

CHEM 4114: Determination of Ca by Atomic Emission Spectroscopy (AES)

Objectives: To determine the concentration of Ca in a groundwater sample using atomic-emission spectroscopy (AES).

Reference: Principles of Instrumental Analysis, 5th Edition, Skoog, Holler, and Nieman, Chapter 8.

Instrumentation: Perkin Elmer Model 3110 flame atomic-absorption spectrometer (in Dav. 123).

Chemicals: 1000 ppm Ca solution, 10,000 ppm La solution, 0.1 M HCl, groundwater sample, instructor or student-supplied water sample from home.

WARNINGS: You will be working with a premixed air/acetylene flame. Light and extinguish the flame only with the TA present. Do not attempt to adjust the flame conditions. Failure to follow the correct procedures can result in a flashback - an explosion in the spray chamber. The instrument is designed to contain such an explosion, but conditions that can lead to flashback should be avoided.

When using the instrument be sure that the drain tube from the spray chamber leads into the waste container, and verify that the drain trap (Tygon tubing) is filled with water. Aspirate DI water into the instrument when no sample is being measured. When finished for the day, aspirate DI water for 5 min. Avoid looking directly into the flame, it is a source of UV radiation. You may safely view the flame through the darkened glass window.

Procedures:

Sample Preparation:

Use 100.0 mL volumetric flasks to prepare the following Ca standards: 100, 50, 25, 10, 5, and 1.0 ppm Ca. Each standard solution should contain 1000 ppm La. Obtain an unknown groundwater sample from the TA. Transfer 50.0 mL to a 100.0 mL volumetric flask, add 10.0 mL of La solution, and dilute to the mark with 0.1 M HCl. Prepare an untreated sample by the same dilution but without addition of the La solution. Prepare your home tapwater sample in the same way as the treated groundwater sample.

Instrument Operation:

Be sure that the following parameters are set (Press the Param Entry button to access the menus):

- INT. TIME (sec) : 0.3
- REPLICATES : 5
- READ DELAY (sec) : 5
- PRINT CALIB : N

Set the following:

- MODE : EM
- wavelength: 422.7 nm for Ca (refer to the Perkin Elmer methods manual for other elements)
- slit width: 0.2
- slit height: high

Start the instrument only with the TA present. Open the air and acetylene cylinder valves. Adjust regulator pressures to air = 50 psi, acetylene = 10-15psi. On the AA gas control panel, turn the switch to the "AIR" setting (you should hear a hissing noise), then adjust the flow rates to approximately: "FUEL"=2, and "OXIDANT" =4. Ensure that all safety conditions are met, then push the Ignite Flame button.

Aspirate 100 ppm Ca into the flame and manually scan the wavelength to verify that the monochromator is centered on the atomic emission line. Adjust the position of the burner to optimize the signal. Rescale the count rate with the Gain button as necessary. After you have optimized the instrument, have the TA check the signal level.

Aspirate DI water into the nebulizer for 2 minutes. Depress the CONT and A/Z buttons to autozero the instrument. To take a first reading depress the DATA, PRINT, and READ buttons. For subsequent readings make sure the DATA button is lit and press PRINT and READ.

Measurements:

(Record 3 sets of 5 replicates for each sample and A/Z after every 3 measurements.)

- Measure the signal of the blank.
- Measure the signal of the 1.0 - 100 ppm Ca standards (from lowest concentration to highest).
- Measure the signal of the treated and untreated groundwater unknowns and your home water sample.

Data Treatment:

1. Plot a working curve of emission signal versus Ca concentration.
2. Determine the Ca concentration in the treated and untreated unknown samples. Report the mean and 95-% confidence interval.
3. Determine the Ca concentration in your "home" sample. Report the mean and 95-% confidence interval.
4. Determine the Limit of Detection (LOD) and the Limit of Quantitation for the measurement.