Chemistry 201 laboratory   Summer, 2006

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 the <u>standards</u> as follows:

Weigh out accurately 0.2 g of copper(II)sulfate pentahydrate 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 spectroscope approaches linear sensitivity to Cu somewhere in the range of 1 to 20 ppm. Therefore, dilute the stock standard solution to obtain 20 ppm. (This can be obtained by adding an accurately measured 50 mL portion of the stock solution to a 250 mL volumetric flask and diluting to volume with distilled water.)

Additional standards can be obtained from this diluted by serial dilution (each time by a factor of 2) to obtain 4 more standards: 10.0, 5.0, 2.5, and 1.25 ppm Cu. (This is the recommended way of preparing your standards.)

As an alternative method for preparing standards, you may prepare 20, 10, 5, 2.5, 1.25 ppm directly from the stock standard solution. For example, to prepare the 20 ppm standard, take 20 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: (final concentration) =(initial concentration)(initial volume/final volume). And so, in this example, # ppm= (100 ppm)(20 mL/100 mL) = 20 ppm.

Prepare the <u>unknown</u> as follows:

Weigh out accurately two portions (about 0.2 g each) of the <u>dried</u> 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 4 mL concentrated nitric acid, cover with a watch glass with glass hooks and heat in the fumehood until the sample dissolves. Cool, add about 4 mL 1:1 sulfuric acid to each beaker (prepared by adding 5 mL concentrated sulfuric acid to 5 mL of distilled water). Heat until dense cloudy 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 mLs 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 (#42 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 this solution by a <u>dilution factor of 10</u>. (For example, take 25.0 mL of the solution and dilute that to 250 mL volumetric flask and making it up to volume 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. (Note that the line may curve at higher ppm values and as such, it may not always be appropriate to draw a straight line all the way.)

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

For example: suppose your graph results show that your unknown solution (e.g. total vol. = 250 mL, dilution = 10) contains 8.36 ppm Cu, using a 0.2130 g unknown solid sample:

%Cu =(8.36mg Cu/L)(1g/1000mg)(0.250L)(10)(100%)/(0.2130g)= 9.81%

Informal report:
Experiment: Determination of Copper in ore:

Name: Values obtained: (1) _____ %

Locker #   (2) _____ %

Average value = _____ %

(if applicable) Standard deviation= _____ %

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grams $CuSO_4·5H_2O$ used for stock solution =_____ g

ppm Cu^{2+} in **stock solution** =
(g $CuSO_4·5H_2O$) (AW Cu/FW $CuSO_4·5H_2O$)(1000 mg/g)/(0.500 L)

= _____ ppm stock solution

% Cu unknown (show calculation here) =

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

	ppm Cu	Signal
	(1.25)	_____
	(2.5)	_____
	(5.0)	_____
	(10.0)	_____
	(20.0)	_____
unk#1 _____		_____
unk#2 _____		_____

FW (Cu) = 63.546 g/mol   FW ($CuSO_4·5H_2O$) = 249.68 g/mol