Research Project
Flame Photometry

“If we knew what it was we were doing, it would not be called research, would it?”

—Albert Einstein
Physicist

Abstract
This is a spectrophotometric technique for determining \([\text{Na}^+]\) and/or \([\text{K}^+]\) in solution. A sample is aspirated into a flame and the intensity of the light emitted at selected wavelengths is measured. Measurements are made individually on the samples and on a series of standards. A calibration plot is generated from the absorbance readings for the standards. The sample concentrations are read from this plot.

Application
The determination of sodium and potassium ion concentrations has traditionally been a difficult analytical problem. This is due to the chemical nature of these ions. They cannot be easily reduced. They have no acid/base properties and they form no insoluble compounds. Standard analytical techniques, like titrations, are out of the question. ISEs are possible, but not very reliable due to the general un-reactivity of these ions. Fortunately, there is one technique that does work. Sodium and potassium, like all the alkali metals, have strong emission spectra. The sodium spectrum is dominated by the sodium D line which produces the characteristic orange color of sodium vapor lamps. These emissions can be used to make spectral measurements.

Sample Issues
Because the sample is aspirated into a flame the color of the sample is not relevant. The only significant interference to worry about is the small size of the capillary tube that feeds the sample into the instrument. Anything that would clog the tube is bad. Please pay attention to the following two points.

- The samples must not have any suspended solids in them. If the sample is cloudy at all, consult your instructor. Suitable micro-filters are available.
- The capillary feed tube must be rinsed after each use. When you are done running a sample, put the capillary feed tube in a beaker of nano-pure water and leave it there.

Background
Flame photometry is the most widely used emission spectroscopy technique. Flame photometers are designed to determine the amounts of metal ions present in samples. The instrument consists of a high temperature burner flame, a detector aligned on the flame and a series of filters between the flame and...
detector. The filters are selected to limit the wavelengths of light to those characteristic for the metal ion being measured.

A liquid sample is aspirated into the flame and the light emitted is measured. The tricks are maintaining a constant temperature flame and feeding the sample solution into the flame at a constant rate. A good instrument operated correctly will produce results accurate to three significant figures. The instrument available for your use (the Buck Scientific PFP-7 flame photometer) is very similar to one commonly used in clinical labs for the determination of [Na⁺] and [K⁺] in blood plasma and urine samples.

**Procedure**
The activities required to efficiently collect data using a flame photometer can be summarized as follows:

1. Make standard solutions.
2. Aspirate each sample and standard into the flame photometer and record the emission values.
3. Generate a calibration plot.
4. Read values from the plot.

**Using the Instrument**
The instrument is very quick and easy to use. About the only problem likely to be encountered is the plugging up of the capillary tube used to aspirate the sample. Should this happen, notify your instructor.

The values generated by the instrument have no absolute meaning. Like absorption spectroscopy, a calibration curve must be generated by taking intensity readings from the display for known samples then plotting those readings against the sample concentrations. Once a calibration curve has been generated, then samples can be measured and the concentrations read from the calibration plot.

**Reagents & Other Materials**
Aside from your samples, the only chemicals needed are a sodium standard, a potassium standard and the nano-pure water. Solid, reagent grade NaCl is provided for sodium standards and solid, reagent grade KCl for potassium standards. It is recommended that you make a 1.0 x 10⁻³ M solution as your most concentration standard, then perform a serial 1 to 5 dilution to generate more standards.

In addition to the normal lab items found in your locker and in the room, the materials that will be provided by the preproom are detailed in the Research Project Materials List.

You are responsible for providing your own samples to evaluate. Additional, non-sample materials may be requested of the preproom. Such requests must be submitted by your TA.

**Procedure Outline**
The following is a description of the steps required to obtain results for a generic sample containing sodium and/or potassium ions in concentrations within the effective range of the spectrometer. This procedure will need to be modified, and more detail added, based on the nature and number of samples to be evaluated.

**Zero the Instrument**
Locate the small capillary tube on the right side of the instrument. If it is not immersed in a beaker of "nano-pure" water, do so now. Adjust the “blank” knob on the front panel until the display reads 0.

**Measuring the First Standard**
Move the capillary tube from the “nano-pure” water to a beaker containing your most concentrated standard. Allow the instrument to run for a few minutes while the sample is drawn up the capillary tube into the flame. Once the reading on the display has stabilized, adjust the sensitivity controls until the display reads something near to 1000. Record this value along with the standard concentration.

**Measuring the Remaining Standards & Samples**
Return the capillary tube to the beaker of “nano-pure” water. Let the instrument run for a minute or so to clear the sample from the capillary tube. Now transfer the capillary tube to a beaker containing your least concentrated standard. When the display reading stabilizes, record the value. Repeat for all remaining standards and samples.