

**Reading:** Skoog, Holler and Crouch; Chapter 8 and Chapter 9, sections 9A-9D.

## A. INTRODUCTION

The phenomenon of atomic absorption (AA) was first observed in 1802 with the discovery of the Fraunhofer lines in the sun's spectrum. It was not until 1955 however that Walsh proposed that atomic absorption be used for quantitative chemical analysis [1]. Atomic absorption analysis involves measuring the absorption of light by vaporized ground state atoms and relating the absorption to concentration. The incident light beam is attenuated by atomic vapor absorption according to Beer's law.

To obtain the best results in AA, the instrumental and chemical parameters of the system must be geared toward the production of neutral ground state atoms of the element of interest. A common method is to introduce a liquid sample into a flame. Upon introduction, the sample solution is dispersed into a fine spray, the spray is then desolvated into salt particles in the flame and the particles are subsequently vaporized into neutral atoms, ionic species and molecular species. All of these conversion processes occur in geometrically definable regions in the flame. It is therefore important to set the instrument parameters such that the light from the source (typically a hollow-cathode lamp) is directed through the region of the flame that contains the maximum number of neutral atoms. The light produced by the hollow-cathode lamp is emitted from excited atoms of the same element which is to be determined. Therefore the radiant energy corresponds directly to the wavelength which is absorbable by the atomized sample. This method provides both sensitivity and selectivity since other elements in the sample will not generally absorb the chosen wavelength and thus, will not interfere with the measurement. To reduce background interference, the wavelength of interest is isolated by a monochromator placed between the sample and the detector.

## B. EQUIPMENT

The instrument used for this experiment is the PERKIN-ELMER AA MODEL 4000. A brief description of the operation the instrument is placed on the bench on the left hand side of the instrument.

To set the aspiration rate (uptake rate), proceed as follows (if it was not done by the instructor):

- a) Rotate the knurled of the nebulizer assembly counter-clockwise until bubbles develop (blow back) in the water.
- b) Gradually rotate the knurled clock wise until the bubbles just stop.
- c) Now rotate the knurled knob one-eighth of a turn
- d) Remove the aspirator tube from the deionized water (*excessive amounts of water sprayed into the burner head when the flame is out will cause the slots in the burner head to clog*).

## C. EXPERIMENT SUMMARY

In this experiment:

1. Sodium in bottle water will be determined using atomic absorption
2. the effect of potassium on sodium absorption will be studied
3. the interference of aluminum on the determination of magnesium will be studied and a method to minimize this interference will be demonstrated.

## D. EXPERIMENTAL PROCEDURES

Turn on the main power switch on the AA when you arrive at the lab. Prepare the solutions and instrument as instructed below. The instructor will brief you on the AA experiment.

### ***D.1 Determination of Na in bottled water using Acetylene-Air flame.***

1. Prepare a stock solution of  $1000 \text{ mg L}^{-1}$  Na.
2. Prepare 100 mL each of calibration standards containing 0, 25, 50, and 100  $\text{mg L}^{-1}$
3. Prepare 100 mL each of calibration standards containing 0, 25, 50, and 100  $\text{mg L}^{-1}$  containing each 350  $\text{mg L}^{-1}$  K
4. Prepare a 100-mL of a dilute sample (2x) of bottled water.
5. Prepare a 100-mL of a dilute sample (2x) of bottled water containing 350  $\text{mg L}^{-1}$  K.
6. Use the instruction manual to set the parameters for the determination of sodium using air-acetylene flame.
7. Use deionized water to zero the instrument.
8. Measure the absorbance of the standard solutions and of the dilute bottle water solutions.
9. If the absorbance of the dilute water solution falls outside the working range of your calibration curve, prepare a more dilute or concentrated sample as required.

### ***D.2 Matrix Effects: Air-C<sub>2</sub>H<sub>2</sub> Flame***

Aluminum and silicon are among the most serious interferences in the atomic absorption determination of magnesium. The effect of Al on the absorbance of Mg will be observed and the addition of an excess of Sr will be used to control this interference. All solutions needed here are provided.

1. Set the instrument zero reading with water.
2. Optimize the instrument with the 1-ppm Mg solution. If the signal level of the 1 ppm Mg solution is less than then 0.6, use the burner height control to get a signal level higher than 0.6.

3. When done, switch the nebulizing tube to water.
4. Reset the zero reading with the blank solution
5. Switch nebulizer to Mg standard solution 1 and read the absorbance
6. Determine the absorbance of the 12 solutions by repeating the 2 last steps.
7. Once you are finished all 12 solutions, switch the tube to water.

Concentration (ppm)					Concentration (ppm)			
Solution	Mg	Al	Sr		Solution	Mg	Al	Sr
A.	1	0	0		G.	1	0	1000
B.	1	10	0		H.	1	10	1000
C.	1	50	0		I.	1	50	1000
D.	1	100	0		J.	1	100	1000
E.	1	250	0		K.	1	250	1000
F.	1	500	0		L.	1	500	1000

## E. DATA PROCESSING AND QUESTIONS

1. Determine the concentration of Na in bottle water using the calibration curves obtained in absence and presence of K.
2. Why did you add 350 mg L<sup>-1</sup> K to your standard Na solutions?
3. Is the calibration curve for Na perfectly linear? If not explain why.
4. Plot the Mg absorbance as a function of the Al concentration for the data obtained with AND without Sr present in the Air-C<sub>2</sub>H<sub>2</sub> flame.
5. Write and discuss a chemical reaction that might occur in the flame to explain the effect of Al on the Mg absorbance in the Air-C<sub>2</sub>H<sub>2</sub> flame?
6. Using reactions, discuss how Sr affects the Mg-Al system? What can Sr be called in this experiment? (reference 9 page 221)

## F. REFERENCES

1. A. Walsh, Spectrochim. Acta., 7, 108, 1955.

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9. Skoog, Holler and Crouch, Chapter 9.