Construction of a Small Vacuum Ultraviolet Spectrograph

Dale A. Eding
Western Michigan University

Follow this and additional works at: https://scholarworks.wmich.edu/masters_theses
Part of the Optics Commons

Recommended Citation
https://scholarworks.wmich.edu/masters_theses/2841

This Masters Thesis-Open Access is brought to you for free and open access by the Graduate College at ScholarWorks at WMU. It has been accepted for inclusion in Master's Theses by an authorized administrator of ScholarWorks at WMU. For more information, please contact maira.bundza@wmich.edu.
CONSTRUCTION OF A SMALL
VACUUM ULTRAVIOLET
SPECTROGRAPH

by

Dale A. Eding

A Thesis
Submitted to the
Faculty of the Graduate College
in partial fulfillment
of the
Degree of Master of Arts

Western Michigan University
Kalamazoo, Michigan
April, 1971
ACKNOWLEDGEMENTS

I would like to express my appreciation to the members of the Physics Department of Western Michigan University for the guidance and instruction they provided in my undergraduate and graduate work.

I especially thank my thesis advisor, Dr. Stanley K. Derby, for his help, encouragement, and patience. I also thank Dr. Nathan Nichols and Mr. Jacob Dewitt for their many suggestions, as well as Mr. Richard Durbin of the machine shop for his help in various aspects of the construction.

Finally, I wish to thank my wife, Gert, for her faith throughout this work, and for typing the manuscript.

Dale A. Eding
MASTER'S THESIS

EDING, Dale Allan, 1942-
CONSTRUCTION OF A SMALL VACUUM ULTRAVIOLET SPECTROGRAPH.

Western Michigan University, M.A., 1971
Physics, general

University Microfilms, A XEROX Company, Ann Arbor, Michigan
# TABLE OF CONTENTS

<table>
<thead>
<tr>
<th>CHAPTER</th>
<th>PAGE</th>
</tr>
</thead>
<tbody>
<tr>
<td>INTRODUCTION</td>
<td>1</td>
</tr>
<tr>
<td>I</td>
<td>DIFFRACTION GRATING SPECTROGRAPHS</td>
</tr>
<tr>
<td></td>
<td>The Normal Incidence Mount</td>
</tr>
<tr>
<td></td>
<td>The Grazing Incidence Mount</td>
</tr>
<tr>
<td></td>
<td>The Ebert-Fastie Mount</td>
</tr>
<tr>
<td>II</td>
<td>PROBLEMS IN DETECTING VACUUM ULTRAVIOLET SPECTRA</td>
</tr>
<tr>
<td>III</td>
<td>CONSTRUCTION OF THE VACUUM SPECTROGRAPH</td>
</tr>
<tr>
<td></td>
<td>Monochromator Alterations</td>
</tr>
<tr>
<td></td>
<td>The Vacuum Chamber</td>
</tr>
<tr>
<td></td>
<td>The Light Source</td>
</tr>
<tr>
<td></td>
<td>Creating A Vacuum</td>
</tr>
<tr>
<td>IV</td>
<td>PHOTOGRAPHING THE SPECTRA</td>
</tr>
<tr>
<td>V</td>
<td>CONCLUSION</td>
</tr>
<tr>
<td>APPENDICES</td>
<td></td>
</tr>
<tr>
<td>I</td>
<td>WELDING OF ALUMINUM</td>
</tr>
<tr>
<td>II</td>
<td>DEVELOPMENT PROCEDURE</td>
</tr>
<tr>
<td>BIBLIOGRAPHY</td>
<td>50</td>
</tr>
<tr>
<td>FIGURE</td>
<td>PAGE</td>
</tr>
<tr>
<td>--------</td>
<td>------</td>
</tr>
<tr>
<td>1</td>
<td>Cross Section of a Blazed Grating ... 6</td>
</tr>
<tr>
<td>2</td>
<td>Normal Incidence Mount ............... 8</td>
</tr>
<tr>
<td>3</td>
<td>Grazing Incidence Mount ............ 10</td>
</tr>
<tr>
<td>4</td>
<td>Photograph of Jarrell-Ash Monochromator 12</td>
</tr>
<tr>
<td>5</td>
<td>Optical Diagram of Spectrograph, Ebert-Fastie Mount ............. 13</td>
</tr>
<tr>
<td>6</td>
<td>Photograph of Adjustable Slit with Bushing Adapter ............ 20</td>
</tr>
<tr>
<td>7</td>
<td>Photograph of Photoplate Holder ........ 21</td>
</tr>
<tr>
<td>8</td>
<td>Photograph of Vacuum Chamber and Lid .... 22</td>
</tr>
<tr>
<td>9</td>
<td>Photograph of Light Source Housing .... 25</td>
</tr>
<tr>
<td>10</td>
<td>Photograph of Light Source Support with Electrode Holders and Electrodes .... 26</td>
</tr>
<tr>
<td>11</td>
<td>Cross-sectional View of Electrode Holder ............... 28</td>
</tr>
<tr>
<td>12</td>
<td>Photograph of Spark Source ............ 29</td>
</tr>
<tr>
<td>13</td>
<td>Photograph of Complete Apparatus ....... 31</td>
</tr>
<tr>
<td>14</td>
<td>Circuit Diagram of Spark Source ........ 33</td>
</tr>
<tr>
<td>15</td>
<td>Photograph of Jig Used for Cutting Plates ............... 36</td>
</tr>
<tr>
<td>16</td>
<td>Composite Diagram of Photographed Spectral Lines of Aluminum Electrodes 41</td>
</tr>
<tr>
<td>17</td>
<td>Diagram of the TIG Welding Process .... 46</td>
</tr>
<tr>
<td>18</td>
<td>Photograph of Developing Apparatus .... 49</td>
</tr>
</tbody>
</table>
INTRODUCTION

The vacuum ultraviolet region of the electromagnetic spectrum covers the wavelength region of 2 A to 2000 A. This corresponds to photon energies from about 6000 electron volts at 2 A to 6 electron volts at 2000 A. The photon energies are progressively smaller for the normal ultraviolet region (2000 A - 4000 A) and the visible region (4000 A - 7000 A). Specifically, the photon energies at 4000 A are 3 eV and at 7000 A are 1.7 eV. It is apparent that the vacuum ultraviolet energy range is much wider and higher than other regions in the spectrum.

The higher excitation energies required in the vacuum ultraviolet region have contributed to this being one of the least explored regions in the electromagnetic spectrum. Probably, the major reason why investigators have shunned this region is because the vacuum ultraviolet photon energies are absorbed by the molecules of the air. Therefore, to explore this region, a vacuum is needed, resulting in the term "vacuum ultraviolet".

Viktor Schumann first investigated this region in 1893, using a vacuum spectrograph having a fluorite prism. Later, Lyman, using a diffraction grating instead of a prism, showed that Schumann had reached down to about 1250 A. Recently the lowest wavelength recorded has been 4.7 A.

This region of the spectrum has come into importance for a number of reasons. Many elements emit very intense spectral
lines at these wavelengths, resulting in the possibility of very sensitive detection of these elements when concentrations are very low.

In addition, photon energies are quite high, often coming from high ionization states of the elements. This provides a method of probing deeper into atomic structure. It also provides a way of examining the properties of plasmas, which are being investigated considerably for the possibilities of controlled nuclear fusion.

The sun and the stars obtain their energy through fusion reactions. With the increased activity in space exploration, an analysis of vacuum ultraviolet radiation from the sun and the stars is becoming possible without the interference of our atmosphere. Hopefully, this information will help us to better understand the properties of the stars and their fusion reactions, as well as the properties of interstellar gases through which this light passes.

Absorption spectroscopy has also become important in the vacuum ultraviolet. All molecules absorb somewhere in the vacuum ultraviolet, and some organic chemicals of biochemical interest have their principle absorption in this region. This field has not been studied extensively, and with newly improved optics and light sources, this should be an important field of interest. Tousey\(^1\) gives a very good account of the past and future in vacuum ultraviolet spectroscopy.
The purpose of this paper is to report on the construction of a vacuum ultraviolet spectrograph, and the problems that one must consider in this construction. It is believed this spectrograph was built with a minimum of cost. This might be important to others, with limited budgets, who wish to investigate the vacuum ultraviolet.
CHAPTER I

DIFFRACTION GRATING SPECTROGRAPHS

One of the breakthroughs in separating light into its various colors so that the light could be analyzed, was achieved by Sir Isaac Newton\(^2\) in 1672. Newton used a triangular prism for this purpose and the prism is still used to the present day. Prisms used in spectrographs, however, have limitations as to useful wavelength range, rapidly changing dispersion, absorption, and curved spectral lines.

About 100 years after Newton, Thomas Young experimented with the most elementary form of a diffraction spectroscope in what is now known as the famous double slit experiment.\(^3\) About 100 years ago it became possible to rule many closely spaced lines on a piece of material. This is called a diffraction grating. There are transmission gratings and reflection gratings. In most modern spectrographs reflection gratings are used. Reflection spectrographs have large wavelength ranges, slowly changing dispersion, lower absorption, and quite often, straight spectral lines. There are problems of astigmatism and wastefulness of light (some light is always thrown into orders not being used.), but these problems have been largely taken care of by special optical arrangements and by blazing the grating.

A grating is blazed by forming the grooves with one steep face and one shallow face as shown in Figure 1. In this way, the
light will reflect from the grating such that the multiple slit interference pattern lies in the middle of the single slit diffraction pattern. One notices from the figure that
\[ \alpha - \theta = \beta + \theta \]
and thus the angle of maximum diffraction intensity is
\[ \beta = \alpha - 2\theta \]
where \( \alpha \) is the angle of incidence and \( \theta \) is the angle of blaze.

The relation governing most gratings is
\[ m \lambda = d(\sin \alpha + \sin \beta) \]
where \( m \) is the order, \( \lambda \) is the wavelength, \( d \) is the grating spacing, \( \alpha \) the angle of incidence and \( \beta \) is the angle of diffraction. \( \beta \) is positive if on the same side of the normal as \( \alpha \).

Reflection diffraction gratings are usually ruled by a diamond on a thin coating of aluminum deposited on some stable substance such as glass. Rulings are now available with up to 90,000 lines per inch.\(^4\) George Harrison\(^5\) has developed an interferometric control for ruling engines by which he has ruled some of the largest and most accurately spaced gratings.

The Normal Incidence Mount

In 1882, H. A. Rowland developed the idea of combining the optics (mirrors) and grating of a grating spectrograph into a single unit.\(^6\) A grating was ruled on a concave mirror. By this technique, the light passing through the spectrograph had to make only one reflection before being detected. Less light loss took
CROSS SECTION OF A BLAZED GRATING

C  Central Image
B  Diffracted Beam at the Blazed Wavelength
N  Normal to the Grating
N' Normal to the Groove Surface
S  Slit

FIGURE 1
place as a result. This is a very attractive feature for the vacuum ultraviolet, where reflectivities naturally tend to be low.

Rowland found that this arrangement provided accurate and simple focusing techniques. If the slit, detector, and grating are put on a circle of diameter equal to the radius of curvature of the grating, the spectra will always be in focus. This circle is called the Rowland circle.

A commonly found application of Rowland's idea is the normal incidence mount. Figure 2 illustrates this mount. This is called a normal incidence mount, because the light is received by the photoplate almost perpendicular to its length. As a result, $\beta$ is very close to zero. If we let $l$ represent distance along the photoplate, then it is seen from the figure that

$$\frac{d l}{R} = d \beta$$

Then differentiating the grating equation gives

$$\frac{d \lambda}{d \beta} = \frac{d \cos \beta}{m}$$

and solving these two equations gives the reciprocal dispersion,

$$\frac{d \lambda}{d l} = \frac{d \cos \beta}{Rm}$$

Since $\beta$ is close to zero, the cosine function changes very slowly and thus, the reciprocal dispersion is practically constant.

This is a very convenient feature in determining unknown spectral line wavelengths by making measurements of distances from known spectral lines.

There are other variations of this mount in use such as the

Reproduced with permission of the copyright owner. Further reproduction prohibited without permission.
NORMAL INCIDENCE MOUNT

S Slit
P Photographic Plate
N Grating Normal
B Diffracted Light
C Central Image
R Rowland Circle (Diameter R)
G Concave Grating

FIGURE 2
Eagle mount, but the basic features are the same. Most of these types of mounts can be used well in the vacuum ultraviolet down to about 300 Å.

The Grazing Incidence Mount

Below wavelengths of 300 Å reflectivities of gratings become very poor. As a result, the normal incidence mount becomes quite ineffective. The grazing incidence mount is another Rowland circle application which is used mostly below 300 Å (see Figure 3). Since reflectivity increases as incidence angle increases, the grazing incidence mount gives the necessary reflectivities at these short wavelengths. Using angles of incidence of 68°, wavelengths as low as 4.7 Å have been recorded.6

Other than providing the necessary reflectivities, the grazing incidence spectrograph seems to have more drawbacks than advantages. Astigmatism is extreme, because it becomes greater with larger angles. Focusing is difficult, because the focal distance varies rapidly at the large angles. The reciprocal dispersion also varies rapidly at these large angles.4 (See the equation on the previous page.) As a result, unknown wavelengths are hard to calculate.

The Ebert-Fastie Mount

The vacuum spectrograph, with which this thesis deals, uses the Ebert-Fastie mounting. This mounting was first developed by Ebert in 1889. However, it was forgotten and not used.
GRAZING INCIDENCE MOUNT

\( S \) Slit
\( G \) Concave Grating
\( CI \) Central Image
\( P \) Photoplate
\( R \) Rowland Circle
\( \alpha \) Angle of Incidence
\( \beta \) Angle of Diffraction
\( N \) Grating Normal
\( B \) Diffracted Light

FIGURE 3
until Fastie\textsuperscript{7} rediscovered and modified it in 1952. The particular version of this mounting which was used, is sold as a monochromator by Jarrell-Ash.\textsuperscript{8} The monochromator is adjusted for different wavelengths by rotating the plane grating. The radius of curvature of the large mirror is 0.25 meter and the grating, entrance mirror, and exit mirror are flat. A picture of the monochromator and a diagram of its optical layout are shown in Figure 4 and Figure 5, respectively.

It is to be noticed that this mount uses a single spherical mirror to perform two separate reflections, before and after the light hits the plane grating. As a result, spectral images are stigmatic and the aberrations produced by the first reflection are mostly cancelled by the second reflection. Originally, Ebert had the optical distance from the grating to the mirror the same as from the slits to the mirror, resulting in a curved focal plane. The modification developed by Fastie put the grating about \( \frac{4}{5} \) of the way to the mirror, resulting in a flat focal plane. This modification is used in the Jarrell-Ash monochromator. One problem of using this mount in the vacuum ultraviolet is that at wavelengths below 1200 \( \AA \), the five reflections absorbing light render the mounting useless.
PHOTOGRAPH OF JARRELL-ASH MONOCHROMATOR

Approximate Scale

1 foot =

FIGURE 4
OPTICAL DIAGRAM OF SPECTROGRAPH
EBERT-PASTIE MOUNT

P Photoplate
M Plane Mirrors
G Plane Rotating Grating
C Concave Mirror
S Slit
L Light Source

FIGURE 5
CHAPTER II

PROBLEMS IN DETECTING VACUUM ULTRAVIOLET SPECTRA

Although some problems of detection in the vacuum ultraviolet have already been referred to, the problems are actually much broader in scope. The fact air absorbs wavelengths below 2000 A is a combination of a number of factors. First, oxygen has absorption bands that start just below 2000 A and continue down to about 1300 A. Maximum absorption of oxygen takes place at 1450 A. Here a path length of 0.0014 cm is sufficient to reduce the light intensity to one-half at standard temperature and pressure. From 1300 A to 1100 A oxygen is quite transparent, but from 1100 A to less than 300 A oxygen again has absorption bands.

Nitrogen has absorption bands beginning at 1450 A which extend all the way down in the vacuum ultraviolet. Water vapor also has superimposed absorption bands beginning at 1780 A and 1340 A. Many other more rare gases in the air have their own absorption bands.

As a result, one notices that the complete region of the vacuum ultraviolet is covered by some absorption band. Vacuums, therefore, of at least $10^{-3}$ mm of Hg should be used in this region. Usually, vacuums of $10^{-4}$ to $10^{-6}$ mm of Hg are used, which require the use of diffusion vacuum pumps.

Another problem in the vacuum ultraviolet is that most solid materials are opaque and below about 1100 A virtually all solid
materials are opaque. Lithium fluoride\textsuperscript{10} has this low transmission limit. As a result, prism spectrographs cannot be used below this point. Only reflective optics can be used.

In reflection instruments problems in reflectivities also come into play. Ordinarily, aluminum is considered to be very reflective, but in the vacuum ultraviolet, the reflectivity drops off drastically. Coating aluminum with magnesium fluoride has been shown to dramatically improve the reflectivity down to 1000 A.\textsuperscript{11} The coating protects the aluminum from oxidation especially in the presence of vacuum ultraviolet light. Below 1000 A the best reflector is platinum followed closely by gold, but even at best, the reflectivities of these substances are on the order of 10 to 20%.

Since light sources used in the vacuum ultraviolet also must be located in the vacuum, certain restrictions come into play. The vacuum spark is probably the most versatile source of light in this region, especially for spectra due to highly ionized atoms. The problems involved with the vacuum sparks are described in Chapter III.

If gas spectra are desired in the vacuum ultraviolet, special problems develop. Discharges through gases must take place without windows (especially at wavelengths below 1100 A) between discharge and the spectrograph, and the spectrograph must remain evacuated. A number of different designs have now been developed and can be found in the literature.\textsuperscript{6} Besides electrical discharges...
in gases, coils driven by radio frequencies, and microwave
cavities can be used for exciting gases.

Detection of vacuum ultraviolet radiation also presents
problems. Below 2200 Å ordinary photographic emulsions are not
sensitive, because the gelatin absorbs the light before it can
sensitize the silver halide grains. A variety of ways have been
developed to give sensitivity to emulsions. One way is to coat
the plate with various fluorescent substances such as oil, lacquer,
sodium salicylate, sodium chloride, various plastics, and many
others. The substances convert the light to longer wavelengths
to which the emulsion is sensitive. Samson⁶ gives a good account
of the properties of many of the materials. Upon development
these substances must first be washed off by appropriate solvents.
Another solution to the photographic problem is to use emulsions
with very little gelatin, such as the Kodak SWR emulsion.¹² This
is the detector used in this investigation. Its properties are
described in Chapter IV.

Photomultiplier tubes can also be used to detect vacuum
ultraviolet radiation, but it has much the same problems as photo-
plates. Since the tubes have windows, these windows serve to cut
out the short wavelengths and thus fluorescent materials must be
put on the tube window just as for photoplates.

An alternative for photomultiplier tubes is to remove the
window completely. The instrument is then sensitive to wavelengths
all the way through the vacuum ultraviolet region. The tube will
still operate without a window, because detection is done in a vacuum. Actually, this method has the advantage of being able to use photosensitive cathodes in the photomultiplier tube which have higher work functions. This limits sensitivity only to the vacuum ultraviolet (Scattered longer wavelengths do not interfere.), which in turn cuts dark currents to a minimum.

Besides photomultipliers, photodiodes are being used with increasing frequency in the vacuum ultraviolet. They are compact and their sensitivity increases as wavelengths get shorter. A good description of all of these detectors, and a good list of references pertaining to these detectors are given by Samson. 6
CHAPTER III
CONSTRUCTION OF THE VACUUM SPECTROGRAPH

Some considerations that were kept in mind in the construction of the spectrograph were as follows:

1. The instrument should be as light and portable as possible.

2. The process necessary to record a spectrum should not be too cumbersome or time consuming.

3. A good quality spectrum of sufficient dispersion should be obtainable.

4. Apparatus and materials which were "on hand" should be used as much as possible.

Monochromator Alterations

In order to build a proper vacuum chamber, the components inside the chamber had to be decided upon first. To disperse the light, a quarter-meter Jarrell-Ash monochromator was used (see Figure 4). The optics of this instrument were described in Chapter I.

The basic monochromator was altered in two ways to transform it into a spectrograph. First, the entrance slit had to be replaced, because the existing slit consisted of an aluminum covered quartz plate upon which the slit was etched. Since quartz does not transmit in the vacuum ultraviolet, an open slit had to take its place. An adjustable open slit was found, and by use of a bushing, was adapted to fit the monochromator. The bushing was
constructed on the lathe from a 1 1/4 inch diameter piece of aluminum stock (see Figure 6).

The second alteration was to replace the exit slit with a holder for a photographic plate. This was done by retaining the original sleeve which held the exit slit. A piece of metal was then mounted on the front of this sleeve to form a slot which would hold the photo plate (see Figure 7).

The Vacuum Chamber

The vacuum chamber to be made had to house this converted monochromator and the light source. The consideration, as noted above, of lightness and portability made aluminum the logical construction material.

In designing this chamber, the volume had to be kept at a minimum to keep pump down time short, and the shape had to make it somewhat self-supporting against atmospheric pressure. It was decided a cylindrical shape could best meet these needs (see Figure 8).

The side of the cylinder was constructed from 1/8 inch sheet aluminum, which was cut 63 in. x 10 in. This was rolled smoothly into a cylinder 20 inches in diameter and 10 inches high. The two ends were then welded together to form a smooth, airtight seal (see Appendix I).

The bottom of the cylinder consisted of 1/4 inch aluminum sheet which was cut in a circle 22 inches in diameter. This
PHOTOGRAPH OF ADJUSTABLE SLIT
WITH BUSHING ADAPTER

Approximate Scale

1 inch = ——

Left to Right: Adjustable Slit, Bushing Adapter,
Monochromator

FIGURE 6

20
PHOTOGRAPH OF PHOTOPlate HOLDER

Approximate Scale

1 inch = ——

FIGURE 7

21
PHOTOGRAPH OF VACUUM CHAMBER AND LID

Approximate Scale

1 foot = ---

FIGURE 8

22
circular plate was welded to the bottom of the cylinder. Aluminum of this thickness was considered necessary, because both the top and the bottom of this cylinder would have to support an atmospheric force of

\[ F = PA = P\left(\frac{\pi D^2}{4}\right) = 4560 \text{ lbs} = 2 \frac{1}{4} \text{ tons} \]

where \( P \) is the atmospheric pressure (14.7 lbs/in\(^2\)), \( A \) is the area, and \( D \) is the diameter (20 in).

The top of the cylinder had to be removable to service the spectrograph. A circular lid was cut 20 1/2 inches in diameter from the same sheet as the bottom of the cylinder. In order to attach the lid to the cylinder, a circular ring 20 1/2 inches outside diameter and 16 inches inside diameter was cut from 1/2 inch aluminum sheet. This ring was mounted on the lathe to cut an "o" ring groove 1/4 inch wide and 18 1/4 inches in diameter. Twenty-four 1/4 inch holes were then drilled and tapped part way into the aluminum ring just outside the "o" ring groove with twenty four matching holes in the lid. The aluminum ring was then welded to the top of the cylinder. Small triangular struts were also welded to the ring and cylinder walls inside the chamber to limit flexing of the ring. When the lid is then screwed onto the ring, the "o" ring groove provides the vacuum seal.

To house the light source, a five inch diameter aluminum pipe was cut to the curvature of the side of the vacuum chamber. A three inch diameter hole was cut in the side of the vacuum chamber,
The pipe was then welded over this hole (see Figure 9). An aluminum flange, five inches inside diameter and seven inches outside diameter, cut from 1/2 inch aluminum sheet, was welded to the other end of the pipe. This flange was previously cut with an "o" ring groove 1/8 inch wide and 5 1/4 inches in diameter along with eight 1/4 inch tapped holes just outside the "o" ring groove. A 3/4 inch nipple was also welded to the side of the light source housing so that the vacuum pump could be connected.

Upon completion, the entire inside of the chamber was sprayed with a velvet black paint to prevent unwanted reflections of light. The monochromator itself was covered with a black, velvet-like flock paper\(^{13}\) for the same purpose. This paper is usually used for lining telescope tubes to prevent internal reflections.

The lid for the light source housing was made of 1/4 inch plexiglas. This material was chosen because it is a good insulator upon which the light source could be mounted. It was cut and drilled to match the flange of the light source housing. Two other 1/4 inch holes, 2 1/2 inches apart, were drilled symmetrically in the lid. Quarter-inch aluminum rods, six inches long and threaded on both ends, were put half way through these holes. Epoxy glue was then used to fasten and seal these rods into place (see Figure 10). These rods have the purpose of supporting the light source that would be used and also conducting current to that light source. This configuration allows for usage of a variety of light sources.
PHOTOGRAPH OF LIGHT SOURCE HOUSING

Approximate Scale

1 inch = ——

FIGURE 9

25
PHOTOGRAPH OF LIGHT SOURCE SUPPORT
WITH ELECTRODE HOLDERS AND ELECTRODES

Approximate Scale

1 inch = ——

FIGURE 10
The Light Source

For the light source, it was decided to use a high voltage spark. As a result, electrode holders had to be fashioned to fit on the ends of the conducting rods which were described in the preceding paragraph. The holders were constructed of two blocks of aluminum which were cut to uniform size on a mill. Quarter-inch holes were drilled and tapped in the ends of the blocks so they could be screwed onto the conducting rods. Holes slightly larger than 1/4 inch were drilled vertically completely through the blocks for inserting the electrodes. To hold the electrodes in place, a hole was drilled and tapped for an 8-32 screw from the opposite end of each block such that this screw hole intersected the electrode hole (see Figure 11). In this way, any size electrode up to a quarter inch can be accommodated by the holder. These aluminum blocks also act somewhat as heat sinks when sparking takes place.

The spark source that was initially chosen was a Bausch and Lomb model X-37 spark generator. It consisted of a 15,000 volt, thirty milliampere transformer, a 0.005 microfarad high voltage capacitor in parallel with the transformer secondary, and an inductance in series with the analytical spark gap which was variable from zero to 20.65 microhenries. The capacitor was protected from excess voltage by a 3/8 inch safety gap across its terminals (see Figure 12).

However, the first time a vacuum spark was attempted, it was
CROSS-SECTIONAL VIEW OF ELECTRODE HOLDER

Approximate Scale
1 inch =

E Electrode
B Aluminum Block
R Support Rod
S Screw

FIGURE 11
28

Reproduced with permission of the copyright owner. Further reproduction prohibited without permission.
PHOTOGRAPH OF SPARK SOURCE

Approximate Scale

1 foot =

Left to Right: High Voltage Leads, Variable Inductor
Capacitor with Safety Gap, Transformer,
and Second Transformer Added in Series.

FIGURE 12

29
apparent this source had to be altered. At the low pressures, a
glow discharge at the electrodes leaked off all the current the
transformer could deliver. Therefore, no spark took place. This
was partially corrected by placing an external control gap in
series with the analytical gap (see Figure 13). In this way, cur-
rent could not leak away until the control gap broke down. As a
result, the capacitor could store charge and build up a potential
so that a high voltage, high current density spark took place
across the analytical gap.

High current densities are necessary for good light inten-
sities from lines of higher ionization states. If one considers
the energy stored in a capacitor
\[ E = \frac{1}{2} CV^2 \]
where \( E \) is the energy, \( C \) is the capacitance and \( V \) the voltage
across the capacitor, one sees that when the spark occurs this
energy is transferred to the inductance of the circuit
\[ E = \frac{1}{2} LI^2 \]
where \( E \) is the energy, \( L \) the inductance and \( I \) the current.
Solving these two expressions for current gives
\[ I = V(C/L)^{\frac{1}{2}} \]
From this expression, it can be seen that high current densities
result from high voltage, high capacitance and low inductance.
Therefore, all spectra in the vacuum ultraviolet were taken with
a minimum inductance.

The situation still was not ideal, however. The production
Shown in this photograph are the control gap on the left, the light source on the front of the vacuum chamber, the spark source below, and the vacuum pump on the bottom.

FIGURE 13
of spectra in the vacuum ultraviolet requires multiple ionization of the atoms in the discharge of the analytical gap. This requires very high voltages. With the set up thus far, the control gap could be opened only about 1/2 millimeter. As a result, voltages less than a thousand volts were achieved. Beyond that, a glow discharge would take place between the analytical electrode, on the opposite side of the control gap, to the housing of the vacuum chamber. A method suggested by Wahr\textsuperscript{15} of grounding both this electrode and the housing, eliminated the glow discharges, but did not improve the voltages. The problem was corrected by using a second transformer in series with the original one (see Figure 14). This way the control gap could be opened to over a millimeter and thus the necessary voltages were obtained.

Creating A Vacuum

A vacuum was produced using a Genco Megavac Pump\textsuperscript{16} which has a free air capacity of 57 liters per minute and an ultimate vacuum of 0.1 micron. The volume of the chamber is about 65 liters. As a result, it requires about half an hour to pump down to under 1 mm of Hg. Typical pump down times, however, were chosen to be about one and one half hours so that operating pressures were estimated at about $10^{-2}$ mm of Hg. Connection was made from pump to chamber by way of a 3/4 inch vacuum hose to a 3/4 inch nipple on the side of the light source housing.
CIRCUIT DIAGRAM OF SPARK SOURCE

FIGURE 14

S Switch
T Transformers (136:1)
C Capacitor (0.005 microfarad)
L Inductor (0 to 20.65 microhenries)
B Control Gap
A Analytical Gap
A preliminary investigation of the characteristics of the converted monochromator was made in the near ultraviolet using a high intensity mercury vapor lamp and an aluminum spark. This investigation was made in air using Kodak SA-3 (Spectrum Analysis No. 3) and Kodak SWR (Short Wave Radiation) plates. Exposure times were ten seconds with the sources located about one-half meter from the slit.

First, this investigation showed the relative sensitivities of the two types of plates. Above 2400 Å, each emulsion gave about the same results. Below 2400 Å, SA-3 sensitivity dropped rapidly showing no sensitivity below 2200 Å. However, SWR plates gave good pictures down to less than 2000 Å. At this point the air began to absorb the light. One disadvantage of SWR plates is their sensitivity to abrasion. Extreme care in handling had to be exercised. A slight touch of the finger would later be developed into a fingerprint.

The reason for the lower wavelength response of SWR plates is that the emulsion is made with much less gelatin. As a result, light passes through much less gelatin before exposing the silver halide grains within the gelatin. This emulsion is supposed to be sensitive all the way down to 75 Å. 12
Second, the investigation showed that the advertised reciprocal dispersion of 33 Å/mm for the monochromator was not quite accurate. By identifying some common spectral lines, the calculated reciprocal dispersion was very close to 31.8 Å/mm. This knowledge was quite helpful later when lines in the vacuum ultraviolet had to be identified.

Third, the investigation showed the range covered per exposure was about 600 Å. Thus, when the monochromator was set, for example at 2800 Å the range recorded on the photoplate began at 2500 Å and ended at 3100 Å.

Fourth, resolution of two closely spaced lines was shown to be about two angstroms. Since this was as good as the advertised specifications of this monochromator, the instrument was considered to be in good focus.

To photograph spectra in the vacuum ultraviolet, SWR plates were used exclusively. These plates normally come from Kodak in the size 4 in. x 10 in. Since this was much too large for the spectrograph constructed, each plate was cut into six pieces of about 1 3/4 in. x 4 in. A special jig, covered with velvet, was constructed for this purpose according to suggestions from Kodak (see Figure 15). A diamond point glass cutter was used to scratch the glass opposite the emulsion. This cutting arrangement works very well with ordinary glass plates that are not very old. However, these SWR plates were a few years old and as a result, quite brittle. This caused the break to not always follow the scratch.
PHOTOGRAPH OF JIG USED FOR CUTTING PLATES

Approximate Scale

1 inch = — —

This picture shows a velvet covered board with a hardwood rail. A diamond point glass cutter and a wooden straight edge are lying on the velvet.

FIGURE 15

36

Reproduced with permission of the copyright owner. Further reproduction prohibited without permission.
Another problem was that the extreme sensitivity to abrasion of these plates led to small markings of the developed plate from the velvet.

The converted monochromator was properly placed in the chamber by using a laser to align the electrodes with the optic axis of the monochromator. This was done simply by shining a laser beam between the electrodes onto the slit. The monochromator was set for the zero order and positioned so that the beam came out the center of the exit.

Once the setup was complete, pictures in a vacuum were taken. The first pictures were taken at a setting of 1800 A. Aluminum electrodes, set with a 3 mm gap, were used for sparking. Initial exposure times of 10 seconds were soon shown to be quite short. The only spectral lines found were the intense 1854.7 A and 1862.7 A lines of doubly ionized aluminum and the 1862.3 A line of singly ionized aluminum. Actually, the 1862.7 A and 1862.3 A lines were outside the resolution limit of the instrument, so that the three lines appeared as a doublet.

All vacuum ultraviolet lines were identified in this investigation by using the reciprocal dispersion (31.8 A/mm) and the published wavelength tables. For example, an unknown line's position was first measured to 0.02 mm from a previously identified line's position by using a seven power comparator. Multiplying the distance by the reciprocal dispersion, gave the wavelength difference between these two lines. Adding or subtracting this
difference from the known line wavelength, gave the approximate wavelength of the unknown line. A line was found in the tables which closely matched this, and thus the identity of the line was determined. Qualitative matching of relative intensities, as given in the tables, also helped in the identification.

When exposure times were increased to one minute, two more aluminum lines were recorded in a vacuum. These were the doubly ionized 1935.9 A line and the singly ionized 1990.5 A line. It is to be noted here that these lines were also detected and were actually more intense when the spectra were taken in air. The reason is, although the oxygen in the air probably absorbs these lines somewhat, the spark which occurs in air has a much higher intensity.

To find shorter wavelength aluminum lines in a vacuum, much longer exposures had to be taken. The best results were obtained with five minute exposures. These were taken in five one-minute intervals to prevent overheating of the electrodes. Longer exposures than this fogged the plate too much due to internal reflections within the monochromator.

With the monochromator set at 1550 A and using five one-minute exposures, a number of other lines were photographed. First was a band of ten lines between 1760 A and 1770 A. These lines are more or less evenly spaced and therefore, almost looked like one broad line. These lines belong to neutral and singly ionized states of aluminum.
The next observation was a doublet at approximately 1743 Å. As it turned out, these lines were not of aluminum, but of nitrogen. Apparently, there was still enough nitrogen of the air left in the chamber to produce these spark lines. Their wavelengths were 1742.7 Å and 1745.3 Å. These result from neutral nitrogen atoms.

At even shorter wavelengths, another bright aluminum doublet appeared. The wavelengths were 1721.3 Å and 1725.0 Å belonging to the first ionization state of aluminum. Actually another line, 1719.4 Å, of first ionized aluminum should have appeared, but its low intensity made it unobservable.

The last aluminum line to be identified was the 1670.8 Å line of first ionized aluminum. This is a very intense line, but on the photographs taken, it showed quite weakly. The reason is thought to be the fact that in this region, the oxygen absorption is fairly close to maximum. Even though the chamber was evacuated possibly down to 10⁻² mm of Hg, the oxygen left still had an effect. For better results, pressures of 10⁻⁴ mm of Hg should be attained.

There were two more doublets in these photographs that were observed. One doublet was centered at about 1334 Å and the other, which was very faint, at about 1372 Å. These lines, however, could not be identified positively either as aluminum or some impurities. The 1379.7 Å and 1384.1 Å lines of doubly ionized aluminum came fairly close to the calculated value of the second doublet, but not close enough for a positive identification. To
check on impurities, the aluminum electrodes were analyzed with a direct current arc through a medium quartz spectrograph. Impurities of silicon, magnesium, copper, and iron were found in small amounts. Just a trace of carbon was found. Of these impurities, however, carbon would be the most likely suspect, because it has some lines in the 1330 Å region. However, some trials of carbon electrodes on the vacuum spectrograph failed to show these lines. It is the feeling of the author that these lines are actually of aluminum. Not all lines are in the published tables and possibly these are a case in point. Furthermore, the aluminum spectrum is characterized by many doublets all the way from the vacuum ultraviolet to the visible. The appearance of these doublets gives one the same impression as other aluminum doublets (see Figure 16). Further investigation should be done in identifying these lines.

When the carbon electrodes were run on the vacuum spectrograph, a couple of lines were identified. They were the 1657.0 Å and 1561.4 Å lines of the neutral carbon atom. These lines were quite diffuse, probably because there were many other unobservable, less intense lines in the vicinity. These two lines were also difficult to observe, because of the heavy continuous background that was generated by these electrodes.

Because of the fact that some closely spaced lines of aluminum could not be resolved well (such as the 1760 Å - 1770 Å band), an attempt was made at photographing these lines in the second order.
COMPOSITE DIAGRAM OF PHOTOGRAPHED SPECTRAL LINES OF ALUMINUM ELECTRODES

Wavelength Scale in Angstroms

FIGURE 16

Reproduced with permission of the copyright owner. Further reproduction prohibited without permission.
However, this did not work well at all, because the first order lines in the vicinity covered these second order lines completely. The reason was the second order lines needed long exposures to be detected and thus, the first order lines blackened much of the photoplate.
CHAPTER V
CONCLUSION

It has been shown that it is possible to construct a vacuum ultraviolet spectrograph with a minimum of cost. All the materials used were already available in the physics department. An exception was the large "o" ring, which was purchased locally.

The spectrograph recorded lines of aluminum, which were identified down to 1670 Å, and other lines down to 1372 Å. Spectral lines were sharp and resolvable to about two angstroms.

Some problems were encountered. Some of these were solved. Others might be topics for future investigations. The unidentified lines with aluminum electrodes could be studied, as well as a search made for lines which did not appear. Other electrode materials could also be studied.

Since very long exposures were needed to bring out spectra, some longer wavelength internal reflections inside the monochromator were fogging the plate. A modification that would help this would be the usage of a diffusion pump to bring pressures down to the range of $10^{-4}$ mm of Hg. Possibly, baffles positioned inside the monochromator at various positions could help cut down these internal reflections, also.

Investigations could be made using other types of light sources as described in Chapter II. If a continuum source is used, absorption spectroscopy could be accomplished, because the
vacuum chamber was constructed with ample room to mount absorbing materials.

Another alteration, which might be tried, would be the use of some other detector, such as a photomultiplier in conjunction with a scintillator. In this way, a motor drive could be attached to the monochromator, allowing one to view the whole spectrum in one pass.
APPENDIX I

WELDING OF ALUMINUM

Even though aluminum is one of the most weldable metals, some special techniques must be used in the welding. When exposed to air, high temperature aluminum readily combines with the oxygen and nitrogen. Therefore, aluminum must be protected from the atmosphere when it is welded.

There are a number of ways this is done. The welds, on the vacuum chamber in this investigation, were made using the inert-gas-shielded arc welding process, often called "Heliarc" welding. The particular form of this process which was used, is called TIG (Tungsten Inert Gas). Figure 17 shows the general design of the welding gun.

A high current, low voltage, electric arc is struck between the non-consumable tungsten electrode and the metals to be welded. This melts some of the metal. At the same time, argon gas flows around the tungsten electrode displacing air all around the melted aluminum. To fill the gap between the pieces of metal to be welded and to form most of the weld, a filler rod is fed into the arc. The filler rod is of a similar composition as the welded metals.

This welding process gives a good looking weld with no residual slag as is the case with normal welding of steel. Jefferson gives a good detailed description of most welding processes.

45
T  Tungsten Electrode
F  Filler Material
G  Flowing Argon Gas
M  Materials to be Welded Together
A  Electric Arc
W  The Weld
APPENDIX II

DEVELOPMENT PROCEDURE

The SWR plates used were processed as are most spectroscopic plates. The process consists of treating the plates with developer, stop bath, fixer, and wash water. The developer used most was Kodak D-19 diluted 1:1 with water. Although recommended development time was two minutes, the author found that this was too long. The plates tended to be excessively fogged. Development times of 1 1/2 minutes were found to be much more suitable. In this way, spectral lines were still brought out fully, with a minimum of fog.

Dupont 24-D developer was also tried on these plates with good success. This is a fast working developer, since development times of less than one minute were needed.

Development with these plates was much more rapid than for Kodak SA-3 plates. The reason for this is, as mentioned before, the much lower amount of gelatin in the emulsion. As a result, when the emulsion is placed in the developer, the developing agent can penetrate the emulsion and thus reduce the exposed silver halide grains in considerably less time.

The stop bath consisted of a two per cent solution of acetic acid. The plate was immersed in this for 30 seconds. This process quickly neutralizes the basic developer and allows one
to control development times quite accurately. The useful life of the fixer is also increased by the use of the stop bath.

Kodak Rapid Fixer was used for the fixing process for times of about 2 1/2 minutes. This process dissolves unreduced silver halide grains, leaving only the silver grains to form the developed picture.

Plates were washed about 30 minutes in running tap water. A final rinsing was made in distilled water to eliminate water marks upon drying.

The developer, stop bath, and fixer are contained in glass trays which in turn are mounted in a metal tray as shown in Figure 18. An important feature of this set up is that water from a constant temperature source can flow through the metal tray bathing the glass trays, and thus keep the solutions at constant temperature. This provides for uniform development of plates at all times.
PHOTOGRAPH OF DEVELOPING APPARATUS

Approximate Scale

1 foot = 

Left to Right: Developer, Stop Bath, Fixer, and Wash

On the extreme upper right is a constant temperature source of water. This water runs through rubber tubing into the metal tray. Here it bathes the glass developing trays and flows into the wash tray.

FIGURE 18

49

Reproduced with permission of the copyright owner. Further reproduction prohibited without permission.
BIBLIOGRAPHY


