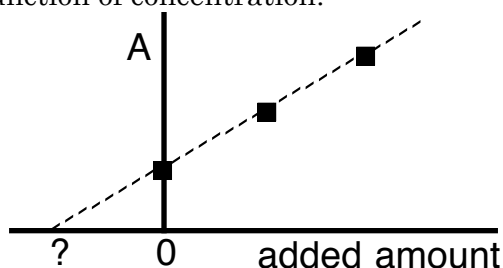

Varian AA240FS Atomic Absorption Spectroscopy

Put the sample into a solution that can be easily aspirated through the nebulizer. Everything in the solution must be dissolved; filter the sample solution if necessary. The concentration of the metal to be determined should be within *5-25 microgram per milliliter*. About 10 mL of solution should be available for each measurement (i.e., even including extra, you don't need to mix up liters).

Prepare standard solutions for a *calibration curve* using known concentrations of the metal to be determined. Use the same matrix as that of the sample, including the same amounts of any additional material that is present in the sample. For example, physical requirements mean the sample and standards must have the same solvent and the same acid concentration. Standard concentrations should bracket the expected concentration of the sample. Very dilute standards should be freshly prepared from a more concentrated stock solution.

If there are possible unknown interferences, *standard addition* should be used instead of a calibration curve. Known amounts of standard are added to solutions of the unknown and the concentration of unknown determined by extrapolation of the plot of absorbance as a function of concentration.



In standard addition the negative *x*-intercept indicates the amount present in the sample. For best results, each additional increment should be approximately the same as that anticipated for the diluted sample.

Take your sample and standard solutions together to the atomic absorption spectrometer. They must all be run at the same time. Have instructor install and align the lamp, adjust instrument to 248.3 nm for determination of iron, and light flame (next page).

Select File Load, then New from... "iron." You must pick a file name that has not already been used or OK will be dimmed. It is easiest to just click on the most recent file which then increments the file name. Under Develop/Edit Methods/Standards enter the concentrations of the standards (not including zero) so you can check the graph as you record data. Click on the Analysis tab.

Start. Click on the traffic light. The computer will request presentation of the solutions.

For CAL ZERO, use the zero concentration standard.

Supply standards, then samples as requested.

Record all absorbance values (top of screen) in your notebook when the next dialog box appears.

Type values in the spreadsheet at

<http://chemistry.beloit.edu/classes/excel/calibration.xlsm>

http://chemistry.beloit.edu/classes/excel/std_addition.xlsm

to plot the absorbance of your standards as a function of their known concentration. The least squares best fit equation for the standards will be calculated and used to calculate the concentration of the unknown. Be sure to include the graph and table in your report.

Directions for Varian AA240FS

Turn on hood

Open main valves for air and acetylene (in use air should be 50 psi, acetylene 10 psi; when the flame is not lit the values may be higher)

Turn on instrument power (front left)

Computer password BCAA240FS (all caps)

Software is called SpectrAA

Click on Worksheet

Select New from "lead in soil" (My Computer/C:/Varian/sp100/DATA)

Develop

 Edit Methods

 Type/Mode

 Element Pb

 Absorbance

 Use SIPS (off)

 Optical

 Lamp position (Pb currently in 2)

 Wavelength 283.3 nm

 Slit width 0.5

 Background Correction ON

 SIPS

 Standards

 Enter concentrations of all standards to be used (not including zero)

 Labels

 Enter sample names (cannot be changed when recording)

Hold in (several seconds) the ignite (flame) button on instrument front left

Analysis

 Optimize

 Adjust lamp position (2 knobs) to maximize left green bar

 Cancel window when done

 Start

 The computer will ask for solutions in the order identified above.

 For CAL ZERO, use the zero concentration standard.

 Supply standards, then samples as requested.

For each group

File Load. New from... "lead in soil"

You must pick a file name that has not already been used or OK will be dimmed.

 Start

 The computer will request presentation of the solutions.

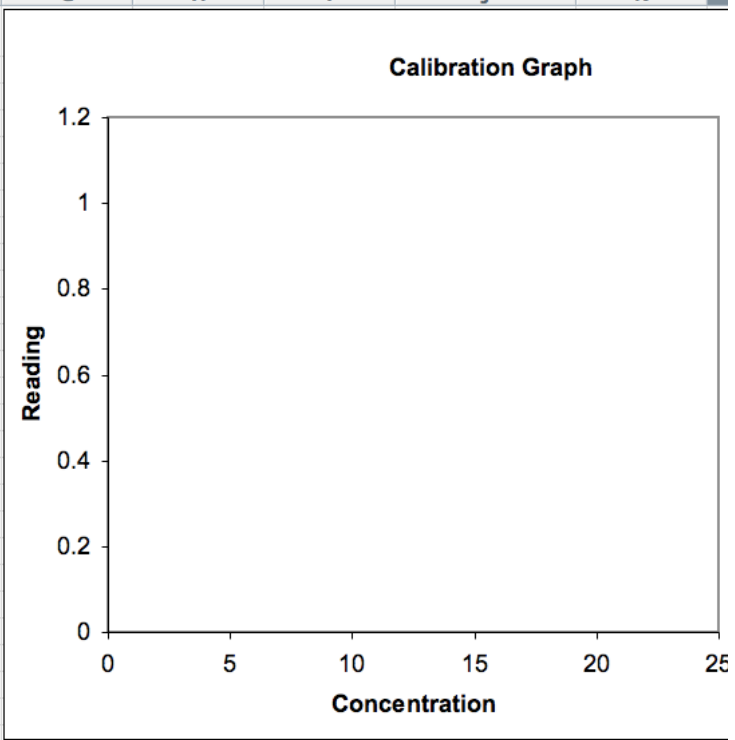
 For CAL ZERO, use the zero concentration standard.

 Supply standards, then samples as requested.

 Record all absorbance values in your notebook.

Type values in spreadsheet at <http://chemistry.beloit.edu/classes/excel/aa.xlsm> to do the calculations.

	A	B	C	D	E	F
1	Standard Concentration	Standard Reading	Sample Name	Sample Reading	Sample Concentration	Standard Deviation
2		0			0.00	0.00
3		3			0.00	0.00
4		6			0.00	0.00
5		12			0.00	0.00
6		20			0.00	0.00
7					0.00	0.00
8					0.00	0.00
9					0.00	0.00
10					0.00	0.00
11					0.00	0.00
12					0.00	0.00
13					0.00	0.00
14					0.00	0.00
15					0.00	0.00
16					0.00	0.00
17					0.00	0.00
18					0.00	0.00
19					0.00	0.00
20					0.00	0.00
21					0.00	0.00
22					0.00	0.00
23					0.00	0.00
24					0.00	0.00
25					0.00	0.00
26					0.00	0.00
27					0.00	0.00
28					0.00	0.00
29					0.00	0.00
30					0.00	0.00
31					0.00	0.00
32					0.00	0.00
33					0.00	0.00



Clear

Remove Line

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