

Colorado State University
CHEM 431
Instrumental Analysis Laboratory

Notes for
Atomic Absorption (AA) Spectroscopy

The following is a set of short notes to outline the experiment in question and to provide helpful guidance to those executing the experiment.

- A. The central goal of this experiment is to convincingly demonstrate the operation and use of Atomic Absorption Spectrophotometry to determine the concentration of calcium in laboratory and "real world" samples.
- B. Prepare a calibration curve for calcium (using calcium nitrate) that demonstrates (a) the lower limit of detection, (b) the linear range of operation, and (c) the saturation region or upper limit of detection in a single figure that clearly displays all of these behaviors. Ensure that the linear portion of this calibration curve is **well populated with data points**. (Suggestion: rather than making up a substantial number of samples then measuring them all at once consider the superior method of making up one sample, measuring its absorbance, tabulating it in your notebook, plotting it on the graph paper in your notebook or electronic appliance and repeating until you have a well-represented dataset.)
- C. Determine the concentration of calcium in commercial milk and report it in comparison to the advertised concentration reported on the container. Report as molarity (reported and measured) and using the units on the container. Confer with teaching staff on the operation of the AA spectrophotometer.
- D. There are large number of chemical components in milk that will clog up the flow apparatus of the AA spectrophotometer. The milk must be "worked up" or digested prior to being analyzed. Study and understand the chemistry of the web site references for the method of high temperature oxidation using nitric acid and hydrogen peroxide relevant to the digestion process. Confer with teaching staff **prior** to performing this work.
- E. Phosphate ions are common in milk and interfere with the calcium measurement. There are two common ways to correct for this interference: (a) standard addition and (b) lanthanide competition. Perform both measurements.
- F. Report your calcium concentration measurements compared to the advertised (don't use silly terms like "true") value.
- G. Use the flow chart presented in the figure to efficiently perform all these measurements.

