The preparation of Aqueous Solutions for the Determination of Sodium and Potassium

Introduction
This protocol describes the general preparation of samples for determination of sodium and potassium in the flame photometer. The range covered is 0.05 to 5 ppm Na, 0.1 to 10 ppm K (in sample solution) with an accuracy of ±1% (95% prob.) at half to full range.

Materials Required

Equipment
- Platinum crucibles (10ml capacity)
- JENWAY Digital Flame Photometer
- Volumetric Glassware
- Hotplate

Reagents
- Sodium Standard Solution – 1000ppm (Jenway Part Number 025 021)
- Potassium Standard Solution – 1000ppm (Jenway Part Number 025 023)
- Deionised Water
- Aluminium – spectroscopic grade
- Hydrofluoric Acid – 40% analar
- Sulphuric Acid – SG 1.84

Method

Reagent preparation
1. 5ppm Sodium Standard - Dilute 5ml of the 1000ppm sodium standard solution to 1000ml with deionised water. Store in an air-tight polythene bottle.

2. 10ppm Potassium Standard - Dilute 10ml of the 1000ppm potassium standard solution to 1000ml with deionised water. Store in an air-tight polythene bottle.

3. Hydrofluoric-Sulphuric Acid Mixture - Dissolve 3.0g of aluminium in a mixture of 180ml of hydrofluoric acid (40%) and 20ml of sulphuric acid (SG 1.84).

4. Other Sodium and Potassium Standard Solutions – prepare 4, 3, 2 and 1ppm sodium solutions and 8, 6, 4 and 2ppm potassium solutions by serial dilution.

Calibration
1. Set up the flame photometer for either sodium or potassium following the instruction manual.

2. Standardise the Flame Photometer to give a reading of 100 when aspirating either the 5ppm sodium or 10ppm potassium standard solution and zero when aspirating deionised water.

3. Aspirate the other standard solutions and from the readings, construct a calibration graph.

Sample Preparation and Analysis
1. Weigh 0.04g of sample into a platinum crucible.

2. Add 2ml of the hydrofluoric-sulphuric acid mixture.

3. Heat the crucible gently on a hot plate until white fumes of sulphuric acid are evolved.
4. Cool and add 5 to 10ml of deionised water.

5. Warm the crucible to bring the residue into solution.

6. Transfer the contents to a 50ml volumetric flask and dilute to volume with deionised water.

7. Aspirate the sample and note the reading. If the reading is above the range of the calibration graph, dilute the sample solution and re-aspirate.

8. Determine the sodium and potassium in the solution using calibration charts.

Notes
1. A ‘blank’ using the hydrofluoric-sulphuric acid and deionised water only, should be performed for each batch of the acid mixture prepared. If care is used in ensuring freedom from contamination, e.g. from the atmosphere, there is no need to carry out a blank determination with each batch of determinations, just when a new batch of acid mixture is prepared.

2. Samples expected to contain more than 40% of calcium and less than 0.5% sodium and potassium should be treated with 2ml of a reagent containing 6g of aluminium in 200ml of hydrofluoric-sulphuric acid mixture.

3. When calibrating the instrument, the standards should be evaporated to fumes of sulphuric acid with 2ml of the hydrofluoric-sulphuric acid reagent.

4. Samples containing high concentration of calcium will give a white residue of calcium sulphate, which may not completely dissolve on dilution to 50ml. The presence of this insoluble material whether in suspension or separated by decantation, will not affect the accuracy of sodium or potassium result.