Brooklyn College
Department of Chemistry

Instrumental Analysis (Chem 42/790) ***Atomic Absorption Spectroscopy***

An atomic absorption spectrometer is used in this experiment to analyze a copper-base alloy for copper, lead, and zinc.

In the first part of the experiment, the alloy is dissolved and the sample solution is diluted to the proper concentration range for each element. Suitable standard solutions for each element are prepared using 1000 ppm stock solutions. The Perkin Elmer AAnalyst 100 atomic absorption spectrometer is then used to prepare calibration curves and analyze for each element.

PREPARATION OF SOLUTIONS

Reagents.

Hydrochloric acid, HCl, (1 : 1) with deionized water.

Hydrochloric acid, HCl, 5% (v/v) in deionized water.

Nitric acid, HNO_3, (1 : 1) with deionized water.

Sample preparation.

Work under the hood. Dissolve an accurately weighed 0.5 g sample of the alloy in 10 mL of (1 : 1) HCl by using a minimum volume of (1 : 1) HNO_3. Use a 50 mL beaker covered with a watch glass to avoid loss of sample. Warm the sample using a hotplate to aid the dissolution process. When the sample is dissolved (there may be some undissolved white salts remaining), boil to remove oxides of nitrogen. Cool. Add an additional 10 mL of (1 : 1) HCl, transfer to a 100 mL volumetric flask and dilute to mark with deionized water.

Sample solutions.

Prepare solutions for AA analysis. Use your sample solution and this Table as a guide. Use 5% HCl for all dilutions.

Element	Approx. Concentration in your Undiluted Sample (%)	Approx. Concentration in your Undiluted Sample (mg/L)	Dilution	Approx. Conc. in Diluted Sample Solution (mg/L)
Cu	70	3500	1/500	7
Pb	12	600	5/250	12
Zn	12	600	1/250	2.4

Standard Solutions.

Prepare suitable standard solutions for each element by dilution of the 1000 ppm stock solutions. Use 5% HCl for all dilutions. Use this Table as a guide:

Element	Wavelength (nm)	Characteristic Concentration Check (mg/L for ≈ 0.2 a.u.)	Suggested concentrations (**mg/L**) and dilutions for three standards (mL sample/ mL total)
Cu	324.8	4.0	**4** (0.1/25), **12** (0.3/25), **30** (3/100)
Pb	283.3	20.0	**20** (2/100), **50** (5/100), **100** (5/50)
Zn	213.9	1.0	**1** (0.5/500), **3** (0.15/50), **6** (0.3/50)

ANALYSIS FOR COPPER, LEAD AND ZINC USING THE AA SPECTROMETER

Turn on the spectrometer (switch is located on the right side of the spectrometer)**.**

Self test mode will be run.

Recall mode (Y/N) - select NO.

How to set up parameters.

To specify individual parameters enter the Parameter Entry Mode by pressing [Param Entry]. The AAnalyst 100 then guides you through the entry of the required information using the display.

To enter a parameter, type in the value using the numeric keypad, then press [Enter].

To accept a default value, press [Enter].

To choose an option, type in the option number, then press [Enter].

In order to quickly pass through the parameter entry screens while in the Param Entry mode, press [Param Entry] until you reach desired screen.

To move to the previous field press [Option], [←], and to move to the next field press [Option], [→] or [Enter].

<u>ANALYSIS FOR COPPER</u>

Parameters for Cu analysis.

Select the lamp - 1.

Since this is non-coded lamp you can not use default parameters (available only for coded lamps, e.g., Pb or Zn lamps), and you have to define several parameters (Lamp current, Slit, Height, Wave length) for this lamp.

Lamp current - 15 mA.

Slit - 0.7.

Full height - Yes.

Wave length - 324.8 nm.

The spectrometer will adjust the lamp and check all lamp parameters you have defined.

Int. *(Integration)* Time - 1 (sec).

Replicates - 5.

Cal. *(Calibration)* - (1) Nonlin. (nonlinear).

Measurement Mode - (1) Hold.

You are not going to use calibration mode for your experiment. Therefore, skip Standard parameters entry.

To skip Standard - push Enter.

Read delay - 0.

Print Calib. (Y/N) - No.

Graph Size - push Enter.

After defining all parameters you should go back to the list of lamps.

Flame ignition.

Be sure that there is water in the 200 mL beaker.

ASK YOUR INSTRUCTOR FOR ASSISTANCE.

Turn on the vent.

Turn on the air compressor.

Open the tank with acetylene.

Turn on the gases (spectrometer).

Gases will be turn off automatically after 3 sec.

The acetylene flowmeter should show 2.5, the air flowmeter should show 4.

Ignite the flame.

Correction for a blank solution.

Aspirate a blank solution (usually deionized water).

Push Data, then Read.

You will see ABS data for your blank solution.

Zero the display by pushing the A/Z key.

Right now ABS reading should be 0.

Remember to repeat this procedure for each element.

Calibration Curve and Unknown Analysis for Copper.

Aspirate each of your 3 Cu standard solutions and your Cu unknown solution, and record their absorbance values.

To record data push Data, then Read.

After several sec. you will see your ABS value. This is the average value from 5 replicates. Write down this result.

Push Read to start next reading. Repeat readings 5 times for each sample of Cu, and use the average value of ABS for all calculations.

Repeat this procedure for each sample of Cu (three standards and one sample of unknown).

Aspirate deionized water for 1 minute between samples.

Remember to do it for 2-3 minutes after each experiment.

Push Param. Entry *(you should see the lamps list on the screen).*

Turn off the flame.

ANALYSIS FOR LEAD

Parameters for Pb analysis.

Select the lamp - 2: Pb.

This is a coded lamp which means that such parameters as lamp current, slit, height and wave length are already defined in the spectrometer memory. Therefore, you can use default parameters for Pb lamp.

Use default conditions (Y/N) - Yes.

Int. *(Integration)* Time - 1 (sec.).

Replicates - 5.

Cal. (Calibration) - nonlinear (NONLIN).

Measurement Mode - (1) Hold.

Again, skip Standard parameters entry.

Read delay - select 0.

You should go back to the list of lamps.

Ignite the flame.

Correction for a blank solution.

Follow the procedure described for Cu analysis.

Calibration Curve and Unknown Analysis for Lead.

Aspirate each of your 3 Pb standards and your Pb unknown, and record their absorbance values.

To record data push Data, then Read.

After several sec. you will see your ABS value. This is the average value from 5 replicates. Write down this result.

Push Read to start next reading. Repeat readings 5 times for each sample of Pb, and use the average value of ABS for all calculations.

Repeat this procedure for each sample of Pb (three standards and one sample of unknown).

Aspirate deionized water for 1 minute between samples.

Remember to do it for 2-3 minutes after each experiment.

Push Param. Entry *(you should see the lamps list on the screen).*

Turn off the flame.

ANALYSIS FOR ZINC

Parameters for Zn analysis.

Select the lamp - 3: Zn.

This is a coded lamp, therefore, you can use default parameters.

Follow the procedure for Pb.

Aspirate deionized water for 2-3 minutes.

Turn off the flame.

Turning off the spectrometer.

ASK YOUR INSTRUCTOR FOR ASSISTANCE.

From the Parameters Entry menu select lamp number 5 (there is no lamp in this position).

Turn of the spectrometer.

Close the tank with acetylene (use only the main valve).

Turn off the air compressor.

Turn of the vent.

REPORT

Prepare Beer's Law plots for copper, lead and zinc. Determine the concentration of each element in your unknown from the graph.

Calculate the concentration of each element in your unknown solution and its wt% in the original unknown alloy sample. Also, determine the sensitivity for copper, lead and zinc.