

## CH 424 INSTRUMENTAL ANALYSIS LAB

### DETERMINATION OF ALKALI AND ALKALINE EARTH METALS BY FLAME EMISSION PHOTOMETRY

(Reproduced with modification from CH 424 Instrumental Analysis Course Manual, Spring 1996 written by Jim Peterson)

#### INTRODUCTION

Flame photometric methods are widely used for the determination of alkali metals and the alkaline earths in samples that are easily prepared as aqueous solutions. In principle, the intensity of emission is proportional to the concentration of analyte atoms present. However, a number of factors, such as flame temperature and various interference effects, can have a strong influence on the intensity observed.

For some samples, it is difficult to prepare standards which are sufficiently similar. This may occur when the samples contain high and variable concentrations of matrix materials, or when the samples contain solids whose effect on absorption is hard to duplicate. In such instances, the method of standard additions should be used.

In this experiment, the sodium concentration of an unknown is determined by flame emission photometry using both the direct intensity method and the method of standard additions to calibrate the instrument. The effects of ionization suppressants and releasing agents are also investigated.

#### REAGENTS

Prepare 500 ppm sodium stock solution by dissolving sodium chloride in 0.5% (w/w) hydrochloric acid. Dilute this stock solution in *ca.* 0.5% (w/w) hydrochloric acid to prepare working solutions: 5, 10, 20, and 40 ppm.

Obtain an aqueous solution of unknown sodium ion concentration from your instructor. Using a buret, dispense 25.0 mL of this unknown into each of four 100 mL volumetric flasks. Next pipet i) 1.00 mL, ii) 2.00 mL, iii) 4.00 mL and iv) 8.00 mL, respectively, of the stock *ca.* 500 ppm sodium solution into the four volumetric flasks. Finally, top the four flasks up to their marks with 0.5% hydrochloric acid.

#### PROCEDURE

Set the instrument monochromator to 589.0 nm, or turn the dial to the sodium position if a filter-based instrument is used. Light the flame and aspirate 0.5% hydrochloric acid (the blank) to check that the nebulizer is functioning correctly.

Determine a calibration curve for sodium ion using the direct-intensity method as follows. First setting the instrument gain to read 0 %T while aspirating 0.5% hydrochloric acid, set the instrument gain to read 0 %T, and to read 100% T (or 50 to 100 if only a sensitivity selection switch is available) while aspirating the 40 ppm sodium. Determine the %T for 5, 10, 20 ppm sodium solutions. Calculate the best straight line through these data by a least squares regression analysis.

Dilute 25 mL of the unknown solution into 100 mL in *ca.* 0.5% (w/w) hydrochloric acid, and measure the emission of this diluted sample at the same time that the direct-intensity calibration curve mentioned above at the same instrument conditions. Based on calibration curve determined above, estimate the concentration of your unknown as supplied to you in ppm and report the result in terms of 95% confidence limits.

Perform a standard addition method (see below, addendum) for the determination of sodium unknown as follows. Measure the emission of solutions i-iv and prepare a calibration curve, and calculate the best straight line through these data by a least squares regression analysis. Estimate the concentration of your unknown as supplied to you in ppm and report the result in terms of 95% confidence limits.

## QUESTIONS

- i) Lithium can serve as an ionization suppressant in flame photometry. What does this mean, how does it work and why would it be necessary?
- ii) Lanthanum can serve as a releasing agent in flame photometry. What does this mean, how does it work and why would it be necessary?
- iii) Why is flame emission photometry *particularly* useful for the determination of alkali and alkaline earth metals?

## ADDENDUM

It is also possible to analyze a sample by the method of standard additions and to calculate the results, rather than plotting an analytical curve. The equation to be used for the calculation is shown below. The following three criteria must be met in order for the calculation to be valid:

1. All solutions are made to the same final volume.
2. A plot of concentration vs. absorbance is linear over the entire concentration range covered.
3. All readings are either in absorbance or in some value directly proportional to absorbance.

$$C_s = \frac{C_a \cdot R_s \cdot V_a}{V_s(R_a - R_s)}$$

Where

$C_s$  = concentration of the element of interest in the original sample solution in  $\mu\text{g/ml}$ .

$C_a$  = concentration of the element of interest in the standard solution used to "spike" the diluted sample solution, in  $\mu\text{g/ml}$ .

$V_s$  = volume of original sample solution used in mL.

$V_a$  = volume of standard solution used in mL.

$R_s$  = reading obtained for diluted sample solution.

$R_a$  = reading obtained for "spiked" sample solution.

## REFERENCES

Perkin-Elmer Application Notes: "Analytical Methods for Atomic Absorption Spectrophotometry" (1973).

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Donald T. Sawyer, William R. Heineman, and Janice M. Beebe, "Chemistry Experiments for Instrumental Methods," John Wiley & Sons (1984) pp. 260- 264.