Brooklyn College
Department of Chemistry
Chemistry 41

Flame Photometry

Determination of Sodium and Potassium

The P&I Clinical Flame Photometer is used in this experiment to analyze an aqueous solution for its sodium and potassium content. This instrument is widely used in clinical laboratories for the determination of sodium and potassium in blood serum and other biological samples. Atomic spectroscopy is covered in Chapter 26 of your text. In particular, Section 26B-4 and Feature 26-1 discuss flame photometry. This material should be studied in preparation for this experiment.

This experiment provides a good example of the use of an **internal standard** in analysis. The light energy emitted by sodium and potassium is proportional to its concentration, but it is also dependent on the flame temperature and rate of sample injection. These are difficult to maintain constant and lead to variations in the energy reaching the photometer. In the internal standard method, a third cation of known concentration is incorporated in the sample solution. Variations are automatically compensated for by comparing the light energy emitted by sodium or potassium with the light energy emitted by the internal standard. A lithium salt is used for this purpose. The instrument has three detectors tuned uniquely for sodium, potassium and lithium, and electronic circuitry that compares the energy emitted by sodium or potassium with the energy from the lithium standard.

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. All solutions must contain a constant concentration of the lithium internal standard.

PREPARATION OF SOLUTIONS.

- *Glassware.* You will need three 250 mL volumetric flasks, five 100 mL volumetric flasks, 25 mL pipette, one adjustable pipette P1000 with a plastic tip.
- *Mixed Na and K stock solution.* Dry approximately 4 g of NaCl and 1 g of KCl. Using oven-dried salts, accurately prepare a 250 mL solution containing both NaCl (0.20 M, approx. 3.0 g, use an analytical balance) and KCl (0.010 M, approx. 0.20 g, 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.

- *Lithium stock solution.* Prepare 250 mL of a 0.060 M Li_2SO_4 solution (approx. 1.9 g). Do NOT dry this salt. It is not necessary to prepare this solution with the same precision as the Na/K solution.
- *Standard solutions.* Pipet 25 mL of the Li stock solution into each of four 100 mL volumetric flasks. Add, respectively, 1.00, 0.75, and 0.50 mL of the Na/K stock solution to three of these volumetrics. Use an adjustable pipet P1000. The fourth volumetric contains no Na or K and will be used to establish the zero or background reading. Mark all volumetric flasks (e.g., 1, 2, 3, 4). Carefully dilute each volumetric flask to the 100 mL mark. The result should be four solutions with the following nominal concentrations.

No.	Sodium [mM]	Potassium [mM]	Lithium [M]
1	2.0	0.10	0.030
2	1.5	0.075	0.030
3	1.0	0.050	0.030
4	0.0	0.0	0.030

- *Unknown stock solution.* The unknown is a mixture of NaCl and KCl. Prepare a 250 mL stock solution of the unknown in a volumetric flask.
- *Solution of unknown for analysis.* Pipet 25 mL (clean your pipette well, do not transfer any Na+K solution) of the Li stock solution into a 100 mL volumetric flask, add 1.00 mL of the unknown stock solution and dilute to the mark.

PROCEDURE.

- Consult with your instructor before starting-up the instrument. The procedure can be summarized as follows:
  - Turn power switch ON (up position).
  - Open propane valve (turn fully counterclockwise).
  - Ignite flame by holding switch on right side of the instrument, in front of the chimney, down until the green light comes on.
  - Adjust propane pressure to 15 psi (black knob above gauge).
  - Warm up the instrument for 3 minutes.
- Full scale adjustment. Using a small beaker, put your most concentrated standard in the sample probe. Adjust the control knobs to obtain a reading of 200 for Na, then a reading of 10 for K. This is not the final adjustment. Since the first sample aspirated into the flame may be diluted or contaminated by the previous sample, it is good practice to make the final scale adjustment using a second portion of the standard. Be careful not to change the settings accidentally.
- Obtain readings for the remaining two standards, first for Na, then for K. Repeat each measurement to insure washing out the previous solution.

- Obtain Na and K readings for the unknown.
- Obtain Na and K readings for the zero solution (only Li).
- To shut-down the instrument. Flush the system by aspirating deionized water for about 3 minutes. Then turn the power switch off (down position). Close the propane valve (fully clockwise).

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.