

Analysis of Cu, Cd, and Cr in Plant Samples by Atomic Absorption Spectrometry

Instrumental Methods for Environmental Analysis—CHEM 463

Sample Digestion

1. Oven dry leaf samples from three plants for at least 24 hrs. in a 65 °C oven.
2. Weigh accurately a 2 g sample of the dried plant material to a small beaker, add 10 mL of trace metal grade concentrated nitric acid, and cover with a watch glass. Allow the mixture to cold soak for 30 min. Steadily raise the temperature to 120 °C and heat for 2 hrs. (This should all be done in the fume hood.) Remove from heat and allow to cool to room temperature.
3. Dilute the solution with 25 mL distilled water. Filter the solution using the apparatus provided by the instructor. Wash the beaker with another 25 mL aliquot of water and filter. Wash again with one more 25 mL aliquot of water and filter. Quantitatively transfer the solution to a 100 mL volumetric flask and dilute to the mark.

Standard Solution Preparation

4. Prepare a zero calibration solution by dissolving 8 mL concentrated nitric in distilled water to a total volume of 1.0 L. (Note: all standard solutions should be prepared using the zero calibration solution to minimize the possibility of contamination from the water supply.)
5. Using the stock 1000 ppm Cu, Cd, and Cr solutions, prepare 25 mL of standard solutions with the following concentrations: 0.0 ppm (blank), 0.5 ppm, 1.0 ppm, 1.5 ppm, and 2.0 ppm.
6. Prepare 50 mL of a 0.1 ppm standard solution—this will be used to determine the detection limits of the instrument for each element tested.

The standard and unknown solutions will be analyzed by atomic absorption spectrometry using the instrument mode that performs background correction with a D₂ lamp. Set up the instrument to perform a set of measurements using the appropriate hollow cathode lamp with deuterium lamp background correction. (The instructor will assist you in making the proper adjustments.)

Determination of unknown concentration

7. Aspirate each of your standard solutions in order to construct a calibration curve. Include as a standard the zero blank solution to establish the zero signal value.
8. Aspirate the unknowns and determine the concentrations from the calibration curve.

Determination of detection limits

9. Using your 0.1 ppm solution, measure the signal value ten times in succession. From these ten measurements, determine the mean, standard deviation, and detection limits for this instrumental mode.
10. Repeat these measurements for chromium and cadmium.