

EXPERIMENT 7

Determination of Sodium by Flame Atomic-Emission Spectroscopy

**USE ONLY *DEIONIZED WATER* (NOT DISTILLED WATER!)
THROUGHOUT THE ENTIRE EXPERIMENT**

Distilled water actually has too much sodium in it. Clean *all* glassware and rinse thoroughly with deionized water both before and after use. There is sufficient sodium in tap water and even distilled water to invalidate your results. Remember also to rinse out your plastic wash bottle several times and then fill it with deionized water.

SAFETY WARNING

CAUTION – Although the natural-gas/oxygen flame is rather small, it has such a high temperature that contact of the flesh with even the outer edge of the flame will instantly produce a third-degree burn. Except for lighting the flame, hands should be kept completely out of the “chimney” or burner housing whenever the flame is burning. In addition, NEVER put your hand above the burner housing.

UNKNOWN

Submit a clean, labeled 100-mL volumetric flask to the instructor so that your unknown sodium solution can be issued. Your name, section number, and your locker number should be written legibly on this flask. The flask does not need to be dry on the inside, but needs to have been rinsed with *deionized* water thoroughly after it has been washed. *The flask must be turned in at least 1 lab period before you plan to do the experiment so that the Teaching Assistants will have time to prepare the unknown. Each student will have his or her own unknown to analyze even if working in pairs.*

BACKGROUND

Flame photometry, now more properly called *flame atomic emission spectrometry*, is a relatively old instrumental analysis method. Its origins date back to Bunsen's flame color tests for the *qualitative* identification of select metallic elements. Probably the most common example of the atomic emission effect is fireworks for 4th of July celebrations and other events. As an analytical method, atomic emission is a fast, simple, and sensitive method for the determination of trace metal ions in solution. Because of the very *narrow* (ca. 0.01 nm) and *characteristic* emission

lines from the **gas-phase atoms** in the flame plasma, the method is relatively free of interferences from other elements. Typical precision and accuracy for analysis of dilute aqueous solutions with no major interferences present are about $\pm 1-5\%$ relative.

The method is suitable for many metallic elements, especially for those metals that are easily excited to higher energy levels at the relatively cool temperatures of typical flames – Na, K, Ca, Rb, Cs, Cu, and Ba. Metalloids and nonmetals generally do not produce isolated neutral atoms in a flame, but rather mostly neutral, polyatomic radicals and ions. Therefore, nonmetallic elements are not suitable for determination by flame emission spectroscopy, except for a very few and under very specialized conditions.

Flame photometry is a highly *empirical*, rather than an *absolute*, method of analysis such as gravimetry. That is, you must *calibrate* the method carefully and frequently. Many different experimental variables affect the intensity of light emitted from the flame. Therefore, careful and frequent calibration is required for good results.

INSTRUMENTATION

Coleman Flame Photometer, Model 21

The Coleman flame photometer represents very old technology indeed. It once found extensive use in clinical labs and even operating rooms to determine sodium, calcium, and potassium in various bodily fluids. This use has been supplanted by more modern, safer, faster, and more accurate methods. The instrument uses a *total-consumption burner* to aspirate aqueous solutions into a natural-gas/oxygen flame. Wavelength isolation is by use of a simple *interference filter* that is designed to transmit only the strong, characteristic sodium-doublet lines at 589.2 nm. [Different filters must be used to transmit the calcium line at 442.7 nm and the two potassium lines at 766.5 and 769.9 nm.] A glass lens is used to focus the light from the flame onto the end of a fiber optic cable, a “light pipe”, which transmits the light onto a photodiode located in the small home-built electronics box by the flame photometer. The electronics therein convert the diode’s output into a digital display.

EQUIPMENT NEEDED

- Wash bottle(s) rinsed several times and then filled with *deionized* water
- One 1-L volumetric flask, from your locker
- 10- and 25-mL volumetric pipets, or a 50-mL burette, from your locker
- Five 100-mL volumetric flasks, available in the locker for this experiment
- Eight to ten small glass containers (aspiration cups) used for aspirating solutions into the flame photometer. These are provided in the locker drawer for this experiment.

PREPARATION OF SOLUTIONS

Standard Sodium Stock Solution, 100.0 ppm

1. Accurately (to 0.1 mg) weigh out by difference 0.1271 g of reagent grade NaCl into a small plastic weighing boat. It is very difficult and time consuming to weigh out this amount exactly. Get it as close as you can, record the exact weight, and correct your concentrations accordingly. **Remember: NEVER transfer chemicals inside an analytical balance.**
2. Carefully transfer the salt *quantitatively* into a 500-mL volumetric flask. Use a few squirts of deionized water from your wash bottle on the weighing boat and the sides of the flask to wash all of it down into the flask. [0.100 g Na/L = 100 mg/L = 100 μ g/mL = 100 ppm Na).
3. Add about 100 mL of deionized water to the flask, swirl several times, and dissolve **all** of the salt before diluting to volume with deionized water. **This is critical.**

Sodium Standard Calibration Solutions

1. Pipet 10.00, 20.00, 30.00, 40.00, and 50.00 mL of the standard 100-ppm sodium solution into the first, second, third, fourth, and fifth 100-mL volumetric flasks, respectively.
2. Dilute carefully to the mark with deionized water and mix thoroughly.
3. Use deionized water for the “blank”.

Unknown Solution

Obtain the unknown from the instructor and carefully dilute to the 100-mL mark with deionized water. Mix thoroughly.

PROCEDURE

Carefully follow the instructions provided you for use of the instrument and determine the emission intensity for each standard, the blank (deionized water), and the unknown.

1. When ready to take emission readings, call the Teaching Assistant to light the flame, stabilize the flame photometer, and instruct you in its proper and safe use.
2. Aspirate deionized water from a small glass container that is designed to fit into the instrument for at least 2-3 minutes to clean out and stabilize the unit. While aspirating deionized water, set the **ZERO** knob on the digital readout unit to 0.0 units. There will be some “bounce” or fluctuation in the meter reading.
3. Aspirate the most concentrated standard solution, and use the **GAIN** knob to set the reading to 100.0 units.

4. Recheck the zero setting with deionized water and the 100 setting again with the most concentrated standard. Repeat if necessary. The instrument may drift substantially while its temperature is stabilizing, and more slowly throughout the experiment.
5. Now aspirate all the standards and unknowns in turn, starting with the least concentrated standard solution. Aspirate deionized water in between each Na solution to clean the unit out. The burner displays a “memory effect”. It takes a while to clean it out the previous solution aspirated.
6. Repeat steps 2-4 a second time to get a good duplicate set of measurements, then repeat the whole sequence a third time.
7. When done, aspirate deionized water for at least 2-3 minutes.
8. Call the Teaching Assistant to shut down the photometer.
9. Clean your work areas up.

HAZARDOUS WASTE DISPOSAL

Essentially there is NO hazardous waste involved in this experiment. All we are dealing with are dilute solutions of ordinary table salt. Empty ALL the solutions down a sink drain with cold water running. Rinse all glassware with distilled water and then rinse the 100-mL volumetric flasks and the glass aspiration cups with deionized water.

DATA TREATMENT

Prepare a calibration curve by plotting the emission intensities as a function of Na concentration. Determine the concentration of sodium in the unknown sample by reading the concentration of the sample which corresponds to its emission intensity from the calibration curve.

Depending on the drift in the instrument and other factors, it may be better to average all three values for each solution and obtain one final value for the unknown, or to get three separate values for the unknown, each using its “own” calibration curve, and average the three values. Try both to see if one approach seems to be better than the other. If the plot appears to be linear, or at least that portion of it that includes your unknown, use some software to run a linear-least-squares fit to the data.

Report the “best estimate” for the average concentration of sodium in ppm ($\mu\text{g/mL}$) and the associated standard deviation of the value.

TEXT REFERENCE

D. A. Skoog, D. M. West, F. J. Holler, and S. R. Crouch, *Analytical Chemistry: An Introduction*, 7th ed., Chapter 23, pp. 594-631.

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