

The Flame Photometric Determination of Alkali Metals in Cement

■ Introduction

When using a flame photometer for the determination of sodium, difficulty is experienced when lime (calcium) is present in appreciable amounts of more than a few per cent. This is due to the calcium emitting some radiation, which passes through the sodium filter to a measurable extent. This error must be taken into account or it can, in the case of cements, cause errors of up to 100% in the reported sodium content.

Methods that remove the calcium from the sample, or methods that add equivalent amounts of calcium to the instrument calibration solutions are available, but in this method the interference is suppressed by the addition of aluminium to the sample.

■ Materials Required

Equipment

Jenway flame photometer
Accurate balance weighing to 0.0005g
Volumetric flasks
Whatman filter paper – No.40
Ice bath

Reagents

Aluminium – Metal
Mercury (II) Chloride – 5% solution
Nitric Acid 70%w/w
Hydrochloric Acid 37%w/w
Sodium Standard – 1000ppm (Part number 025 021)
Potassium Standard – 1000ppm (Part number 025 023)
Deionised Water

■ Method

Aluminium nitrate solution preparation

1. Whilst cooling in an ice bath, dissolve 30.0g of aluminium in 400ml of 1:1 nitric acid in a 1.0L volumetric flask. Add a few drops of 5% mercury (II) chloride solution to facilitate the dissolution of the aluminium.
2. Allow the solution to cool to room temperature and dilute to volume with deionised water.

Standard preparation

1. Accurately pipette 10.0ml of the 1000ppm sodium standard and 10.0ml of the 1000ppm potassium standard into a 1.0 L volumetric flask containing 400ml of deionised water.
2. Add 10ml 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, prepare 100.0ml of 7.5, 5 and 2.5ppm standard solutions using deionised water as diluent.

Sample preparation

1. Mix 0.5g of cement, accurately weighed, with 45ml of deionised water and 5ml of concentrated hydrochloric acid.
2. Heat the solution to boiling point and filter through a no. 40 filter paper into a 500ml volumetric flask.
3. Wash the filter paper with 6 x 30ml aliquots of hot deionised water.
4. Add 100ml of aluminium nitrate solution and allow the solution to cool.
5. Once cool, dilute the solution to volume with deionised water.

Method

1. Set up the flame photometer as detailed in the instruction manual.
2. Aspirate deionised water and set the zero.
3. Aspirate the standards into the flame photometer.
4. Plot a standard curve of sodium/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.

$$\text{Sodium content of cement (\%)} = \frac{\text{sample (ppm)} \times 500 \times 100\%}{1000000 \times \text{sample weight (g)}}$$

$$\text{Potassium content of cement (\%)} = \frac{\text{sample (ppm)} \times 500 \times 100\%}{1000000 \times \text{sample weight (g)}}$$