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Variation of sensitized fluorescence with penetration depth of incident 2537A radiation

George Grayson Robinson

VARIATION OF SENSITIZED FLUORESCENCE WITH PENETRATION DEPTH OF INCIDENT 2537A RADIATION

BY

GEORGE GRAYSON ROBINSON

AN

ABSTRACT

Submitted to the faculty of the

SCHOOL OF MINES AND METALLURGY OF THE UNIVERSITY OF MISSOURI

in partial fulfillment of the requirements for the

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An investigation of the sensitized fluorescence of a thallium and mercury mixture using a variable depth cell and photomultiplier type of detection is discussed. Data are given to indicate the variation of the intensity of the 3519A thallium line as a function of thallium temperature, as a function of mercury temperature, and as a function of cell depth. Explanations of results require the use of generally accepted ideas concerning energy transfer, emission, and absorption.

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Approved by

Richard Anderson (Advisor) John D. Stettler

Ralph E. Lee S J Pagano

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I. INTRODUCTION

A. <u>History of Sensitized Fluorescence</u>. Sensitized Fluorescence was discovered in 1923 by Cario and Franck.[1] Early research on sensitized fluorescence uncovered three basic items of information:

1. When a mixture of two monatomic gasses, A and B, is illuminated by light which only A can absorb, fluorescent light may be emitted by B atoms. If the excitation energy of B is less than that received by A in absorbing the light, the intensity of B fluorescence is much greater than in the reverse case when the excitation energy of B is greater than the energy of light absorbed by A. Hence, atoms may transfer excitation energy on collision, the energy difference going into or resulting from thermal energy.

2. B atom fluorescent intensity depends upon the energy difference between that of excited A atoms and excited B atoms. The intensity is greater for smaller energy difference. This effect for sodium and mercury is credited to Beutler and Josephy.[2] Hence, the probability of transfer is greater for smaller energy difference.

3. Wigner[3] first stated the rule concerning the intensity of lines in sensitized fluorescence, that those transitions are most probable for which the change in electron spin angular momentum for one atom is equal and opposite

to that of the other atom.

Others have made additional contributions to the knowledge of sensitized fluorescence. For example, Winans[4] extended Wigner's selection rule. The rule stated by Wigner for sensitized fluorescence may be written as $\Delta S=0$, where S is the total spin angular momentum for the quasi-molecule formed by the two atoms on collision. Winans showed that the probability of transfer is greater if $\Delta S=0$ and $\Delta J=0$, where J is the resultant total angular momentum of the quasi-molecule formed by the two atoms on collision.

B. Events Leading to the Problem. In an experiment by Swanson and McFarland[5] on sensitized fluorescence, concerning mainly the role of mercury metastable atoms in a mercury-thallium mixture, the temperature dependence was investigated. In order to follow the discussion, an energy level diagram of mercury and thallium is shown in Figure 1, page 4. Their investigations showed the following results:

1. The resonance lines of thallium, 3776A, 2768A, and 2580A were seen with low intensities at very low mercury temperatures. Their intensities increased with temperature, reached a maximum between 300-350°C, and then decreased above 350°C.

2. Thallium lines 5350A, 3519A, 3529A, 3230A, and 2921-18A increased in intensity at low mercury temperatures, (120°-170°C), and decreased at higher temperatures, where the previous lines reached a maximum intensity.

Swanson and McFarland explained the behavior of the intensity of the thallium lines as follows:

1. The initial rise in intensity of the thallium spectral lines is anticipated with increasing mercury vapor pressure, because all incident resonance radiation penetrates deeply into the absorption cell, and is absorbed by the mercury.

2. The decrease in intensity of the non-resonance thallium lines, 5350A, 3529A, 3519A, 3230A, and 2921-18A is

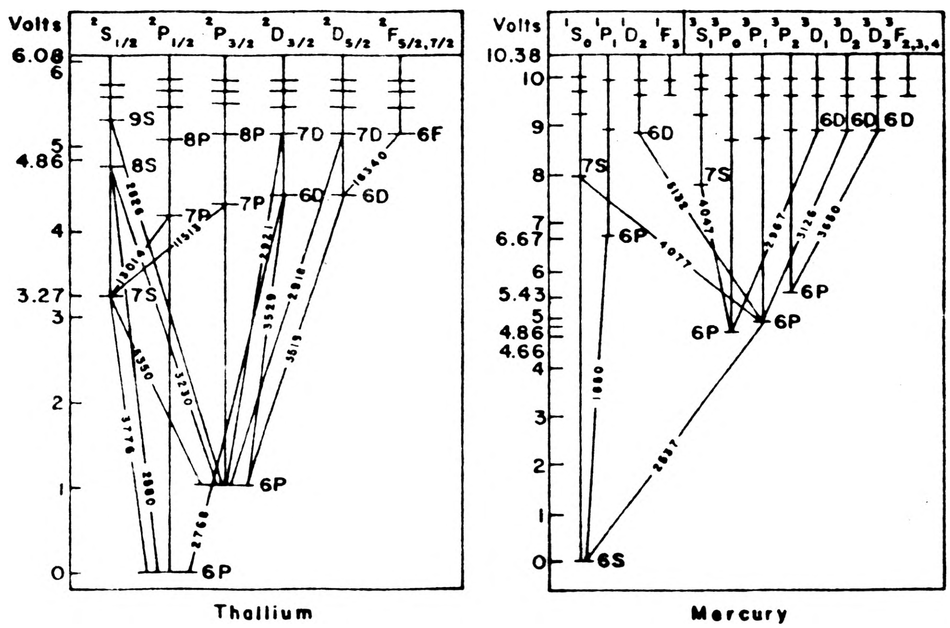


Figure I Reproduced from Anderson and Mc Farland[9] due to the effects of specular reflection in the mercury vapor as well as to an unfavorable ratio in the number of mercury atoms available to the number of thallium atoms present.

Using Winan's partial selection rule[4], the $7^2S_{\frac{1}{2}}$ thallium level would be preferentially excited by metastable mercury atoms. The increased population of 6^3P_0 atoms with increased mercury temperatures would decrease the number of 6^3P_1 atoms thus accounting for a decrease in intensity of the 3519A, 3529A, 3230A, and 2921-18A lines which are in close energy resonance with the 6^3P_1 level or are preferentially excited by this mercury level according to Winan's $\Delta J=0$ rule. There is a slight leveling off of the 5350A line in its fast decrease which can be accounted for by increased metastable atom concentration, since this line also originates from the $7^2S_{\frac{1}{2}}$ level.

3. Increased metastable atom formation in the cell at higher temperatures increased the intensity of the 3776A thallium line. If the $7^2S_{\frac{1}{2}}$ level is excited by direct collisions with metastable atoms a large amount of energy is converted to kinetic energy which will give a Doppler broadening to the 3776A line, so only its central portion is absorbed and the line is enhanced.

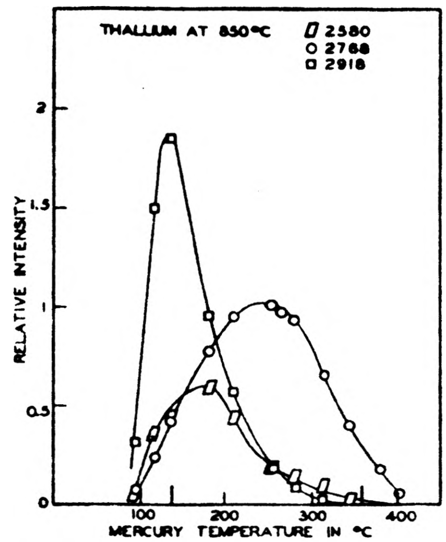
Also in the investigation by Swanson and McFarland[5], there was observed to be a shift in the maximum intensity of the 2768A and 2580A thallium lines toward higher mercury temperatures which cannot be explained by metastable atom formation; for the levels from which these lines originate are preferentially excited by 6^3P_1 mercury atoms. Therefore, the shift in the maximum intensity of these lines toward higher mercury temperatures has never been satisfactorily explained.

C. <u>Statement of the Problem.</u> If concentration factors, imprisoned resonance lines, specular reflection, metastable atom formation, and Doppler broadening are introduced, (with proper controls), the results obtained by Swanson and McFarland,[5] presented on pages 3, 5, and 6, can be explained more satisfactorily and completely with the proper experimental support. If metastable atom formation and Doppler broadening are of less significance, and changes in concentration, imprisonment of resonance radiation and specular reflection prove dominant, then the results obtained in all other experiments on sensitized fluorescence will be altered. The following analysis of these effects was proposed by Dr. Richard Anderson.[6]

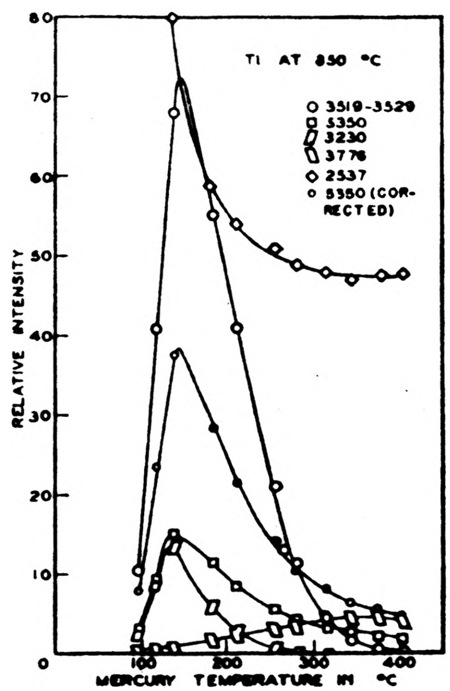
The expected initial increase in intensity of the 5350A, 3519A, 3529A, 3230A, and 2921-18A thallium lines will be caused by a mercury concentration effect. At low mercury temperatures, (hence pressures), the incident 2537A radiation penetrates deeply into or through the absorption cell, since the mercury atom concentration is not great enough to absorb all the incident radiation. As the mercury temperature increases, the concentration of mercury atoms reaches a point where all incident radiation is absorbed in the cell. Here the 3529A, 3519A, 3230A and 2921-18A lines will reach a maximum intensity since these lines originate from levels in close resonance with 6^3P_1 mercury atoms.

The 5350A thallium line should also show a maximum intensity. Though this line originates from the $7^2S_{\frac{1}{2}}$ level lying out of resonance, the $7^2S_{\frac{1}{2}}$ thallium level is populated by cascade transitions from the 7^2P and 6^2D thallium levels, which are in direct energy resonance with 6^3P_1 mercury atoms. The 3776A line should not exhibit a large intensity at low temperature, even though it originates from the $7^2S_{\frac{1}{2}}$ level, as it is a resonance line of thallium and is strongly self-absorbed when deep penetration occurs. This absorption depends on the thallium temperature. The 2768A and 2580A thallium lines originate from energy levels preferentially excited by 6^3P_1 mercury atoms. These lines should have weak intensity at low pressures because they are resonance lines and are also strongly self-absorbed at low mercury temperature. In order to follow these transitions, refer to Figure 1. The decrease in intensity of the 5350A, 3529A, 3519A, 3230A, and 2921-18A lines comes about because the incident radiation can not now penetrate as deeply into the cell before it is absorbed due to the increased mercury concentration. Therefore, the number of thallium atoms which can be excited is greatly reduced and the above spectral lines should be reduced in intensity. As the excitation region comes closer to the surface of the cell, the 3776A, 2768A, and 2580A thallium lines exhibit an increase in intensity, since the escape path length is decreased for these resonance lines, and they may escape with decreased self-absorption. The 2768A and 2580A lines should exhibit a maximum intensity when the energy exchange reaction occurs as a surface effect. Beyond this point, these lines should drop in intensity for the mercury pressure becomes so great that the incident radiation is totally reflected from the absorption cell as if the surface were a mirror, (specular reflection). The 3776A thallium line should show similar behavior, but the point at which this line decreases in intensity is at a slightly larger mercury temperature than for the 2768A and 2580A lines. The intensity behavior of this line and the slowing of the decrease in intensity of the 5350A thallium lines at high mercury temperatures is thought to be caused by an increased concentration of metastable mercury atoms at high temperatures. This line originates from the $7^2S_{\frac{1}{2}}$ level with a large energy difference with the 6^3P_0 mercury state, and as a result is Doppler broadened in the excitation process. This can account for its longer persistence. Figure 2 is a reproduction of graphs of Swanson and McFarland[5] which shows these effects.

The purpose of this experiment is to verify the above hypotheses which differ from those of Swanson and McFarland.[5] One step in this direction is the performing of an experiment to study the sensitized fluorescence spectrum as a function of penetration depth of the resonance radiation. An absorption cell containing a plane window through which the excitation radiation is incident and the fluorescent radiation is emergent was used. The cell contained a baffle to



Sensitized fluorescence intensities of the three least intense lines observed.



Sensitized fluorescence intensities as a function of mercury temperatures with the thallium temperature at 850°C. (5350 line corrected for phototube response.)

Figure 2 Reproduced from Swanson and Mc Farland[5] restrict the depth of penetration. This cell was used to repeat the work on the temperature dependence of the sensitized fluorescent spectrum of a mercury-thallium mixture.

Swanson and McFarland[5] used a cell at a constant depth, varied the mercury temperature and kept the thallium temperature constant. The author used a cell of variable depth, varied the mercury temperature and cell depth, and kept the thallium temperature constant. A plot of the relative intensity of thallium lines versus different cell depths at different mercury temperature should produce different results for different thallium lines. For the 3519A, 3529A, 3230A, and 2921-18A, thallium lines, the intensity is expected to increase initially, then to break or drop in the rate of intensity increase, and possibly level off to a constant intensity value. Depending on the mercury temperature, the rate of intensity increase will vary, and the cell depth where the break in intensity occurs will vary. If the mercury temperature is high, the rise in intensity of these lines should be rapid, and the break will occur at small cell depths. The intensity of the line will be low. At low mercury temperature the rise in intensity may be slower. The intensity of the line where the break occurs may be higher, depending upon the mercury temperature. The break in intensity will then occur at a deep penetration. Using the data of Swanson and McFarland[5], our graphs for these lines should appear as shown in Figure 3.

The 5350A thallium line will probably exhibit a different behavior than the 3519-29A, 3230A and 2921-18A lines. Its intensity is controlled by cascade transitions into the $7^2S_{\frac{1}{2}}$ level, and by excitation of this level by mercury metastable atoms. Its intensity may rise more rapidly than the other lines, and the break in the intensity will probably be less pronounced because of metastable atom concentration. The anticipated behavior of the 5350A intensities are shown in Figure 4.

The resonance lines 2768A and 2580A will have low intensities except at high mercury temperatures. Their intensities will break and become constant at nearly the same cell depth for all mercury temperatures, for they are self-absorbed by the constant thallium vapor pressure in the cell. Their intensity will depend upon the mercury temperature, and the break point depth upon the thallium concentration. Applying Swanson and McFarland's[5] data to our proposed problem, the curves anticipated appear in Figure 5.

The 3776A line should have an intensity behavior similar to the 2768A and 2580A lines, except that the break in intensity should be less pronounced, since this line originates from the $7^2S_{\frac{1}{2}}$ level. This level is predominately excited by metastable mercury atoms, and this will lead to a Doppler broadening of the 3776A line. Thus, even though the line is a resonance line of thallium, the intensity may continue to rise above the break point of the 2768A and 2580A lines. In the case of the 3776A line, the break point may be more pronounced at low mercury temperatures where the concentration of mercury metastable atoms is negligible. At low mercury temperature, the 3776A line will probably exhibit intensity behavior similar to the other resonance lines as shown in Figure 6.

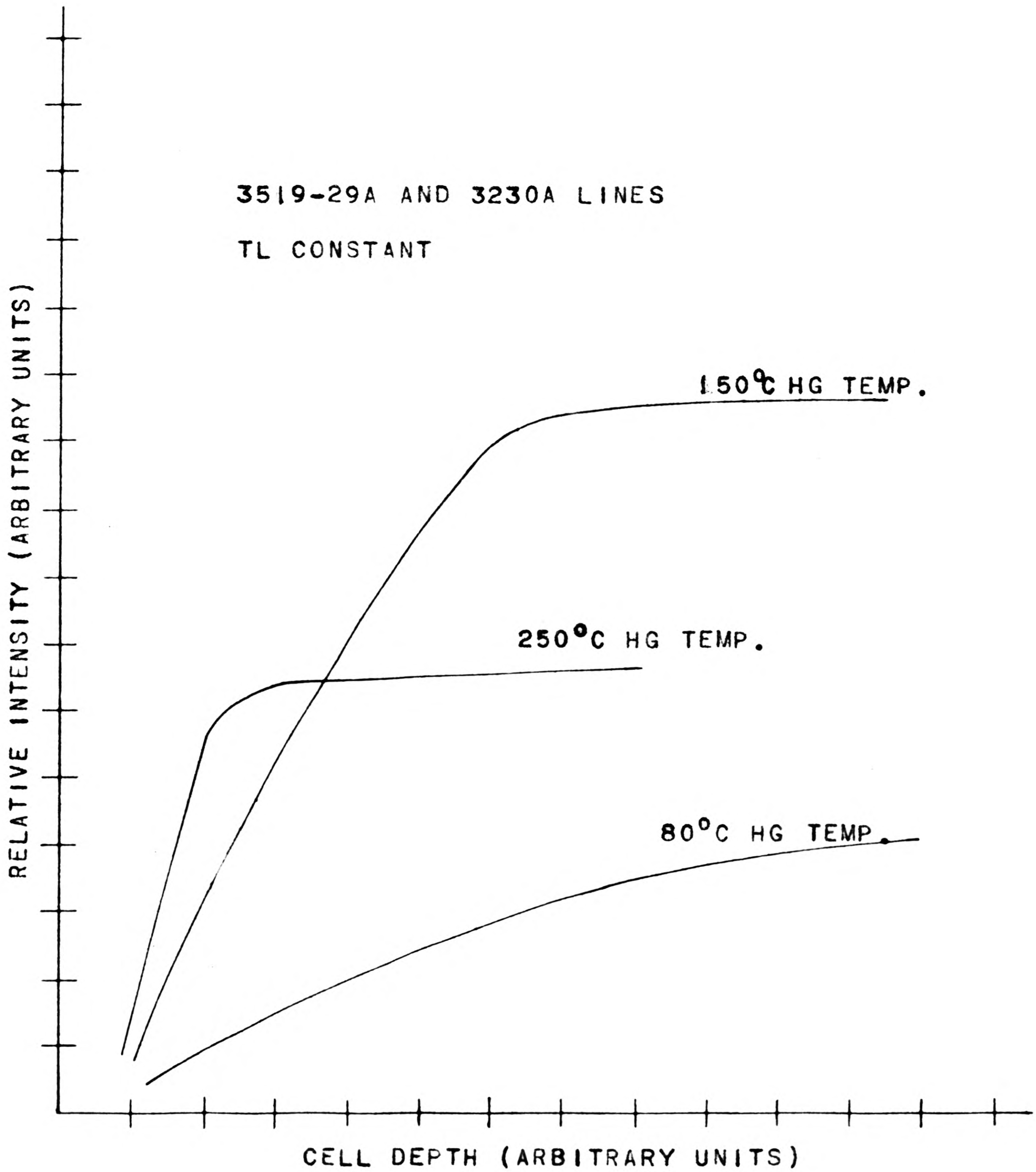


FIGURE 3. EXPECTED RESULTS BASED ON DATA OF SWANSON AND McFARLAND[5]

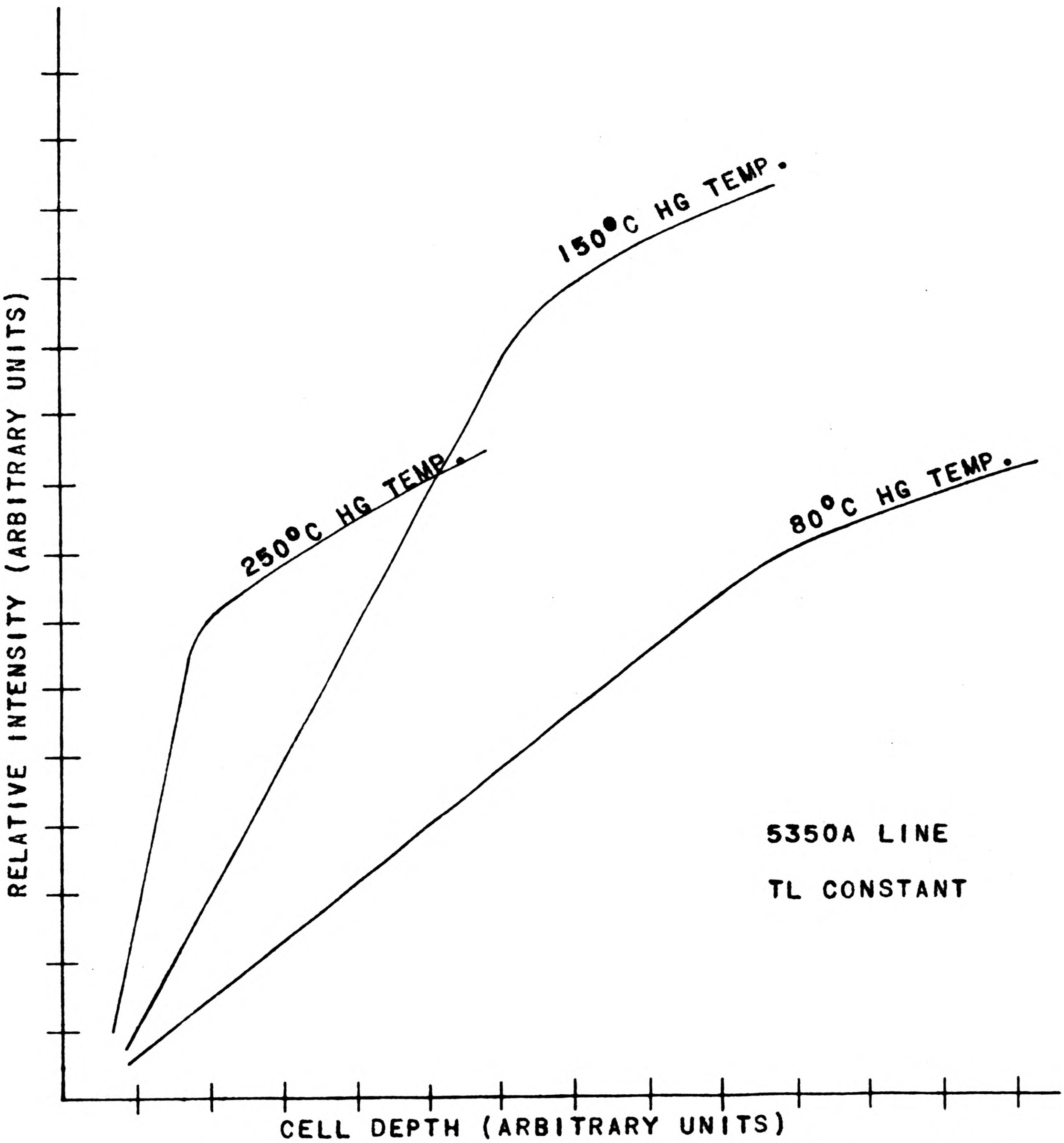


FIGURE 4. EXPECTED RESULTS BASED ON DATA OF SWANSON AND McFARLAND[5]

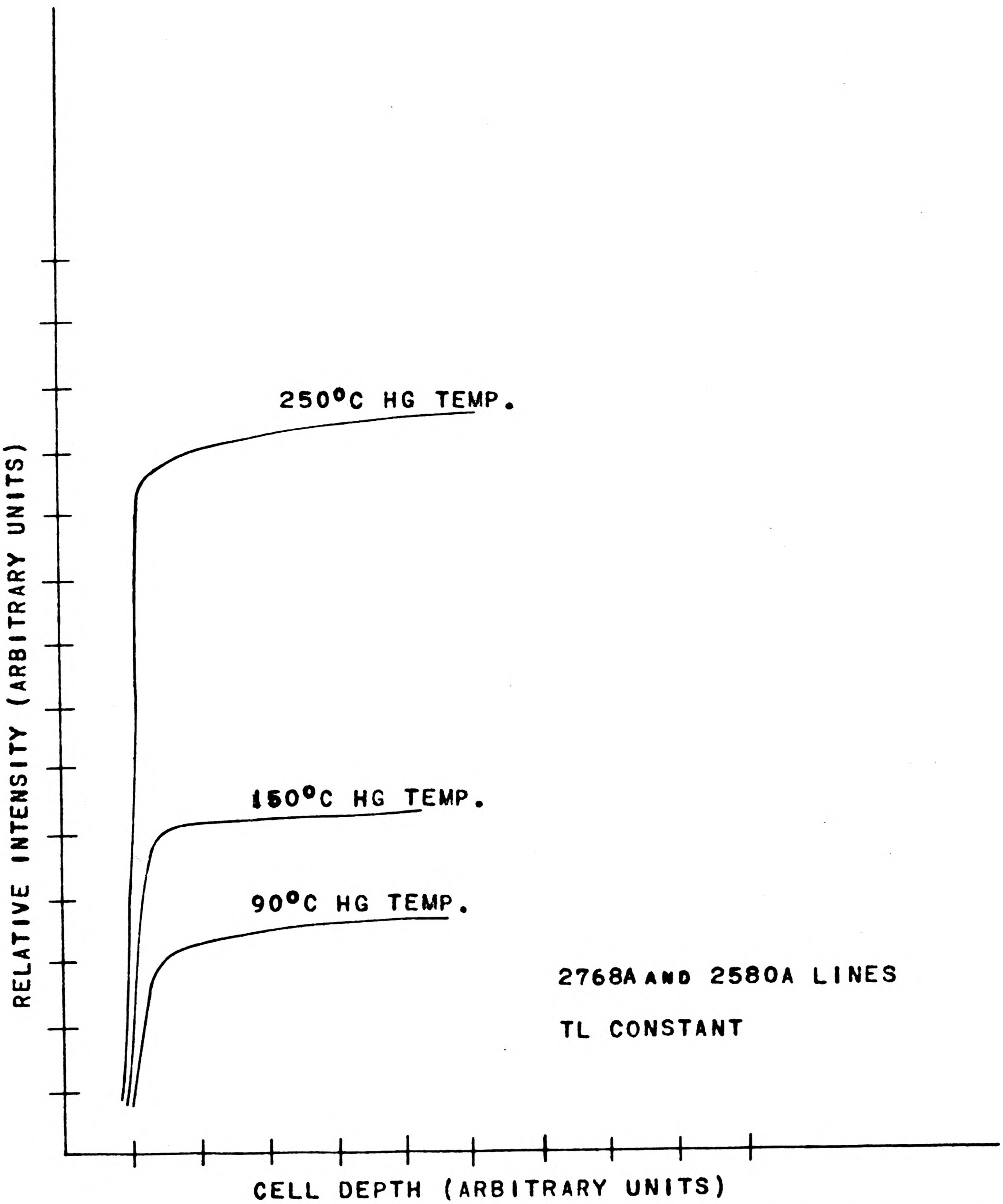


FIGURE 5. EXPECTED RESULTS BASED ON DATA OF SWANSON AND McFARLAND[5]

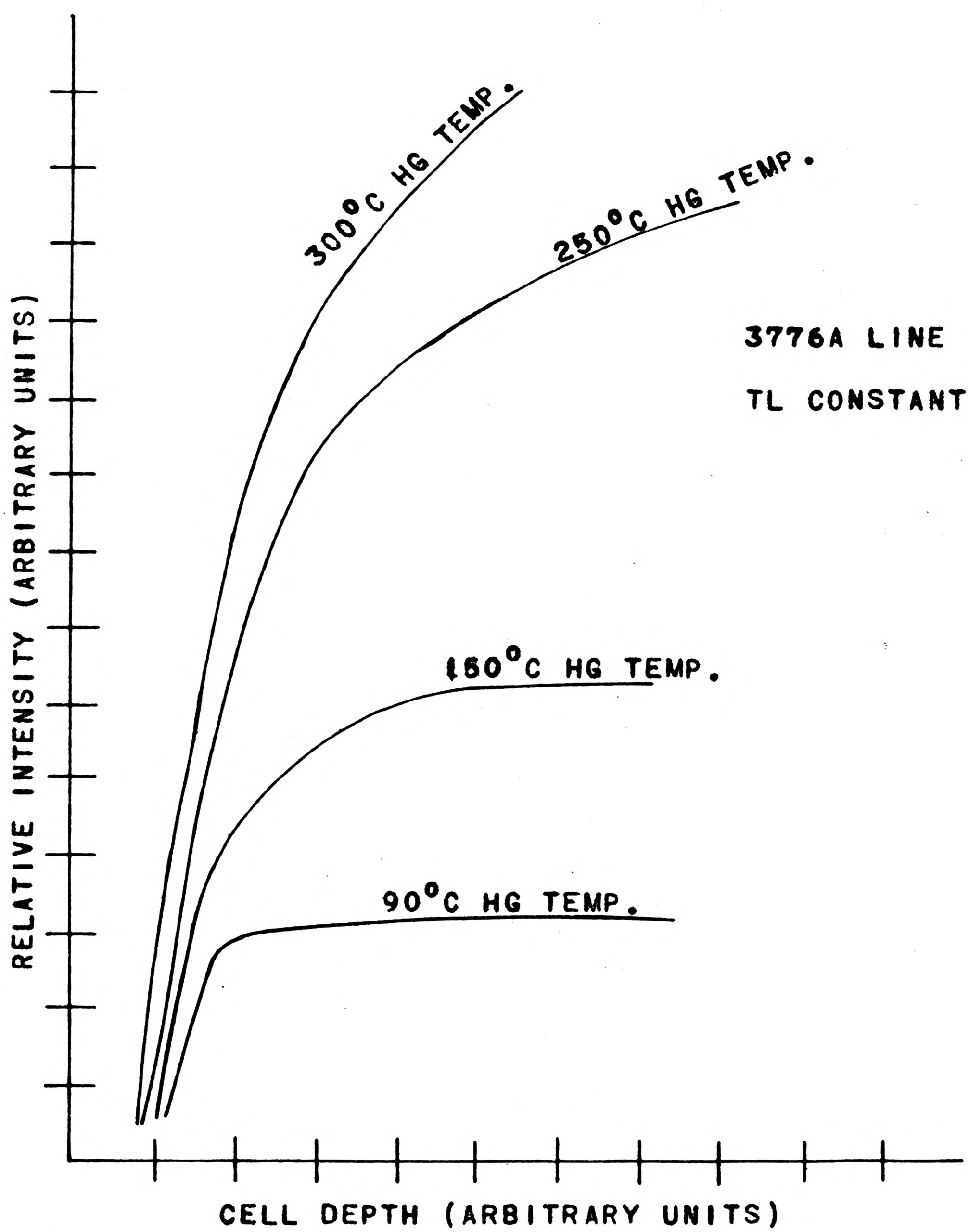


FIGURE 6. EXPECTED RESULTS BASED ON DATA OF SWANSON AND McFARLAND[5]

II. EXPERIMENTAL PROCEDURES

A. Construction of the Vacuum System. Plates I, 2, 3, pages 19, 20, and 21, show the vacuum system constructed and used by the author in connection with the experiment. The following items of equipment were used:

a. H. F. Martin mercury diffusion pump,

b. A. S. Aloe Duo Seal vacuum fore pump,

c. Consolidated Electrodynamics ionization vacuum gauge,

d. Consolidated Electrodynamics thermo-couple vacuum gauge.

A dry ice and acetone mixture was used in dewar flasks on the cold traps.

B. Design and Construction of the Quartz Cell. A number of considerations determined the final cell structure:

I. The cell had to be long enough to provide for movement of an interior baffle over a desired range of at least eight centimeters. Various positions of the baffle would then be possible throughout the eight centimeter range for data purposes.

2. The mercury and thallium reservoirs were located on each side of the main cell for practical convenience in oven design.

The cell was constructed of I" diameter quartz tubing and ½" quartz tubing was used for the reservoirs.

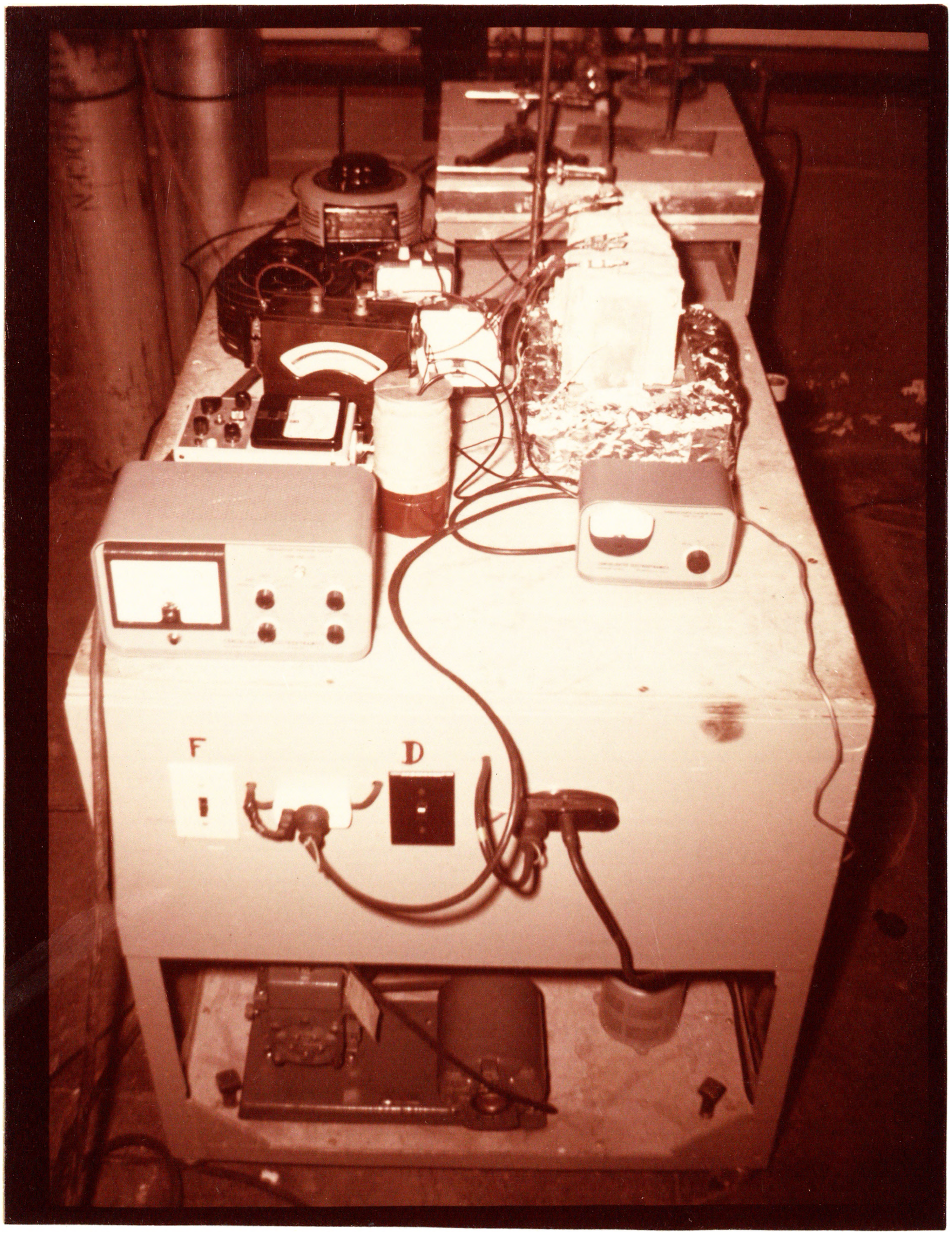


Plate 1. Vacuum system, front view, when cell is being outgassed

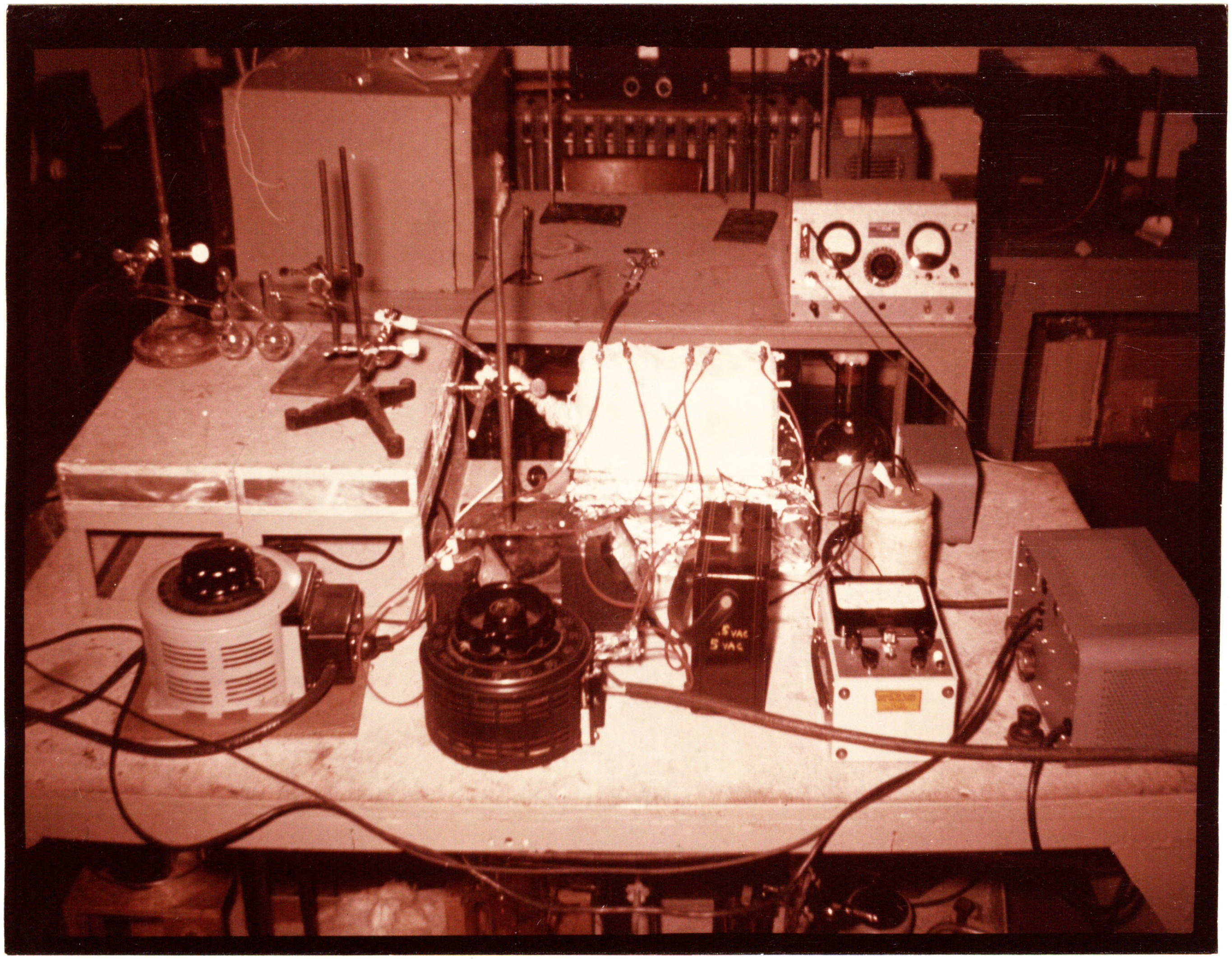


Plate II. Vacuum system, side view, when cell is being outgassed

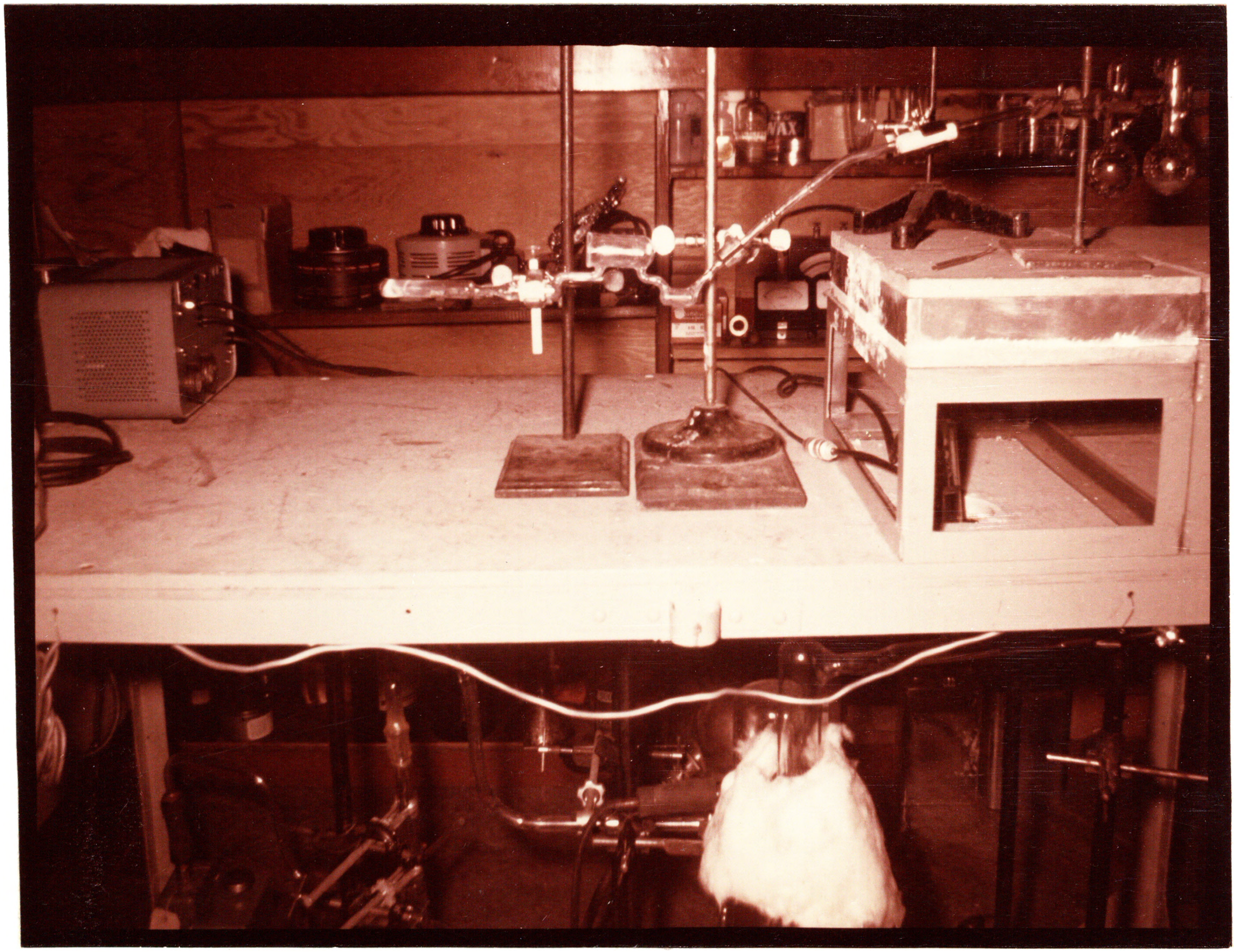


Plate III. Vacuum system with cell attached

C. Design and Construction of the Baffle. Pure molybdenum sheet metal $\frac{1}{2}$ mm. thick was selected for the construction of the baffle, since molybdenum has a much higher melting temperature than was used in the experiment, and does not react with the mercury and thallium metals and their respective vapors.

Gloves, washed tools, and distilled water and alcohol washes were used to prevent contamination of the metal.

Two short iron slugs,(about $\frac{1}{2}$ mm.), were embedded individually in quartz. These were then placed within the baffle, sealed in by the ends of the baffle cylinder. The baffle was then placed inside the main cell. Finally, the quartz end-window was sealed onto the front of the cell.

The baffle was moved with a magnet to any desired location within the cell, thereby making possible a cell of variable thickness throughout a range of 10.8 cm.

D. Design and Construction of the Main Oven and Oven Mount. Plates 4 and 5 illustrate the design features incorporated into the main cell oven in order to provide for rigid clamping of the cell externally from the oven, and at the same time to allow the oven to be removed from over the cell without moving or disturbing in any way the position of the cell.

The author began construction by selecting a ceramic core of suitable size, which he wound with lathe-made wire elements. After tying them with string to hold them in

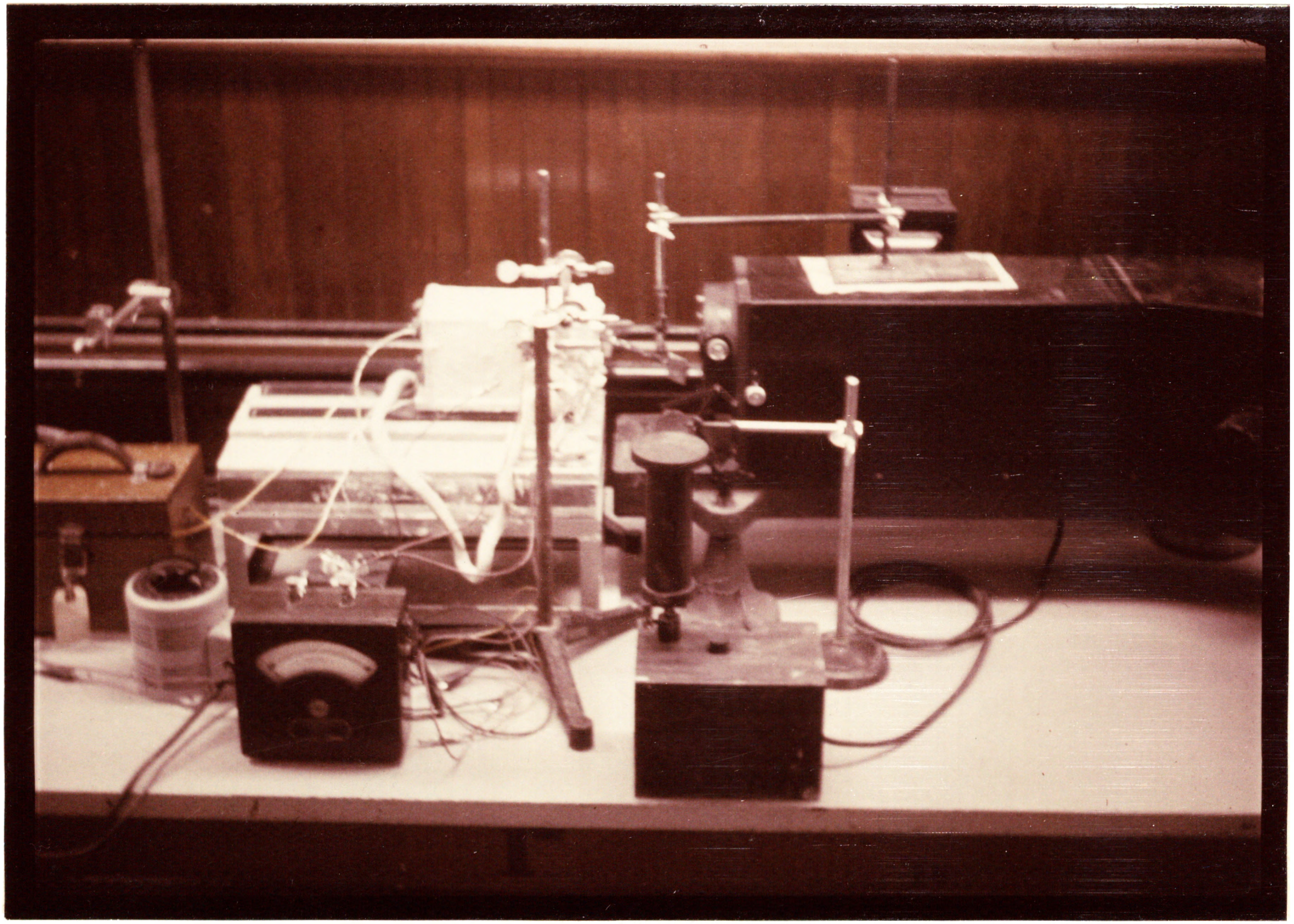


Plate IV. Cell set-up on spectrograph

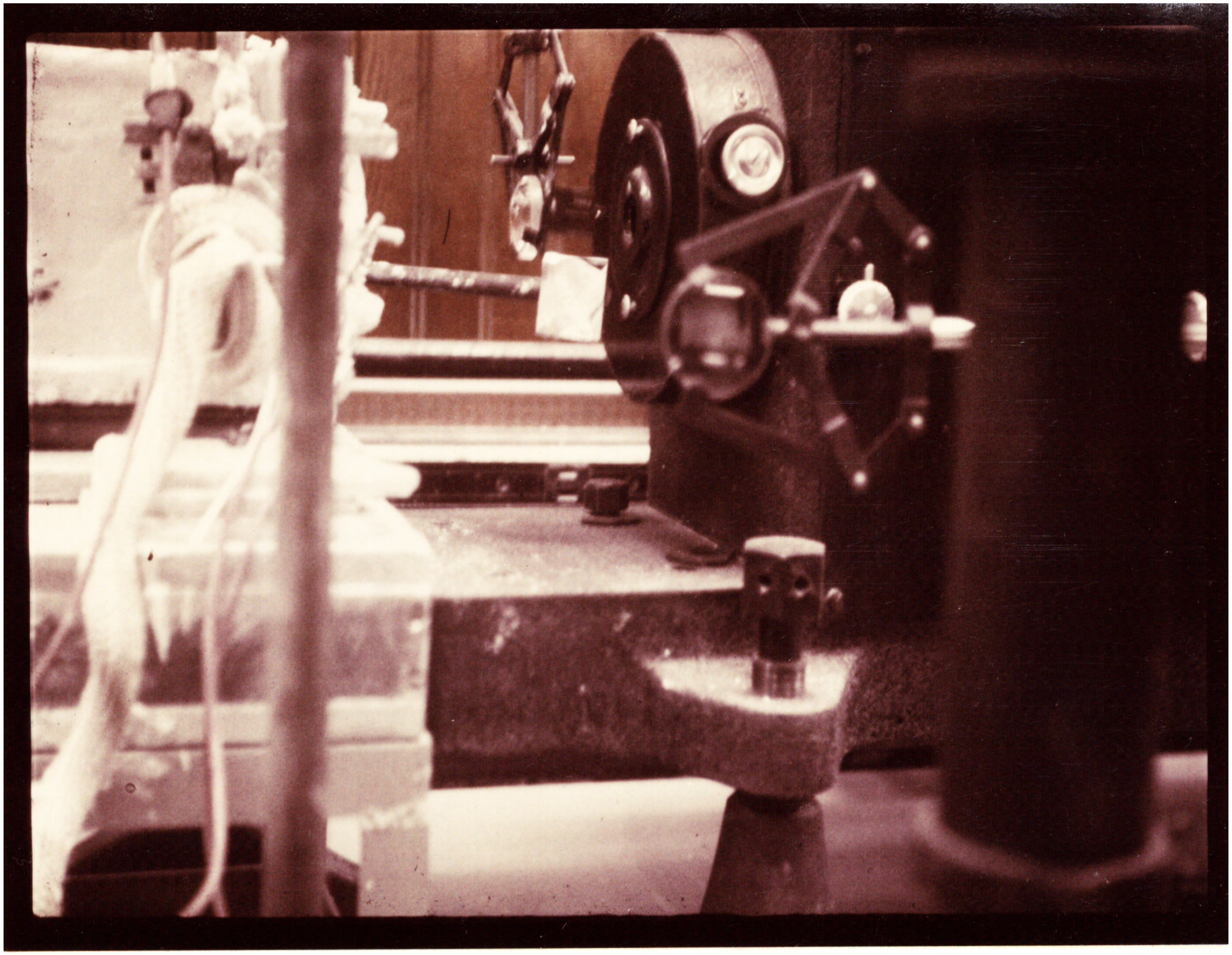


Plate V. Light system set-up

place temporarily, they were washed thoroughly, first with detergent and then alcohol to remove all grease and oil in order to prevent the elements from deteriorating at high temperatures. Then, the elements were covered with a layer of Hiloset refractory cement to protect them from alkali damage because of the 85% magnesium cement used to insulate and finish the oven construction. Oven ends were made of transite held together by steel bolts and nuts. Short springs behind the nuts helped to prevent heat expansion damage to the transite end-pieces.

The oven mount consisted of a transite-topped platform on which were placed moveable slides such as used for mounting phonograph turntables. The oven was attached to the slides, and thus could be easily and quickly moved back and forth.

E. Operational Procedures. The cell was first attached to the vacuum system, and a vacuum of 10^{-5} mm. was obtained. The cell was then outgassed by covering it and its reservoirs with ovens, and heating the ovens to about 1000°C for fifteen hours. An oxygen-gas torch was used to outgas all exposed quartz tubing adjacent to the cell. The reading of the pressure at the end of outgassing was below 10^{-5} mm., using a gauge which read to 10^{-5} mm. It was estimated on the basis of the gauge reading and previous experience as being in the neighborhood of 6×10^{-6} mm. After shutting down the vacuum system, the end of the thallium reservoir was opened, (see Plate 3); 99.6% thallium metal pieces were deposited inside, and the reservoir was resealed. The top of the distillation flask was opened and triple-distilled mercury was poured into it, then resealed. The thallium metal was vacuum distilled into the cell from its reservoir. This operation was performed by connecting the ovens as for outgassing, except that a heating tape was used over the exposed mercury reservoir. The distillation required about twelve hours at approximately 900°C. Then, the mercury was distilled into the mercury reservoir with a torch, and the cell was severed from the vacuum system. The vacuum at cut-off was between one and two $X\ 10^{-5}$ mm. The cell was then ready for mounting before the spectrograph and monochromator slit for data gathering purposes.

F. Cell Set-up on the Spectrograph and Monochromator. Plates 4 and 5 show the cell set-up used for taking data in the experiment. The cell was rigidly clamped in front of the spectrometer or monochromator slit. The main oven was mounted on slides on a platform. The freely sliding oven facilitated quick removal of the oven off of the cell. After positioning the baffle within the cell by an external magnet, the oven was positioned over the cell without disturbing its position. The incident lighting arrangement was likewise not disturbed by this procedure. Thus, a great deal of data covering a great many cell thicknesses could be gathered under identical experimental conditions.

The incident light system used is shown in Plate 5. The oven window and all lenses used were quartz. A quartz lens was used to focus the emitted light on the spectrometer or monochromator slit in order to condense the feeble flourescent light rays.

In the first four one hour exposures using a prism spectrograph, positive results were obtained. Unfortunately, baffle movement caused by vapor pressure changes within the cell during heating up prevented having the desired control over the cell thicknesses. Heating up the oven very slowly failed to prevent baffle movement. After other attempts, the problem was solved by allowing the mercury to remain in the mercury reservoir; then the main oven was heated to several hundred degrees before raising the temperature of the mercury reservoir. Many other problems were encountered, and eventually solved.

III. RESULTS AND DISCUSSION

In Figure 7, the thallium temperature increases from 750°C to 830°C, while the mercury temperature remained constant. This caused a constant increase in the intensity of the 3519A thallium line at a rate of increase of .17 volts per 10°C. Thus, an increase in thallium atom concentration proportionate to mercury concentration produced higher intensity.

In the case of Figures 7 and 8, the excitation depth was limited to 2 cm. and 3.5 cm. respectively, which is shorter than the potential unrestricted depth of penetration of the incident radiation as discussed in a later paragraph on Figure 13. Whereas, in Figures 9 and 10, the cell depth is 4.5 and 5.7 cm. at thallium and mercury temperatures in the relative range of the data of Figure 13. Thus, it is assumed the cell depth is greater than the true penetration depth of the incident radiation. Nevertheless, Figures 7, 8, 9, and 10 show that irregardless of whether cell depth is less than or greater than the depth of penetration of the incident radiation, the increase in intensity of the 3519A line is constant within the data range limits. Figure 10 has the lowest rate of increase of intensity, .1 volt per 10°C, but the highest mercury range of temperature, (143-152°C). Figure 9 has the next lowest rate of increase of intensity, .13 volts per 10°C, and the second highest range of mercury temperature,

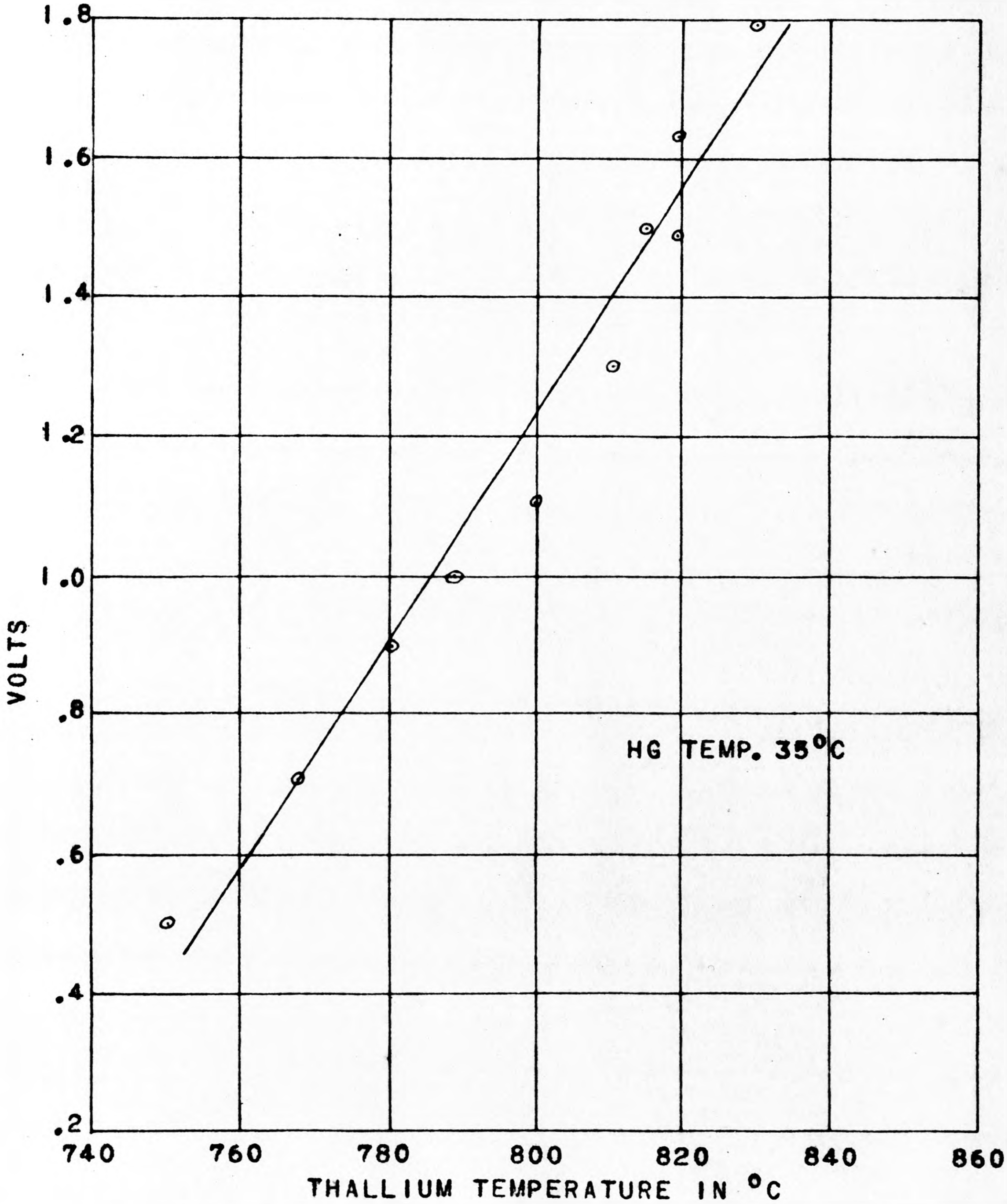


FIGURE 7. CELL DEPTH 2 cm, 3519A TL LINE

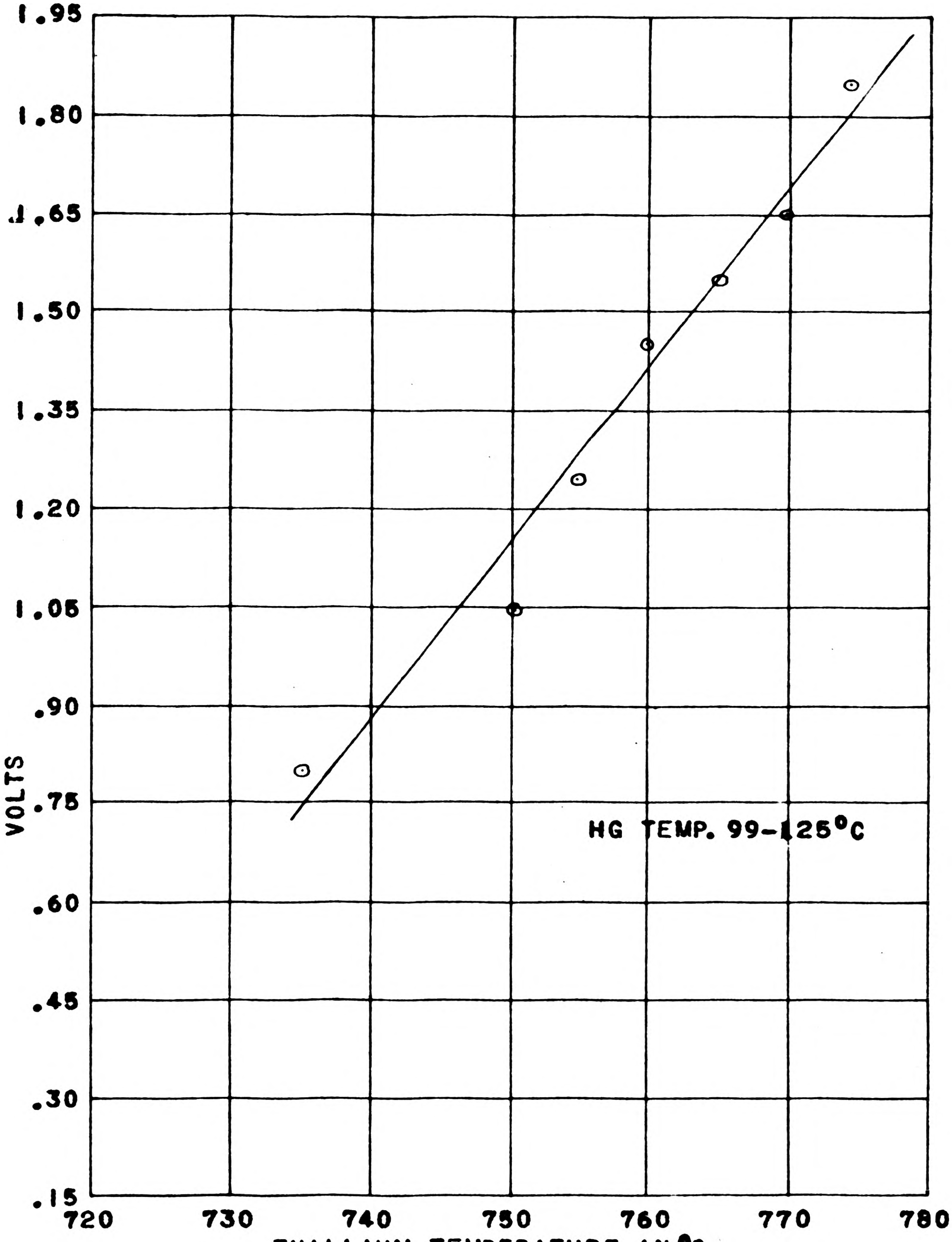


FIGURE 8. CELL DEPTH 3.5 cm, 3519A TL LINE

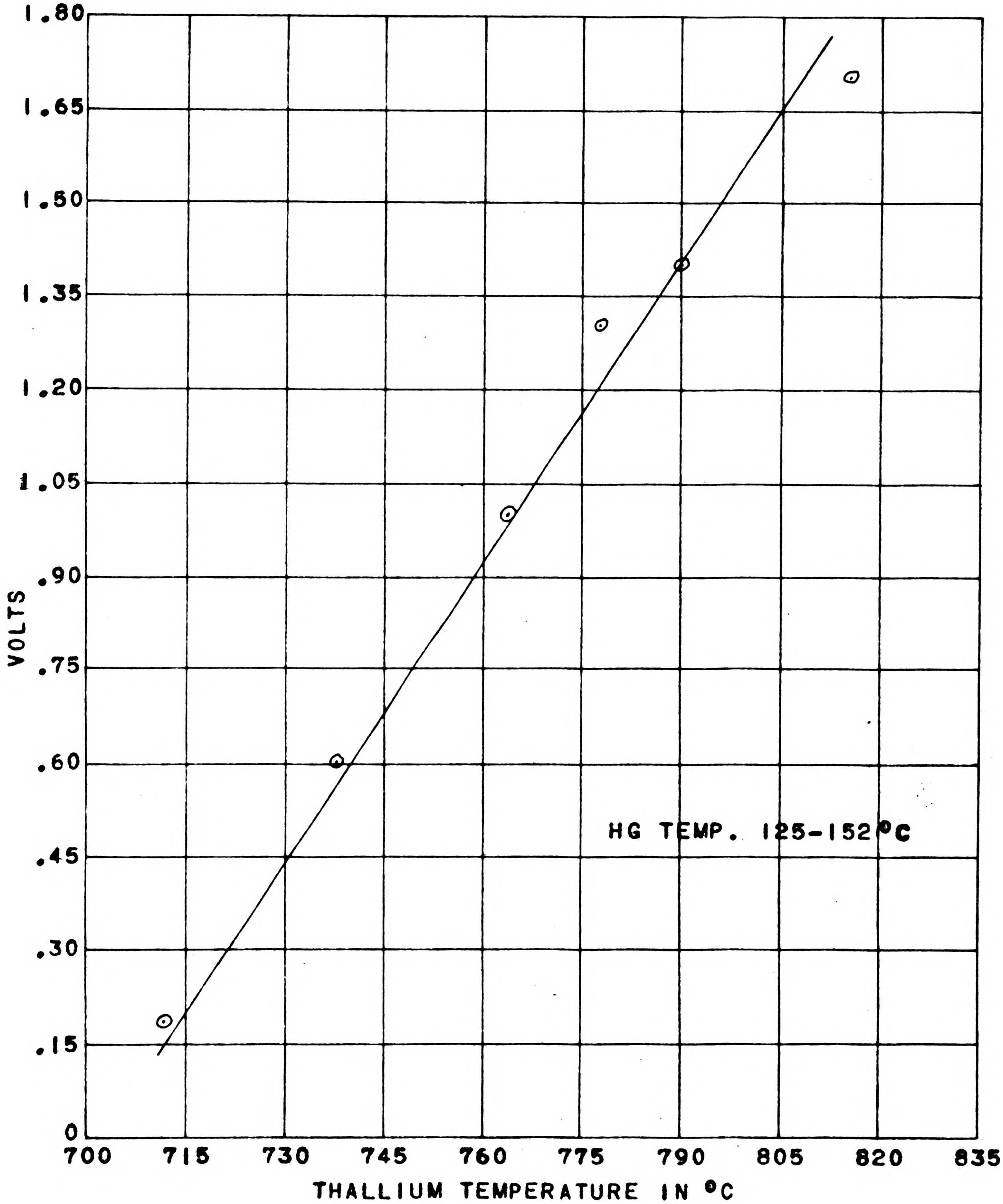


FIGURE 9. CELL DEPTH 4.5 CM, 3519A TL LINE

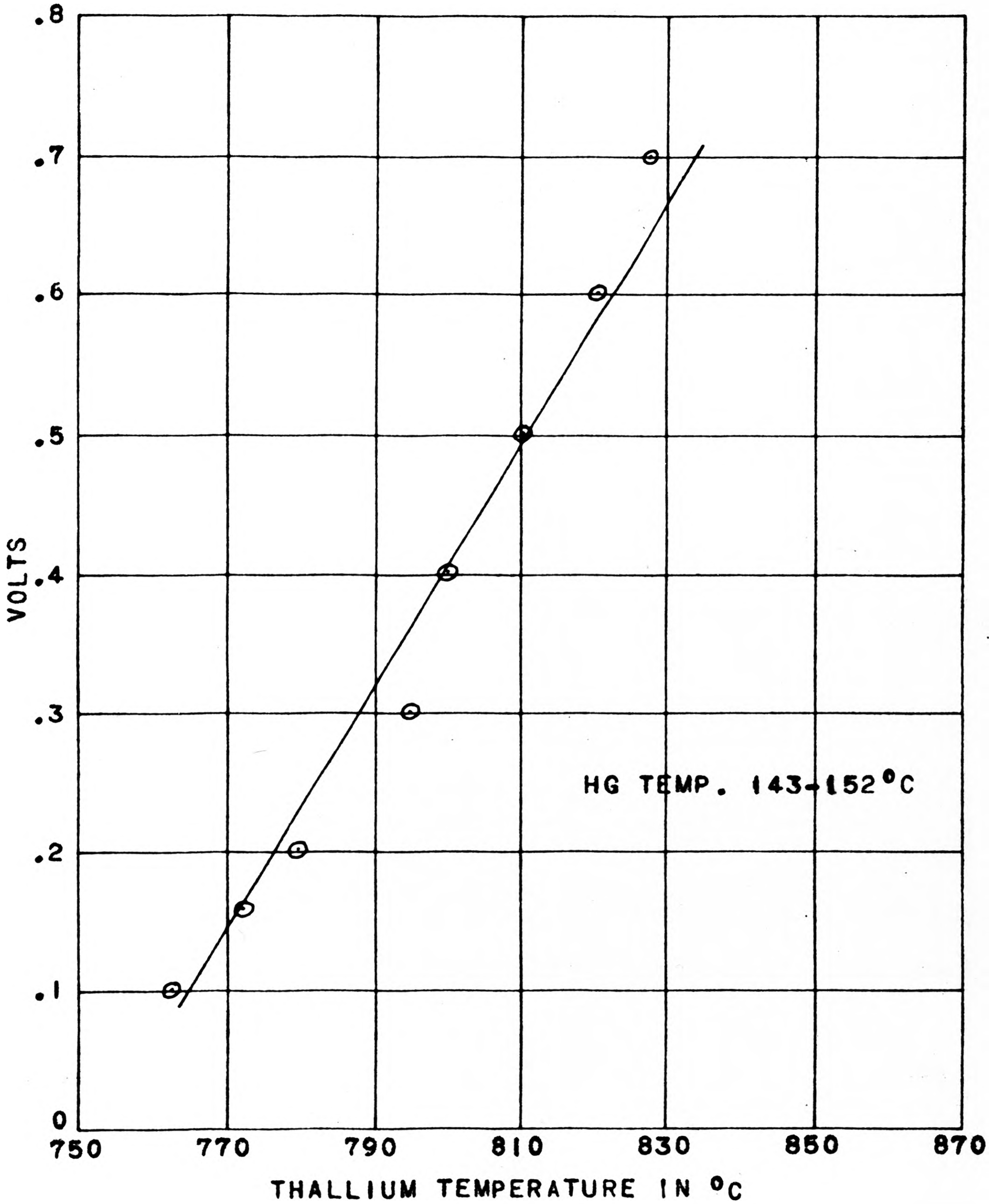


FIGURE 10. CELL DEPTH 5.7 CM, 3519A TL LINE

(125-140°C).

Figure 8 has the highest rate of increase of intensity, .2 volts per 10°C and the second lowest range of mercury temperature, (99-125°C). Figure 7 has a slightly lower rate of increase of intensity than Figure 8, .17 volts per 10°C, and the lowest range of mercury temperature, (35°C).

These results show the rate of increase of intensity as the density of thallium increases is directly dependent on the thallium temperature or density, and nearly inversely dependent on the mercury temperature in the temperature ranges considered. The data taken at a mercury temperature of 35°C was also obtained at the smallest cell depth, and so the incident radiation was not totally absorbed in the cell as was the case for the other figures. It would have been better to have taken this data versus various mercury temperatures at one constant cell depth. This was impossible because of experimental difficulties which will be mentioned at the end of the discussion.

In Figure 11, the intensity of the 3519A thallium line shows an increase during the initial rise of mercury temperature to about 190°C, and then drops down as the mercury temperature continues to increase. This data was taken at nearly constant thallium temperatures. This supports the results of prior experiments.[5] The initial rise is because of the increasing mercury concentration when full absorption takes place. The point of maximum mercury

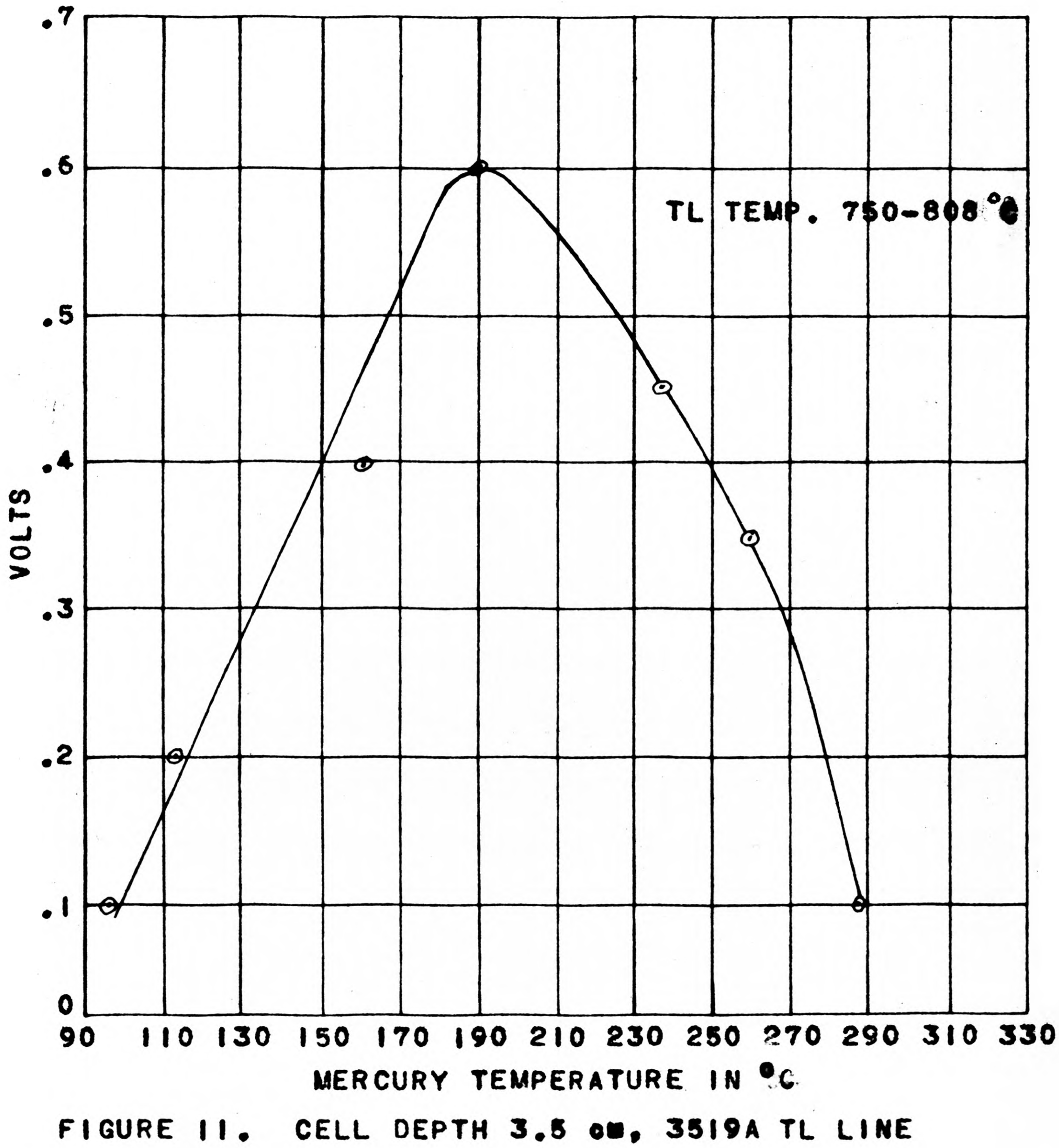


FIGURE 11. CELL DEPTH 3.5 cm, 3519A TL LINE

density with full penetration of the incident radiation is reached at the peak of intensity. From this point onward, the depth of penetration of the incident radiation decreases until the mercury density becomes so great that the radiation is absorbed at the surface of the cell, and the intensity of the 3519A line decreases to the threshold of detection.

Figures 12 and 13 show four different sets of data for the intensity of the 3519A line which have changing cell depth. In Figure 13, the center line, (long and short dash), hidden line, (dash), and object line, (solid line), curves were all made using data taken at a similar thallium temperature. The object line and hidden line curves were made using data taken at a similar thallium temperature range, and they coincide quite well. The object line curve was made using data taken at slightly higher mercury and thallium temperatures than the hidden line curve. As the mercury and thallium pressures rise slightly, the intensity becomes slightly greater, which accounts for the increased intensity of the object line curve. This fact is evident from Figures 7, 8, 9, and 10. The mercury temperatures are well below the critical temperature at which the concentration effect begins to act inversely upon the intensity, as is seen in Figure 11. Therefore, it is expected that the hidden line curve would be lower than the object line curve. The center line curve, on the other hand, represents data taken at a higher mercury temperature range than the

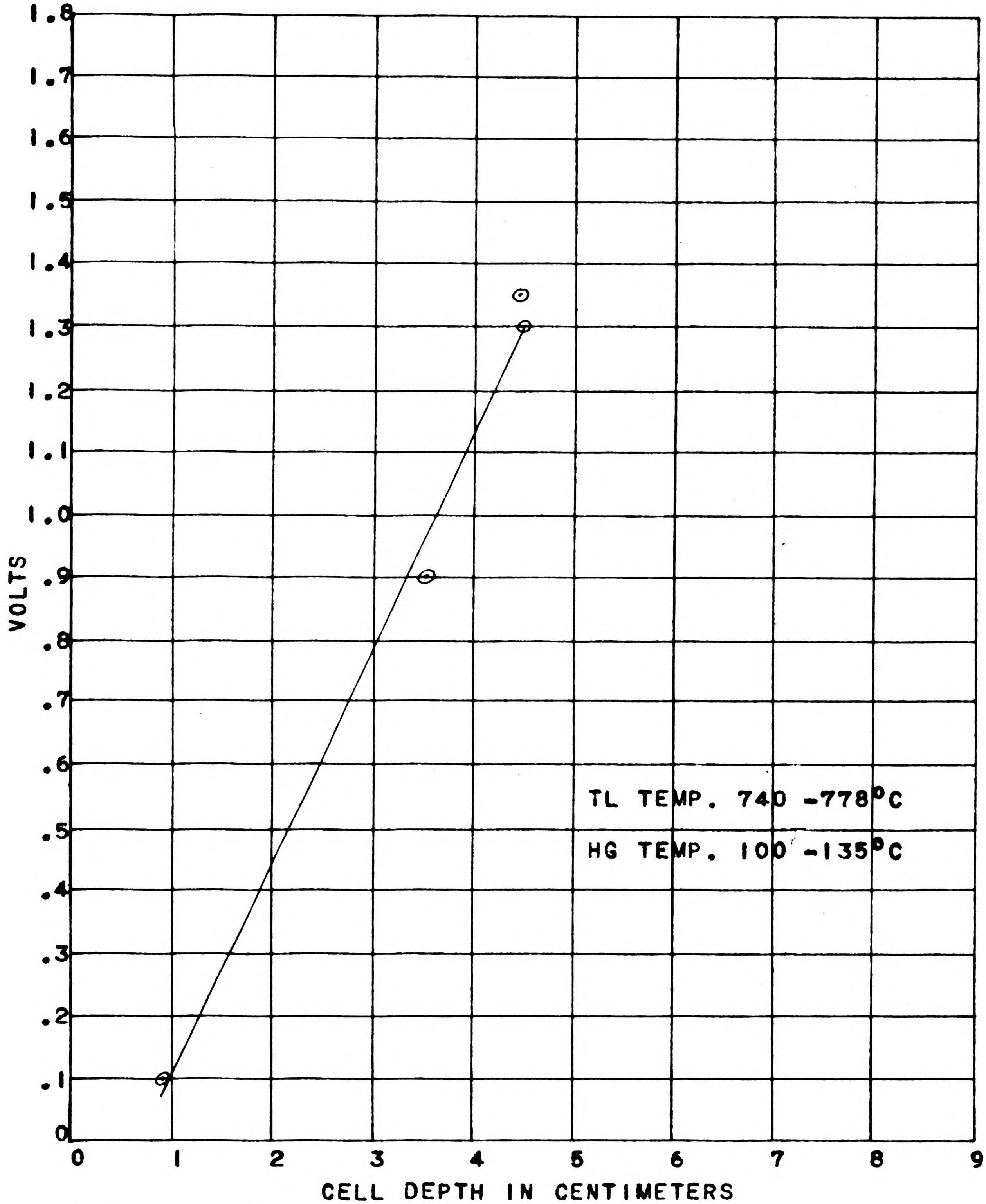


FIGURE 12. 3519A TL LINE

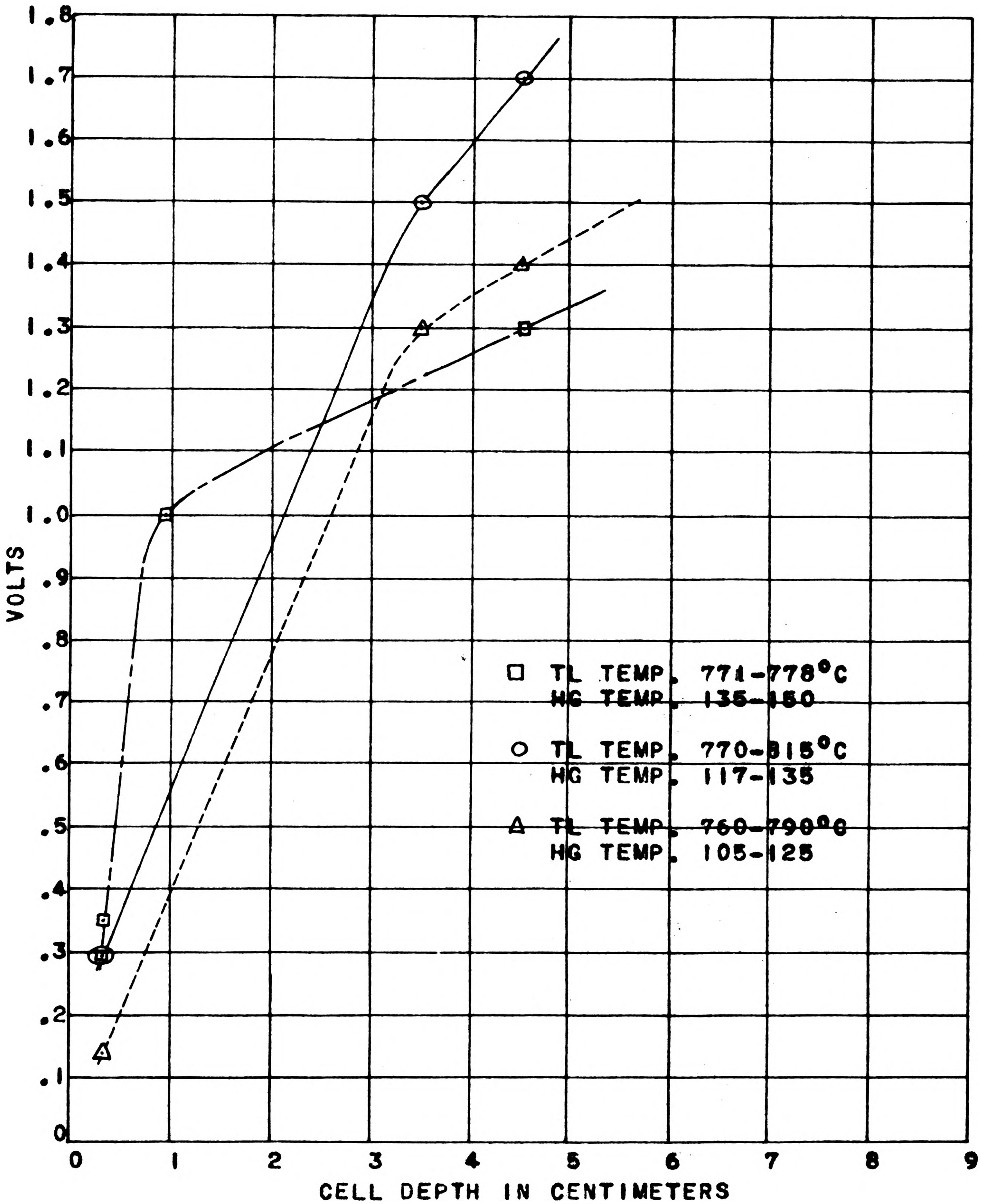


FIGURE 13. 3519A TL LINE

object line or hidden line curves. The data of the center line curve has the most constant thallium temperature within the lower part of the thallium temperature range of the other two curves.

In the hidden line curve and object line curve, the mercury temperatures are in the same general range, and the breaking point in the rise in intensity occurs at about 3.5 cm. This would indicate that the majority of the incident radiation is absorbed by the mercury atoms through this depth. The center line curve, however, was made at higher mercury temperatures and shows that the penetration depth is less due to higher mercury pressures. The rise in intensity with increasing cell depth is much larger for the center curve due to the higher mercury pressure than for the hidden line and object line curves. The rate of rise is less after the break in the center line curve compared to the hidden line and object line curves due to an unfavorable ratio in the number of mercury atoms available to the number of thallium atoms present.

With the use of the department monochromator, the cell exhibited only sufficient intensities for the study of the 3519A spectral line of thallium. It was thought that the monochromator might have insufficient light gathering ability for the experiment. Additional data taking was then postponed until the arrival of a new Bausch and Lomb monochromator. The author had planned to take additional data using the new monochromator to obtain more complete graphs on the 3519A line. It was also planned to use the new monochromator to attempt to observe other thallium lines. However, the cell became contaminated during the intervening six month period, and negative results were obtained. The cell was about nine months old at this time, and helium and hydrogen had no doubt entered the cell due to the porousity of the quartz, and quenched the fluorescence.

IV. CONCLUSION

The experiment exhibits three conclusions with respect to the 3519A fluorescent line of thallium on the three types of data obtained:

1. From Figures 7, 8, 9, and 10, it is concluded the rate of increase of intensity, as the density of thallium atoms increase, is directly dependent on the thallium temperature, or density, and nearly inversely dependent on the mercury temperature in the temperature ranges considered, 35-152°C.

2. From Figure 11, it is concluded that increasing the mercury temperature to about 190°C will increase the intensity of the fluorescent line. After which the increasing mercury density decreases the fluorescence mainly because of specular reflection and also due to metastable atom formation.

3. From Figure 13, it is concluded, when using a cell of variable depth, a constant thallium temperature, and a varying mercury temperature, the intensity of the 3519A line initially increases at a constant rate, then breaks to a lower final rate of increase. The rate of increase of intensity varies according to the mercury temperature at different cell depths. The break in intensity is indicative of the penetration depth of the incident resonance radiation, and depends on the mercury

temperature. The rise in intensity becomes more rapid with higher mercury temperature, and the break in intensity occurs with decreased cell depth. The intensity of the spectral line also decreases with increased mercury temperature.

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VITA

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