

Experiment II.2: **Flame Photometry: Determination of Sodium and Potassium**

The PFP7 Flame Photometer (Jenway) is used in this experiment to analyze an aqueous solution for its sodium and potassium content. This instrument has been widely used in clinical laboratories for the determination of sodium and potassium electrolytes in blood serum and other biological samples. Atomic spectroscopy is covered in Chapter 28 of your text and in particular in section 28B-3. This material should be studied in preparation for this experiment.

The principle of the method is based on the light energy emitted by sodium and potassium that is proportional to its concentration. Concentration levels are also dependent on instrumental parameters such as flame temperature and rate of sample injection. These are difficult to maintain constant and lead to variations in the energy reaching the photometer. In an effort to circumvent these variables, several instrument manufacturers make use of an internal standard. Variations are thus automatically compensated for by comparing the light energy emitted by sodium or potassium with the light energy emitted by an internal standard cation of known concentration added to the sample (*e.g.*, a lithium salt). The instrument has three detectors tuned uniquely for sodium, potassium and lithium, and electronic circuitry that compare the energy emitted by sodium or potassium with the energy from the standard. For this laboratory, however, no internal standard will be used.

Concentrations of Na and K are proportional to the readings on the digital readout of the flame photometer. A calibration curve is needed to convert these readings to concentration. Three standards containing **both** Na and K are used to establish the calibration curve.

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Preparation of Solutions

- *Glassware.* You will need five 100mL volumetric flasks, one adjustable pipette P1000 with plastic tips and plastic cuvettes.
 - *Mixed Na and K stock solution.* Using oven-dried salts, accurately prepare a 100mL solution containing both NaCl (40mM approx. 0.24g, use an analytical balance and KCl 60mM, approx. 0.45g, use an analytical balance). Note that you are going to use this solution for preparation of standard solutions. Use a powder funnel and weigh by difference. It may be helpful to transfer and wash the salt(s) into the volumetric flask in several steps to avoid clogging the funnel. The concentrations of Na and K should be known to an accuracy of ± 2 ppt.

MIX ALL SOLUTIONS THOROUGHLY. AVOID CONTAMINATION OF YOUR SOLUTIONS AND GLASSWARE WITH SALT FROM YOUR HANDS. USE ONLY DEIONIZED WATER.

- *Standard solutions.* Using an adjustable pipette transfer 1.00, 0.50 and 0.25 mL of the Na/K stock solution to three labeled 100.00 mL volumetric flasks. Carefully dilute each volumetric flask to the 100mL mark with deionized water. The result should be three solutions with the following nominal concentrations:

No.	Sodium [mM]	Potassium [mM]
1	0.40	0.60
2	0.20	0.30
3	0.10	0.15

- *Unknown stock solution.* The unknown is a mixture of NaCl and KCl. Prepare a 100 mL stock solution of the unknown in a volumetric flask.
- *Solution of unknown for analysis.* Pipet 1mL of unknown stock solution into the 100 mL volumetric flask and dilute to the mark with deionized water.

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Procedure

- Consult with your instructor before starting-up the instrument. Follow this procedure:
 1. Plug-in the air compressor and the instrument.
 2. Wait for pressure on the air compressor gauge shows about 2.5psi.
 3. Turn the FUEL ADJUST knob 5 turns anti-clockwise.
 4. Turn ON gas supply.
 5. Turn POWER switch ON.
 6. Depress ignition switch and hold down. Watch the FLM indicator in the display window. If the FLM indicator does not light within approximately 20 seconds, release the switch. Repeat several times if necessary.
 7. Set FILTER SELECT control to **Na**.
 8. Aspirate deionized water for 10 min to allow the instrument to warm-up.
 9. Set the readout to Zero by adjusting the BLANK control.
 10. Set COARSE sensitivity knob on the second position from the left.
 11. Aspirate the most concentrated standard. Adjust the FINE knob to a reading of 100.
 12. Aspirate water, re-adjust Zero if necessary.
 13. Then spray the same solution again. Make final adjustment after 2-3min of aspiration
 14. Aspirate the remaining standard solutions. Make sure to aspirate water between samples. Record the readings. (Perform measurements three times)
 15. Obtain Na reading for the unknown.
 16. Change FILTER SELECT control to **K**. Aspirate water for 2-3 min.
 17. Aspirate the most concentrated standard solution.
 18. Adjust the FINE to read 200.
 19. Repeat steps 12-14.
 20. Obtain K reading for the unknown.
- To **shut-down** the instrument:
 1. Flush the system by aspirating deionized water for about 10 minutes.
 2. Turn the POWER switch off.
 3. Close the fuel supply valves (fully clockwise).
 4. Unplug the instrument and the air compressor.

Data Analysis and Report

- Prepare graphs of your data using a computer. Do **NOT** fit the data to a straight line. A non-linear calibration curve is more likely in the concentration range being used. Determine the Na and K concentrations by interpolation between the two points that bracket each component of the unknown. Convert these concentrations to the original sample using dilution factors and formula weights. **The report consists of your graphs and the weights of NaCl and KCl in the unknown.**