

## CHAPTER 5

# Atomic Absorption Spectroscopy

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## 5.1 INTRODUCTION

Atomic absorption spectroscopy is an analytical technique which has been developed primarily for the determination of metals at low levels of concentration.<sup>1</sup>

It is based on the absorption of radiation by free atoms. Each chemical element in the atomic state absorbs only radiation of well-defined wavelengths characteristic of the particular element involved. It is the reverse physical process to that involved in flame photometry and emission spectroscopy. The relationship between these processes is illustrated in Fig. 5.1.



FIGURE 5.1: Relationship between atomic emission and atomic absorption.

Atomic absorption takes place when unexcited atoms absorb energy and become excited atoms. Absorption therefore is carried out by unexcited atoms, whereas emission arises from excited atoms. This relationship leads to the principle that all experimental conditions that affect the total atom population affect the emission and absorption signal in a similar fashion. All variables that increase the percentage of excited atoms (such as an increase in atomizer temperature) increase the intensity of the emission signal, but decrease the absorption signal (very slightly). All variables that affect the number of unexcited atoms affect only the atomic absorption signal. It is important to remember this principle when developing new procedures based on atomic absorption spectroscopy.

## 5.2 ADVANTAGES OF ATOMIC ABSORPTION SPECTROSCOPY

### A. WIDESPREAD APPLICATION

Atomic absorption spectroscopy has been used for the quantitative determination of most of the metals in the periodic table. This illustrates the widespread application of the method to quantitative elemental analysis. In this respect it compares very favorably with other methods of elemental analysis, such as X-ray fluorescence and emission spectrography.

### B. HIGH SENSITIVITY

It has been shown experimentally to be very sensitive compared to other techniques for the determination of trace quantities of metals. Many elements can be determined at the part-per-million concentration level and some can be determined at the part-per-billion level. These facts illustrate the high sensitivity of the method.

### C. FREEDOM FROM INTERFERENCE

A third advantage of the method is its high degree of freedom from interference. No two elements absorb at the same resonance wavelength as each other. As a result, the presence of one element does not directly interfere with the absorption of radiation by another element. Conversely the measurement of absorption is generally a direct measure of the concentration of the absorption element, irrespective of the other elements present.

### D. INDEPENDENT OF FLAME TEMPERATURE AND ABSORPTION WAVELENGTH REGION

Absorption is by unexcited atoms. The ability of the atom to absorb is independent of atomizer temperature and the spectral region of the absorption wavelength. As a consequence, atomic absorption is free from interference from atomizer temperature or the wavelength region of the absorption lines. This is in direct contrast to emission methods, where each of these variables directly affects the emission signal. For example, the sodium resonance line is 5890 Å. This element emits and absorbs strongly at this wavelength. However, the zinc resonance line is at 2138 Å. It absorbs strongly at this wavelength, but the emission is extremely weak.

With these advantages atomic absorption spectroscopy is capable of providing accurate and precise answers when the analysis is performed properly. Further, inasmuch as the equipment is simple to operate, routine analytical laboratories find it an attractive tool.

## 5.3 DISADVANTAGES OF ATOMIC ABSORPTION SPECTROSCOPY

### A. DOES NOT DETERMINE NONMETALS

In common with all methods of analytical chemistry atomic absorption spectroscopy is subject to some disadvantages. At present, it has not been found useful for the direct determination of nonmetallic elements such as halides, oxygen, and nitrogen. This is because the resonance line of these elements is in the vacuum ultraviolet region of the spectrum and therefore

cannot be used with present-day equipment. To operate in this region requires that the optical path be in a vacuum. This itself presents a difficult problem in practice. However, the problem is made much more difficult to solve because the common atomizers use flames and flames absorb strongly in the vacuum ultraviolet. It would be difficult to distinguish between the flame absorption and the absorption by the sample. As a result, no equipment is available to use in this spectral region.

#### B. DOES NOT ANALYZE SOLID OR GAS SAMPLES DIRECTLY

Another problem in atomic absorption is that it is very useful for analyzing liquid samples, but has not been successfully used for the direct analysis of solids or gases. The difficulty arises in the atomizer stage. All commercial equipment uses atomizers built to handle liquid samples, but not any other phase. Research equipment has been developed to handle solid samples, but these are not commercially available. In routine work, difficulties were encountered in the use of this research equipment and in the interpretation of data obtained using this equipment. Commercial manufacturers have not been encouraged to develop suitable atomizers for handling solid or gas samples.

To carry out a determination, all solid samples must be decomposed or dissolved in a suitable solvent. Similarly, gas samples must be washed or scrubbed so that the metal components are trapped in a suitable liquid. This step may be time-consuming and is always a source of error. However, with proper care these steps can be performed and accurate results obtained.

#### C. DETERMINES ONLY ONE ELEMENT AT A TIME

The equipment which is currently available is built to determine only one element at a time. This is no problem if a number of samples must be analyzed for a single element. But if one sample must be analyzed for several elements, a difficulty may arise. Each element must be determined separately. This means the use of a different hollow cathode, a change of wavelength, and possibly different flame conditions for each element. These equipment changes are not difficult to perform, but they are time-consuming. It should also be remembered that a separate portion of sample must be used for the determination of each element. This may be expensive in terms of the quantity of sample used. About 1 ml of sample is required for each determination.

#### D. ANIONIC INTERFERENCE

The major source of analytical interference is from the anions present in the sample. The anions do not absorb the atomic radiation, but they affect the population of free atoms formed in the atomizer.

When a liquid is introduced into a flame atomizer, the liquid is normally in the form of a droplet. The droplet evaporates, leaving a solid residue. The

residue is then decomposed by the flame and free atoms are liberated in the process. The last step is directly affected by the predominant anion. The predominant anion determines the chemical form of the metal in the residue. Some chemicals are more easily decomposed than others. For example, a residue of calcium chloride is relatively easily decomposed, but calcium phosphate is much more difficult to break down. More free calcium would be formed in the atomizer from a solution of calcium chloride than from a solution of calcium phosphate of equal calcium concentration. The two solutions would give different absorption signals and therefore different analytical results.

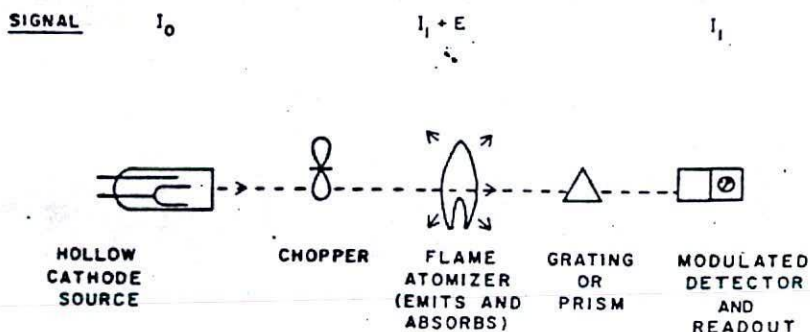


FIGURE 5.2: Schematic diagram of equipment.

The problem can be overcome by using a calibration curve prepared from solutions with the same predominant anion as the sample, or by complexing the metal with an organic reagent. In general, the interference is not as severe as in the situation illustrated. However, when setting up a routine procedure, steps should always be taken to eliminate any possible interference from a change in the predominant anion from one sample to the next.

## 5.4 EQUIPMENT

The equipment used is basically the same as for other spectroscopic absorption methods. It consists of a radiation source, a monochromator, a sampling device, and a detector. A schematic diagram of the equipment is shown in Fig. 5.2.

The individual components are as follows.

### A. RADIATION SOURCE

Atoms absorb at a characteristic wavelength and over a very narrow wavelength range. The width of the absorption lines is about  $0.01 \text{ \AA}$ , even under the most adverse conditions.

As a result "continuous" wavelength sources such as the hydrogen lamp or the tungsten lamp are at a severe disadvantage in this equipment. Even under optimistic conditions of resolution and slits, a wave band about  $2 \text{ \AA}$  wide falls onto the detector. The sample atoms can only absorb out a bandwidth of  $0.01 \text{ \AA}$ , consequently, when the atoms absorb all the radiation they can absorb, a large proportion of the radiation still passes unabsorbed to the detector. The method would be insensitive under these conditions and analytical application would be severely limited. The detector would only find a slight change in total radiation intensity, even though the concentration of atoms was high enough to remove all the radiation over its absorption wave band.

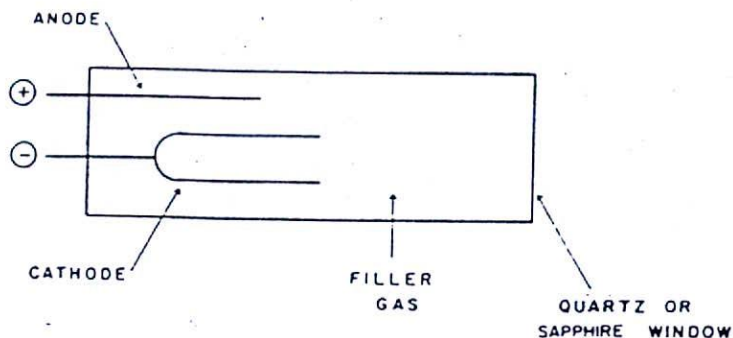


FIGURE 5.3: Diagram of a hollow cathode.

To overcome these problems, the hollow cathode has been developed by Walsh<sup>1</sup> and co-workers as a radiation source. A schematic diagram of this source is shown in Fig. 5.3.

The filler gas used is ionized at the anode. The charged ion is attracted to the cathode. On arrival, it strikes the metal surface and liberates free excited atoms from the surface of the metal. The atoms emit at their own characteristic wavelength. The width of the emission lines emitted by the metal atoms is very narrow and matches the absorption line width of the absorbing atoms from the sample.

If the absorption line is isolated from the rest of the spectrum, it alone will fall on the detector. The absorption line width is similar to the emission line width, hence all the radiation falling on the detector can be absorbed by the atoms. A great increase in sensitivity results.)

Recently, high-intensity hollow cathodes have been developed. A diagram is shown in Fig. 5.4. As described in the simple hollow cathode, a cloud of atoms is formed by bombardment of the cathode by filler-gas ions. This creates a cloud of atoms at the mouth of the cathode. Some of these atoms are excited and emit characteristic radiation, but a large proportion are

unexcited and do not contribute to the signal. A beam of slow-moving electrons passes through the atom cloud and causes them to become excited. The resonance line is greatly enhanced compared to other spectral lines. The lamp is therefore able to emit at the resonance wavelength with a high intensity.

This lamp is very useful if the resonance line is similar in wavelength to other unabsorbed lines originating from the cathode. For example, the

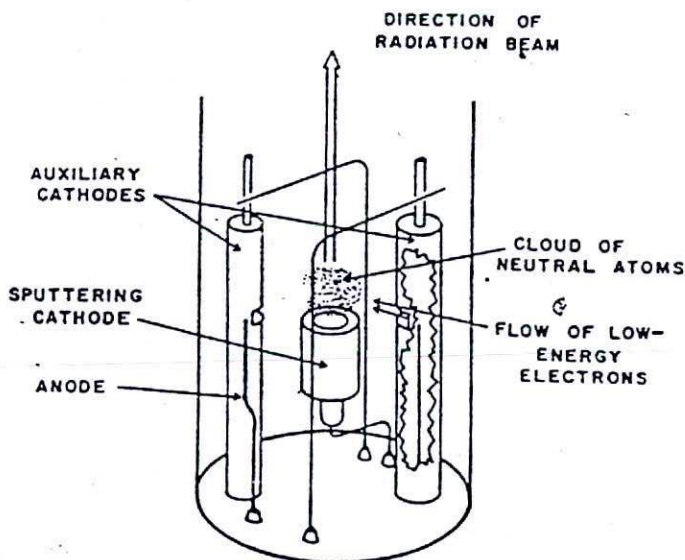


FIGURE 5.4: Schematic diagram of a high intensity lamp.

wavelength of the nickel resonance line is  $2320 \text{ \AA}$ , but the nearby  $2321.4 \text{ \AA}$  is not a resonance line.

Both lines fall on the detector. Even if all the  $2320 \text{ \AA}$  lines were absorbed, the detector would still be exposed to the  $2321.4 \text{ \AA}$  line. Since the  $2321.4 \text{ \AA}$  line is not absorbed, its presence results in a loss of sensitivity of the procedure. Using a high intensity lamp greatly reduces this problem. However, it should be pointed out that high intensity lamps are only useful under these special circumstances. Normally, if the resonance level is strong and isolated from other lines, the advantage of high intensity is limited.

One difficulty involved in using the hollow cathode source is that the cathode must be made of the same element as that being determined. As a consequence, only one element at a time can be determined. As mentioned earlier, multielement hollow cathodes have been developed which will enable three elements to be determined simultaneously. However, in general these

sources are not very stable and have a shorter operational life than conventional single-element hollow cathodes.

## 5.5 MONOCHROMATOR

The optical range of this analytical field is between 2000 and 8000 Å. The function of the monochromator is to select radiation of the correct wavelength and eliminate other radiation from the light path. For many elements such as sodium, potassium, and copper the spectrum is simple and only a low resolution monochromator is required. However, for certain other elements, particularly the transition elements, high resolution is necessary to prevent unabsorbable emission lines originating either from the cathode or the filler gas from falling on the detector. Commercial equipment is usually fitted with a high resolution prism or grating monochromator. Such a prism can be used whether high resolution or low resolution equipment is necessary.

There is little difference in performance between the prism and the grating. Commercial equipment tends to favor the grating.

The use of filters has not been widespread, although it would appear that for very repetitive analysis for a few elements, filters would have been used as they are used in flame photometry. This simple monochromator has not yet been used commercially for atomic absorption spectroscopy.

## 5.6 DETECTORS

Photomultipliers are used exclusively on commercial equipment. In practice, it is the function of the detector to measure the intensity of radiation before and after absorption by the sample. From this we can calculate how much radiation has been absorbed from the intense beam. If the amount of absorption is small, then the detector must compare an intense beam and a slightly less-intense beam. This measurement cannot be done with film, with any degree of accuracy, but can be done using a photomultiplier.

## 5.7 OPTICAL SLIT SYSTEM

Two slits are included in the optical system, an entrance slit and an exit slit. The entrance slit serves to obtain a narrow, parallel beam of light from the source. Other radiation, including stray radiation, is physically blocked out by the walls of the slit. The exit slit is used to select radiation of the correct wavelength after it emerges from the monochromator. Other radiation is blocked out and not allowed to continue down the light path. The function of the slits is illustrated in Fig. 5.5.



Because the hollow cathode emits very narrow lines, the actual slit width used is not critical. However, when high resolution is required to eliminate unabsorbed radiation from nearby nonresonance lines, narrow slit widths are necessary.<sup>2</sup> This situation only occurs for a few elements such as iron and nickel. For other elements the slits can be left at a standard width. Most analytical procedures<sup>2,3</sup> specify the slit widths to be used for the particular determination described.

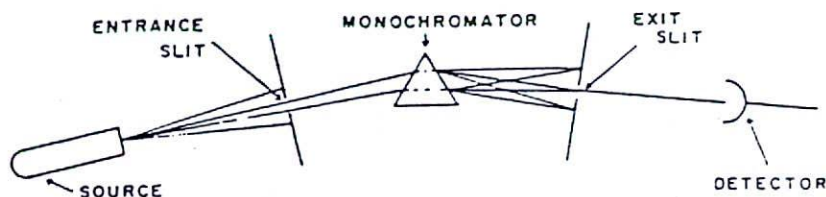


FIGURE 5.5: Function of the slit.

## 5.8 MODULATION

The wavelength at which atoms are absorbed is usually the "resonance wavelength." Absorption at this wavelength causes a transition from the ground state to the first excited state of the atom. Unfortunately, the atoms of many elements in a flame also emit radiation at this same wavelength. This is particularly so if the resonance wavelength is longer than 3000 Å. Radiation is usually very intense from atoms such as sodium, potassium, lithium, and calcium, which emit at comparatively long wavelengths.

This emission will create a problem in measuring the degree of absorption by the sample. For example, examine the following circumstances:

Intensity of radiation from lamp	Intensity of radiation after absorption by sample	Emission intensity by sample at same wavelength
100 units	50 units	20 units

The "observed" absorption would be:  $100 - (50 + 20) \text{ units} = 30 \text{ units}$ .  
The true absorption was  $100 - 50 \text{ units} = 50 \text{ units}$ .

The emission from the sample in the flame can be, therefore, a serious source of error. It can be overcome by modulating the instrument. This entails using an alternating light source and a detector tuned to the same frequency.

Under these circumstances, the detector "sees" the alternating light from the source and can detect any absorption of that light, however, the dc light

from the flame does not give rise to a signal from the tuned detector. The emission signal is thereby eliminated as a source of error and only the absorption signal is measured.

The principal manufacturers of atomic absorption equipment all produce modulated equipment. However, to avoid any unnecessary source of error this point should always be checked when buying a new instrument.

#### A. ATOMIZER

The function of the atomizer is to convert the combined atoms of the liquid sample into free atoms. The most common atomizer is the flame. In practice

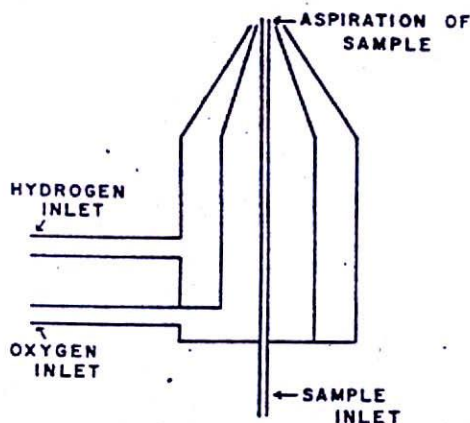


FIGURE 5.6: Total consumption burner.

the liquid sample is introduced into the flame in the form of a droplet. The droplets evaporate and leave a solid residue. The residue, which contains the same atoms, is decomposed by the flame and free atoms are liberated. These free atoms absorb the radiation which is measured in this procedure. The free atoms exist in the flame for varying periods of time, but in most instances quickly become oxidized to the metal oxides, which do not absorb.

The rate of formation and the rate of oxidation of the atoms depends to a large extent on flame conditions such as the fuel used, flame temperature, and fuel-to-oxygen ratio. This series of reactions enters a state of dynamic equilibrium and a steady number of atoms exist at any one time. To get quantitative results it is important that this number remains constant and is reproducible.

Two types of flame atomizers are available, the total consumption burner and the Lunde-gardh burner. The whole sample is aspirated into the base of the flame and then atomized. The burner is illustrated in Fig. 5.6 and employs a preevaporation chamber which evaporates the sample before it

reaches the burner. This burner tends to be more sensitive than the total consumption oration chamber, as illustrated in the Beckman or preheat burner shown in Fig. 5.7.

The total consumption burner is most useful if the sample contains several solvents. It assures that all the sample reaches the flame. The Lundegardh burner is physically quieter and frequently enables more sensitive results to be obtained. Its efficiency is improved because the evaporation step occurs prior to reaching the burner. This helps to conserve the energy of the burner for atomizing the sample.

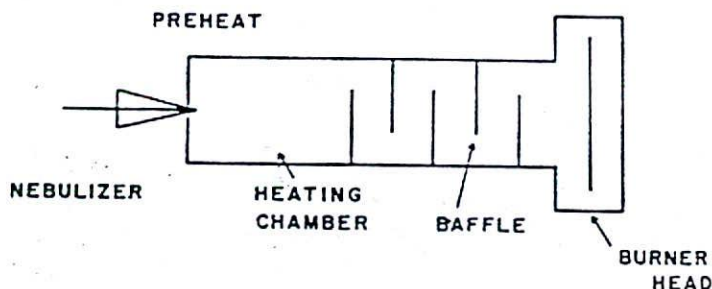


FIGURE 5.7: Preheat burner.

## 5.9 ANALYTICAL VARIABLES

The *quantitative* application of atomic absorption spectroscopy is based on two relationships. First, the degree of absorption is a function of how many free atoms are present in the light path. Second, the number of free atoms is a function of the concentration of the metal on the original sample.

Consequently, the degree of absorption is proportional to the original concentration of metal. For this relationship to be meaningful all the variables concerned must be carefully controlled. Each step will be considered separately.

The degree of absorption is described mathematically by

$$K_{\nu} d_{\nu} = \frac{\pi e^2}{mc} N f \quad (5.1)$$

- where  $K_{\nu} d_{\nu}$  = total amount of radiation absorbed at resonance frequency  $\nu$   
 $e$  = charge of the electron (a constant)  
 $m$  = mass of the electron (a constant)  
 $c$  = speed of light (a constant)  
 $N$  = number of atoms of the light path  
 $f$  = oscillator strength of the absorption line ( $f$  varies from element to element, but is constant for a particular element at the resonance frequency)

From this equation it can be seen that for a given line

$$K, d, = \text{constant} \times N \quad (5.2)$$

Hence, the degree of absorption is a physical property of the system and depends on the number of atoms in the light path.

However, the relationship between  $N$ , the number of free atoms produced in the atomizer, and the concentration of the element in the sample depends on the atomizer efficiency.

The process of atomization depends on several variables. Included are the type of burner used, the rate at which the sample is introduced into the flame, the type of flame used, such as oxyhydrogen or air-acetylene, and the type of solvent used. Each of these variables noted affects the efficiency of atomization.

### 5.10 BURNER DESIGN

The burner design is of prime importance, as can easily be imagined. The function of the burner is to evaporate the liquid component of the sample and to atomize the element being determined. The efficiency with which a burner carries out these functions depends on its design. It varies greatly from one type of design to another. There is even a significant variation in efficiency between two burners of the same design. It can be seen therefore that for reproducible results to be obtained, the identical burner should be used for analyzing the samples as that used for preparing the calibration curves.

### 5.11 SAMPLE FLOW RATE

Any given burner atomizes a sample best at the optimum sample flow rate. If the flow rate is too low, too few atoms are formed, if the flow rate is too high, the flame becomes swamped. Between the extremes is the optimum flow rate. In practice, the manufacturers of the burners determine the optimum flow rate of their burner and design it accordingly. No control over this variable is left to the operator. However, should the burner become clogged or distorted, a change in efficiency occurs and erroneous answers are obtained. Care should always be taken to prevent burner clogging during operation.

### 5.12 TYPE OF FLAME

The type of flame directly affects atomization efficiency. Hot flames decompose the sample more easily than cool flames. Hence, hot flames such as oxyhydrogen flames have a higher efficiency than air-propane flames.

The free atoms formed generally become oxidized shortly after formation.

However, if there is an excess of fuel over oxygen in the flame, the lifetime of the free atoms is extended. By the same reasoning, if there is an excess of oxygen in the flame, the free atoms quickly become oxidized and no longer absorb at their resonance wavelength.

Some elements such as aluminum, titanium, tungsten, and molybdenum form very stable oxides in the flame. Until recently, it was difficult to detect these elements by atomic absorption. However, it has been found by Willis and Amos<sup>1</sup> that by using a nitrous oxide-acetylene flame, the oxide formation is delayed and these elements can be detected at the part-per-million concentration level.

### 5.13 SOLVENT

The type of solvent used in the sample has a profound effect on atomization efficiency. Aqueous solvents require energy to be evaporated. The residue is usually a hydrated inorganic salt and this requires energy to be decomposed. The whole process of atomization is endothermic and proceeds relatively slowly.

However, if the solvent is organic, it will burn in the flame instead of evaporating. Further, the residue is usually in the form of an organic compound and this burns and decomposes. In short, the reaction is exothermic and atomization is more efficient than with aqueous solvent. The net effect is that when organic solvents are used, a greater absorption signal is observed than when aqueous solvents are used with the same metal concentration.

To obtain reproducible results all these variables must be controlled. In practice, it is not as difficult a task as it appears, but it does require attention in order to avoid errors.

### 5.14 WAVELENGTH CHOICE

For each element there are several absorption lines originating in the ground state. The most sensitive line is associated with the transition from the ground state to the first excited state. However, a less sensitive absorption line is associated with the transition from ground state to the second excited state. For many elements there are several absorption lines available. If high sensitivity is required, the most sensitive line is used. However, this line could not be used for the determination of a high concentration of the element because most of the radiation will be absorbed by the large concentration of atoms produced from such a sample. Large changes in concentration between samples would result in only small changes in absorption and this would lead to imprecise answers. For high concentrations therefore a less strongly absorbed line is used. The analytical range of the various lines is generally given in the analytical procedure when it is described in the literature.

### 5.15 QUANTITATIVE METHOD

Equation (5.1) shows the relationship between the degree of absorption and the number of atoms in the light path. However, the variable efficiency of most flames in producing free atoms from the sample makes it impossible to calculate the concentration of the metal in the sample from the determination of  $N$ , the number of atoms in the light path.

The conversion of atoms existing in the sample as molecules to free atoms is a very inefficient process. A valid arithmetic calculation of  $N$ , and therefore

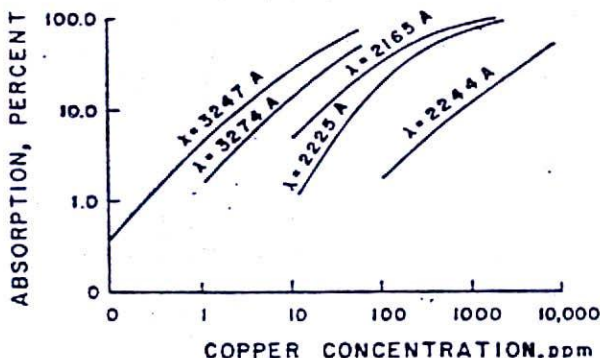


FIGURE 5.8: Calibration curves for copper determination.

the concentration of the element in the sample, cannot be made. Variables such as the efficiency of atomization negate the calculation. The relationship between absorption and concentration is therefore usually determined experimentally by using a calibration curve.

#### A. PREPARATION OF CALIBRATION CURVES

A series of solutions of the sample element are prepared. The concentration of the element in each solution is predetermined by weighing known amounts of a salt of the metal into a known volume of solution.

The atomic absorption spectrometer is set at the desired resonance wavelength for the metal, and each solution is introduced successively into the flame atomizer. The absorbance is measured for each solution, and the relationship is then plotted out. The plot constitutes a "calibration curve." A typical calibration curve is shown in Fig. 5.8.

This curve indicates the relationship between absorbance and concentration of copper in the standard samples.

When a sample of unknown concentration is to be analyzed, it is introduced into the atomizer and the absorbance measured. From the calibration curve

the concentration corresponding to the measured absorbance is read off. From this the analysis of the original sample can be determined.

The analytical range of the procedure is determined primarily by the sensitivity limits of the method. Calibration curves can be prepared starting at concentrations about ten times as great as the sensitivity limit. Quantitative determination with lower precision can be obtained at lower concentrations.

The sensitivity of detection of various elements is shown in Table 5.1.

TABLE 5.1: Analytical Sensitivities of the Various Elements\*

Element	Wavelength, Å	Sensitivity, ppm
Al	3092	2.0
Sb	2175	0.5
As	1937	3.0
Ba	5536	5.0
Be	2349	0.2
Bi	2231	1.0
B	2497	250
Cd	2288	0.04
Ca	4227	0.1
Cs	8521	0.15
Cr	3579	0.15
Co	2407	0.5
Cu	3247	0.1
Ga	2944	4
Au	2428	0.1
Fe	2483	0.3
Pb	2170	0.5
Li	6708	0.07
Mg	2852	0.02
Mn	2798	0.01
Hg	2357	10.0
Mo	3133	0.5
Ni	2320	0.2
Pd	2474	1.0
Pt	2148	2.0
K	7665	0.1
Rb	7800	0.2
Se	1961	0.5
Ag	3281	0.1
Na	5890	0.03
Sr	4607	0.05
Tl	2767	1.0
Sn	2863	5.0
Ti	3653	12.0
V	3184	7
Zn	2138	0.05

\* The sensitivity is here defined as that concentration of the element which results in the absorption of 1% of the resonant radiation passing through the atomizer.

These sensitivities are reported by different workers in the field and may vary somewhat with equipment and experimental conditions.

The actual experimental conditions to be used for the quantitative determination of each of these elements should be ascertained from the literature.

## 5.16 CONCLUSIONS

Atomic absorption spectroscopy is an analytical procedure used for the determination of the metals and metalloids. It is sensitive compared to other analytical methods. It enjoys a higher degree of freedom from interference than most methods and is therefore capable of giving accurate and precise analytical results.

The cost of commercial equipment varies from \$3500 to \$7000. Training of personnel is relatively simple. Also, installation is easily carried out. For highly routine analysis it is a most attractive method.

## EXPERIMENTS

- E5.1. Prepare a calibration curve for copper salts using copper sulfate in water for the standard solution. Make solutions containing  $\text{Cu}^{2+}$  in the range 2 to 20 ppm. Measure absorbance of the resonance line at wavelength 3247 Å. Plot absorption curve for the same solution at the resonance line 2024 Å.
- E5.2. Prepare a calibration curve for lead salts. Use lead solution (as nitrate) with a concentration range between 4 and 40 ppm. Measure the absorption at 2170 Å. Plot absorbance vs. concentration. Add chloride to the lead solutions; note the depression of the absorption. Add EDTA to the chloride solution; note the absorption depression is removed.

## QUESTIONS

- Q5.1. Why is it necessary to use a hollow cathode lamp as a light source in atomic absorption spectroscopy?
- Q5.2. Why is atomic absorption equipment modulated?
- Q5.3. What are chemical interferences?
- Q5.4. The data for preparing a calibration curve for gold was as follows:

Gold Conc. ppm	Absorbance
3	0.12
6	0.24
12	0.48
15	0.60
18	0.71
21	0.81



- a. Plot the curve relating absorbance and concentration of gold.
- b. Does this curve deviate from Beer's law? Several gold solutions of unknown concentration were analyzed. The absorbance was 0.31, 0.51, 0.74, respectively. What were the concentrations of the unknown solutions?

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