

Experiment: Determination of Copper in an Aqueous Sample by Atomic Absorption

Upon atomization in a flame, metals absorb at well-defined wavelengths. Atomic absorption flame spectrophotometry (AAS) is a convenient and sensitive method for the determination of dissolved metals such as copper. The presence of other elements in an aqueous sample is not expected to interfere with the signal from copper because absorption peaks are discrete and non-overlapping.

Procedure:

Prepare standards:

Weigh out accurately 0.20 g of copper(II)sulfate pentahydrate ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$) and dissolve it by adding to it about 100 mL of distilled water in a 250 mL beaker. Transfer the solution quantitatively to a 500 mL volumetric flask and make it up to volume and mix well. Calculate its concentration in terms of ppm Cu (show that the final concentration is about 100 ppm Cu. The atomic absorption spectrometer approaches linear sensitivity to Cu somewhere in the range of 7 ppm. Prepare standard solutions with the following concentrations: 6 ppm, 4 ppm, 3 ppm, 2 ppm, and 1 ppm. For example, to prepare the 6 ppm standard, take 6 mL of the stock (i.e. 100 ppm) solution and dilute to 100 mL with distilled water in a 100 mL volumetric flask. Note that in general:

$$(\text{final concentration}) = (\text{initial concentration})(\text{initial volume}/\text{final volume})$$

And so, in this example, # ppm = $(100 \text{ ppm})(6 \text{ mL}/100 \text{ mL}) = 6 \text{ ppm}$.

Note: You do not need many 100 mL volumetric flasks. Just reuse the volumetric flask and don't use it as your storage container.

Prepare the unknown:

Weigh out accurately two portions (about 0.1g each) of the dried unknown ore into two 250 mL beakers (drying time is 90-120 minutes using the convection oven). In the fume hood, to each of the beakers add about 2 mL concentrated nitric acid, cover with a watch glass and heat in the fumehood until the sample dissolves. Cool and then add about 2 mL 1:1 sulfuric acid to each beaker (prepared by adding 1 mL concentrated sulfuric acid to 1 mL of distilled water). Heat until dense white fumes of sulfur trioxide evolve. The solution may bump at this point and so use a stirring rod and watch carefully at this point. Sulfuric acid displaces nitric acid which may interfere later in the filtration process. Cool and dilute to about 50 mL with hot distilled water. Since you are adding water to acid, be careful and pour the water down the side of the beaker with vigorous stirring.

Filter both solutions through fine filter paper. Collect the filtrates in two separate 100 mL beakers, if available. For each unknown, wash the residue with three 10 mL aliquots of hot distilled water. Transfer the filtrate quantitatively to a 250 mL volumetric flask and make up to volume with distilled water. Dilute the each solution by a dilution factor of 25. For example, take 10.00 mL of the solution and make up to volume in a 250 mL volumetric flask with distilled water.

Submit your standards and unknown solutions CLEARLY MARKED as to locker # and identity of solutions for analysis by AAS. Carry out the atomic absorption spectroscopic measurements on the diluted standards and the two diluted unknowns.

Analysis of your results:

(1) Draw a graph of signal vs ppm Cu standard. This is your standard curve. Using this curve, determine what value of ppm corresponds to the signal for your unknown sample.

(2) Determine the %Cu in your original powder sample:

Example: suppose your results showed that your unknown solution (e.g. total vol. = 250 mL, dilution = 25) contains 2.36 ppm Cu, using a 0.2130 g solid sample:

$$\%Cu = (.00236g\ Cu/L)(0.250L)(25)(100\%)/(0.2130g) = 6.92\%$$

Informal report:

Experiment: Determination of Copper in ore:

Name: Values obtained: (1) _____ %

Locker # (2) _____ %

Average value = _____ %

(if applicable) Standard deviation = _____ %

g dried $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ used for stock solution = _____ gppm Cu^{2+} in stock solution = $(\text{CuSO}_4 \cdot 5\text{H}_2\text{O}) (\text{AW Cu}/\text{FW CuSO}_4 \cdot 5\text{H}_2\text{O})(1000 \text{ mg/g})/(0.500 \text{ L})$

= _____ ppm stock solution

% Cu unknown (show calculation) =

Calibration Data: (**attach a graph of signal(I) vs ppm Cu^{2+})

	ppm Cu	Signal
	(0.75)	_____
	(1.50)	_____
	(3.00)	_____
	(6.00)	_____
	(12.0)	_____
unk#1	_____	_____
unk#2	_____	_____

FW (Cu) = 63.546 g/mol

FW ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$) = 249.68 g/mol