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THE HOLLOW CATHODE SOURCE

By

RAYMOND CLARE LLOYD

A thesis submitted to the Graduate Committee of
South Dakota State College of Agriculture
and Mechanic Arts in partial fulfill-
ment of the requirements for the
degree of Master of Science.

1951

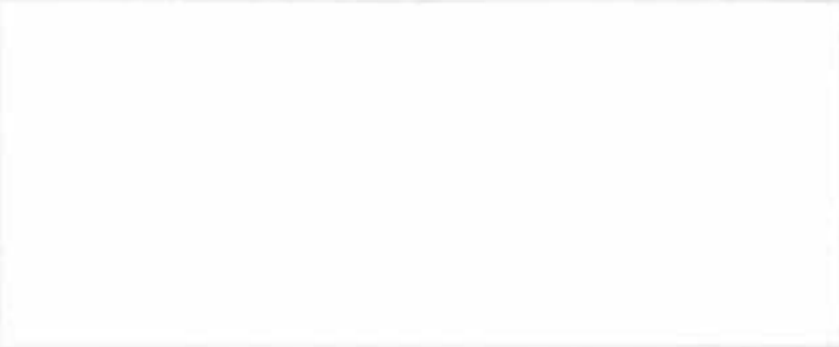
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Raymond Clare Lloyd

This thesis is approved as a creditable independent investigation by a candidate for the degree, Master of Science, and acceptable as meeting the thesis requirements for this degree, but without implying that the conclusions reached by the candidate are necessarily the conclusions of the major department.



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THE HOLLOW CATHODE SOURCE

CHAPTER I

INTRODUCTION

The hollow cathode glow was discovered by Paschen in 1916. He recognized its value as a source for general high resolution spectroscopy and did considerable work with it. Since his original work, many modifications in the details of the hollow cathode source have been made, but the essential discharge conditions as first found by Paschen remain unchanged. Schüler's change in the experimental arrangement to permit drastic cooling is the most important of these modifications. He was first to apply the source intensively to the study of hyperfine structures. The Schüler type source has contributed much to the rapid development of hyperfine structures.

Choice of a Source for Spectroscopic Study

The nature and rarity, atomic weight, vapor pressure, boiling point, ease of sputtering, chemical activity, region in which the spectrum lies, excitation and ionization potentials, and other factors concerning the

material to be studied have considerable influence upon the choice of an appropriate source. The discharge should be stable and constant over reasonably long periods of time so that study can be critical or a photograph of the spectrum can be taken with a spectrograph. The ideal light source should produce lines which are sharp, intense, and of constant brilliancy. The two main causes of the broadening of spectral lines in a gaseous discharge are the Doppler effect and the Stark effect.

The Doppler effect is due to the random motion of the emitting atoms. Neglecting other effects, if the atoms were at rest, the emission would be quite monochromatic with a frequency f . If v is the velocity of the atom and θ is the angle between the direction of v and the direction of observation, the frequency of the light will be changed by an amount df given simply by the classical expression

$$\frac{df}{f_0} = \frac{f-f_0}{f_0} = \frac{v \cos \theta}{c} = \frac{u}{c}$$

where f_0 is the frequency at $v = 0$, f is the observed frequency, $u = v \cos \theta$ is the component of the velocity in the direction of observation and c the velocity of light. Assuming a Maxwellian distribution of the velocities and that the intensity probability varies as the velocity probability, it can be shown that the half-intensity width will be given by the expression^a

a. See Appendix, p. 25.

$$w = .71 \times 10^{-6} \sqrt{\frac{T}{M}} f_0 \text{ cm}^{-1}$$

where T is the absolute temperature, M is the molecular weight and f_0 is the frequency at $v = 0$. This equation shows that the Doppler broadening is (1) proportional to the square root of the absolute temperature, (2) proportional to the frequency f_0 and (3) inversely proportional to the square root of the molecular weight.

The Stark effect is the splitting of the energy levels of an atom in an electric field. This splitting of the energy levels of an emitting atom results in a splitting of the spectral line. However, in an actual experiment using a gaseous discharge source, a broadening is usually observed since the various emitting atoms are in regions of different electric field strengths and the splitting for any given atom is dependent on the field strength.

Properties of the Hollow Cathode Source

The hollow cathode source is probably of more importance than any other for large scale analysis of the hyperfine structures. It is capable of yielding very intense and extensive spectra in which line widths can be reduced to moderately small values.

This source is also useful in the study of scarce

or rare samples since only a small amount of the solid sample is placed on the walls of the cathode to produce the emission lines of the sample. After studying the sample, it can be recovered from the cathode.

The mechanics of the discharge can be studied to explain why the hollow cathode source is quite free from the Stark effect. Cathode electrons, which have passed through the cathode drop in potential, continually create rare gas ions. These electrons being much lighter than the positive ions will have higher mobility, thus causing a resultant positive charge to remain in the region of the cathode. Therefore, this positive charge, the cathode drop of potential, and the mean free path of the cathode electrons are quite interdependent. In the hollow cathode discharge, the space charge is created by electrons from all azimuths, causing the resultant cancellation of the impressed field to be more uniform, giving a large field free space. This result has been found to exist experimentally by Schüler. Since there is a large field free space, the discharge results in emission lines which are almost entirely free from Stark broadening.

If the cathode is cooled by liquid air, this will lower the Doppler width and sharp lines will be radiated. This cooling can be accomplished efficiently when using

the Schüller type hollow cathode source since the metal cathode is part of the exterior portion of the source where it is accessible to the cooling agent.

Molecular spectra are absent in the liquid air-cooled source due to removal of the molecule from the discharge region through the process of adsorption at the cold metal cathode. This is especially true for the carbon monoxide molecules and to a limited extent for other molecules. Of course, a reasonably tight source is a necessity.

Ease in changing the cathode makes it possible to study and compare spectra of several different samples without great loss of time and movement of equipment.

CHAPTER II

PROBLEM

Since work is in progress at State College on the isotope shifts in atomic spectra it seemed desirable to construct such a source and investigate some of its properties for the excitation of atomic spectra. The problem was to build the source and the associated vacuum system to successfully operate for the study of spectral lines. To further check the operation of the source, it was used in the excitation of carbon and especially for study of the transition of carbon II $2s\ 2p^2\ ^2P_{\frac{3}{2}} - 2p^3\ ^2D_{\frac{5}{2}}$. This transition was of interest because an earlier study of the isotope shift in carbon¹ indicated that it might not be produced in a hollow cathode source. The lines that are expected from this transition in carbon II are $\lambda 2509.11$, $\lambda 2511.71$, and $\lambda 2512.03\ \text{\AA}$. The following energy level diagram shows the levels and possible transitions mentioned above.

1. Clyde R. Burnett, "Isotope Shift in the Atomic Spectrum of Carbon," Physical Review, LXXX (November, 1950), p. 494.

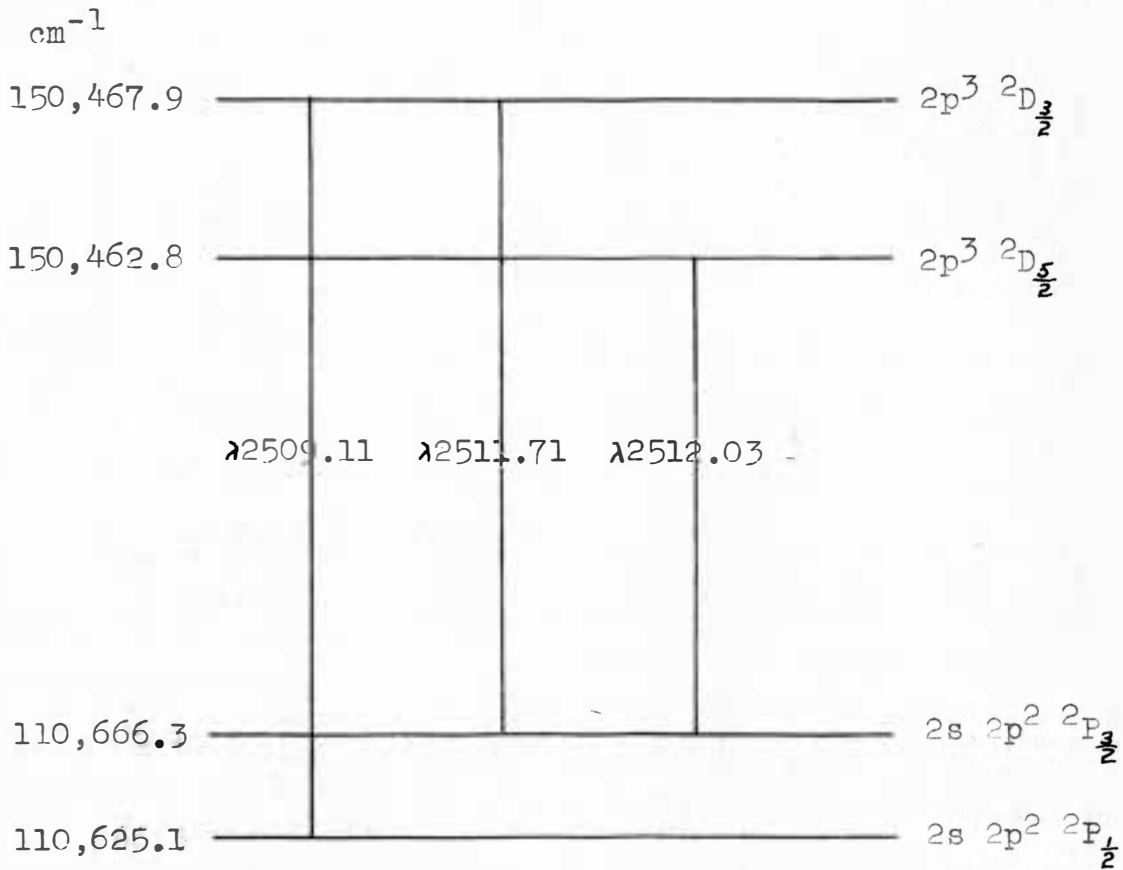


FIGURE I. ENERGY LEVEL DIAGRAM OF THE CARBON II
TRANSITION $2s\ 2p^2\ 2P_{\frac{3}{2}} - 2p^3\ 2D_{\frac{5}{2}}$

CHAPTER III

EQUIPMENT AND OPERATION

Description of the Source and System

The project was to design, construct, and test the source and associated vacuum system. The metal parts of the system were drawn up and the machine work carried out in the shop. The glass portion of the equipment was made in the laboratory and the entire system assembled. The glass portion of the system was checked for small leaks by using the tesla coil. If the pressure can be decreased sufficiently that a discharge can be started inside the glass system, the spark will follow any inlet of air so that a hole in the glass is easily noticed. The metal portion of the system was checked for leaks, in each section that was separated by valves, by first evacuating and then letting stand to see if air had leaked in. It was possible to detect large leaks by the sound of the pump.

The diagram of the source is shown in Figure 2. The source has an aluminum cathode A which is held against an iron knife edge B making a vacuum tight connection. A pyrex glass cylinder C two centimeters in diameter is inserted inside the iron cylinder D to shield the iron from the discharge. The glass is

sealed at E with black Apiezon hard wax where vapor pressure is of the order of 10^{-6} millimeters of mercury at room temperature. A quartz window F is placed on top of the glass cylinder and is also sealed with the sealing wax. Since the light of the source must pass through this window for study, a quartz window is used instead of a glass one so the ultra-violet light will not be absorbed. The anode N is a small aluminum cylinder fitted inside the glass tube at about a centimeter from the cathode, and a nichrome wire lead is brought out of the source through the glass T at G and sealed. Another glass connection H is used to connect the source to the rest of the system.

Figure 3 shows the vacuum system. The coil of glass I was used in order that the system would have a greater flexibility without breaking. The three-way stopcock J makes it possible to change cathodes without subjecting the charcoal trap K to the atmosphere, by evacuating the source through the by-pass line. This feature saves time since it takes about one-half hour to outgas the charcoal. At low temperatures charcoal has high absorption qualities. The small tank L holds a supply of helium, the carrier gas. A system of valves provides adequate control of the rare gas.

Vacuum System

As a general rule, vacuum systems for spectroscopic sources are quite elaborate so as to prevent contamination of the source with other spectra. The hollow cathode source uses a charcoal trap for this purpose. In cooling the source a purity of the spectrum was noted, since the clean-up effect of the discharge is accelerated at low temperatures of the cathode.

The system must be of a static type--that is, one which holds vacuum for long periods of time without constant pumping. It has been found that with a reasonably tight system and a small charcoal trap to absorb the residual air, a clean discharge can be maintained without forced circulation. The charcoal trap is outgassed by heating it to a low red heat for about one-half hour with constant pumping to remove the absorbed air which has been driven off by the heating. This allows more efficient absorption at low temperatures.

The rare gas must be regulated, so that it passes through the charcoal trap very slowly, in order to best purify it and more accurately control the source pressure. This regulation was accomplished by the use of Hoke needle valves. The bellows type valve

is more desirable for regulation of the source pressure because of less possibility for a leak to the atmosphere than in the packing type valve. The three-way stopcock is added to this simple system at the source entrance so that the cathode can be changed and pumped through the by-pass without exposing the charcoal trap to the atmosphere.

Source Operation

The source operates best at two to three millimeters of pressure and with a potential drop of 250 to 300 volts. A current of 100 to 200 milliamperes was generally used. When the current density in helium is over thirty milliamperes per square centimeter the cathode potential fall becomes "anomalous", giving rise to the Stark effect. The cathode area can be made larger for using higher currents. A cathode with inner area of about seven square centimeters would be needed to use 200 milliamperes.

The glow is concentrated in the hollow cathode space. Its brilliancy depends, in a critical manner, upon the gas pressure. When the pressure is lowered below the point of maximum brilliancy of discharge, the light intensity falls until the hollow cathode suddenly stops glowing. At this changeover position the tube resistance increases, the current falls to

a small value, and only a feeble positive column glow is emitted from the gas-filled space between the cathode and the anode.

The glow within the cathode is not uniformly distributed, but has an outside ring known as cathode glow, then a ring of Crookes dark space and a center of negative glow. The negative glow is brightest on the axis and falls off slightly towards the dark space.

Helium was used as a carrier gas for the discharge. To free the system of any impurities, the helium was filtered through the charcoal, which was kept at the temperature of dry ice by placing the trap in a bath of acetone and dry ice. The following table shows the absorption qualities of charcoal for several gases at 0° and minus 185° centigrade. Listed is the volume of the gas at standard conditions of temperature and pressure absorbed by a unit volume of charcoal.

TABLE I

ABSORPTION CAPACITY OF CHARCOAL²

<u>GAS</u>	<u>0°C.</u>	<u>-185°C.</u>
Helium.....	2	15
Hydrogen.....	4	135
Argon.....	12	175
Nitrogen.....	15	155
Oxygen.....	18	230
Carbon Dioxide.....	21	190

2. John Strong, Procedures in Experimental Physics, p. 105.

Charcoal purifies the helium since it absorbs other gases much more readily than it does helium. The source was placed in a cooling bath of acetone and dry ice to help with the absorption. When this is not done carbon monoxide bands appear with considerable intensity.

For taking spectrograms of the carbon spectra a thin coating of barium carbonate was packed on the wall of the hollow cathode space. The system was evacuated and the helium allowed to seep in slowly until a bright discharge appeared. The atoms which enter the discharge are removed from the cathode wall by ion bombardment, and to a much less extent by evaporation. A study of the maximum energy from cathode atoms and ions and the rare gas atoms has been made by Sawyer³. A process of secondary collisions is generally assumed between rare gas atoms or ions and the cathode atoms or ions. These ions entering the negative glow suffer collisions with rare gas atoms. The presence of rare gas atoms in the metastable state, and the transfer of the energies of the metastable rare gas ions to the cathode atoms fix the limiting conditions of excitation for the spectrum of the cathode material. In order for the excitation of the cathode ions or atoms to be possible, the available energy of the gas atoms

3. Ralph A. Sawyer, "Excitation Processes in Hollow Cathode Discharge," Physical Review, XXXVI (July, 1930), p. 44.

should be greater than the energy of the excited state of cathode atoms if secondary collisions are to cause the excitation.

The energies from metastable states $2s\ 3S_1$ and $2s\ 1S_0$ of helium are 19.72 and 20.51 electron volts respectively and the ionization energy is 24.58 electron volts. The ionization potential of carbon I is 11.26 electron volts. The energy of the excited state, $2p^3\ 2D_{3/2}$ in carbon II is 18.56 electron volts. If the ionized carbon were to collide with the metastable helium ions there would be enough energy to excite the carbon II state. The energy of the metastable helium is not sufficient to ionize and excite the carbon in one process, since this would take 29.82 volts. This process is probably accomplished in two parts: first, ionization, and second, excitation by secondary collisions.

The Spectrograph

The spectrograph used was a medium quartz spectrograph manufactured by Bausch and Lomb Optical Company. The lens is 50 mm. in diameter and has a focal length which varies from 498 mm. at $\lambda 2100\ \text{\AA}$ to 697 mm. at $\lambda 7000\ \text{\AA}$. The speed of the spectrograph is thus about $f/10$ at $\lambda 2500\ \text{\AA}$. The speed of the source is $f/7$. This allows a small amount of magnification, which is limited because

of the desirability of a minimum loss of light.

The Photographic Process

Kodak spectroscopic plates of type II-0 and size 2 x 10 inches were used for recording the data. This type of plate is sensitive in the ultra-violet region. Plates were developed for three and one-half minutes in Kodak D-19 developer, then rinsed thirty seconds, and placed in Kodak F-5 fixer for thirty minutes. This fixer is an acid hardening fixing bath. The plate is then rinsed in water for an hour and placed out to dry.

CHAPTER IV

DATA

Reproduction of the plates given in Figure 4 shows the transition of carbon as compared to the iron and helium spectra. Measurements were taken using the traveling microscope comparator. Dispersion was found by linear interpolation between known iron lines. The following table compares the measurements taken from the plates to the standard values.

TABLE II

WAVE LENGTHS FOR THE CARBON TRANSITION
 $2s \ 2p^2 \ 2P_{\frac{3}{2}, \frac{1}{2}} - 2p^3 \ 2D_{\frac{5}{2}, \frac{3}{2}}$

<u>STANDARD</u>	<u>MEASUREMENT AS COMPARED TO HELIUM SPECTRA</u>	<u>MEASUREMENT AS COMPARED TO IRON SPECTRA</u>
2509.11	2509.22	2509.25
2511.71	none observed	none observed
2512.03	2512.22	2512.21

Discussion of the Data

Variations in the current between 100 and 200 milliamperes were tried with no apparent difference in the intensity of the lines under consideration. Exposure times between ten and thirty minutes were tried with the longer exposures having the greater intensity.

The source was water cooled and the charcoal trap was not used for photographing the carbon spectra, since good results appeared without it. In obtaining the helium spectra, it was necessary to pump out the source for several hours and filter helium through the charcoal trap, with the trap and the source at the temperature of dry ice. If this procedure were not followed carbon monoxide bands would appear and the helium line $\lambda 2511.22 \text{ \AA}$ would not appear.

The carbon line $\lambda 2511.71 \text{ \AA}$ which was expected in this transition did not seem to appear. Its brightness was not sufficient to be photographed on this plate. When applying the sum rules^b for line intensities to this transition the relative intensities for the lines $\lambda 2509.11$, $\lambda 2511.71$ and $\lambda 2512.03 \text{ \AA}$ are found to be 5, 1, and 9 respectively. The plates show that the longer wave length line is about twice as intense as the shorter wave length line. Since the middle line is much less intense it is quite possible that it would not be visible on the plate. The carbon line $\lambda 2478 \text{ \AA}$ was about fifteen times, and the $\lambda 2837 \text{ \AA}$ line about three times as intense as the $\lambda 2512.03 \text{ \AA}$ line in the transition being studied.

b. See Appendix, p. 27.

CHAPTER V

SUGGESTED IMPROVEMENTS IN SOURCE

After experience with this source, it appears that changes in the design of the system would provide advantages in checking for leaks. If the metal system could be pressurized to check for leaks, one metal to glass connection used instead of two, and if this connection were placed farther from the charcoal trap where it would not become heated when outgassing the charcoal trap, the system would be more efficient. This could be accomplished by using metal tubing to a point above the source. In this way by disconnecting the system at the metal to glass connection, the metal part of the system could be pressurized and checked for leaks by painting it with a soap solution.

CHAPTER VI

SUMMARY AND CONCLUSIONS

1. The hollow cathode source and the associated vacuum system was planned and constructed.
2. The system was checked for leaks.
3. The source was operated with barium carbonate in the cathode to produce the carbon lines to be studied. Helium and iron spectra were photographed on the plates next to the carbon spectra for comparison of wave lengths.
4. Measurements were made by comparing the carbon lines to helium and iron lines as standards.
5. This data shows that carbon lines of the transition $2s\ 2p^2\ ^2P_{\frac{1}{2}} - 2p^3\ ^2D_{\frac{3}{2}}$ can be produced using the hollow cathode source.

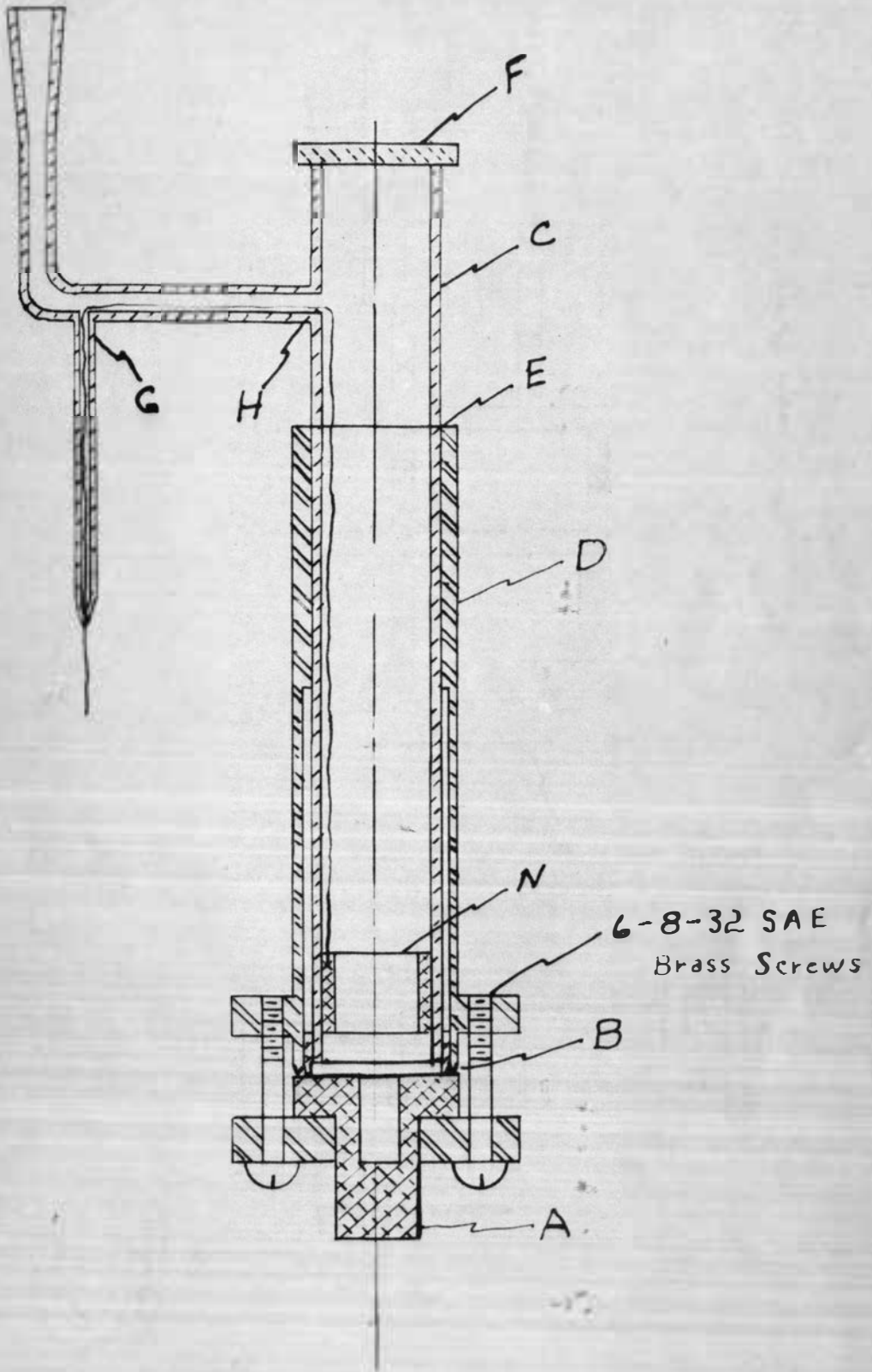
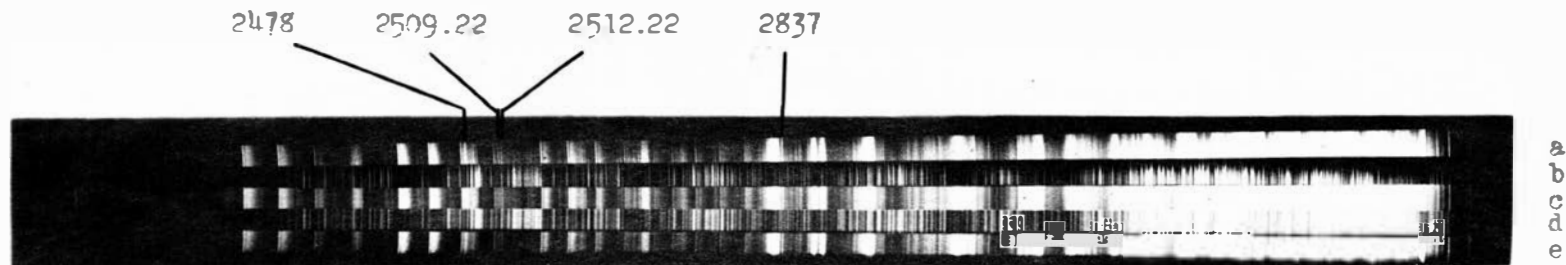


Fig. 2 THE SOURCE

- a. Carbon spectrum showing the carbon transition $2s\ 2p^2\ 2P_{1,1} - 2p^3\ 2D_{3,3}$ with lines $\lambda 2509.22\ \text{\AA}$ and $\lambda 2512.22\ \text{\AA}$. Also carbon lines $\lambda 2478\ \text{\AA}$ and $\lambda 2837\ \text{\AA}$ are marked. Exposure time is 15 minutes.
- b. Iron comparison spectrum.



- c. Carbon spectrum, 25 minutes exposure.
- d. Iron comparison spectrum.
- e. Carbon spectrum, 20 minutes exposure.
- f. Helium comparison spectrum with line $\lambda 2511.22\ \text{\AA}$ marked.
- g. Carbon spectrum.
- h. Helium comparison spectrum.
- i. Carbon spectrum.
- j. Helium comparison spectrum.

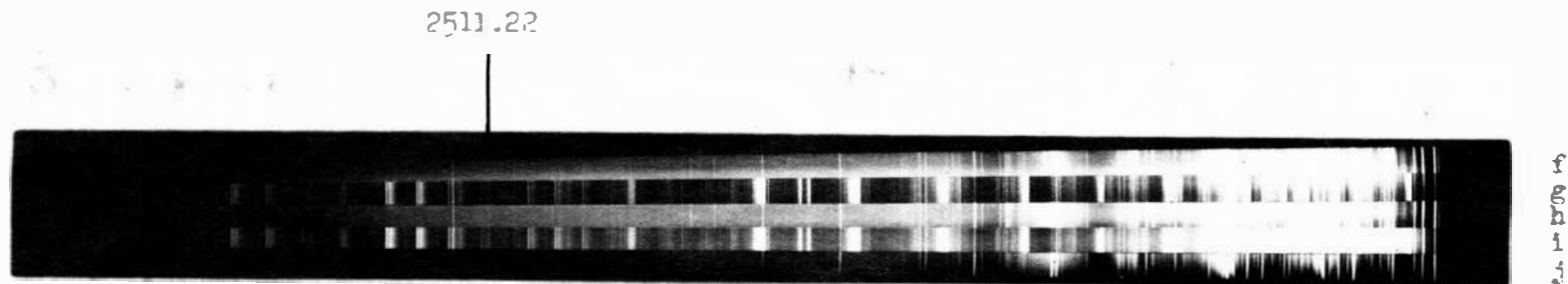


Figure 4 SPECTROGRAMS

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APPENDIX

a.

The Theory of the Doppler Broadening

If v is the velocity of the atom and θ is the angle between the direction of v and the direction of observation, the frequency of the light will be changed by an amount df given simply by the classical expression

$$\frac{df}{f_0} = \frac{f-f_0}{f_0} = \frac{v \cos \theta}{c} = \frac{u}{c}$$

where f_0 is the frequency at $v = 0$, f is the observed frequency, and $u = v \cos \theta$ is the component of the velocity in the direction of observation and c the velocity of light.

Assuming a Maxwellian distribution of the velocities the probability that the velocity will be between u and $u + du$ is given by

$$dv = \sqrt{\frac{M}{2\pi RT}} e^{-\frac{Mu^2}{2RT}} du$$

where M is the molecular weight, R the universal gas constant, and T the absolute temperature.

The probability of the observed frequency being between f and $f + df$ will vary as the probability of the velocity and can be written as the intensity

$$I = \text{const.} e^{-\frac{Mu^2}{2RT}} du$$

Substituting $u = c \frac{(f-f_0)}{f_0}$ gives the intensity as a function of frequency f .

$$I(f) = \text{constant } e^{-\frac{Mc^2 (f-f_0)^2}{2RTf_0^2}}$$

To find where intensity falls to half its maximum value the exponential term is set equal to one-half. Solving this for $f-f_0$ and multiplying by two we get the half-intensity width.

$$e^{-\frac{Mc^2 (f-f_0)^2}{2RT f_0^2}} = 1/2$$

$$\frac{Mc^2 (f-f_0)^2}{2RT f_0^2} = \log 2$$

$$(f-f_0) = \frac{f_0}{c} \sqrt{\frac{2RT}{M} \log 2}$$

$$w = 2 (f-f_0)$$

$$w = 2 \sqrt{\log 2 \frac{2RT}{M}} f_0 \text{ cm}^{-1}$$

Substituting for known constants gives

$$w = .71 \times 10^{-6} \sqrt{\frac{T}{M}} f_0 \text{ cm}^{-1}$$

b.

The Sum Rules

(1). The sum of the intensities of all lines of a multiplet which start from a common initial level is proportional to the quantum weight $(2J + 1)$ of the initial level.

(2). The sum of the intensities of all lines of a multiplet which end on a common level is proportional to the quantum weight $(2J + 1)$ of the final level.