or K sample (ppm)} \times 200 \times 100}{1000000 \times \text{sample wt (g)}}$$