Examination of heavy metal pollution in water around Falu copper mine
(Commenius project)

In this practical you will investigate watersamples from the area around Falu copper mine. Copper has been mined in Falun for thousand years and waste products from the mining activities can be found in many places in the area surrounding Falun, especially where there is water. Lakes were used to transport the ore, and whereever there was a small river or creek, the energy was used run smelters where the ore was converted to copper metal. From these waste products, metals are slowly beeing released into the environment causing problems for the environment.

In order to investigate the metal levels in different waterbodies around Falun we will sample water from lakes and rivers both upstream and downstream the mine. Samples will be analysed using an Atomic Absorption Spectrophotometer.

Atomic absorption spectrophotometry analyzes the concentration of elements in a liquid sample based on energy absorbed from certain wavelengths of light (usually 190 to 900 nm). Atomic absorption spectrophotometers typically include a flame burner to atomize the sample (most commonly a hollow cathode lamp), a monochromator, and a photon detector. Depending on the model, some atomic absorption spectrometers are equipped with a turret or fixed lamp socket that can hold multiple lamps (up to eight) to reduce downtime between samples or allow for sequential analysis.

Typical sensitivity for an atomic absorption spectrometer using a flame burner is in the parts per million range. For trace analysis, a graphite furnace can be used in place of a flame burner to increase the sensitivity by several orders of magnitude (in the parts per billion range). Atomic absorption spectrophotometers are used in many industries including environmental testing, metal analysis, semiconductor manufacturing, petroleum and chemical production, and in pharmaceuticals, for example.

Principle of Atomic Absorption /Emission Spectroscopy

ATOMIC EMISSION-THE FLAME TEST

When a small amount of a solution of a metal ion is placed in the flame of a Bunsen burner, the flame turns a color that is characteristic of the metal ion. A sodium solution gives a yellow color, a potassium solution results in a violet color, a copper solution gives a green color, etc. Such an experiment, called the flame test, has been used in conjunction with other tests in many qualitative analysis schemes for metal ions. Whatever color our eye perceives indicates what metal ion is present. When more than one metal ion is present, viewing the flame through a colored glass filter can help mask any interference. Figure 1 shows this experiment.

The phenomenon just described is an "atomic emission" phenomenon. This statement may seem inappropriate, since it is a solution of metal ions (and not atoms) that is tested. The reason for calling it atomic emission lies in the process occurring in the flame. One of the steps of the process is an atomization step. That is, the flame converts the metal ions into atoms. When a solution of sodium chloride is placed in a flame, for example, the solvent evaporates, leaving behind solid crystalline sodium chloride. This evaporation is then followed by the dissociation of the sodium chloride crystals into individual ground state atoms -a process that is termed atomization. Thus sodium atoms are actually present in the flame at this point rather than sodium ions, and the process of light emission actually involves these atoms rather than the ions .
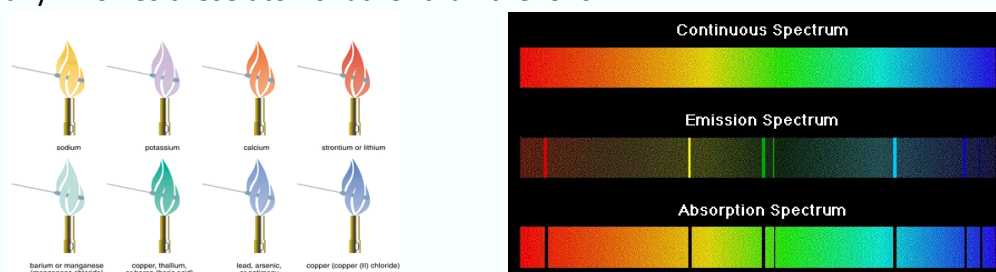

Figure 1. The flame test: observation of the flame color (a) without colored glass and (b) through prism
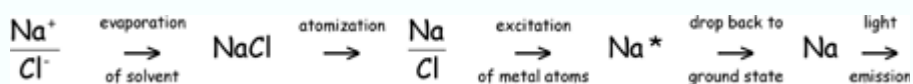

Figure 2. The sequence of events occurring in the flame test.

As with fluorescence, the atomic emission is a result of electrons dropping from an excited state to lower states. The difference is that (1) atoms are involved here, rather than molecules, and (2) light is not absorbed prior to this atomic emission. Following atomization, a small percentage of the atoms absorb sufficient energy from the flame (as opposed to a light beam) so as to be promoted to an excited state. As with molecules in fluorescence, these atoms quickly return to a lower state, and light corresponding to the energy that is lost in the process is generated. It is this light that our eye perceives. The complete sequence of events is depicted in Figures 2 and 3.

Since there are no vibrational levels in atoms, the energy of emission is a discrete amount of energy corresponding to the difference between two electronic levels. Also, since there are usually a number of electronic levels to which an electron in an atom can be promoted, there are a number of possible discrete energy jumps back to the lower energy states. These represent a number of distinct wavelengths of light to be emitted. What is actually emitted by the atoms in a flame is then a line emission spectrum as indicated in Figure 4. Figure 5 depicts an explanation of the atomic emission phenomenon. When atoms fall back to lower energy states following the absorption of energy from a flame, a line spectrum is emitted which our eye perceives as a particular color of light. Each kind of atom is different in terms of the separation between energy levels and the line emission spectra are therefore different. Because of this, different elements are found to emit the different.
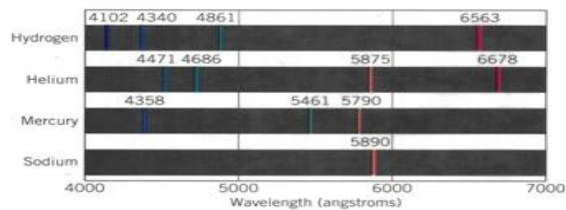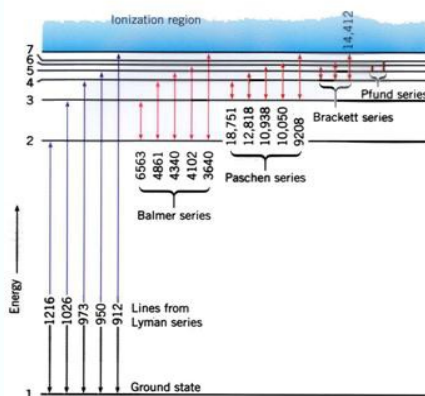

Figure 4. The atomic emission phenomenon: (a) Allowed energy jumps back to lower levels. (b)
A line spectrum representing the wavelengths of the emitted light corresponds to the jumps in (a).

FLAME PHOTOMETRY

The transition from the simple flame test to an instrumental technique is easily described. In the instrumental technique of flame photometry (FP), a monochromator replaces the colored glass filter, and a photocell detector/readout replaces our eye. Also, the burner design is more sophisticated in that the sample is continuously fed into the flame by aspiration. A schematic of this design is shown in Figure 6.
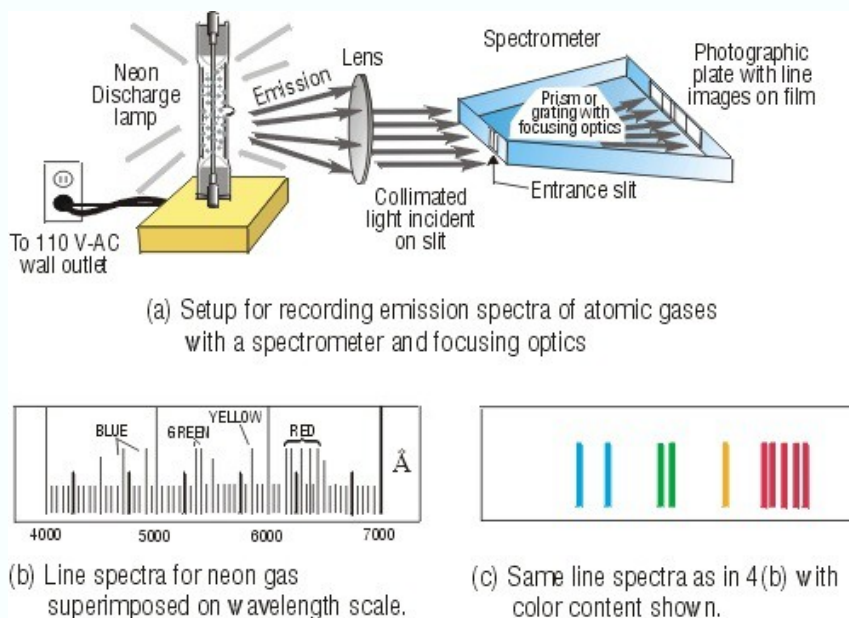

Figure 6. A schematic diagram of a flame photometer.

Since each element emits its own characteristic line spectrum, qualitative analysis can be performed here by observing what wavelengths are emitted and comparing these with various standards. However, since the detector is capable of measuring light intensity, quantitative analysis, as well as qualitative analysis, is possible. The intensity of the emitted light increases with concentration, and the relationship is usually linear:

$$I = K c$$

Thus, unknown concentrations can be determined by comparison with one or a series of standards in the same manner as described for the molecular techniques in UV-Vis Absorption spectroscopy.
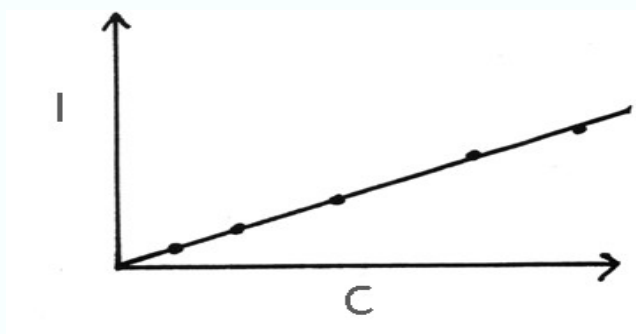

Figure 7
A plot of intensity (I) vs concentration (C)
representing quantitative analysis.

In short, flame photometry (FP) is an atomic technique which measures the wavelength and intensity of light emitted by atoms in a flame resulting from the drop from the excited state (formed due to absorption of energy from the flame) to lower states. No light source is required since the energy imparted to the atoms comes from the flame. Thus, FP is different from atomic absorption spectroscopy (AA), which is described in the next section.

ATOMIC ABSORPTION SPECTROPHOTOMETRY

As implied previously, only a very small number of the atoms in the flame are actually present in an excited state at any given instant. Thus there is a large percentage of atoms that are in the ground state and available to be excited by some other means, such as a beam of light from a light source. AA takes advantage of this fact and uses a light beam to excite these ground state atoms in the flame. Thus AA is very much like molecular absorption spectrophotometry in that light absorption (by these ground state atoms) is measured and related to concentration. The major differences lie in instrument design, especially with respect to the light source, the "sample container," and the placement of the monochromator. The basic AA instrument is shown in Figure 8. Below is a brief discussion of the essentials of each.
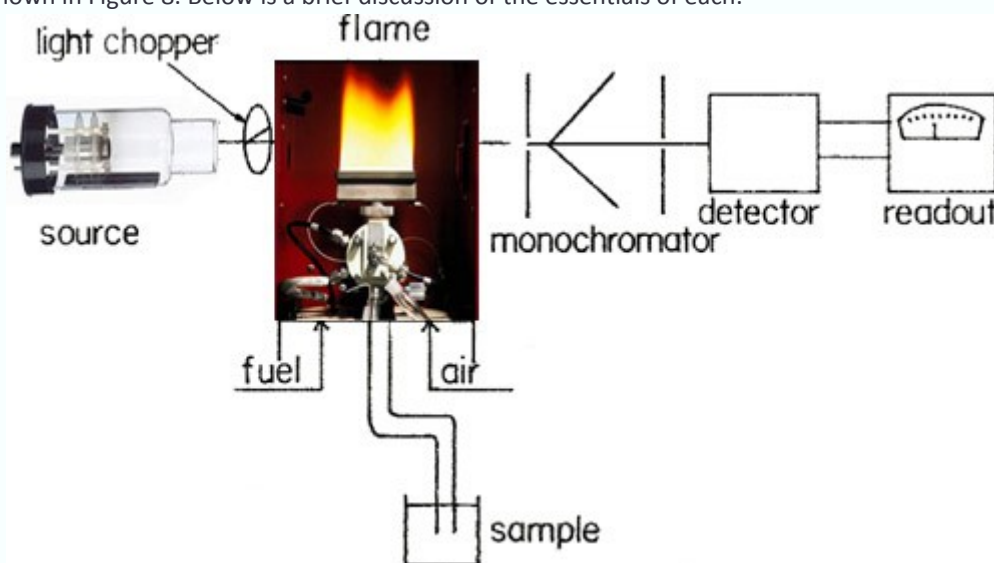
The light source, called a hollow cathode tube, is a lamp that emits exactly the wavelength required for the analysis (without the use of a monochromator). The light is directed at the flame containing the sample, which is aspirated by the same method as in FP. The flame is typically wide (4-6 inches), giving a reasonably long pathlength for detecting small concentrations of atoms in the flame. The light beam then enters the monochromator, which is tuned to a wavelength that is absorbed by the sample. The detector measures the light intensity, which after adjusting for the blank, is output to the readout, much like in a single beam molecular instrument. Also as with the molecular case, the absorption behavior follows Beer's Law and concentrations of unknowns are determined in the same way. All atomic species have an absorptivity, a, and the width of the flame is the pathlength, b. Thus, absorbances (A) of standards and samples are measured and concentrations determined as with previously presented procedures, with the use of Beer's Law ($A = a b c$).

beam
split

beams
joined
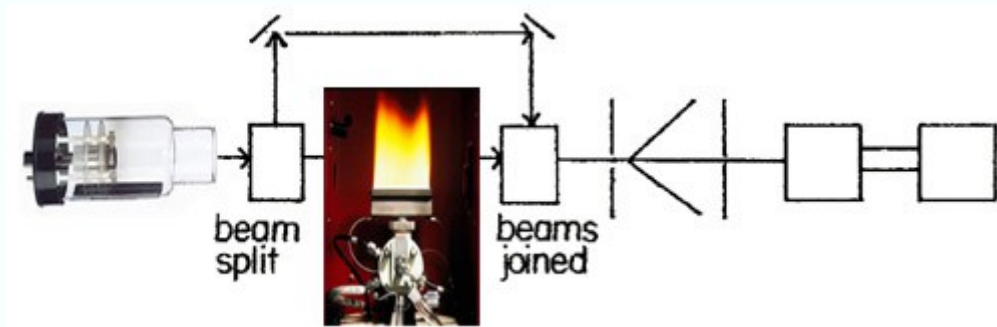
A question arises here as to how the detector can measure only the intensity of light that does not get absorbed (which is what a detector must do in any absorption experiment) and not measure the light emitted by the same atoms in the flame, since both sources of light are present and are of the same wavelength. The answer has to do with the light chopper in Figure 8. The light is "chopped" with a rotating half-mirror so that the detector sees alternating light intensities. At one moment, only the light emitted by the flame is read since the light from the source is cut off, while at the next moment, the light from both the flame emission and the transmission of the source's light is measured since the source's light is allowed to pass. The electronics of the detector is such that the emission signal is subtracted from the total signal and this difference then, which is T, is what is measured. Absorbance, A, however, is usually what is displayed on the readout.

Table 1. Comparison of **AA and FP**

|	AA	FP
Process Measured	absorption (light absorbed by unexcited atoms in flames)	emission (light emitted by excited atoms in a :flame)
Use of Flame	atomization	atomization and excitation
Instrumentation	light source	no light source (independent of flame)
Beer's Law	applicable	not applicable ($I=kc$)
Data Obtained	A vs. c	I vs. c

Double beam instruments are also in use in AA. In this case, however, the second beam does not pass through a second sample container (it's difficult to obtain two closely matched flames). The second beam simply bypasses the flame and is relayed to the detector directly. See Figure 9. This design eliminates variations due to fluctuations in source intensity (the major objective), but does not eliminate effects due to the flame (cuvette) or other components in the sample (blank components). These must still be adjusted for by reading the blank at a separate time.

INSTRUMENT COMPONENTS

Hollow Cathode Lamp

As indicated in the previous section, the light source in the AA instrument is called a hollow cathode lamp. As stated before, the light from this lamp is exactly the light required for the analysis, even though no monochromator is used. The reason for this is that atoms of the metal to be tested are present within the lamp, and when the lamp is on, these atoms are supplied with energy, which causes them to elevate to the excited states. Upon returning to the ground state, exactly the same wavelengths that are useful in the analysis are emitted, since it is the analyzed metal with exactly the same energy levels that undergoes excitation. Figure 10 is an illustration of this point. The hollow cathode lamp therefore must contain the element being determined. A typical atomic absorption laboratory has a number of different lamps in stock which can be interchanged in the instrument, depending on what metal is being determined. Some lamps are "multielement," which means that several different specified kinds of atoms are present in the lamp and are excited when the lamp is on. The light emitted by such a lamp consists of the line spectra of all the kinds of atoms present. No interference will usually occur as long as the sufficiently intense line for a given metal can be found which can be cleanly separated from all other lines with the monochromator.
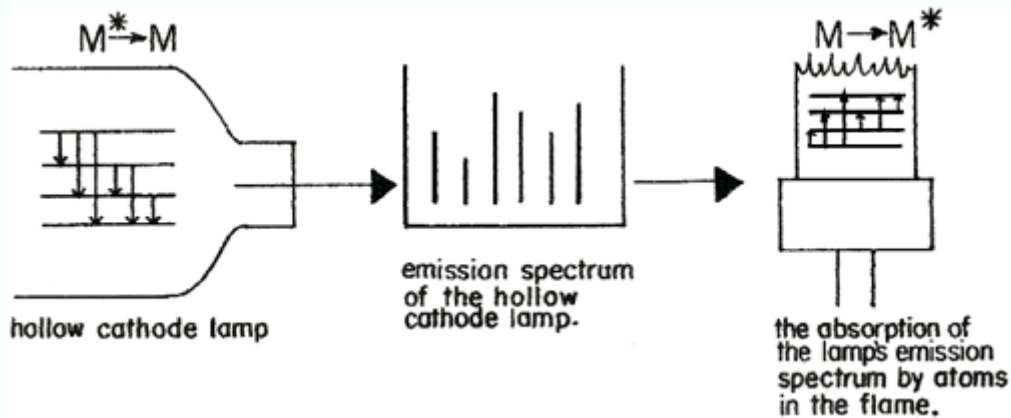
The exact mechanism of the excitation process in the hollow cathode lamp is of interest. Figure 11 is a close-up view of a typical lamp and of the mechanism. The lamp itself is a sealed glass envelope filled with argon or neon) gas. When the lamp is on, argon atoms are ionized, as shown, with the electrons drawn to the anode (+ charged electrode), while the argon ions, Ar+, "bombard" the surface of the cathode (- charged electrode). The metal atoms, M, in the cathode are elevated to the excited state and are ejected from the surface as a result of this bombardment.
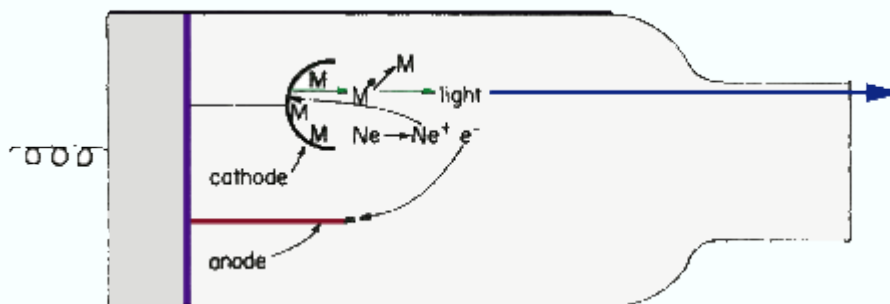
When the atoms return to the ground state, the characteristic line spectrum of that atom is emitted. It is this light, which is directed at the flame, where unexcited atoms of the same element absorb the radiation and are themselves raised to the excited state. As indicated previously, the absorbance is measured and related to concentration.

Flames

All flames require both a fuel and an oxidant in order to exist. Bunsen burners and Meker burners utilize natural gas for the fuel and air for the oxidant. The temperature of such a flame is 1800 K maximum. In order to atomize and excite most metal ions and achieve significant sensitivity for quantitative analysis, however, a hotter flame is desirable. Most AA and FP flames today are air-acetylene flames-acetylene the fuel; air the oxidant. A maximum temperature of 2300 K is achieved in such a flame. Ideally, pure oxygen with acetylene would produce the highest temperature (3100 K), but such a flame suffers from the disadvantage of a high burning velocity, which decreases the completeness of the atomization and therefore lowers the sensitivity. Nitrous oxide (N20) used as the oxidant, however, produces a higher flame temperature (2900 K) while burning at a low rate. Thus, N2O-acetylene flames are fairly popular. The choice is made based on which flame temperature/burning velocity combination works best with a given element. Since all elements have been studied extensively, the recommendations for any given element are available from literature sources or reference books (see pages 451-453) or in applications literature from instrument manufacturers. Air-acetylene flames are the most commonly used.

Burners

There are two designs of burners for the flame atomizer that are in common use. These are the so-called "total consumption burner" and the "premix burner." In the total consumption burner (Figure12), the fuel, oxidant, and sample all meet at the base of the flame. The fuel (usually acetylene) and oxidant (usually air) are forced, under pressure, into the flame, whereas the sample is drawn into the flame by aspiration. The rush of the fuel and oxidant through the burner head creates a vacuum in the sample line and draws the sample from the sample container into the flame with a "nebulizing" or mixing effect. This type of burner head is used in flame photometry and is not useful for atomic absorption. The reason for this is that the resulting flame is turbulent and non-homogenous-a property that negates its usefulness in AA, since the flame must be homogeneous, for the same reason that different sample cuvettes in molecular spectrophotometry must be closely matched. One would not want the absorption properties to change from one moment to the next because of the lack of homogeneity in the flame.
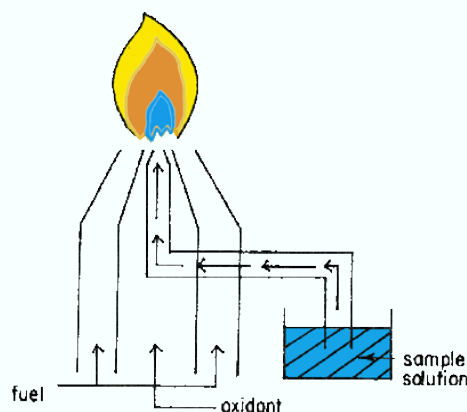
Figure 12. The cross-sectional view of a total consumption burner (nebulizer). The fuel, oxidant and sample all meet at the base of the flame.

The premix burner does away with this difficulty and is the burner typically used in AA. The sample is nebulized and mixed with the fuel and oxidant prior to introduction into the flame, with the use of a series of baffles. Figure 13 is a diagram of this design. Here also the sample is drawn from the sample container via the vacuum created by the rushing fuel and oxidant (aspiration). A drain line is required in this design in order to remove sample solution droplets that do not make it all the way to the flame. See the section entitled "Important Experimental Considerations" for a discussion of a potential safety hazard associated with this.
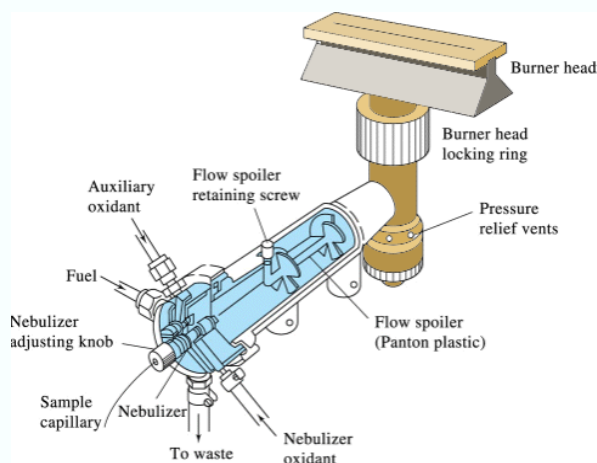

Figure 13.
The cross-sectional view the premix burner (nebulizer). The fuel, oxidant and sample all meet at the base of the flame ad is premixed prior to entering the flame.

Most instruments are equipped to accept a variety of fuels and oxidants. As the gas combinations are varied (see previous discussion), it is usually necessary to change the burner head to one suitable for the particular combination chosen. A faster burning mixture would require a burner head with a smaller slot so as to discourage burning (explosion!) inside the head of a premix burner.

Readout Components
As with molecular spectrophotometry, the readout of the absorbance and transmittance data can consist of either a meter, a recorder or digital readout. The meter can be calibrated in either % transmittance (or % absorption 100 %T) or absorbance, or possibly both. If %T or % absorption are displayed, these of course must first be converted to absorbance (-log T) before plotting.If a recorder is used, it is not the atomic spectrum that is recorded but rather the wavelength is fixed, and the absorbance (or %T or % absorption) is recorded vs. time as the various solutions are aspirated. Such a recording for a series of standards would have the appearance shown in Figure 14. The advantage of such a readout would be to make it easier to obtain a good average value for each solution when electrical (background) "noise" is a problem, as indicated by serious fluctuations in the readings. This problem is very common in *AA,* especially when the sample concentration is low and the sensitivity is set at a high level.
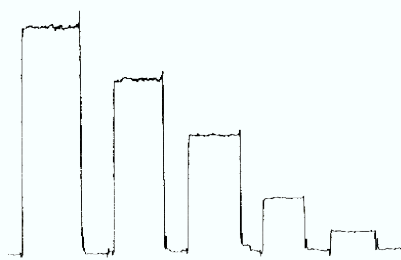

Figure 14 Strip chart recording of the absorption values of a series of standard solution as measured by an AA instrument.

IMPORTANT EXPERIMENTAL CONSIDERATIONS

Safety

There are a number of important safety considerations regarding the use of AA equipment. First of all, the fuel that is used most often is acetylene. The flammability of acetylene itself poses a safety problem, as does the presence of combustion products in the laboratory. For this reason, an independently vented fume hood is placed above the burner to remove burned and unburned fuel from the area. This hood should be on at all times while the instrument is in use. (See Figure 15.)

Also, the danger of so-called flashbacks is ever-present, especially with older instruments. Flashbacks are minor explosions due to improperly mixed fuel and air. These can occur for a variety of reasons, and therefore precautions are taken to prevent their effects as well as their causes. Thus, burner heads and other parts are securely fastened down with safety cables or shields. Some specific causes of flashbacks are: (1) air drawn back through the drain hole in the mixing chamber of the premix burner; (2) shutting off all air to the burner before the fuel has been shut off; and (3) improper proportioning of air and fuel while adjusting the fuel or air flow rates. The solution to these latter two points is proper operator or proper supervisor instruction. Air drawn back through the nebulizer drain hole is avoided by connecting a 6-foot long tube to the drain hole and forming the tube into a loop, which is then filled with water. The other end is then placed into a container of water. (See Figure 15.) Because of the danger of flashbacks, safety glasses must be worn at all times while operating the instrument.
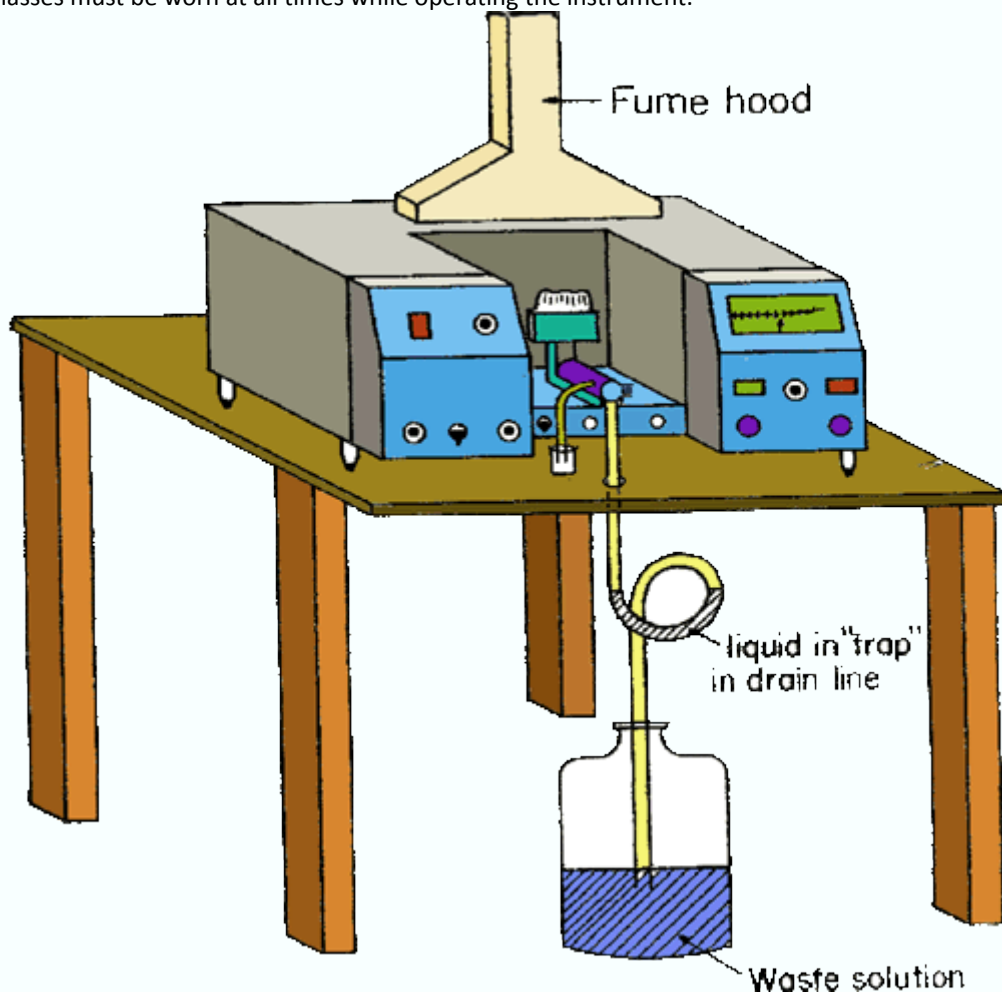
The plotting procedure and the use of the graph for obtaining the sample concentration is altered somewhat, however, The Beer's Law plot is a graph of A vs. concentration *added*. Since the element being determined is present in the sample from the start, a bona fide absorbance reading is measured for the sample to which nothing has been added. As more and more analyte is added, the absorbance reading simply increases (linearly) so that a graph, which does not intersect zero (at zero added concentration) is plotted. (See Figure 16.) Extrapolation of the graph to zero absorbance, as shown, results in a length of x-axis, on the negative *side* of zero added, which represents the concentration in the unknown.
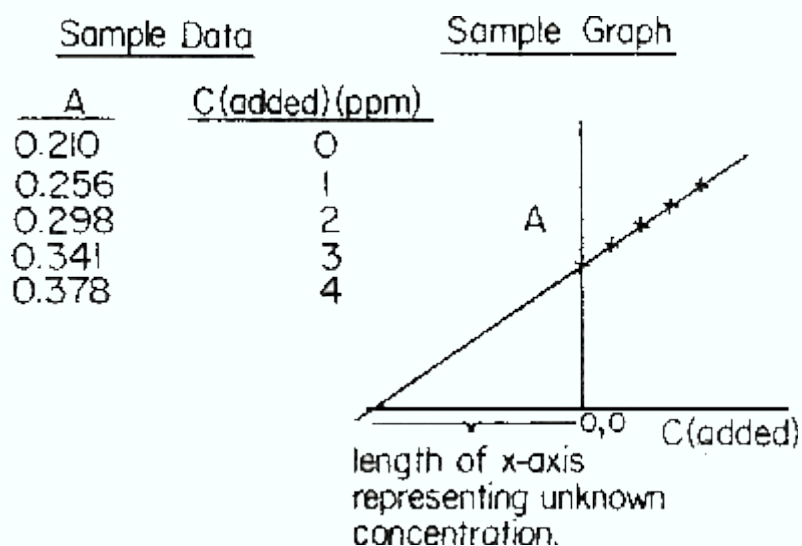

Figure16.
Sample data and graph for a "standard addition" experiment in AA.

This method can be used in cases in which there is some sample preparation as well; for example, in cases in which lanthanum needs to be added. Once the pretreatment establishes the sample matrix, the standard additions can be performed and data obtained and graphed.

Since some sample is consumed by the instrument during each aspiration, and since there can be a significant dilution effect when making the additions, the standard addition method could result in an error due to concentration changes that result. One way to partially compensate is to prepare a series of standard solutions using the sample matrix as the diluent. With either method, volumes of the highly concentrated solution of the analyte (e.g. 1000 ppm) can be quite small (on the order of microliters) so that the dilution effect is negligible. A correction factor for the dilution can also be calculated.

A wide variety of sample matrices are acceptable to AA equipment. These include high concentration of acids as well as organic solvents. The method of standard additions aids in the nullification of any problems associated with such matrices, such as high viscosity or the highly efficient burning of organic compounds. If the method of standard additions is not used, the importance of matching the sample to the standards in terms of organic solvents is paramount.

Maintenance

Finally, periodic cleaning of the burner head and nebulizer is needed to ensure minimal noise level due to impurities in the flame. Scraping the slot in the burner head with a sharp knife to remove carbon deposits and/or removing the burner head for the purpose of cleaning in an ultrasonic bath are two commonplace maintenance chores.

OPERATING INSTRUCTIONS FOR USE OF THE PE AANALYST 2100 ATOMIC ABSORPTION SPECTROPHOTOMETER

The Perkin-Elmer AAnalyst Model 300 Atomic Absorption Spectrophotometer is a sophisticated instrument that allows determination of the concentration of many elements in both organic and aqueous solutions. The basic principles of atomic absorption spectroscopy should be well understood before operating this instrument. You must follow basic safety precautions when working with highpressure gases and high temperature flames and furnaces, and a thorough understanding of the instrument is your best protection. If at any time you have reservations about the use of this instrument, please consult your instructor.

The basic capabilities of the instrument and operating procedures are covered in this document (assuming aqueous samples and standards); advanced operational methods can be found in the instrument manuals and publications available in the laboratory.

* For a very basic overview of AAS, consult the Perkin-Elmer Publication: *Concepts, Instrumentation, and Techniques in Atomic Absorption Spectrophotometry*

* For a more advanced overview of AAS, consult CH 9, "Atomic Absorption and Atomic Fluorescence Spectroscopy" in *Principles of Instrumental Analysis, 6th Ed.* by Skoog, Holler, and Crouch

* For specific sample methods, consult the Perkin-Elmer Manuals:

o *Analytical Methods for Atomic Absorption Spectrophotometry*

o *Analytical Techniques for Graphite Furnace AAS*

o *Graphite Furnace AAS, A Source Book*

* For software related applications, consult the Perkin-Elmer Manual: *AA Winlab Software Guide for Atomic Absorption Spectroscopy*

* For hardware related applications, consult the Perkin-Elmer Manual:

o *AAnalyst 100/300 Atomic Absorption Spectrometer Hardware Guide for Atomic Absorption Spectroscopy*

o *HGA-800 Graphite Furnace System including the AS-72 Autosampler Hardware Guide*

* For questions related to the water circulator, consult the Lauda/Brinkman Manual: *Operating Instructions #132: Lauda/Brinkman Circulator, Model K-2/R, Cat. No. 27 50 150-8 & Model Super K-2/R Cat. No. 27 50 400-1*

BASIC OPERATING PROCEDURES

1. Prepare your samples according to methods appropriate for both the metal ion you wish to study and for the sample matrix from which you wish to extract the metal ion. Note the final analysis medium used—either organic solvent or aqueous solution. Consult the literature and/or the methods manuals for that particular metal to find the recommended instrumental parameters.

2. Prepare a blank and a set of standards within the concentration ranges appropriate for the method you have chosen. Consult the table below to determine the appropriate absorption wavelengths and standard concentrations for measurement of your metal ion analyte.

Element	Wave Length, (nm)	Sens. Check [a] (mg/L)	Linear Range [b] (mg/L)	Min.[c] (mg/L)	Max. [d] (mg/25 mL)	Expected Absorbance Units
Antimony	217.6	15.0	100.0 - 1.5	0.038	2.50	0.10-0.20
Barium	553.6	10.0	50.0 - 1.0	0.10	0.20	0.06-0.12
Bismuth	223.1	10.0	50.0- 1.0	0.025	1.25	0.12-0.24
Cadmium	228.8	0.5	3.0 - 0.05	0.075	0.0013	0.18-0.36
Calcium	422.7	0.5	3.0 - 0.05	0.0013	0.075	0.25-0.50
Cobalt	240.7	2.5	15.0 - 0.25	0.0063	0.375	0.08-0.16
Copper	324.8	1.5	10.0 - 0.15	0.0038	0.250	0.11-0.22
Iron	248.3	2.5	15.0 - 0.25	0.0063	0.375	0.20-0.40
Lithium	670.8	1.0	5.0 - 0.10	0.0025	0.125	0.11-0.22
Magnesium	285.2	0.15	1.0 - 0.015	0.0004	0.025	0.25-0.50
Manganese	279.5	1.0	5.0 - 0.10	0.0025	0.125	0.22-0.44
Molybdenum	313.3	15.0	100 - 1.50	0.038	2.50	0.13-0.26
Nickel	232.0	4.0	0.40 - 0.01	0.50	20.0	0.18-0.36
Potassium	766.5	0.4	2.0 - 0.04	0.001	0.050	0.20-0.40
Silver	328.1	1.5	10.0 - 0.15	0.0038	0.250	0.11-0.22
Sodium	589.0	0.15	1.0 - 0.015	0.0004	0.025	0.25-0.50
Strontium	460.7	2.0	10.0 - 0.20	0.005	0.250	0.35-0.70
Zinc	213.9	0.4	2.0 - 0.04	0.001	0.050	0.20-0.40

a. NOTE: Because this instrument generates an enormous amount of heat in the burner flame or graphite furnace and a substantial amount of air, acetylene, and/or argon are required to run the instrument, it is important that you have all standard and sample solutions and a blank solution ready to analyze and record once you set up the instrument and start the flame or furnace as described in subsequent steps.

b. ALSO NOTE: Please be reminded that whenever you are using compressed gases and high temperature flames or furnaces, you must exercise extreme caution. Proper safety attire (goggles, lab coats, long pants, closed-toed shoes, *etc.*) must be worn as always when working in the laboratory. Flame atomic absorption spectrophotometers can, on rare occasion, flash back through the burner assembly, so you will wish to pay special attention to the step in which you verify that the safety interlock pin is latched and the steel cables connected to the burner head are properly attached. For your safety, you will also wish to locate the nearest fire extinguisher, fire alarm, and fire exit before proceeding.

3. Assure that the solution collection bottle is empty and that the tubing is placed properly inside of it. If the solution collection bottle needs to be emptied, notify the instructor. Also, for flame

measurements, place the aspirator tube into a clean beaker containing deionized water in the tray below the burner head assembly. For graphite furnace measurements, assure that the pipet for the autosampler is connected to the tubing that flows to an appropriate solvent in the solvent bottle to the left of the instrument.

4. Under the supervision of or with permission from an instructor, install in the turret the appropriate hollow cathode lamp (HCL) for the element you wish to analyze. Record the maximum operating current (somewhere between 3 and 15 mA) that is likely printed on the bottom of the bulb, and assure that it is not exceeded when powering up the bulb.

a. NOTE: There are two basic HCL designs used in atomic absorption spectrophotometers—one that is 1.5 inches in diameter and one that is 2.0 inches in diameter. All of the Perkin-Elmer instruments use the two inch bulbs, and the turret for the bulbs, located inside the door on the upper right quadrant of the instrument, is designed to receive only the larger HCLs. There is an adapter available to allow the PE Aanalyst 300 to use the smaller bulbs, but the instrument detection limits may not meet specifications if the smaller source is used.

b. Following the installation of the bulb in the holder, plug in the power cable end fitting into the socket on the turret. If you are using an adapter for the smaller bulbs, you will need to use an additional adapter cable for the power connection.

5. Turn on the exhaust hood using the VENT FAN SWITCH behind the instrument (to the left) on the wall, and assure that the cutoff slide is open. Close the two cutoff slides for the exhaust from the atomic emission spectrometer to the left of the AA. Verify that the air is being pulled into the top of the cone above the AA before proceeding (*i.e.*, assure that a KimWipe is pulled upward).

6. Power up the computer and monitor. Hit Default when prompted for a Password.

7. Turn on the power to the Aanalyst 2100 Spectrometer (on the left-hand side of the instrument). Be certain to record any unusual observations or changes that you make to the instrument.

FOR FLAME MEASUREMENTS:

9. On the computer, select and launch the AA WinLab Analyst Software. The software will initialize and establish communication with the spectrometer. If the initialization step includes a verification that the furnace is on (*i.e.*, you are the first to use the flame set-up after the instrument has been used in graphite furnace mode), you will need to exit the Checking Connections box and select Flame on the Technique menu bar.

10. When prompted, select "Use the basic system procedure for…" and choose the setup designated byyour instructor. Alternatively, you may develop your own setup by consulting the appropriate section in the instrument manual.

11. If necessary,

a. align the lamp according to the procedures outlined in the *AA Winlab Software Guide for Atomic Absorption Spectroscopy*, Section 3-3.

b. adjust the burner height as described in the *AAnalyst 100/300 Atomic Absorption Spectrometer Hardware Guide for Atomic Absorption Spectroscopy*, Section 4-74.

12. Consult the literature and/or the methods manuals to determine the appropriate gas mixture for measurement of your metal ion analyte.

a. Turn on the air supply and adjust the outlet pressure to 60 psi or that which you determined from the literature.

b. Turn on the acetylene supply and adjust the outlet pressure to 13-14 psi or that which you determined from the literature.

13. Double check that you know the location of the fire extinguisher, and then select Flame from the Toolbar, and light the burner. Close the flame control window, and make certain that the Print Log has been selected.

14. Measure Blank, Standards, and Sample(s)

a. Aspirate a blank solution (solvent matrix with no analyte), and select Analyze Blank. NOTE: When you remove the aspiration tube from a solution, insert it quickly into the next solution to prevent significant gas build-up in the tube.

b. Aspirate a standard solution beginning with the least concentrated. When the reading stabilizes, select Analyze Standard. Record the absorbance in your notebook. Repeat this step with each of your standard solutions in order of increasing concentration.

c. Aspirate your sample solution(s), and select Analyze Sample when the absorbance reading stabilizes. Verify that the absorbance for your sample falls between the readings for your standards. If it is too high, you will wish to dilute and re-measurethe sample. If it is too low but within the detection limits of the instrument, you will wish to prepare and measure standard solutions of lower concentration.

d. Aspirate the blank solution again, and record the reading in your notebook. A value that differs significantly from zero is problematic and will require your attention.

e. Record all measured absorbances in your notebook.

15. Close the Manual Analysis window. To view the calibration curve, select Calib. To view the results of the sample analysis, select Results. To retrieve the last page of the printed output, select New Page from the Print command list. Affix printed hard copies into your notebook.

16. SHUTDOWN PROCEDURES

a. After the final solution has been aspirated, allow at least three minutes of pure deionized water to aspirate through the burner head to remove all reagents.

b. Open the Flame Control window, and click on the Flame icon. The system will automatically extinguish the flame safely.

c. Turn off the burner gas supplies to the spectrometer.

d. In the Flame Control window, click on the Bleed Gases button to depressurize the gas lines.

e. Wait at least 10 minutes after you have extinguished the flame, and then turn off the fume ventilation system using the switch behind the instrument.

f. Exit the software and Windows, and turn off the spectrometer, the computer, and the printer.

g. Empty the drain vessel, being certain to dispose of waste properly.

h. Store samples and standards or dispose of them properly. They should not be left near the instrument.

i. Double check that you carried out each of steps 16a-h before exiting the lab.

http://en.wikipedia.org/wiki/Atomic_absorption_spectroscopy
http://www.labcompare.com/Spectroscopy/Atomic-Absorption-Spectrophotometer/
http://faculty.sdmiramar.edu/fgarces/labmatters/instruments/aa/AAS_Theory/AASTheory.htm
http://users.etown.edu/k/kneask/Instrument%20Operating%20Procedures_files/PE_AA.pdf