Colorado State University

CHEM 431
Instrumental Analysis Laboratory

Notes for
Determination of Calcium by
Atomic Absorption 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 (AA) to determine the concentration of calcium in laboratory and "real world" samples.

B. Prepare a calibration curve for calcium (using calcium nitrate) that demonstrates the lower limit of detection, the linear range of operation and the saturation region or upper limit of detection. Prepare 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 data set.)

C. Obtain commercial milk samples and determine the concentration of calcium in them. Because of the large number of species present that will clog up the flow apparatus of the AA spectrophometer the milk must be "worked up" or digested prior to being analyzed. Study and understand the chemistry of the associated references for the method of high temperature oxidation using nitric acid and hydrogen peroxide, both concentrated, relevant to the digestion process. Learn how phosphate ions in the digested milk interfere with the measurement of calcium and how this problem is addressed. Discuss your method plan with teaching staff prior to executing it.

D. Validate your digestion method by performing a standard addition analysis on the milk samples using calcium nitrate. Report on the quality of your analysis methodology.