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## A LIGHT SOURCE TO BE USED

### WITH A VACUUM SPECTROGRAPH

BY

ROY GEORGE WOODLE, Jr.

------

A

THESIS

submitted to the faculty of the

SCHOOL OF MINES AND METALLURGY OF THE UNIVERSITY OF MISSOURI

in partial fulfillment of the work required for the

Degree of

MASTER OF SCIENCE, PHYSICS MAJOR

Rolla, Missouri

1951

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Harold Q Fuller Professor of Physics Approved by -

#### ACKNOW LEDGEMENT S

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R. G. W.

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### INTRODUCTION

A grazing incidence vacuum grating spectrograph
for use in the vacuum ultraviolet was designed
and partially constructed by Mr. L. H. Chapin, 1
1. Chapin, L. H., <u>A Grazing Incidence Vacuum</u> Grating Spectrograph For Far Ultraviolet <u>Spectroscopy</u> , Unpublished Masters' Thesis, Missouri School of Mines and Metallurgy, 1950.
as a research project for a Masters' Degree. This
instrument is designed to photograph the region
of the spectrum from 100 Å to 1000 Å. As Boyce
pointed out in his exhaustive review, 2 there is
<ol> <li>Boyce, J. C., Spectroscopy in the Vacuum Ultraviolet, Review of Modern Physics, Vol. 13, p. 1, Jan. 1941.</li> </ol>
much inportant observational work to be done in
this region.

The author of this paper has undertaken to develop a satisfactory source for the region from  $100 \text{ \AA}$  to  $1000 \text{ \AA}$ . This is part of a project to photograph the empission and absorption spectra of crystals.

#### REVIEW OF LITERATURE

Whenever atoms or molecules possess excess energy, they may radiate light in order to dissipate this energy. The probability of the emission of this radiation depends upon the degree of excitation and the length of time between collisions which may relieve the atom or molecule of its excess energy. The nature of the radiation emitted depends upon the degree of excitation and the characteristics of the excited atoms or molecules.

There are four ways that an atom or molecule can receive energy: (1) by collisions with an electron of sufficient kinetic energy (collisions of the first kind), (2) by collisions with other atoms or molecules which possess appropriate amounts of excess energy (collisions of the second kind), (3) by absorption of radiation of suitable frequency, and (4) by thermal excitation. Light sources rich in ultraviolet are primarily excited by collisions of the first kind, although some are modified by thermal excitation.

The most suitable light sources for the vacuum ultraviolet are the Paschen discharge tube, the Lyman discharge tube, and the vacuum spark.

The Paschen hollow-cathode discharge tube<sup>3</sup>

3.	Paschen,	F,	Annalen	Der	Physik,	Vol.	50,
	p. 901,	19	16.				

is characterized by low potential gradients and the almost pure electron excitation. This source is only useful when excitation of singly or doubly ionized atoms are to be studied.

The Lyman discharge tube<sup>4</sup> radiates a continuum

from the visible down to about 270 Å. The radiation is produced by the discharge of a condenser through a capillary. The long wave length part of the continuum is probably produced by the thermal radiation emitted by minute particles of glass torn from the walls of the capillary and heated to a high temperature by the discharge. The short wave length radiation is probably soft X-Rays produced by electron excitation. Collins

<sup>4.</sup> Lyman, T., The Reversal of the Hydrogen Series in the Extreme Ultraviolet, Science, Vol. 64, p. 89, 1926.

and Price <sup>5</sup> improved the Lyman tube by constructing

5. Collins, George and Price, W. C., A Source
of the Lyman Continuance for Use with
Spectrographs of High Dispersion, Review of
Scientific Instruments, Vol. 5, p. 423, 1934.
it so that the capillary tube could be readily
replaced. Further improvements were made in the
tube by Worley. <sup>6</sup> These consist of making the
6. Worley, R. E., An Improved Source for the Lyman Continuance in the Vacuum Ultraviolet, Review of Scientific Instruments, Vol. 13, p. 67, 1942.
cathode with large fins to eliminate water cooling
of this electrode and to collect most of the
solid products from the discharge.
The vacuum spark, first used by Millikan
and <b>Sawyer</b> , 7 was developed by Edlén <sup>8</sup> to give an
7 Millicon D A and Compan D A Extrame

7.	Millikan,	, R. A	., and	Sawy	er,	R. A	., E:	<i>streme</i>
	Ultra-v:	iolet	Spectra	l of	Hot	Spar	ks ir	n High
-	Vacuum,	and the second se						1

8.	Edlen,	B.,	Zeits	f.	Physik,	Vol.	100,
	p. 62	1, 1	936.		_		

excitation of copper ionized 18 times. The vacuum spark is the only source that will give radiation of the shortest wave length.

In most cases the spark is produced by charging a condenser in parallel with the gap, to from 10,000 volts to 80,000 volts, then discharging the condenser through the gap, either gap so that it will break down at the desired voltage. The condenser serves to increase greatly the current density through the gap. For rapid sparking the condenser may be charged with alternating current and discharged on every half cycle or more rapidly if desired. For a slow rate of sparking, the condenser must be charged with direct current. The charging rate of the condenser may be controlled by a resistor in the circuit, between the transformer secondary and the condenser.<sup>9</sup>

# 9. Sawyer, R. A., Expermental Spectroscopy, Prentice Hall, New York, 1946, pp. 23, 295.

The potential breakdown of a vacuum spark decreases as the pressure drops to a minimum value of about 100 microns, depending on the gas; then increases rapidly as the pressure drops still further.<sup>10</sup> The spark may be operated <u>10. Boyce, J. C., Op. Cit., p. 18.</u> at the potential desired by dropping the pressure to the required value, or by operating the source in the range of the softest spark and controlling the sparking potential by an auxiliary spark gap in series with the source.

Schoen and Hodge<sup>11</sup> of Kodak Research

# 11. Schoen, A. L., and Hodge, E. S., Photographing Spectra in the Vacuum Ultraviolet, Journal of the Optical Society of America, Vol. 40, p.23, 1950.

Laboratories, used a vacuum spark source to test the new short wave radiation film. This source was operated at 100 microns and excited by a 1 MFD condenser charged to 17,000 volts discharging once every two seconds.

The spark potential and rate of sparking were controlled by an auxiliary air spark gap and a resistor in the charging circuit.

The electrodes in the source were about 20 cm. apart and the spark was fired through a quartz capillary of 1/4 inch inside diameter. The quartz tube, surrounded by the water cooled central section of the source tube, was held in place by glass wool.

The 100 micron pressure was maintained by a fine wire leak. They used 500 discharges in air and photographed lines down to 202 Å.

A very intensive study of controlled spectrographic spark sources has been made by Enns and Wolfe.<sup>12</sup>

<sup>12.</sup> Enns, J. H., and Wolfe, R. A., Symposium on Spectroscopic Spark Source. American Society for Testing Materials, pp. 12-25, Philadelphia, 1948.

Vacuum spectrographs are usually operated at pressures of about  $10^{-4}$  mm of mercury, in order to reduce the absorption of radiation by gases in the system. If the source is to be operated in the range of  $10^{-2}$  mm, and controlled by an auxiliary spark gap, an adjustable leak must be incorporated in the source tube to maintain the differential in pressure between the source and the spectrograph. The same leak may be used to admit different gases into the vacuum system for spectral investigations.

Several different types of leaks have been developed to introduce gases into vacuum systems. The porcelain plug type, like the one described by Smythe <sup>13</sup> has been used extensively when a <u>13. Smythe, W. R., R.S. I. Vol. 7, p. 435, 1936</u>. small leak is needed for long periods of time, and traces of mercury vapor are not objectionable. A glass tube is shrunk around a porcelain rod and sealed into the system; then the gas leaks through the porcelain rod. The rate of leakage is controlled by covering different percentages of the rod with mercury.

A capillary tube drawn down to suitable length and fineness is often used to permit a small flow of gas into a vacuum system, and Pyrex tubing shrunk down on a short piece of copper wire allows a small leak. The disadvantage of either of these types of leak is that each leak has only one rate of flow.

Needle valve leaks have been designed which vary in construction from the simple type described by Strong <sup>14</sup> to the fine-adjustment, 14. Strong, J., Procedures in Experimental Physics, p. 98, Prentice Hall, N. Y., 1946. precision-machined valve described by Alcock, Peiser, and Pout, <sup>15</sup> which provides accurate 15. Alcock, T.C., Peiser, H. S., Pout, J. S., A Fine-adjustment Needle-valve Suitable as a Controlled Leak for X-Ray Gas Tube, Journal

of Scientific Instruments, Vol. 25, p. 87, 1948. reproducibility of setting and control of gas flow to within two percent.

Perhaps the simple t and easiest to construct of all devices for controlling the flow of gas into a vacuum system is the one described by Knauss.<sup>16</sup> A length of wire is placed 16. Knauss, H. P., A Device for Controlling the Flow of Gas Into a Vacuum System, R.S.I., Vol. 2, p. 750, 1931.

inside a section of rubber vacuum tubing and the tube is squeezed with a pinch clamp. A small opening remains along the side of the wire which may be completely closed by applying sufficient pressure with the pinch clamp, and may be opened gradually by releasing the pinch clamp pressure. The leak would not be satisfactory however, when accurate resetting without the assistance of pressure gauges might be necessary.

Boyce and Robinson<sup>17</sup> have tabulated many

17.	Boyce, J. C., and Robinson, H. A., Wave- length Identification Lists for the Extreme Ultraviolet, Journal of the Optical Society of America, Vol. 26, p. 133, April, 1936.
of t	he spectrum lines in the extreme ultraviolet
that	have been identified.
	Edlen <sup>18</sup> and Soderqvist <sup>19</sup> have identified
18.	Edlen, B., Wellenlangen und Termsysteme zu den Atomspektren der Elemente Lithium, Beryllium, Bor, Koblenstoff, Stickstoff und Sauerstoff. Nova Acta Regiae Societatis Upsaliensis, Series LV, Vol. 9, No. 6.
19.	Söderqvist, J., Vakuunfunkenspektren der Elemente Natriun, Magnesium, Aluminium und Silicium. Nova Acta Regiae Societatis Scientiarum Upsaliensis, Series IV, Vol. 9, No. 7.

many lines of less than 100 Å. They used a vacuum spark as the light source.

As this brief review indicates, the literature describing light sources for vacuum spectroscopy is very extensive.

It was decided that a source similar to the one used by Schoen and Hodge of Kodak Research Laboratories, using the fine wire leak described by Knauss, would be the most satisfactory for the proposed research to be carried out with the vacuum spectrograph at the Missouri School of Mines and Metallurgy.

The remainder of this paper will be chiefly concerned with the development of this source and with the spectra it produces.

### LIGHT SOURCE

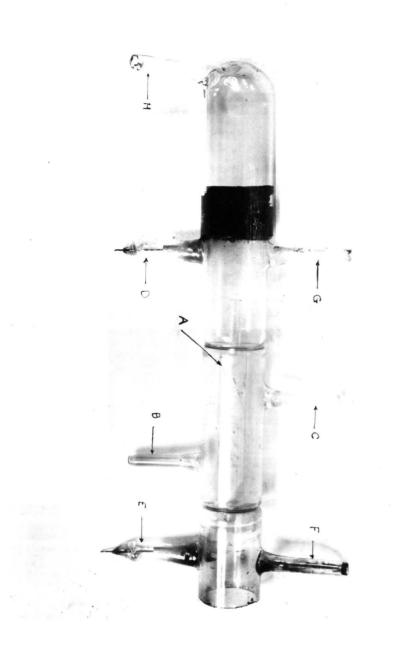


Fig. 1 Source Tube

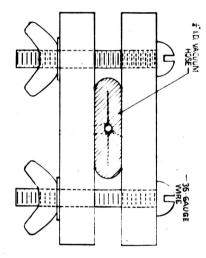
The pyrex source tube, Fig. 1, manufactured at Missouri University, is 26 cm long and 4 cm in diameter. A coaxial tube (A), 2 cm in diameter and 10 cm long is flanged and sealed into the center of the source tube. Two 8 mm diameter side tubes (B and C), are sealed onto opposite sides of the source to provide inlet and outlet for cooling water. Two 1 cm diameter side tubes, (D and E) are sealed to the bottom of the tube through which are sealed the tungsten leads to the aluminum electrodes. Two other 1 cm diameter side tubes (F and G) are sealed onto the top of the source directly opposite the two electrode holders. Side tube (F) is the outlet to the source tube vacuum pump, and to side tube (G), is fixed the fine wire leak. A cap is waxed to one end of the large tube, and a 1 cm diameter tube (H) is scaled to the cap. A length of 1/4" inside diameter vacuum tubing leading to a Stokes McLeod gauge is fixed to (H) so the pressure inside the source tube can be read at any time. A 6 mm quartz capillary about 14 cm long. is

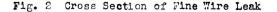
centered between the electrodes and aligned with the axis of the source tube. It is secured by glass wool packed tightly around it. The glass wool also serves to keep the discharge from going around the quartz tube.

The fine wire leak is composed of an 8 inch length of  $\frac{1}{4}$  inch inside diameter hose with a length of 36 B and S gauge wire in the center and a pinch clamp. One end of the wire is looped over the free end of the hose to prevent it from falling into the source tube. Fig. 2 shows a cross section of the leak with the clamp pinched down.

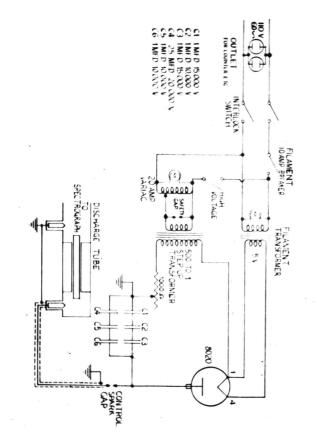
As the rubber tube is pinched down around the wire, the size of the leak (L) around the sides of the wire decreases. Enough pressure can easily be applied to the clanp to close the leak entirely. The leak can be adjusted to give any desired pressure in the source tube.

The source tube is supported by a bracket bolted to the spectrograph, and waxed into a collar directly in front of the slit.





The electrode nearest the spectrograph, the slit cover, the spectrograph and the source tube vacuum pump, are all grounded. The other electrode is connected to the lead from the power supply. This lead is a 20,000 volt cathode ray cable wrapped with two thicknesses of electrical insulating tape (7,000 volts per thickness) inside a grounded shield.



#### Fig. 3 Circuit for Power Supply

#### POWER SUPPLY

Figure 3 is the electrical circuit for the power supply. The entire power supply is mounted in a grounded steel case, 26 inches wide, 21 inches deep and 52 inches high. The case is divided into three sections by two shelves. A heavy wooden frame covered with 1/4 inch hardware cloth makes a door opening the entire front of the case except for the switch panel across the top. This provides excellent observation and ventilation of all three sections. The hardware cloth is grounded to the case through the hinges. The power transformer occupies the bottom section of the case. The middle section of the case contains the condenser bank and the variable resistor. In the top section is the variac, the auxiliary spark gap, the rectifier tube and its filament transformer. A photograph of the power supply is shown in Figure 4.

The 500 to 1 ratio, oil filled power transformer (W. M. Meyer Co., No. 1384-6) had been out of service and open to the air for some **time**; so both core and case were thoroughly cleaned and baked at 70° C. for eight hours, since contamination of transformer oil by as little as one hundred parts per million of water will decrease its dielectric strength by as much as 20%. <sup>20</sup>

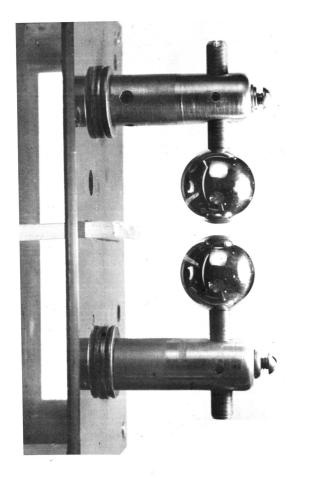
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20. Knowlton, A. E. (Editor) Standard Handbook
for Electrical Ergineers, 7th ed., N.Y.,
McGraw-Hil, 1941, pp. 400.
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Ten gallons of Wemco-C Insulating oil (Westinghouse Electric Corp.) were required to fill the transformer.

The transformer voltage is controlled by a 20 amp. variac (General Radio Co. V-20M) in the primary circuit. The variac is panel mounted on the left side of the case for easy accessibility from the spectrograph.

The line cord runs directly to an interlocking switch which opens when the door is opened. However, the two 110 volt outlets mounted in the switch panel are connected to the line in front of the interlocking switch. When the interlocking switch is closed, the 10 amp. circuit breaker switch can be closed, supplying power to the filament transformer and the power switch.

The filament transformer (United Transformer Co. LS-83) is insulated for 30,000 volts and





isolates the 8020 rectifier tube from the line.

The rectifier tube was selected because of its high peak plate current (.750 amp) and its high maximum plate voltage (40,000 volts).

First the tube was mounted in a conventional 4-pin tube socket insulated from the case by porcelain stand-off insulators, but corona discharge from the points on the tube socket was excessive. The tube socket with the tube mounted was immersed in a 3" x 3" x 1 1/2" wooden box of molten parafin and allowed to cool. The parafin surrounding the tube pins, socket and leads provided adequate insulation.

The auxiliary spark gap was first constructed with 1/4" pointed copper electrodes mounted in a wooden box, but a constant breakdown potential could not be maintained because of corona discharge between the electrode points. Also leakage around the gap through the box was excessive.

A new gap shown in Fig. 5 was constructed using chrome plated steel spheres 1 1/2" in diameter as electrodes. These were mounted on a sheet of 1/4" bakelite which was in turn mounted on four dowels screwed to a wooden base and insulated from contact with the grounded case by a sheet of plate glass. This gap has proved to be very satisfactory; although periodic cleaning with carbon tetrachloride has been necessary to prevent leakage across the surface of the bakelite. A slit 1/4" x 3" cut in the bakelite perpendicular to the axis of the electrodes seemed to decrease leakage by lengthening the surface path between the electrode mountings.

The condenser bank is composed of three one MFD condensers at, 10,000 volts, two one MFD condensers at 15,000 volts and one 0.25 MFD condenser at 20,000 volts. These condensers are all pyranol filled and are grouped to supply 0.5 microfarads at 40,000 volts. They are arranged in the case so that both ends of the bank may be grounded by a shorting bar that falls across them whenever the door is opened.

The condenser shorting device is a brass rod 12" long with a rectangular copper plate fastened at each end to form three sides of a rectangle. One side of a short strap hinge is bolted under the top shelf of the grounded

case and the other side is bolted to the center of the brass rod. When the door is opened the copper plates hang against the end terminals of the condenser bank. This keeps the ends of the bank grounded. When the door is closed a wooden block fastened to the inside of the door pushes under the brass rod, strikes the hinge, pushing it back and swinging the copper plates back and up away from the condenser terminals.

This shorting bar and the interlocking switch cut off all power and voltage in the upper section of the power supply and at the source tube. When the door is opened these parts are immediately accessible for adjustment. However, if any work is to be done in the center section, around the condensers, the middle condensers in each **bank must be grounded by a hot** stick since the condensers are not all just exactly alike.

The sparks are counted by two impulse counters. The first (Cenco No. 73511) counter counts each spark up to 100 sparks, and the second (Cenco No. 73510) counts each 100 sparks.

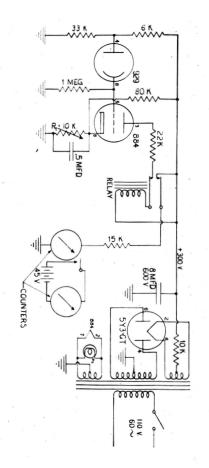


Fig. 6 Circuit for Counter

A 929 photoelectric cell is mounted on the outside of the case to shield it from the intense field around the spark gap. It is directly opposite the auxiliary spark gap and sees each spark through a 3/4" hole in the case. The impulse from the cathode of the photoelectric cell is fed to the grid of an 884 thyratron tube. The voltage applied to the grid is adjusted by the variable resistance  $R_1$  until the impulse from the photoelectric cell will just fire the tube. When the tube fires a current flows in the relay coil. The coil pulls in the armature, first closing the counter circuit and then opening the thyratron plate circuit, quenching the tube. After each 100 counts the first counter closes an external circuit, tripping the second counter. The power for the external circuit is supplied by a 45 volt "B" battery. The counter and battery are mounted in a case on top of the power supply.

The counter circuit is shown in Fig. 6.

#### OPERATION

The power supply for this source must be turned on by closing the switches in the following order. The interlocking switch must be closed first by closing and hooking the door; then the rectifier filament switch. After the rectifier tube has warmed up, the power switch can be closed and the source tube is ready to operate. This order is necessary to protect the rectifier tube from the high voltage until the cathode has had time to reach operating temperature.

The operation of the source is affected by four factors; the secondary transformer voltage, the setting of the resistor, the length of the air gap and pressure in the source tube.

The secondary transformer voltage is controlled by the variac. The scale on the variac reads the voltage applied to the primary winding of the transformer. This reading multiplied by  $500 \times 1.414$  gives the peak voltage across the secondary terminals of the transformer; the

maximum voltage to which the condenser bank may be charged.

The amount of resistance in the charging circuit determines the rate of charging of the condenser. If the resistance is increased, it takes longer for the condenser to build up to the peak voltage.

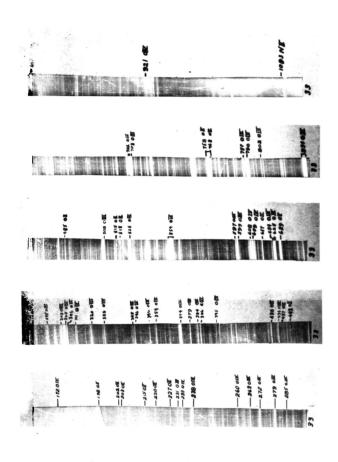
The pressure in the source tube and the length of the air gap limit the potential that the condensers will attain before discharging. If the combined resistance of the source tube and the air gap is too high, the condenser will charge to peak voltage but will not discharge. If their combined resistance is below a critical value, the condenser will discharge before the peak voltage can be attained.

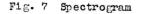
The pressure in the source tube should be maintained as a constant value close to 100 microns. At this pressure, the potential required to break down the gap is lowest and the auxiliary air gap can be adjusted to fire at the desired potential.

The pressure in the spectrograph must be as low as possible (not more than a few microns) to prevent absorption of the radiation. Therefore, in order to maintain a pressure of 100 microns in the source tube, a separate vacuum pump for the tube must be used in conjunction with a small leak. The pump used with this source is a Welch Duo-Seal 1405-H vacuum pump, and the leak is controlled by squeezing a rubber tube around a fine wire. The pump has a free air capacity of 33.4 liters per minute, and the leak is easily adjusted to maintain the constant pressure of 100 microns.

The desired sparking voltage can be attained by adjusting the air gap opening until the condenser will just discharge when the variac is set at the value calculated to produce this potential as peak voltage across the secondary of the transformer. Then the rate of sparking can be increased or decreased by adjusting the resistor.

The degree of excitation depends mostly upon the sparking potential and to a small degree upon the source tube pressure. Spectrograms were obtained showing O IV and O V lines intense from 762 Å down to 272 Å, and O II and O III intense from 834 Å down to 553 Å.





Many much fainter 0 IV and 0 V lines were observed down to 162 Å and 0 II and 0 III lines were observed as low as 303 Å. One of the spectrograms is shown in figure 7. Eastman Kodak Short Wave Radiation film was exposed to 50 sparks with oxygen in the source tube at 125 microns, the gap 8 turns open (0.20 inches), the variac at 25 volts and the resistor at 900 ohms. The measured voltage across the condenser bank was 17,000 volts just before discharge, and the sparking bate was 40 sparks per minute. The lines were identified by comparison with pictures published by Edlén.

### CONCLUSIONS

The light source which has been completed for use with a grazing incidence Vacuum spectrograph operates very satisfactorily at 17,000 volts. It produces intense radiation in the region from 100 Å to 1000 Å.

It is believed that many more O IV and O V lines will be observed below 200  $\mathring{A}$  if the pressure in the spectrograph is reduced.

If the power supply is ever to be operated at more than 20,000 volts it is suggested that all terminals be coated with anti-corona lacquer. Above that range corona discharge is distinctly audible, though not visible.

#### SUMMARY

A vacuum spark light source has been developed to be used with a grazing incidence vacuum spectrograph. The source operates at a pressure of 100 microns. A 17,000 volt spark is fired through a quartz capillary 40 times per minute, to produce intense radiation in the 100 Å to 1000 Å range. The voltage and rate of sparking are controlled by an auxilliary air spark gap, a resistor, and a condenser in the secondary circuit of a transformer.

Spectrograms have been obtained with this source showing lines below 200  $\stackrel{\circ}{A}$ . The degree of excitation that produced the most intense lines is 0 IV and 0 V.

This light source appears to be satisfactory for photographing emission and absorption spectra of crystals.

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#### VITA

Roy G. Woodle, Jr., was born July 5, 1924, at Springfield, Missouri. He attended grade school, junior high school, and in 1942 he was graduated from Senior High School, Springfield, Missouri.

In September, 1942 he enrolled in Drury College, Springfield, Missouri, as a freshman. In February, 1943, he was inducted into the United States Army and attended Niagara University from July 1943, until January 1944, in the Army Specialized Training Program.

After he was honorably discharged from the Army in March, 1946, he re-enrolled in Drury College. In September, 1947, he became a laboratory assistant in the Physics Department, and in June, 1948, he was graduated with a Bachelor of Science degree in Physics and Mathematics.

Since September, 1948, he has been employed as Instructor in Mathematics at the Missouri

School of Mines and Metallurgy, Rolla, Missouri; where, in January, 1949, he enrolled in the Graduate School as a candidate for a Masters degree in Physics.

In August, 1950, he was married to Miss Eugènia Covin of Springfield, Missouri.