Chemistry 425 Dr. Petr Vanýsek, Instructor
Analytical Chemistry II

03

ATOMIC ABSORPTION FLAME PHOTOMETRY OF CADMIUM

Purpose:

To become familiar with the method of flame atomic absorption spectroscopy (FAAS). An instrument using laminar flow burner and a cadmium hollow cathode lamp will be used to construct calibration curve based on known concentrations of cadmium ion solutions. The calibration curve will be used to measure concentration of cadmium ions in issued unknown sample.

Materials:

Spectrophotometer model Perkin Elmer 3100 equipped with hollow cathode lamp, volumetric flasks, pipettes, stock calibration solution and unknown sample.

Procedure:

Start the cadmium hollow cathode lamp as per instructions and allow about 30 minutes for warm-up. Adjust the lamp current setting to 5 mA or the value given by the teaching assistant. Set the slit width to 0.7 nm.

Use the provided stock 10.0 ppm Cd^{+2} solution to prepare standard solutions of 2.00, 1.00, 0.500, 0.200 and 0.100 ppm Cd^{+2}. The standard solutions should be prepared by careful dilution of the stock solution using distilled water. Receive an unknown sample in 100-ml volumetric flask (solution containing $CdCl_2$ of undisclosed concentration) from the TA. Ignite the acetylene-air burner while aspirating deionized water. Adjust the hollow cathode lamp or burner position to obtain the maximum energy.

Wavelength studies:

The absorbance of the standard cadmium samples will be monitored at 228.8 nm. The slit width and the hollow cathode lamp position will be previously adjusted.

While aspirating the 0.500 ppm standard solution, set the wavelength dial to 228.8 nm and adjust for maximum absorbance. Aspirate each of your standards and obtain ten absorbance readings for each solution to prepare a calibration curve. Aspirate your unknown solution and obtain ten absorbance readings as well.

For the 228.8 nm wavelength plot the obtained data (signal vs. concentration, using computer software, *e.g.*, Origin or Excel) using the following fitting:

(1) Zero intercept fit – linear
(2) Zero intercept fit - nonlinear
(3) Calculated intercept (which may or may not be zero)

From each of the three calibration plots, determine the concentration of your unknown in units of ppm Cd^{+2}.

Dispose of any cadmium salts and their solutions properly in the inorganic waste container.

Appendix:

1. When a nebulizer is used, the solvent which is often used often is dilute nitric acid. Why would this be a good choice? (Hint - What are the relevant properties of nitrates?)

2. What was the physical width of the slit used in your particular setting?

3. Name and describe three types of chemical interference that generally occur during FAAS. Then pick one chemical interference and describe in detail how it can be minimized.