

Estimation of Sodium by flame Photometry

AIM- The aim of the experiment is to estimate sodium in the given sample by flame photometry.

BACKGROUND-

Flame photometry, more precisely called *flame atomic emission spectrometry* or "*flame photometry*" is a traditional instrumental analysis method. It is originated a long back to Bunsen's flame-colour tests for the qualitative identification of some selected metallic elements. Most probably a very simple 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 appreciably 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. Detection limits can be quite low. "Good" elements are having detection limits lying between about 1 ng/ml and 1 $\mu\text{g/ml}$. The method is appropriate for many metallic elements, especially for those metals those are easily excited to higher energy levels at the relatively cool temperatures of some flames – Li, Na, K, Rb, Cs, Ca, Cu, Sr, and Ba. Metalloids and non metals generally do not produce isolated neutral atoms in a flame, but mostly as polyatomic radicals and ions. Therefore, non-metallic elements are not preferred for determination by flame emission spectroscopic studies, except for a very few and under very specialized conditions.

Flame photometry is a highly empirical, in comparison to an absolute, method of analysis such as gravimetry. So the method must be calibrated carefully and frequently. Many different experimental variables affect the intensity of light emitted from the flame and that finding its way to the detector.

REQUIREMENTS-

1. The bottle(s) was/were washed and rinsed several times and then filled with deionized water
2. One 500 ml volumetric flask
3. Assorted volumetric and/or graduated transfer pipettes
4. Five 100 ml volumetric flasks for the standards (experiment locker)
5. Eight to ten small plastic containers for aspirating solutions (experiment locker)

PROCEDURE-

Standard sodium stock solution, 100.0 ppm

Accurately (to 0.1 mg) weighed out by difference 0.1271 g of reagent grade NaCl into a small plastic weighing boat. The exact mass was recorded, and corrected the concentrations accordingly.

(Remember: NEVER transfer chemicals inside an analytical balance.)

Carefully the salt was transferred quantitatively into a 500-mL volumetric flask. Then few squirts of deionized water was used from the 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 µg/mL = 100 ppm Na]. About 100 ml of deionized water was added to the flask, swirled several times, and dissolved all of the salt before diluting to volume with deionized water. This is critical.

Sodium standard calibration solutions

Deionized water is used for the “blank”. A series of volume including 1.00, 2.00, 3.00, 4.00, and 5.00 mL of the standard 100-ppm sodium solution were taken into the first, second, third, fourth, and fifth 100-mL volumetric flasks, respectively and separately. Diluted carefully to the mark with deionized water and mixed thoroughly.

Unknown solution

The unknown was obtained from the instructor and carefully diluted to the 100-mL mark with deionized water and mixed thoroughly. Carefully followed the instructions provided for the use of the instrument and measured the emission intensity for the blank (deionized water), each standard, and the unknown(s).

When approaching to begin taking emission readings, utmost steps are followed to light the flame, stabilize the flame photometer, and for its proper and safe use. The instrument should have been turned on and the flame lit for 15 minutes [aspirating deionized water] to ensure stability. Thoroughly rinsed all the equipments used in this experiment, first with lots of distilled water, secondly with deionized water from a rinse bottle. Then filled the tall, 25 ml, capped polyethylene vials with the Blank (deionized water), the five standards (1, 2, 3, 4, and 5 ppm Na) and the unknown solution(s) – in that order – and placed in the plastic holder designed for them. Because water droplets cling to the vials, their insides will need to be pre-rinsed with small amounts of their solutions first. Then put a mL or two into a vial, capped it, shaken the contents into the sink.

Repeat at least 3 times for each vial. Aspirate deionized water until the meter reading stabilizes, this may take 30-90 sec. Then the blank knob was used to set the meter reading to 0.00. Then the highest standard (5 ppm) was aspirated until the meter reading has been stabilized. Then the fine sensitivity knob was used to set the meter reading to 5.00. The coarse sensitivity switch should be in the correct setting and not have to be switched.

Repeated the two-step calibration procedures with deionized water and the 5 ppm standard as many times as it takes to get them both stabilized at 0.00 and 5.00, respectively. The blank, the 5 standards, and the unknown(s) were aspirated in that order. Three replicate readings were done for each solution

once the meter reading has been stabilized. There may be some “bounce” (noise) in the readings, especially at the higher concentrations. For the second calibration run, place the unknown solution(s) between the two standards whose readings bracket that of unknown(s), so that the concentrations of the solutions aspirated now all increase monotonically.

Atomic emission instruments may work best when going from lower to higher concentrations.

The whole process of calibration repeated by taking triplicate readings as before at least 1 or 2 more times. The more data we have to review, the better the analysts will be able to detect and eliminate determinate error – inaccuracy in the final reported value.

CONCLUSION-

The calibration curve was done 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. If the plot appears to be reasonably linear, or at least that portion of it that includes your unknown, use the Excel LINEST function to do a linear-least-squares fit to the data, which will also be provided for some quality parameters for the fit. The “best estimate” was reported for the average concentration of sodium in ppm ($\mu\text{g/mL}$) and the associated standard deviation of the value.

REFERENCES-

1. Skoog DA, West DM, Holler FJ, Crouch SR. Analytical Chemistry: An Introduction, 7th ed., Chapter 23: 594-631.