



Improvement and application of nonflame atomic absorption instrumentation  
by Douglas Edmund Shrader

A thesis submitted to the Graduate Faculty in partial fulfillment of the requirements for the degree  
DOCTOR OF PHILOSOPHY in Chemistry  
Montana State University  
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Abstract:

An optical system applicable to single beam instruments is presented to correct for background absorption found in atomic absorption spectroscopy. The optical system involves two Glan-Taylor air-spaced calcite polarizers.

The hollow cathode light is polarized perpendicular to the reference beam. The polarizers are used to combine and finally separate the two beams after passage through the furnace atomization device and a Beckman DU. Individual photomultipliers are used for the two beams, whose outputs are recorded individually and compared. An improved furnace design is presented. Representative signals for the two channels are presented. Calibration curves for Ag, Au, and Hg were obtained and sensitivities are given.

The design, construction, specifications, and operation of a new dual-wavelength spectrophotometer is presented. The instrument utilizes only one fixed grating and mobile exit slits with photomultiplier light sensors. Two wavelengths can be monitored simultaneously and both channels may be scanned independently. The spectrophotometer has been integrated into an atomic absorption system which includes a Woodruff furnace and Ithaco dual-channel lock-in amplifier. The two channels may be used separately (A and B) or may be ratioed (A/B). Taking the ratio of the intensity of a resonance line of interest and the intensity of a nearby nonresonant line allows background absorption corrections to be made. In the separate channel mode, two elements may be simultaneously determined in a single sample. Results are given for the determination of Ag and Pb in various sample types requiring background correction using the ratio (A/B) mode. Results are also given for the simultaneous determination of Ag and Pb in synthetic samples using the separate channel (A and B) mode. Calibration curves were obtained for the two most sensitive lines of both Ag and Pb.

Applications of furnace atomic absorption are presented. Trace element concentrations of different elements were determined in various types of samples and the results are given and discussed. The average relative standard deviations of the results ranged from 3.1% to 15.4% for amounts of metals in the nanogram and sub-nanogram region. Sensitivities for Ag, Pb, Au, Cd, Cu, Hg, and Mn are presented.

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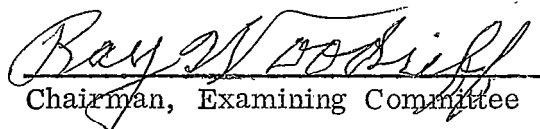
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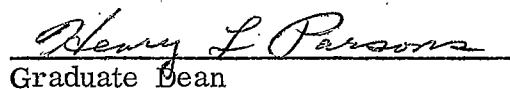
in

Chemistry

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## ABSTRACT

An optical system applicable to single beam instruments is presented to correct for background absorption found in atomic absorption spectroscopy. The optical system involves two Glan-Taylor air-spaced calcite polarizers. The hollow cathode light is polarized perpendicular to the reference beam. The polarizers are used to combine and finally separate the two beams after passage through the furnace atomization device and a Beckman DU. Individual photomultipliers are used for the two beams, whose outputs are recorded individually and compared. An improved furnace design is presented. Representative signals for the two channels are presented. Calibration curves for Ag, Au, and Hg were obtained and sensitivities are given.

The design, construction, specifications, and operation of a new dual-wavelength spectrophotometer is presented. The instrument utilizes only one fixed grating and mobile exit slits with photomultiplier light sensors. Two wavelengths can be monitored simultaneously and both channels may be scanned independently. The spectrophotometer has been integrated into an atomic absorption system which includes a Woodriff furnace and Ithaco dual-channel lock-in amplifier. The two channels may be used separately (A and B) or may be ratioed (A/B). Taking the ratio of the intensity of a resonance line of interest and the intensity of a nearby nonresonant line allows background absorption corrections to be made. In the separate channel mode, two elements may be simultaneously determined in a single sample. Results are given for the determination of Ag and Pb in various sample types requiring background correction using the ratio (A/B) mode. Results are also given for the simultaneous determination of Ag and Pb in synthetic samples using the separate channel (A and B) mode. Calibration curves were obtained for the two most sensitive lines of both Ag and Pb.

Applications of furnace atomic absorption are presented. Trace element concentrations of different elements were determined in various types of samples and the results are given and discussed. The average relative standard deviations of the results ranged from 3.1% to 15.4% for amounts of metals in the nanogram and sub-nanogram region. Sensitivities for Ag, Pb, Au, Cd, Cu, Hg, and Mn are presented.

## INTRODUCTION

Since its introduction in 1955<sup>(1)</sup>, atomic absorption spectroscopy has become a very useful analytical technique and is a part of almost every modern analytical lab. Atomic absorption theory, its application to numerous fields, and the problems involved in its use have been the subject of many publications and texts in the past.

Inherent in flame atomic absorption is a high noise level caused by turbulence in the flame and nebulized sample introduction. This imposes a limit on sensitivity and detection limit, thus relatively large samples are needed. Also, the samples need to be in relatively pure liquid form for aspiration into the burner head. These facts put the analyst at a disadvantage in many fields such as clinical, forensic, and environmental chemistry where the available sample is or should be very small, or in a solid or a complex, viscous form.

The recent concern over the environment and its quality has made necessary the development of new instrumentation in order to improve sensitivities and detection limits. Several nonflame atomization devices for atomic absorption have been introduced during the last few years<sup>(2, 3, 4, 5, 6, 7, 8, 9)</sup>. These devices have the advantage of much greater sensitivity, allowing a smaller sample to be analyzed. Also, in some cases, solids, complex liquids,

and gases may be analyzed directly or with a minimum of sample preparation. This is extremely valuable if the sample size is or should be small, or in some form other than a relatively pure liquid. Even though many of the problems involved in the use of flame atomic absorption have been eliminated by the nonflame devices, more work needs to be done to perfect them.

Another problem found in atomic absorption spectroscopy is that of background absorption<sup>(10, 11)</sup>. The cause of this background absorption is molecular absorption and/or scattering of light due to particles. This non-atomic absorption can cause erroneously high results if not compensated for. Several methods of correction for background absorption have been described<sup>(12, 13, 14, 15, 16)</sup>. These all involve the use of a hydrogen or deuterium continuum in various instrumental arrangements to measure the background absorption at the wavelength of interest and allow for correction. Also, the use of a nonabsorbing wavelength near the resonance line of interest for the purpose of background correction or reference has been reported in several publications<sup>(10, 17, 18, 19, 20)</sup>.

An additional problem, or rather disadvantage, of atomic absorption spectroscopy is that in practice it is generally only useful for single-element determinations. Several instrumental arrangements for the determination of more than one element have been published<sup>(21, 22, 23)</sup>. Very recently Fisher

Scientific Co. and Instrumentation Laboratory, Inc. have introduced spectrophotometers which can be used to determine two elements simultaneously by atomic absorption. These instruments can monitor two wavelengths. Perkin-Elmer, American Instrument Co., and Phoenix Precision Instrument Co. also have instruments with dual-wavelength capabilities but have not applied these to atomic absorption but rather to UV-Visible spectroscopy. In all but one case the instruments are composed of two grating monochromators. The Instrumentation Laboratory instrument utilizes one grating monochromator and an interference filter for the second channel.

This thesis deals with the improvement of instrumentation available for use in atomic absorption spectroscopy. Complete instrumental systems have been developed as well as improvements in the atomization device, the Woodriff furnace. The instrumental systems have been developed in order to provide a method of background correction to compensate for nonatomic absorption. The second instrumental system may also be used for the simultaneous determination of two elements. Applications of furnace atomic absorption have been developed and are part of this thesis. They show that the Woodriff furnace can be utilized to determine trace element concentrations in real samples, taken in connection with problems or projects of current interest, and that reproducible results in the nanogram and sub-nanogram region can be obtained.

## STATEMENT OF PROBLEM

Briefly, the problem was to look into the improvement of instrumentation (complete systems as well as the furnace atomization device) available for use in atomic absorption spectroscopy. This improvement involves applications and evaluation of results with real samples.

As was stated previously, there are several problems which one encounters in atomic absorption spectroscopy. A major breakthrough in solving some of the problems associated with flame atomic absorption came about with the introduction of various nonflame atomization devices. The Woodriff furnace is such a device. Since its introduction to the public in 1966<sup>(24)</sup>, various publications have presented sensitivities, detection limits, and some of the problems encountered in its use. Heater tubes had a very short life (15 hours)<sup>(4)</sup>. Blanks were many times irreproducible<sup>(25)</sup>. A large portion of this was thought to be caused by furnace design and materials. The construction of a second generation furnace with enclosed ends and improved chucks for end cooling and electrical conduction seemed to increase the life of the heater tubes<sup>(14)</sup>. On this basis it was decided that the furnace design needed additional improvement, not only with the goal of increased life of heater tubes and other graphite parts, but also with the goals of increasing reproducibility of results and the development of a design which would promote

safety, simplicity, and efficiency. A third generation furnace and an improvement of it were constructed and are discussed.

The problem of background absorption caused by the scattering of light by particles in the optical path and/or molecular absorption needed to be confronted. The instrumental arrangements previously used to correct for background absorption are in some cases very complicated and either give only the net absorbance or individual absorbances at different times or on different samples. It was thought that a system of background correction involving plane polarized light could be useful. By using two polarizing beam splitters, reference and sample radiation, polarized perpendicularly to each other, could be combined into a single beam and, after passage through the furnace, could be separated and monitored. The use of DC electronics would allow a continuous and simultaneous record of both hollow cathode and hydrogen lamp radiation to be obtained.

Along the same lines but more versatile was an idea for a dual-wavelength monochromator. It would have one fixed grating and two mobile exit slit and photomultiplier tube assemblies. Being able to monitor two wavelengths would allow one to use a nonresonant line, close to the resonant line of interest, as a reference and thus be able to compensate for background absorption. A simultaneous record of both reference and sample beams could

be obtained or the outputs from the two channels could be ratioed, giving the net absorbance. Also, it would have the capability of simultaneously determining two elements by atomic absorption. The versatility would be greater and the construction would be simpler than commercially available dual-wavelength spectrophotometers.

Both instrumental arrangements were developed and incorporated into atomic absorption systems involving a Woodriff furnace. The instruments are presented and discussed.

Finally there arises the problem of applications. Techniques for determining very small amounts of metals are in great demand due to the current emphasis on the environment and its quality. A technique such as furnace atomic absorption can fulfill the requirement of being very sensitive. It is in fact 3 or 4 orders of magnitude more sensitive than conventional flame atomic absorption. However, with this technique, as well as any other technique, real samples need to be analyzed in order to determine its applicability to routine analysis. Applications of nonflame devices available from different instrument companies have been the subject of various publications<sup>(26,27,28,29,30)</sup>. The Montana State University group has published only two applications to real samples<sup>(31,32)</sup>. The opportunity to become involved in the analysis of samples from various

research projects presented itself and was accepted. The methods used and the results obtained in these projects are presented.

## EXPERIMENTAL

### Furnace Atomic Absorption with Reference Channel

Systems mentioned earlier to compensate for background absorption employ alternating sample-reference observation and give only the difference between the two signals or measure the sample signal and reference signal at two times relatively far apart or on two samples. The system described here gives a continuous, simultaneous record of both signals.

#### Optical System and Readout

Figure 1 shows the optical system which was used. The hollow cathode light enters the primary Glan-Taylor polarizer and is polarized parallel to the optical axis of the polarizer. The polarized hollow cathode light then passes down the optical path of the furnace. The hydrogen lamp light enters the primary polarizer through the side window and is divided into two perpendicularly polarized beams which are reflected in such a manner that the portion which is polarized perpendicular to the optical axis of the polarizer follows the same optical path as the beam from the hollow cathode. The other portion of the hydrogen lamp beam leaves the optical path and is absorbed. Before passage through the furnace, the two beams of interest are collimated by means of a quartz lens placed between the polarizer and the furnace.

Both beams, the hollow cathode beam polarized horizontally and the

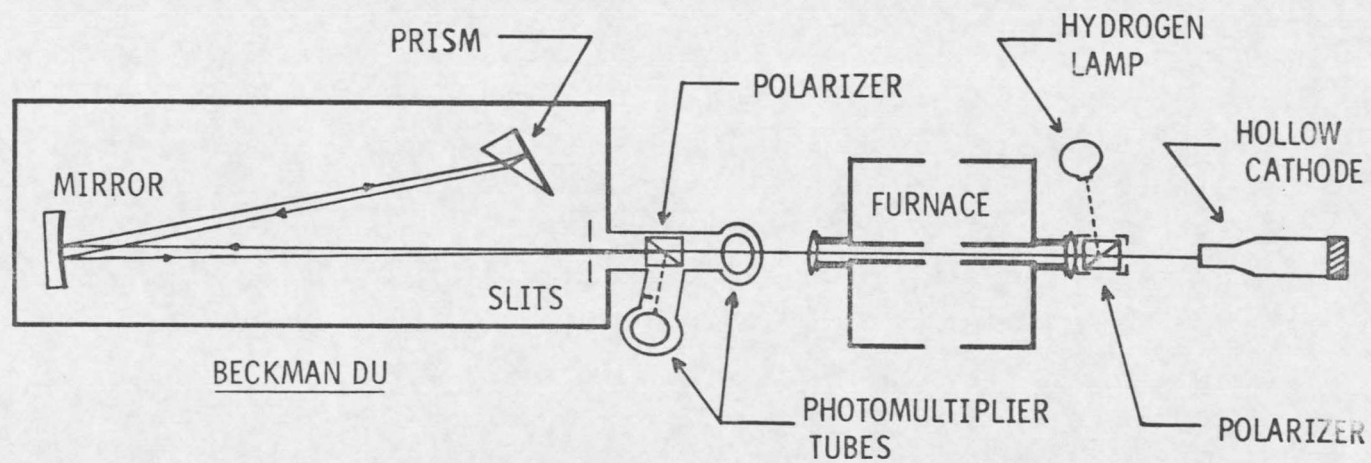


Figure 1. Optical System Diagram.

hydrogen lamp beam polarized vertically, after passing through the furnace are focused on the slit of the Beckman DU with a second lens placed on the end of the furnace. After passing through the monochromator, the combined beams pass through the second slit and fall on the secondary Glan-Taylor polarizer. The polarizer separates the two perpendicularly polarized portions of the beam. The hollow cathode portion is transmitted straight through the polarizer and falls on a photomultiplier tube. The hydrogen lamp portion is reflected through the side window of the polarizer and falls on a second photomultiplier tube.

Enlarged diagrams of both the primary and secondary polarizers are shown in Figure 2. The polarization of the hollow cathode and hydrogen lamp beams is indicated. A horizontally polarized portion is reflected through the side window of the secondary polarizer at a slightly different angle than the vertically polarized hydrogen lamp beam of interest. This portion is kept from striking the reference photomultiplier tube by placing a baffle between the polarizer and photomultiplier tube (see Figure 1). A drawing of the optical bench and accessories which were constructed is shown in the Appendix, page 90.

After the hollow cathode light and hydrogen lamp light fall on their respective photomultiplier tubes (RCA IP28's), the signals are recorded

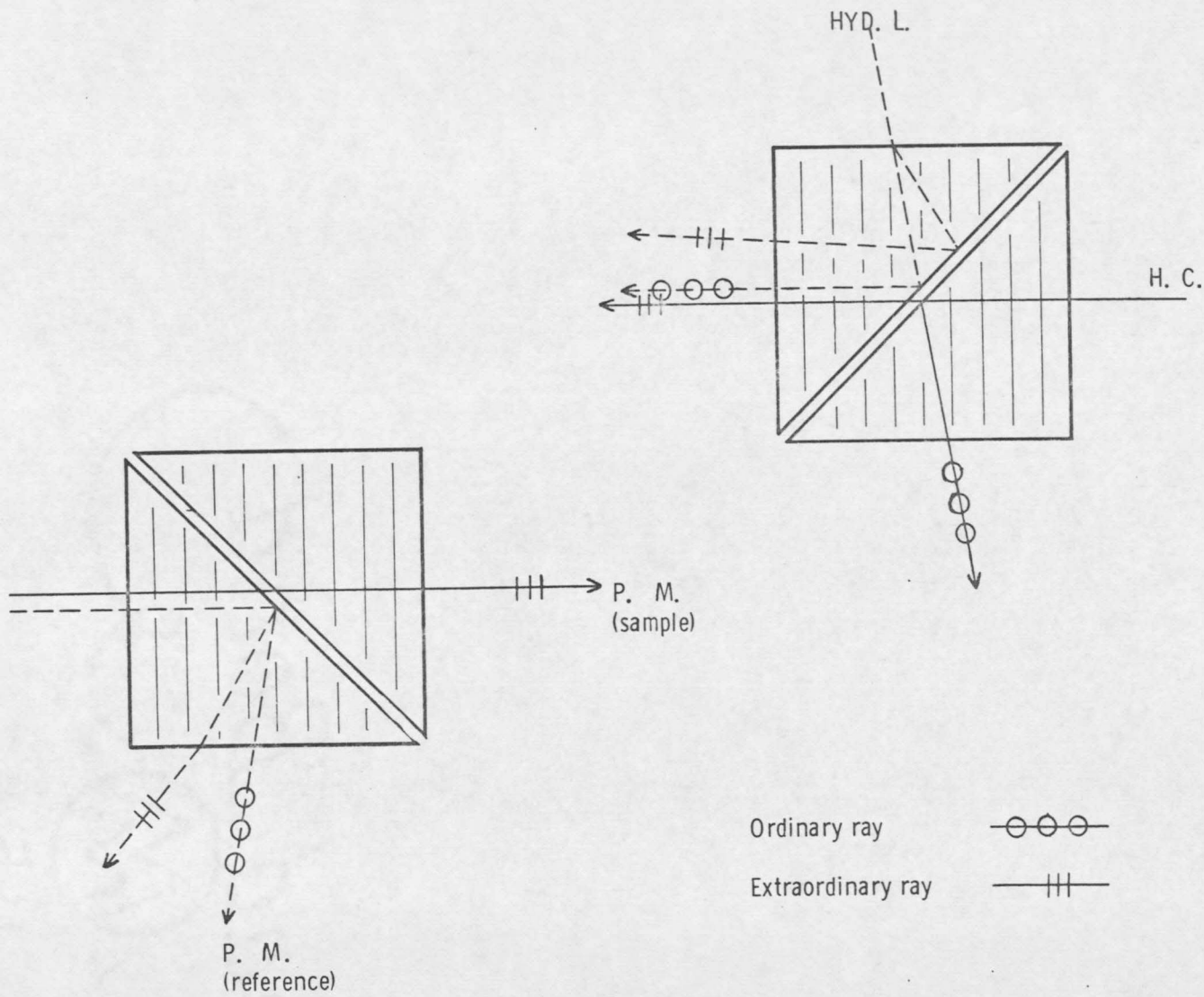


Figure 2. Glan-Taylor Polarizers.

individually. Two Heath Servo-recorders, Model EUW-20, were used to record the results. The two simultaneous records, sample and reference, are then available for comparison.

#### The Furnace

Previous furnace designs have been published<sup>(4, 14)</sup>. The furnace used in this study was a third generation furnace. A schematic drawing of the improved furnace design is shown in Figure 3. The heater tubes are 15.2 cm long, 10 mm o.d., and 8 mm i.d., and make contact in the center with the one-piece combination heat sink and shield tube. The outer ends are connected to a spiral copper tube which fulfills the dual purpose of electrode contact and cooling<sup>(14)</sup>.

The shield tube prevents the graphite felt insulation from coming into contact with the heater tubes and also helps reduce heat loss from the heater tubes to the rest of the furnace. The one-piece heat sink and shield tube makes the optical path more stable and gives a better heat capacity for volatilization of the sample. This change in shield tube design from the previous three-piece construction also gives more uniform temperature by allowing more efficient heat conduction to the central part of the furnace.

The side tube, through which samples are introduced, is 6 mm i.d., is very thin-walled next to the heat sink to reduce heat conduction away from

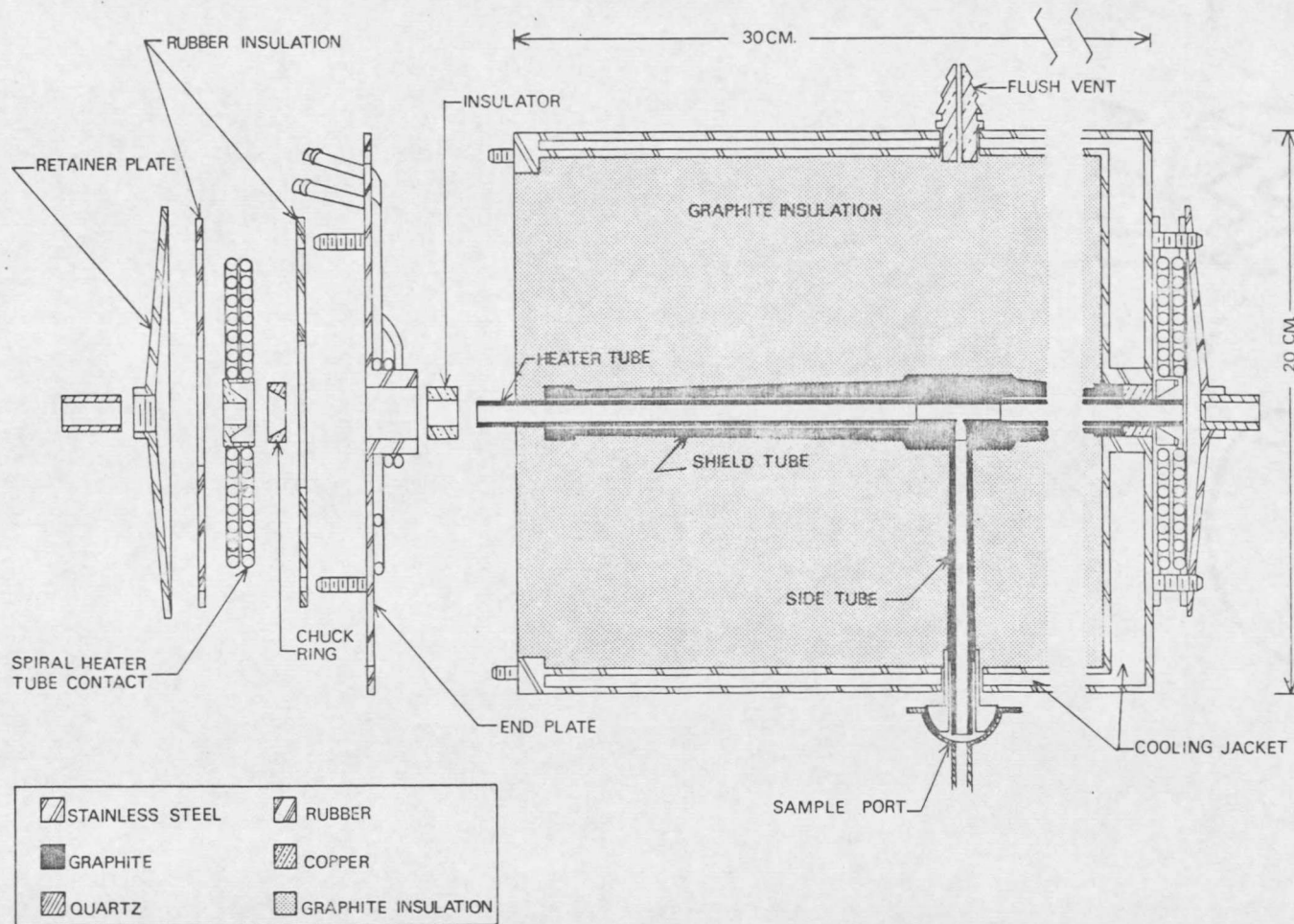


Figure 3. Drawing of the Furnace (third generation).

the interior, and has a thick lip approximately 4 cm from the outer end to hold a spring which provides constant tension on the side tube as it expands or contracts with changing temperature. Argon gas enters the sample port and side tube through small aligned holes in both. A Vycor 18/9 socket is attached to the sample port through which samples are introduced.

In addition to other improvements, this furnace is double-walled to provide effective cooling (with water) of the entire furnace. The furnace is also made of stainless steel rather than iron. This design has proved very satisfactory. Heater tubes needed to be replaced every month or two with the second generation furnace. Sets of heater tubes in this furnace have been used for periods of up to ten months without replacement.

#### Sample Preparation

Standard solutions were prepared from salts of the metal to be investigated. The solutions were made with doubly-distilled water to  $10^{-7}$  g of metal/ml and diluted to  $10^{-8}$  g of metal/ml when necessary. The solutions were kept acidic (pH ca. 2) to reduce the amount of adsorption of metal on the walls of the container<sup>(33, 34, 35)</sup>. This was true of all standard or sample solutions used at any time. In order to prepare calibration curves, 10- to 100- $\mu$ l portions of the appropriate solutions were placed into cups made of high-density graphite and dried under a heat lamp.

Samples are placed into the cups and, after drying or ashing as needed, are inserted directly into the furnace. The cups (6 x 16 mm), either for cleaning or sample introduction, are screwed onto a threaded 1/8 in. carbon rod and inserted through the Vycor socket and side tube into the furnace so that they rest against the heat sink. Any sample present in the cup vaporizes quickly, enters the optical path, and a reading is recorded.

## RESULTS AND DISCUSSION

### Furnace Atomic Absorption with Reference Channel

Some problems were encountered in the use of polarized light in the optical system. These were, to an extent, based on the properties of polarized light. The Glan-Taylor polarizers used are of the birefringence or double refraction type. A double refraction polarizer divides an incident beam into two perpendicularly polarized components and reflects either one or both of them towards the side of the polarizer<sup>(36)</sup>. This was no problem with the hollow cathode light but caused alignment problems with the hydrogen continuum.

Another problem encountered was the mixing of the two perpendicularly polarized light beams. It was not possible to completely isolate the two beams. This was probably due to the following facts<sup>(36)</sup>:

- (a) Most polarizers have some depolarizing tendencies.
- (b) There is partial linear polarization produced when light is passed through a slit. The electric vector tends to align itself with the slit. This would affect light polarized perpendicular to the slit.
- (c) Prisms, mirrors, and gratings have some partial polarization tendencies.

Thus, partial polarization (or depolarization as the case may be) occurs in the prism instrument itself, which has previously been reported<sup>(37,38)</sup>, and the

secondary polarizer may cause some mixing. The partial polarization produced in prism instruments has been reported to vary extensively and cyclically with wavelength<sup>(39)</sup>. It was found that the least amount of mixing occurred when the secondary polarizer was rotated from 20-45° out of plane with the primary polarizer, depending upon the elemental wavelength being used. The small amount of mixing remaining after alignment to peak both signals (approximately 5%) did not seriously affect the results obtained.

A slight limitation is imposed by the use of calcite polarizers in the optical system. As shorter wavelengths are approached, the polarizers tend to absorb increasing fractions of the incident light. Figure 4 shows the absorption curve of one of the polarizers, obtained with the Cary 14 UV-Visible instrument. The signal throughput decreases making necessary increased voltages to the photomultiplier light sensors and/or increased current to the hollow cathode. However, only analyses involving resonance lines such as Pb (217.0 nm), Se (196.0 nm), and As (193.7 nm) would be seriously affected by this property.

The optical system described worked very well in eliminating errors due to background absorption. If the sample exhibits broad-band absorption because of anions or carbonization of organic material, equal light fractions are absorbed from both the hollow cathode beam and the reference beam which

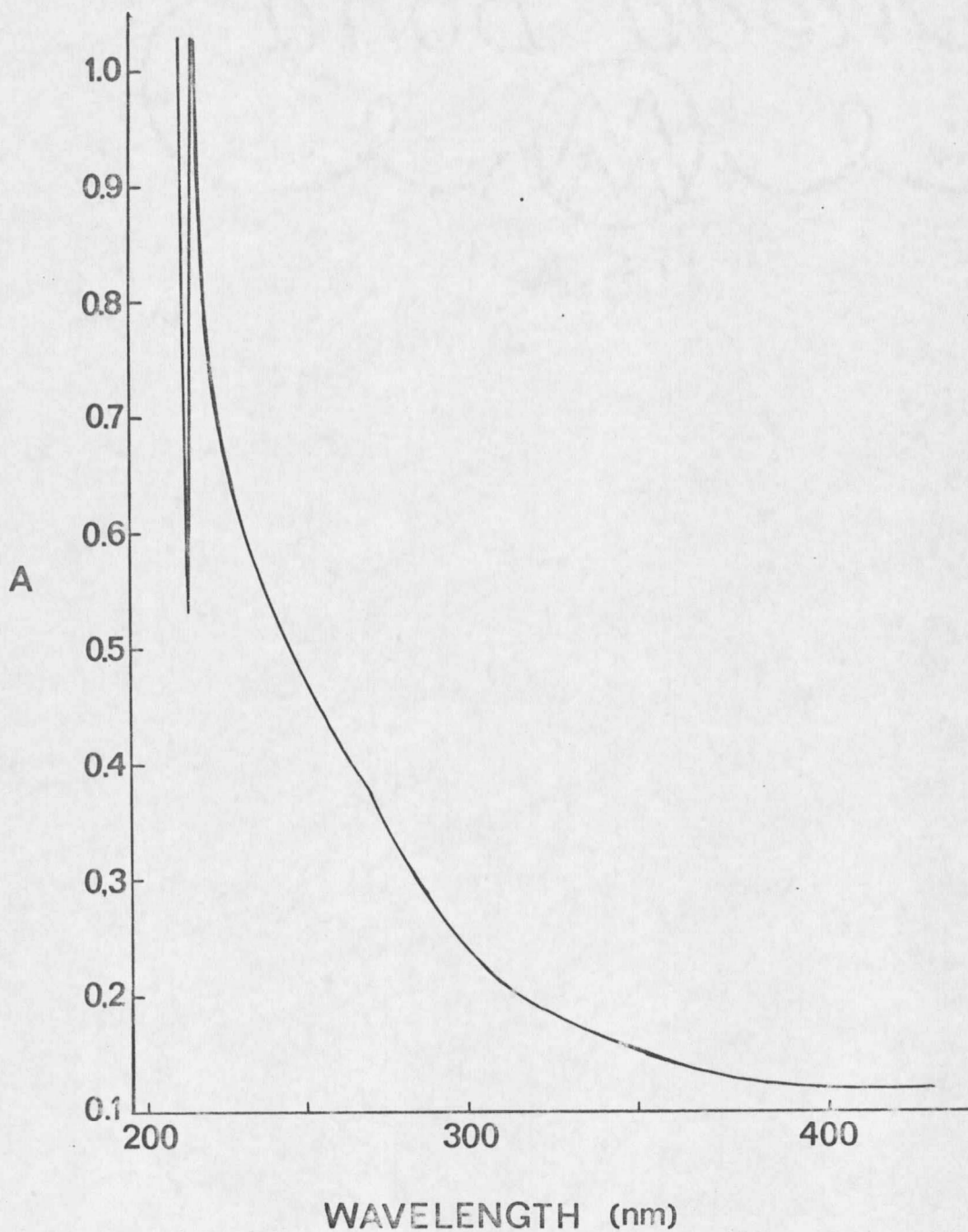


Figure 4. Absorption Spectra of the Glan-Taylor Polarizer.

can then be taken into account. Individual recorders permitted complete quantification of each beam, sample and reference. Different types of samples were run qualitatively to determine the background absorption which may occur.

Many organic samples such as drugs and tissues pyrolyze and give broad-band absorption before the trace elements present are volatilized. An example of the two signals recorded in such a case is illustrated in A of Figure 5. The opposite behavior is shown in B. This type of curve is obtained with Hg in organic matrices under proper conditions. The Hg volatilizes and diffuses into the light path more rapidly than the pyrolyzed organic material. The most common behavior is one where the trace element and the broad-band absorbing material are simultaneously present in the light path as shown in C. If the peak separation in A and B is sufficiently large, and the particular peak due to the element being analyzed is known, determinations may be made without background correction. Even in these cases, the broad-band absorption is ordinarily wide enough to cause some error.

The reproducibility of the blank, especially with solid samples, has been a problem with using the graphite tube furnace technique<sup>(4, 25)</sup>. This problem was encountered initially with Ag. It was found that the interior of the furnace (insulation and side tube) was heavily contaminated. The same was true with the sample cup holders and desiccators. The contamination

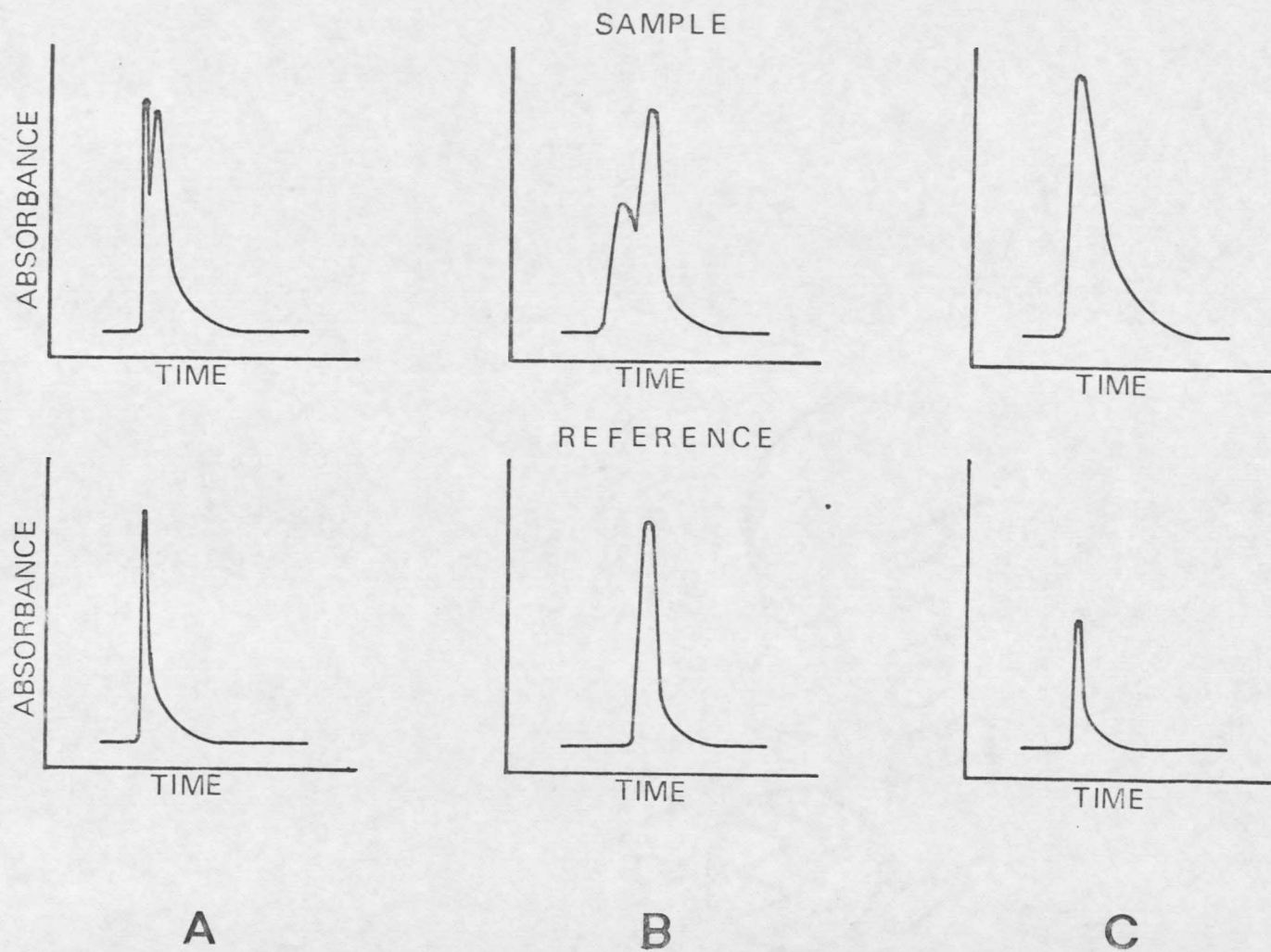


Figure 5. Representative Signals.

problem was reduced in several steps. First graphite felt rather than graphite flake was used for insulation. The felt seemed to be much cleaner. Then the entire furnace was cleaned by prolonged heating, while flushing with large volumes of argon. Second, improved sample preparation was instituted. All cup holders, desiccators, and the Vycor socket are now cleaned regularly with a solution of sodium thiosulfate and/or a mixture of concentrated  $\text{HNO}_3$  and  $\text{H}_2\text{SO}_4$ , and rinsed with doubly-distilled water. The third step was standardizing the sample cups. Very reproducible blanks were finally obtained. Fifteen blanks run on different days were obtained for Ag whose standard deviation equaled an absorption of 0.0022. Defining the detection limit as the amount of element required to give a signal twice the standard deviation of the blank,  $A = .0044$ , it can be seen that the calculated detection limit for this procedure is approximately equal to the measured sensitivity.

Calibration curves were obtained for Ag, Au, and Hg. These curves are shown in Figures 6 and 7. It was found that the reproducibility depended to a great extent upon the size of the sample and the cups used for the samples. The precision for samples of  $10^{-10}$  grams might be greatly improved by better sampling technique, the use of a set of standardized cups, and improved electronics and optics. The precision for larger samples (ca.  $5 \times 10^{-9}$  grams) was approximately 1-2%.

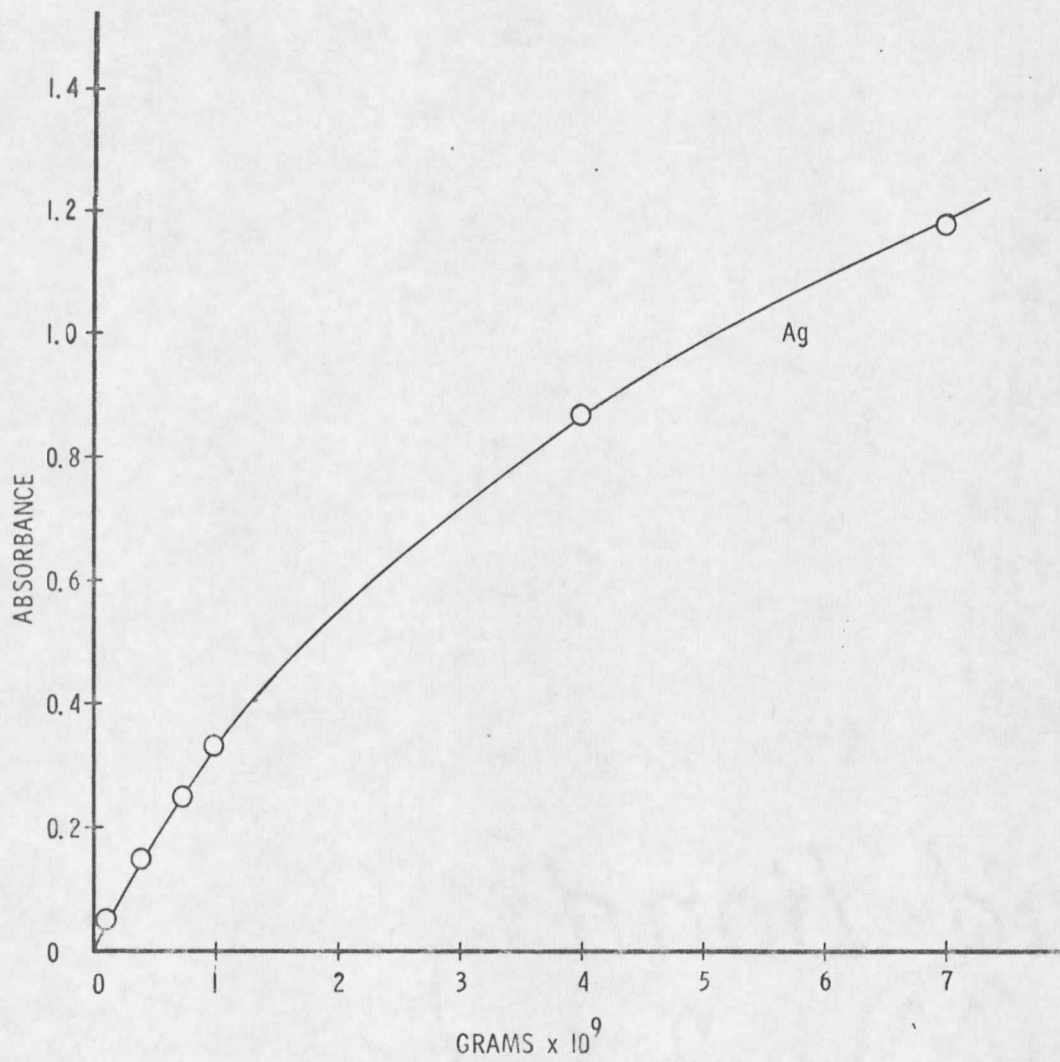


Figure 6. Calibration Curve for Ag.

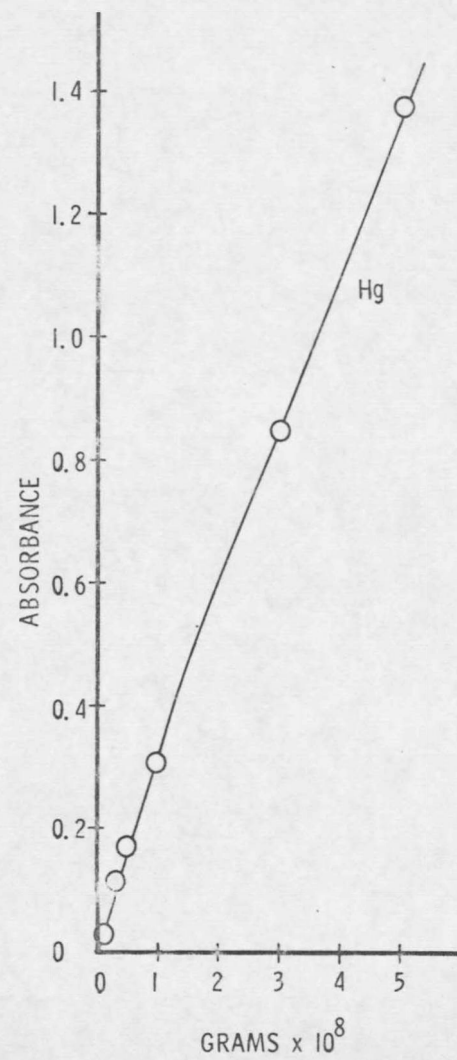
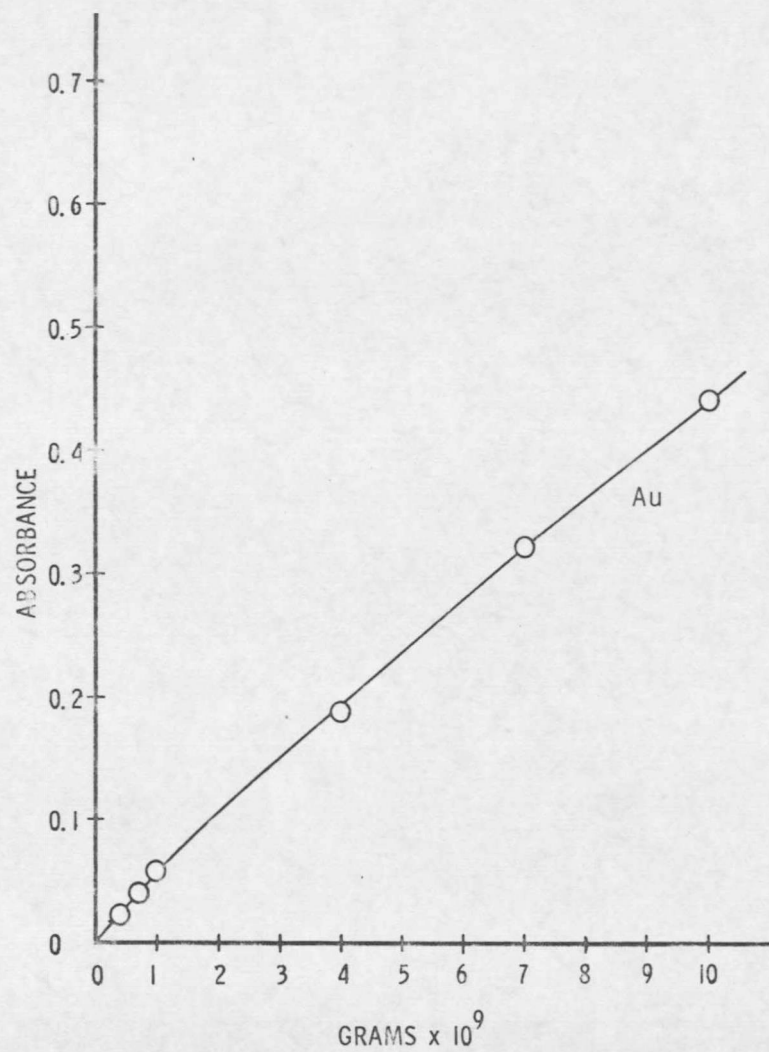


Figure 7. Calibration Curves for Au and Hg.

Table I shows pertinent data for each element including the measured sensitivity. The sensitivity corresponds to the amount of metal which would give a 1% absorbance reading. As was stated previously, the detection limit of this method is approximately equal to the sensitivity.

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Table I: Sensitivity Data for Ag, Au, and Hg.

Element	Wavelength (nm)	Furnace temperature (°C)	Sensitivity
Ag	328.1	1800	$8 \times 10^{-12}$ grams
Au	242.8	2150	$7 \times 10^{-11}$ grams
Hg	253.7	1050	$1 \times 10^{-10}$ grams

---

This continuous, direct current system is applicable to any single-beam instrument. It can be applied to either flame or furnace atomic absorption. The greatly increased sensitivity of furnace atomic absorption over flames permits the analysis of very small samples, and with a minimum of sample preparation, provided any broad-band absorption is corrected for. The equipment involved in the system is comparable to flame atomic absorption with regard to complexity and cost of operation. It is comparable to neutron activation analysis with regard to sensitivity and its precision is much greater

## EXPERIMENTAL

### A New Dual-Wavelength Spectrophotometer

This dual-wavelength monochromator has the capability of correcting for background absorption and also the simultaneous determination of two elements by atomic absorption spectroscopy. In comparison to other dual-wavelength instruments, the monochromator utilizes only one grating. This spectrophotometer has a fixed grating and mobile exit slits with photomultiplier light sensors. The design utilizes the property of concave diffraction gratings whereby light reflected from the grating comes to focus on the Rowland circle. The design and operation of the monochromator as well as the components of the total atomic absorption system and its applications are discussed.

#### Instrument Design

As stated previously, this new dual-wavelength spectrophotometer is a concave diffraction grating instrument capable of sensing two different wavelengths reflected from one grating, simultaneously and independently. The grating is fixed and there are two mobile exit slits with photomultiplier housings encasing two Hamamatsu R106 photomultiplier tubes.

Figure 8 diagrammatically shows the monochromator system. The entrance slit and grating are positioned directly opposite each other on the

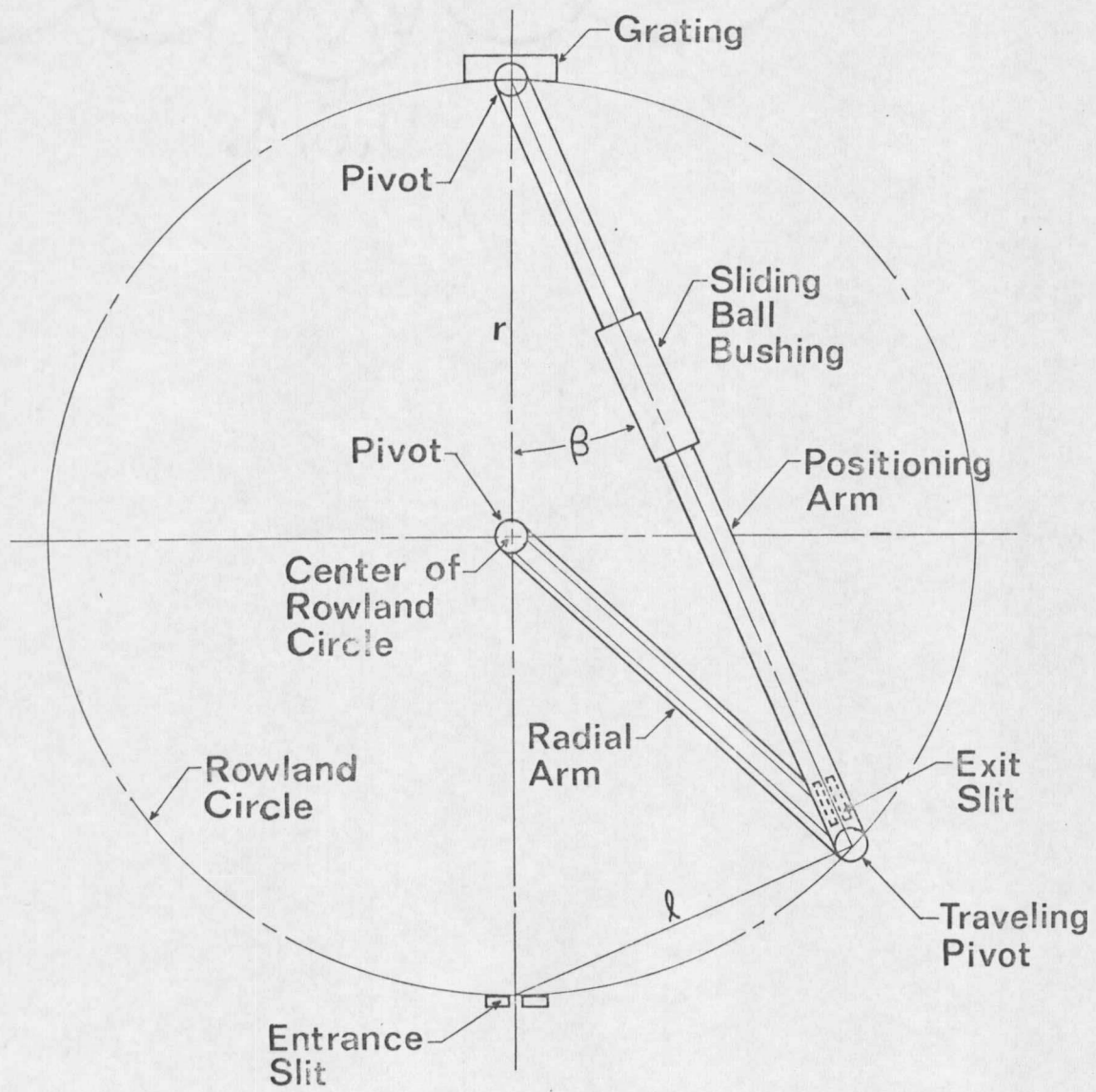


Figure 8. Monochromator System.

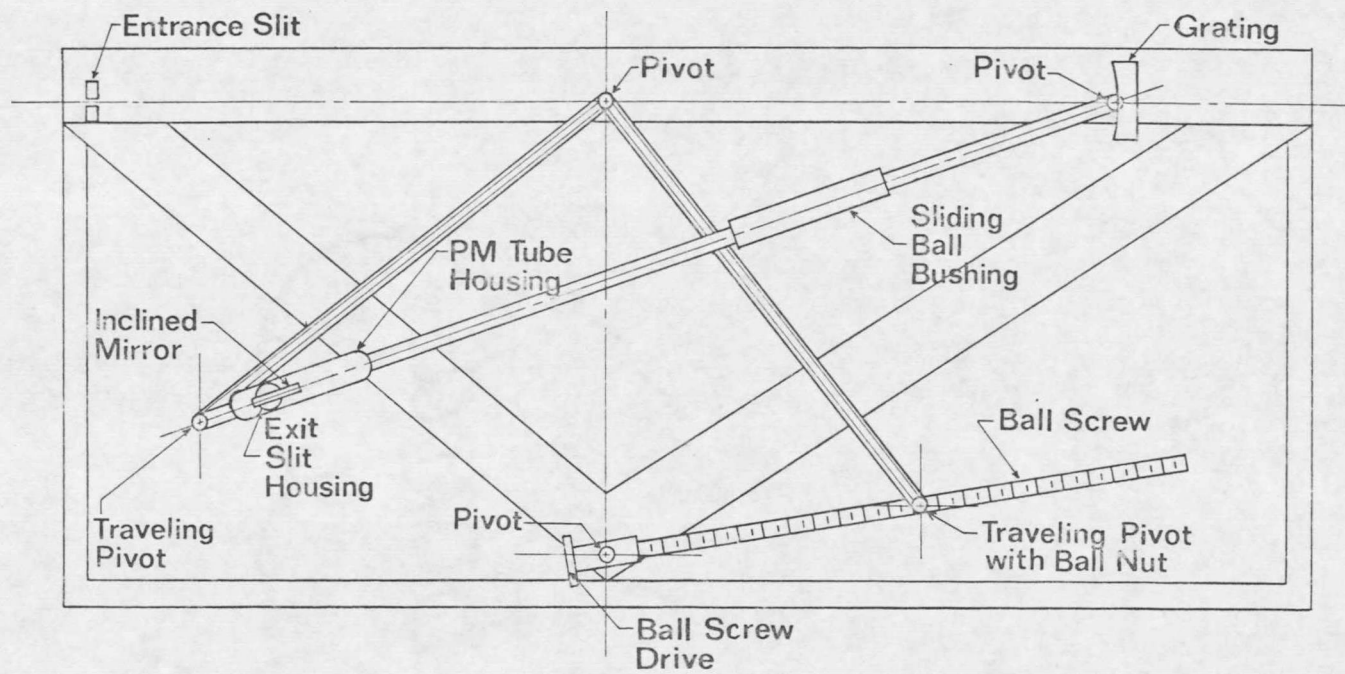
Rowland circle. The detected wavelength ( $\lambda$ ) reflected from the concave grating is directly proportional to the sine of the angle  $\beta$ .

$$\underline{+n\lambda} = d(\sin \alpha + \sin \beta)$$

The angle of incidence  $\alpha = 0$ .  $\beta$  includes the line from the grating to the exit slit, positioned near the traveling pivot, and the line from the grating through the center of the Rowland circle. The diameter ( $r$ ) is constant and is the hypotenuse of the right triangle formed which includes the angle  $\beta$ . Thus, an incremental change in the distance  $l$  will produce a corresponding change in wavelength.

The radial arm has one end fixed at the center of the Rowland circle but free to pivot. Its length is equal to the radius of the Rowland circle. Thus, the free end of the radial arm is always on the focus point of the grating. The positioning arm is collapsible and is attached at the end of the radial arm and beneath the center of the grating, and is free to pivot. An exit slit and photomultiplier tube and housing placed on the positioning arm, parallel to it, are always facing the grating and the slit is maintained at the focus point (see Figures 9 and 10).

Figure 9 shows a top view of the monochromator and the mechanical parts of one of the two channels. The grating, slits, photomultiplier tube housings, and mechanical linkages are shown. The ball screw drive assembly



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Figure 9. Monochromator (top view).

is driven by a small DC motor. The ball screw was machined so that 1 turn corresponds to a wavelength change of  $10 \text{ \AA}$ . This allows a mechanical counter connected to the ball screw with an automobile speedometer cable to be used to denote the wavelength.

Figure 10 shows a side view of the monochromator and the physical placement of both channels. To allow the two radial arms with their sensing devices and associated mechanical parts to pass one another, one above the other, front-silvered mirrors are positioned as shown. The exit slits are positioned at the distances  $c$  and  $d$  such that they are always at the focus point of the grating. In addition, this leaves the Rowland circle clear for the placement of photographic film or photomultiplier tubes for spectrographic or direct reader applications. How close two wavelengths may be and still be monitored successfully depends upon the width of the inclined mirrors used. In the present model, the two wavelengths can be within 5 nm of each other before any significant amount of signal is lost in the second channel.

Figure 11 shows the electrical circuit used to power the scanning mechanisms of each channel. The monochromator is powered by AC rectified to DC. A switch allows either channel to be scanned. The scan speed can be varied with a rheostat. Additional switches on each channel allow forward and reverse scan of each channel independently. Limit switches are included at

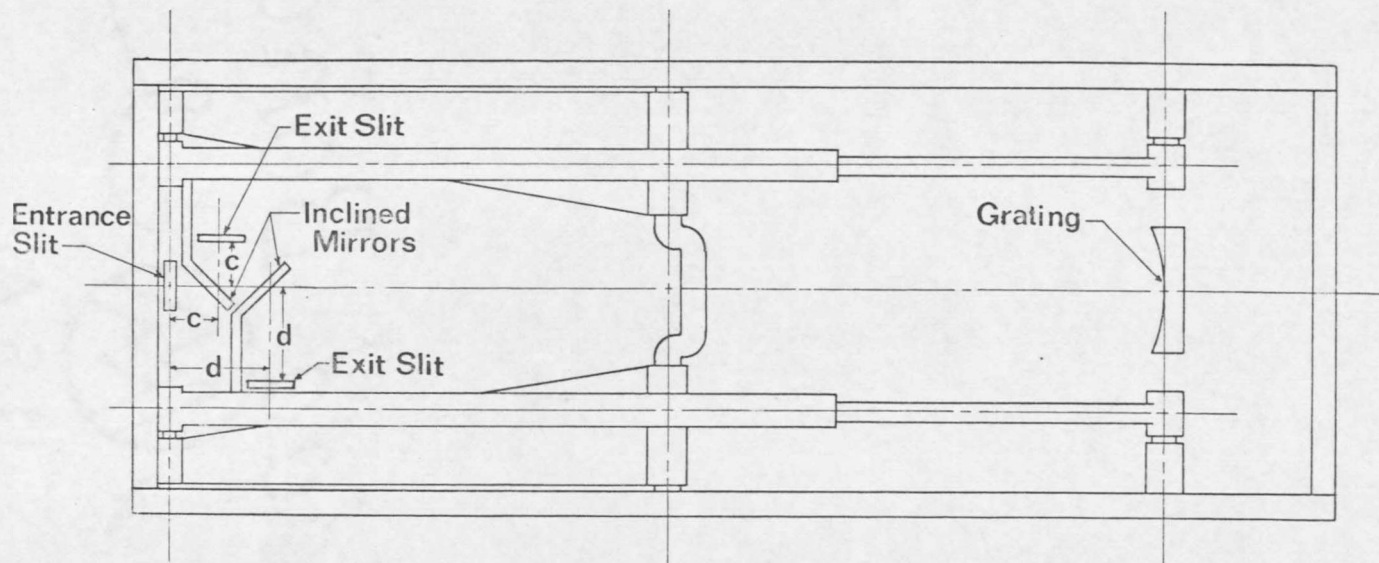


Figure 10. Monochromator (side view).

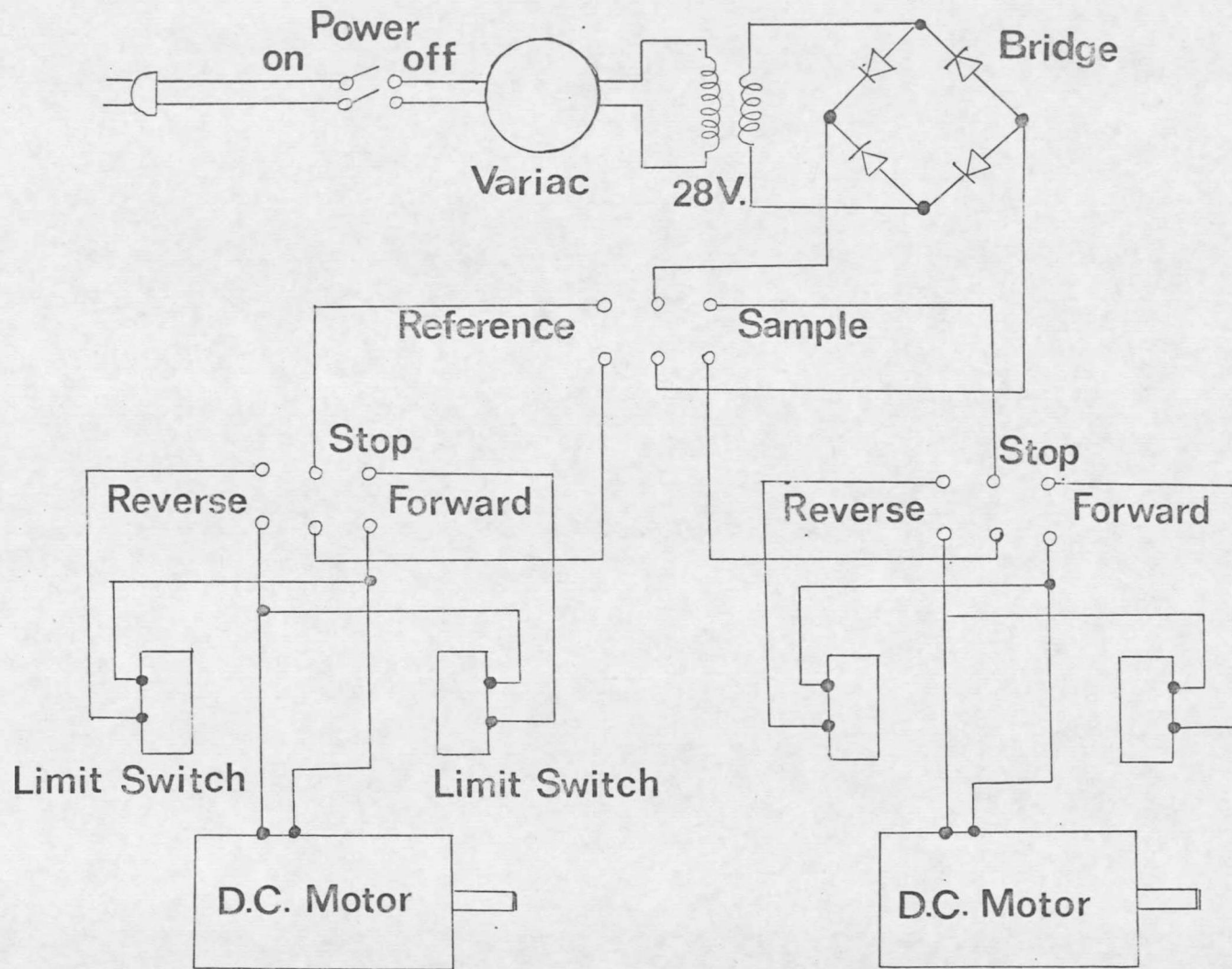


Figure 11. Monochromator Electrical Circuit.

the ends of the scan range to automatically stop each channel. Although the two channels are labeled sample and reference in Figure 11, they will be designated as A and B, respectively, for future reference.

Table II gives the monochromator specifications as calculated or measured. Some of the specifications may not be optimum since the monochromator is a prototype model and was built utilizing a concave grating available in the laboratory and easily obtainable materials. Size, weight, aperture, and dispersion could be improved and made more practical for general usage by using a different grating, lighter materials, and shorter, finer ball screws.

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Table II: Monochromator Specifications

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Grating: concave, 50 x 85 mm, 600 lines/mm

Focal length: 0.5 m

Aperture: f/7

Reciprocal linear dispersion: 1.7 nm/mm

Wavelength range: 185-1100 nm

Scan speed: continuously variable at 3-110 nm/min

Outside dimensions: 1.2 x 0.6 x 0.4 m

Weight: 64 kg

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#### Total System

The dual-wavelength monochromator was integrated into an atomic absorption system. A block diagram of the components is shown in Figure 12.

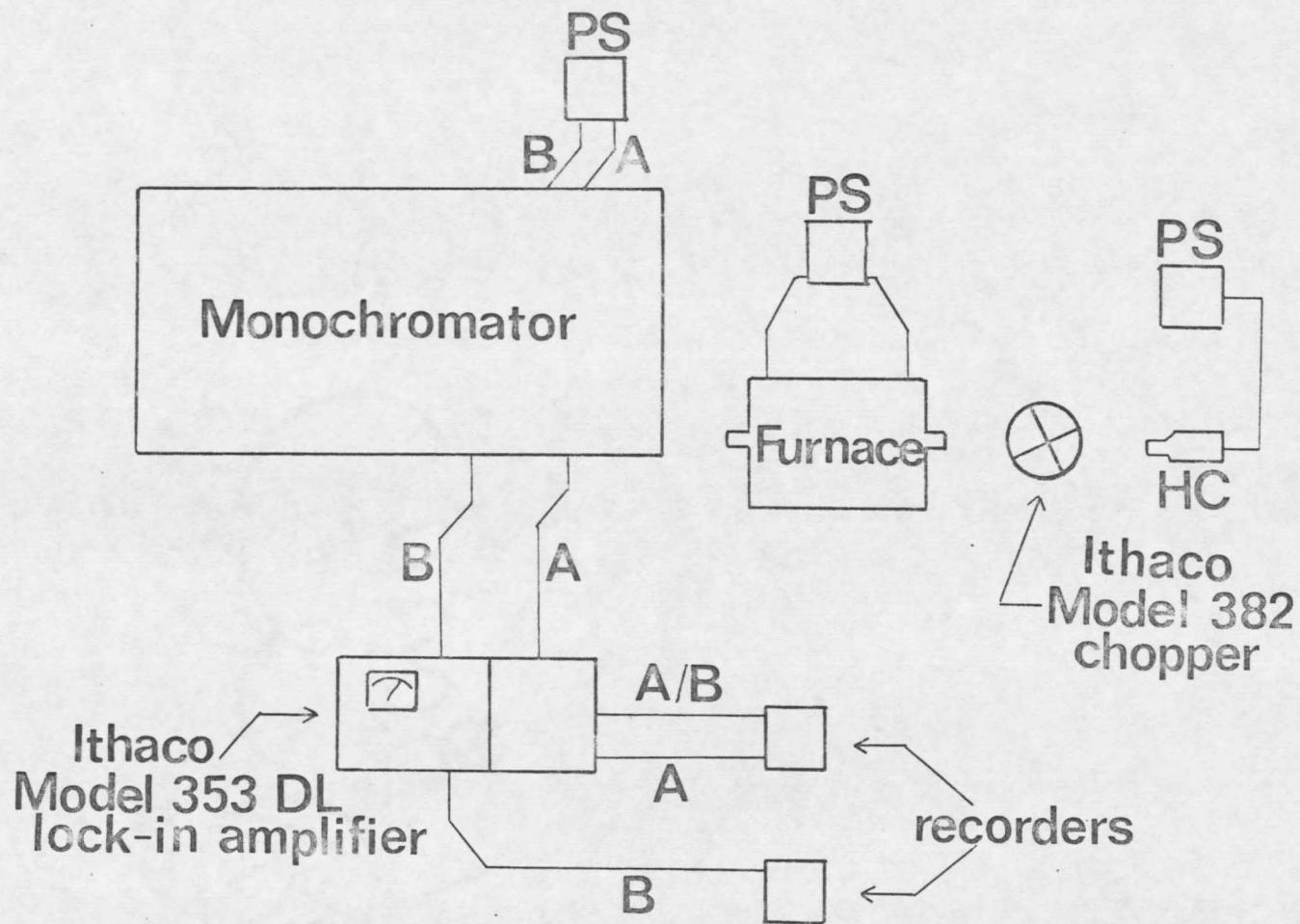


Figure 12. Block Diagram of Components.

A drawing of the optical bench and accessories constructed is shown in the Appendix, page 91. Being primarily concerned with trace element analysis in the lab, and since a nonflame atomization device has been developed over a period of years<sup>(4, 14, 25, 31, 32, 40)</sup>, a Woodriff furnace was included in the system. The furnace is basically like the third generation furnace previously described but with a few improvements. Figure 13 shows a schematic drawing of the furnace.

The cooling jacket design was simplified to make construction easier and improve the cooling characteristics. Rather than having the side tube held against the shield tube by spring tension, it is threaded and screwed into the shield tube, giving a better seal and simplifying the sample port construction. Both gas vents are included in the separate, threaded sample port, decreasing construction costs. The sample port is concave rather than convex, aiding cup introduction and reducing breakage of the Vycor socket. Pages 92 and 93 of the Appendix show diagrams of the water flow and gas flow systems of the furnace.

The spiral heater tube contact was simplified. Figure 14 shows the new design. The chuck ring is one piece of copper with a tapered hole in the middle rather than the previous more complicated design involving two rings and three screws<sup>(14)</sup>. The heater tubes are made with the same taper (12°)



















































































































































