

PHOTOGRAPHIC DI AND INTENSITY MEA THE COPPER HYDRI

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PHOTOGRAPHIC DENSITOMETRY AND INTENSITY MEASUREMENTS IN THE COPPER HYDRIDE SPECTRUM

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A THESIS

Submitted to the Graduate School of Kichigan State College of Agriculture and Applied Science in partial fulfilment of the requirements for the degree of

MASTER OF SCIENCE

Department of Physics

1940

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INTRODUCTION

The need has been felt for several years in the Department of Physics at Michigan State College for a microdensitometer for spectrographic work. It was the purpose of this problem to study the basic photographic, optical, and mechanical principles involved in the construction and operation of a microdensitometer. From the knowledge thus obtained, a working model of such an instrument was designed, constructed, and applied to a specific problem.

The specific problem to which the completed apparatus was applied was a measurement of the intensity distribution in the rotational structure of the O-O band of Copper Hydride. Employment of this measured intensity distribution has yielded information as to the temperature attained in a copper arc operated in an atmosphere of hydrogen.

THE MEASUREMENT OF SPECTRAL LINE INTENSITIES BY PHOTOGRAPHIC MEANS

The problem of measuring spectral line intensities is complicated by the fact that only small amounts of energy are available, measurements must be made over wide wavelength ranges, and that the spacial dimensions of the spectral lines are relatively small. Because these difficulties are most easily overcome by the photographic method, that method has gained the widest acceptance for use in such measurements.

The basis for the measurement of the intensities of spectral lines by the photographic process is the characteristic or calibration curve of a photographic emulsion. Such a curve for spectrographic purposes must be an intensity curve obtained from the measurement of the densities resulting from a standard series of intensities of the same wavelength as of the spectral line to be measured. Therefore, the method of construction of the calibration curve must take into account reciprocity law failure (which includes the intermittency effect) and the spectral response of the emulsion. (1) However, it should be noted that while a calibration curve should be constructed for each wavelength used, it is found in practice that there are long wavelength ranges over which the calibration remains constant.

After the calibration curve has been obtained, the intensities of the spectral lines may be measured by a comparison of their densities with the standard densities by means of a microdensitometer.

One further difficulty of the photographic method is the problem of making background or fog intensity corrections. The method used in this problem is to subtract background intensities as found from the calibration curve. A test of the background intensity equation $I_{C=}I_{T}-I_{B}$ is illustrated in Fig. 1 and Plate I.



Fig. 1. Background Correction Test

The total intensity (I_T) results from the superposition of a continuous background intensity (I_B) upon part of the intensity calibration scale (I). I_B and I_T are found from the calibration curve and their differences compared with the calibration intensities. The results, as shown in the "Degree of Correction" graph, indicate that such a correction method is applicable to the present problem.

The design and construction of a rotating sector wheel for intensity calibrations and of a microdensitometer for spectrographic density measurements will now be considered.

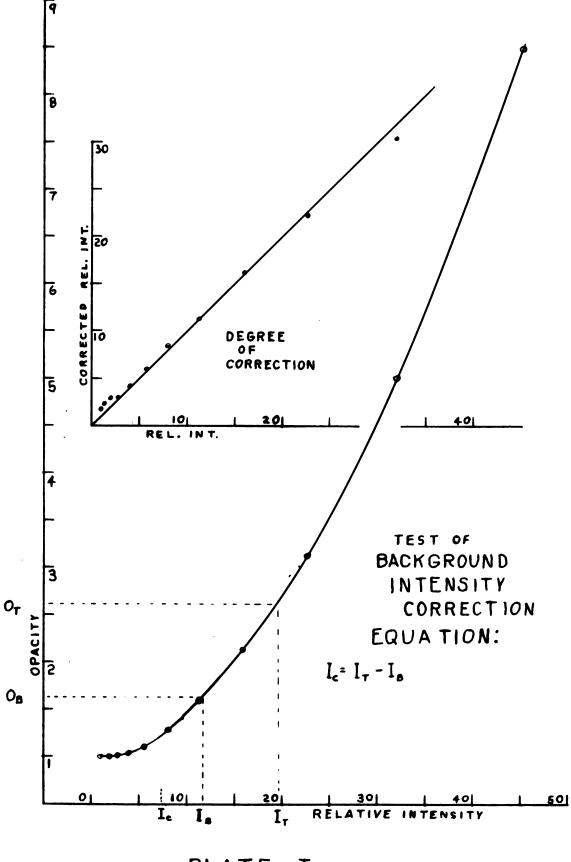


PLATE I

The revolving sector wheel was chosen as the means of calibration because an astigmatic grating spectrograph was used in this problem.

While a sector wheel usually gives a time scale, the justification for its use in the production of an intensity scale is that the speed of revolution of the wheel can be made high enough to give flashes above the minimum critical frequency of intermittency. (1) The flash frequency in this case was approximately 30 per second.

The form of the sector wheel was that of an aluminum disk having 15 open arcs cut in a ratio of the square root of 2 from 1 to 128. See Figs. 2 and 3. For convenience of use the wheel was mounted horizontally under a slot in the top of a two compartment light tight box. One compartment contained the sector wheel and its driving motor. The other contained the light source and transformers. Light from the source passes through a diffusing medium, filter holder, and adjustable diaphragm in the wall between the two compartments. A mirror under the sector wheel reflects the light up through the open arcs and masking slot to the photographic plate which may be laid face The size of the slot and wheel is such down over the slot. that a calibration scale 0.3" by 3" results. See Figs. 1 and 14. In use for long exposures a light tight cover comes down over the plate.

The design of the instrument including the sector wheel



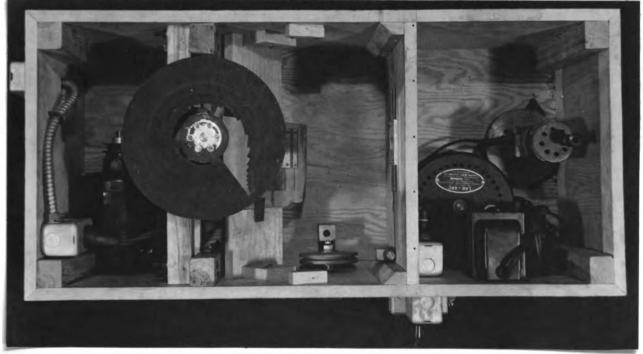


Fig. 2. Sector Wheel Sensitometer Fig. 3. Interior View

is such that its use is not limited to spectrographic intensity measurement but can also be applied to general photographic standardization and measurement.

When the energy of a spectral line falls upon a photographic plate, the observable and measurable result is a blackening or an increase in the optical density of the plate. The density unit is defined as: $D = \log_{10} i_o/i$ where i_o is the intensity of a beam of light transmitted by a clear portion of the plate and i is the intensity of the same beam transmitted by a blackened portion of the plate. The basis of this unit is the Lambert law of absorption. (2) The simple ratio i_o/i is called opacity.

The measurement of density depends upon the measurement of io and i. That is, a densitometer is an instrument combining a beam of light of constant intensity and a photometer for measuring the intensity of the beam after transmission by the photographic plate. Such an instrument with a beam small enough to pass through a single spectral line is referred to as a microdensitometer.

The microdensitometer as constructed as part of this problem has the design as schematically diagramed in Plate II and photographed in Figs. 4 and 5.

Referring to Plate II: (S) is an adjustable slit illuminated by a ribbon filament lamp (F). An image of the slit is projected by the lens (L) and front surface totally reflecting mirror ($\%_1$) upon the emulsion side of the spectrographic plate (?). The light transmitted by

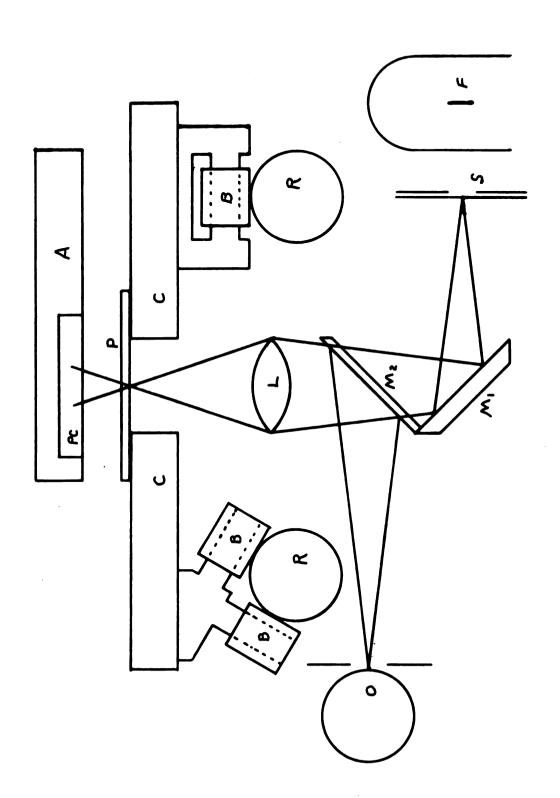
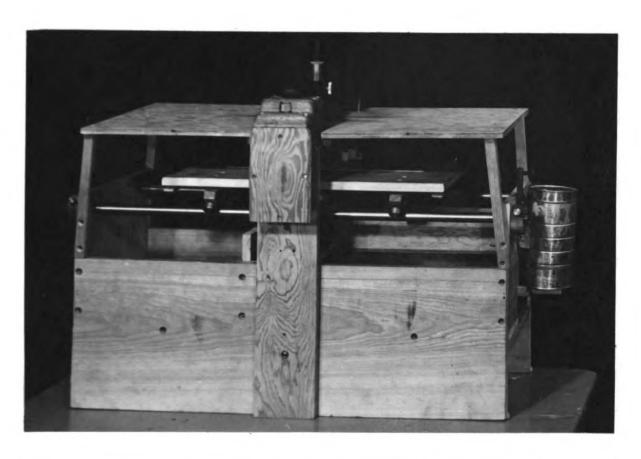
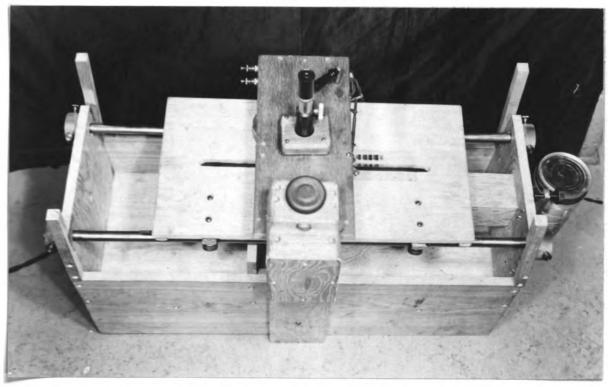


PLATE II MICRODENSITOMETER





Figs. 4 & 5. Microdensitometer (Working Model)

the plate falls upon the photovoltaic cell (PC) whose electrical output is measured by a critically damped gal-vanometer.

In order to keep the plate in the focal point of the beam and to be able to move any required line into the beam, the plate rests upon a movable carriage (C).

The carriage itself moves on steel rods (R). It is held in position on the rods and limited to one degree of freedom by five kinematic constraints in the form of double row radial ball bearings (B).(3) The five bearings are shown in Fig.6. Coarse adjustment for position is done by rack and pinion and fine adjustment by a micrometer screw.

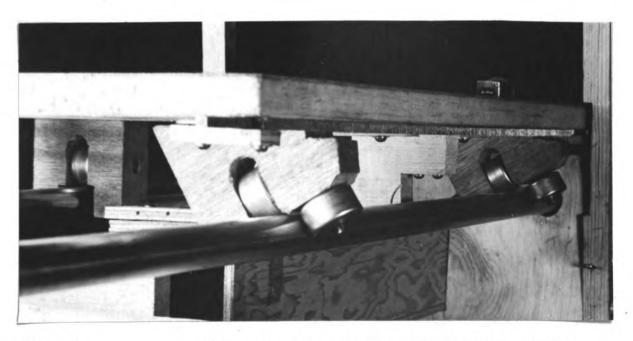


Fig. 6. Kinematic Support for the Plate Carriage

For plate orientation purposes a viewing system has been incorporated in the construction of the microdensitometer. In Plate II (0) is a flashed opal bulb whose surface is projected in the same plane as the image of the slit. This is accomplished by the same lens (L) and a mirror (\mathbb{H}_2) in the form of a sheet of clear glass. In addition there are two illuminating lights placed above the plate on each side of the photo cell.

For the operation of the viewing system, the photo cell is carried in and out of the transmitted beam by the movable arm (A). The movement of the arm operates a switch which controls the viewing lights. The operating cycle is such that the viewing lights are off when the photo cell is in the measuring position and on when it is moved out of position. Direct observation is accomplished either by a simple magnifier which is moved into the viewing position by the photo cell arm or by a fixed viewing microscope above the photo cell.

The sensitivity and reliability of the microdensitometer depends upon the intensity and constancy of the light
source, the sensitivity and constancy of the photo cell and
galvanometer, the efficiency of the projecting lens and
viewing system, and the accuracy of the plate carriage movement.

In order to get high intensity and constancy of the light source, a 6 volt 18 ampere ribbon filament lamp operated through a voltage stabilizer was used. The

necessity for voltage stabilization and the results obtained by stabilization are graphically illustrated by galvamometer movement and recording voltometer record in Figs. 7. 8. and 9.

In the selection of a photo cell for use in this microdensitometer, several cells of different manufacture were tested and the one which had the highest sensitivity combined with the lowest drift or fatigue effects was chosen. This cell was the G-E Visitron F-3. For measuring the electrical output of the photo cell a galvanometer having a short period, high sensitivity, and capable of being critically damped by the internal resistance of the photo cell is the ideal.

A short focus, high aperture anastigmatic lens was used to focus the measuring beam on the photographic plate. In order to gain high intensity combined with narrowness of beam and freedom from diffraction patterns, the lens position was such that a reduced image of a relatively wide slit was used.

The procedure for the measurement of the relative intensities of spectral lines is as follows. The plate is taken from the spectrograph and exposed through the revolving sector wheel to radiation of the appropriate wavelength. The plate, after processing, is placed on the carriage of the microdensitometer with its emulsion side in the focal plane of the photometer beam. The galvanometer deflections

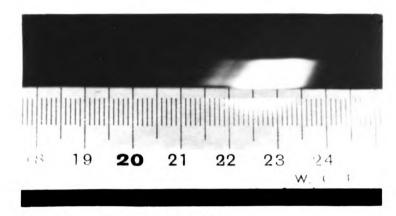


FIG. 7. BEFORE

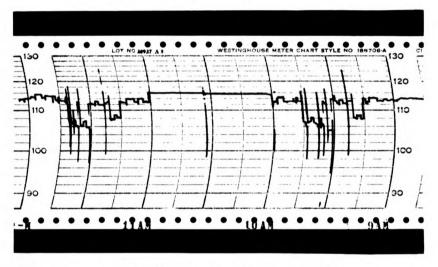


FIG. 8. VOLTAGE VARIATION

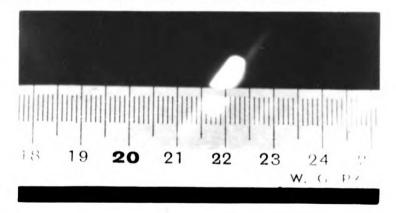


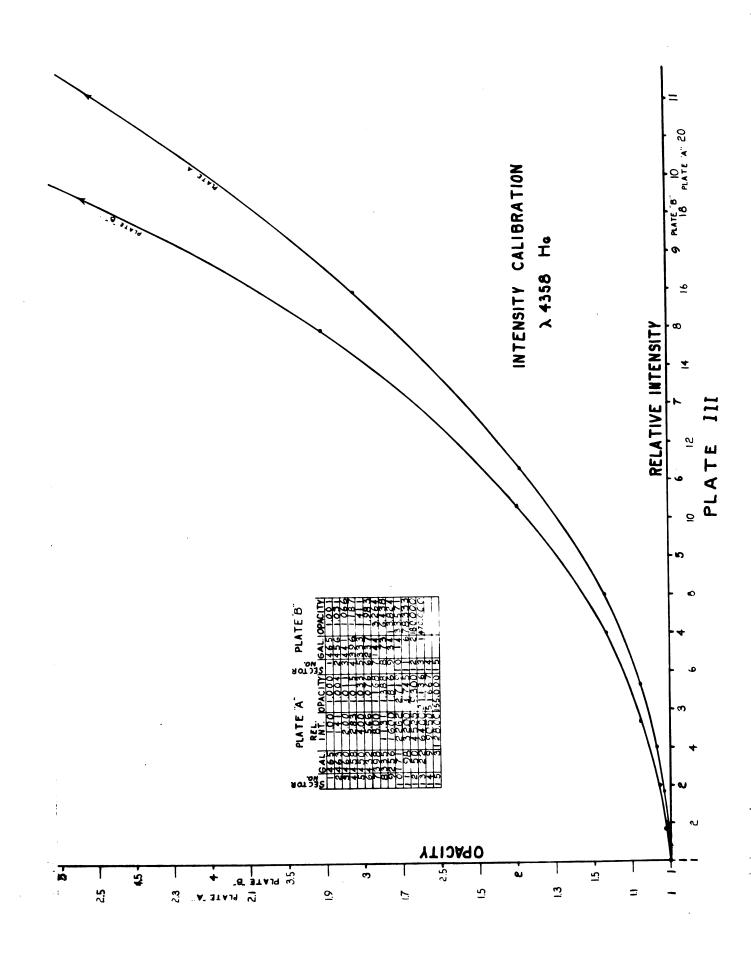
FIG. 9. AFTER

STABILIZATION

are recorded for clear plate, for each step of the calibration sector scale, for each spectral line whose intensity is to be measured, and for background next to each such line. (In case the background is small and relatively constant an average background deflection may be used).

From the recorded data, a calibration curve of density against the common logarithm of the standard intensity ratios (or of opacity against the standard intensities) is drawn. See Plate III. The total line intensities and background intensities are read from the calibration curve and substituted into the background intensity correction formula.

The final result of this method of measurement is the relative intensity of each spectral line in arbitrary intensity units. These units may be calibrated in absolute intensity units if the absolute intensity or energy of the standard is known.

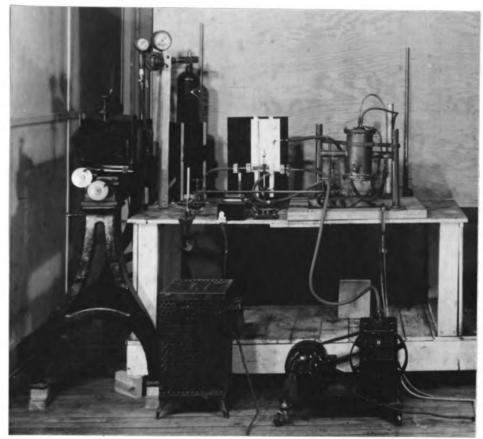


OF THE O-O BAND OF COPPER HYDRIDE

A very efficient source for the production of the spectra of the metallic hydrides may be arranged by operating an electric arc with metallic electrodes in an atmosphere of hydrogen. Such a procedure was adapted here. An enclosed 220 volt d. c. arc with water cooled copper electrodes was constructed such that the arc could be operated in any desired gaseous atmosphere. See Figs. 10 and 11.

Numerous tests were made as to best operating conditions and it was found that the CuH spectrum was obtained with reasonable efficiency when the arc was operated in hydrogen at 10-15 cm Hg pressure with an arc current of 20 amperes. Ordinary tank hydrogen was employed without purification. With this arc current and hydrogen pressure the arc does not maintain itself and continual striking is necessary. The arc was so constructed that it could be started and re-ignited without disturbing the atmosphere in which it was operating.

The spectrograms were taken with a concave grating spectrograph in an Eagle mounting. The grating itself was of 1 meter focal length ruled with 30000 lines per inch over a 4 inch surface.



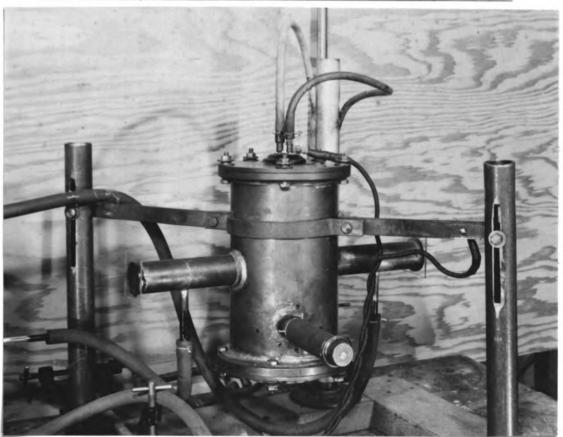


Fig. 10. Grating Spectrograph Fig. 11. Enclosed Arc and CuH Source

The ruling of the grating and the construction of the mounting is such that considerable background intensity is present. This relatively high background intensity in connection with a background which is probably the many line spectrum of hydrogen is the reason for the great attention paid to the method of its correction in this problem.

Because the band whose intensity distribution was studied extended from 4280 to 4420 A° with an average at 4350 A°, the 4358 line of mercury was used for intensity calibration. Eastman Process film used in conjunction with a Wratten filter No. 85 gave perfect isolation of this line. For this reason the spectrograms were taken on Eastman Process film and developed in D-19.

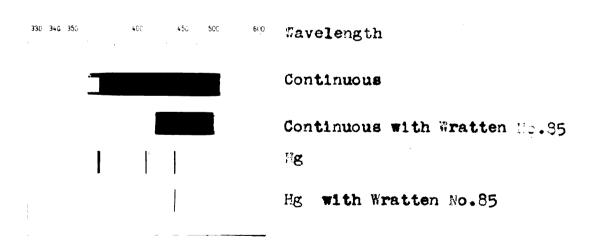


Fig. 12. Isolation of $\lambda 4358$

The C-O Band of CuH

The specific problem to which the completed microdensitometer was applied was a measurement of the intensity distribution in the rotational structure of the 0-0 band of Copper Hydride. The analysis of this band has been given the most completely by Heimer and Heimer (4).

A given line in the band system arises when a transition occurs from an excited state of the molecule with given electronic, vibrational, and rotational energies to a lower state possessing different energies. For a constant electronic and vibrational change, the structure which results is a band due entirely to rotational energy changes in the transition. This energy change expressed in wave numbers $(\sqrt{-\frac{1}{\lambda}})$ is represented by:

for the total energy as a sum of the electronic, vibrational, and rotational energies. For a given band $V_c + V_{\infty}$ is a constant and V_{α} varies for each line in the band. The complete energy expression is given by: (5)

$$V = V_e + V_v + (B' + B'') m + (B' - B'') m^2$$

for a band, such as the 0-0 band of CuH, having a P and an R branch. For this particular band $Ve^{+}V_{\nu} = 23,311.1$ cm⁻¹, B' = 6.75, B' = 7.81 (4).

The factor m is a running number having integral values and is related to the rotational quantum numbers of the lower state. The rotational quantum numbers for the upper or excited state are $J' = 0,1,2,\ldots$ and for the lower are $J'' = 0,1,2,\ldots$

For the P branch of the band, m = -J'' and for the R branch m = J'' + 1. The designation P and R branch comes from the selection rules for possible transitions. The selection rule for this case limits the changes in J between the upper and lower states to + and -1. The spectral lines which result from $\triangle J = +1$, as shown in Fig. 13 forms the P branch of the band. The R branch

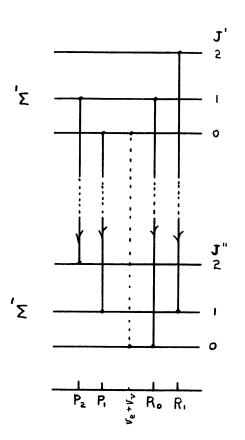


Fig. 13 Energy level diagram for the 0-0 band of CuH

results from a $\Delta J = -1$. Each line of the band is given its appropriate J'' value as an index. Thus the lines of the P branch are P_1 , P_2 , P_3 , P_4 ,... and those of the R branch, R_0 , R_1 , R_2 , R_3 ,....

As a result of the selection rule and the terminology for the branches, the m's
in the energy expression are:
m = -1, -2, -3,.... for the
P branch and m = 0,1,2,3,4,...
for the R branch.

TABLE I. THE O-O BAND OF CuH

	P Bra	nch	R Branch			
J" O	cm-1	A°	cm ⁻¹ 23,324.66	A° 4,236.1		
1	23,295.54	4,291.5	23,336.05	4,284.0		
2	23,277.81	4,294.7	23,345.23	4,282.3		
3	23,257.94	4,298.4	23,352.22	4,291.0		
4	23,235.94	4,302.5	23,357.05	4,280.2		
5	23,211.92	4,305.9	23,359.67	4,279.7		
б	23,185.81	4,311.8	23,360.20	4,279.6		
7	23,157.72	4,317.0	23,358.52	4,279.8		
8	23,127.53	4,322.6	23,354.62	4,280.6		
9	23,095.36	4,328.7	23,348.54	4,281.7		
10	23,061.24	4,335.1	23,340.38	4,283.2		
11	23,025.19	4,341.9	23,329.87	4,285.2		
12	22,937.16	4,349.0	23,317.27	4,287.5		
13	22,947.26	4,356.6	23,302.48	4,290.2		
14	22,905.46	4,364.5	23,285.50	4,293.3		
15	22,861.88	4,372.9	23,266.43	4,296.8		
16	22,816.42	4,381.6	23,245.13	4,300.8		
17	22,769.26	4,390.6	23,221.71	4,305.1		
18	22,720.25	4,400.1	23,196.13	4,309.8		
19	22,669.52	4,410.0	23,168.52	4,315.0		
20	22,617.13	4,420.2	23,138.74	4,320.5		

It should be observed that for the P branch of this band, both the linear and quadratic terms in m are negative. The result is that the wavelengths of the different lines of the P branch continually increase. But for the R branch the linear term is positive and the quadratic term is negative.

This difference in sign causes the wavelengthd of the lines of the R branch to decrease at first and then begin to increase as the quadratic term becomes more effective than the linear term. This reversal of direction causes the lines of the R branch to double back upon the selves and form a so called band head. For the appearance of this band see Fig. 15 and Plate IV. Data as to J^R value and wave number for the individual lines of this band as given by Heimer and Heimer (4) as well as the corresponding wavelengths as converted from wave number are given in Table I. These data are the basis for the wavelength scale in Plate IV.

The Intensity Distribution of the Rotational Structure of the 0-0 Band of Copper Hydride

It may be seen from Fig. 15 that there is a non-uniform distribution of intensity among the individual lines of the band. The intensity of the individual lines of the band depends not only upon the frequency of the energy causing the spectral line, (Energy of the quantum = AV),

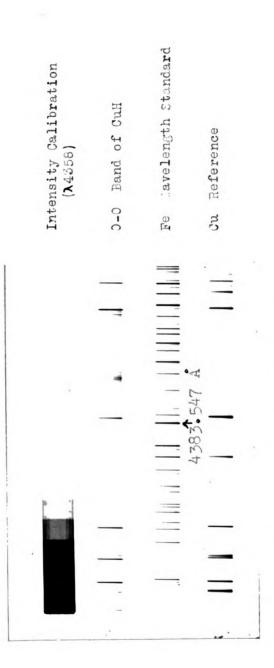


Fig. | 4 Spectrogram of the 0-0 Band of CuH (Natural Size)

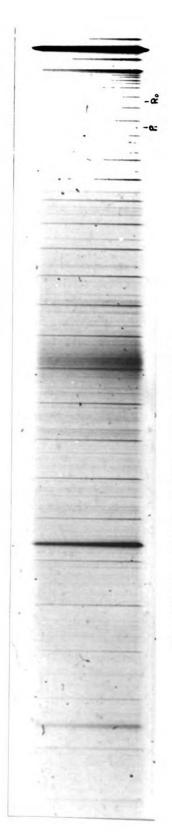


Fig. | 5 The O-O Band of Jopper Hydride

but also upon the probability of occurrence of the transitions which result in the spectral energy, and upon the number of molecules in the initial state.

The distribution of intensity, from emission in the rotational structure of a diatomic molecule, as given by Herzberg, is: (5)

The C in the expression for intensity distribution is proportional to the fourth power of the frequencies in the band. For any particular band C is approximately constant, and therefore may be regarded as a proportionality constant.

The factor (J' + J'' + 1) is an expression of the fact that the intensity distribution depends upon the statistical weight of both upper and lower state.

The dependence of the intensity distribution upon the number of molecules in the initial state which in turn is dependent upon the temperature of the emitting source is expressed by the Boltzmann factor.

Since the P branch arises from successive transitions in which $\Delta J = +1$ and the R branch $\Delta J = -1$, the expression (J' - J'') representing the transition which results in a single line becomes:

$$J' - J'' = -1$$
 for the P branch, and $J'' = J' + 1$
 $J' - J'' = +1$ for the R branch, and $J'' = J' - 1$

Therefore the expression $(J^{t}+J^{t}+1)$ in the intensity distribution becomes:

$$(J'+J''+1) = 2J'+2$$
 for the P branch $(J'+J''+1) = 2J$ for the R branch

The intensity distribution function may now be written for the P branch for example as:

$$I = C(2J'+2)e^{-\left[B'J'(J'+1)Ac/nT\right]}$$

The intensity distribution for both P and R branches of the band as measured by the method outlined in this problem are shown in Plate IV. Shown also are the total intensity and background intensity for the P branch. It is to be noted that the background intensity is from one to three times the intensity of the lines themselves. This fact illustrates the necessity for a reliable method of background correction. The data for this intensity measurement is to be found in Plate III (for Plate A) and Table II.

TABLE II

INTENSITY DISTRIBUTION IN THE O-O BAND OF Cuh

P Eranch			R Branch			
J" O	Total	Fog	Line	Total 11.68	Fog 9.66	Line 2.02
1	11.60	9.55	2.05	13.98	9.31	4.67
2	13.16	9.55	3.61	16.41	9.90	6.71
3	15.69	9.91	5.78	17.99	9.45	8.54
4	16.60	\$•90	6.80	20.65	9.45	11.20
5	18.04	9.55	8.49			
6	18.42	10.38	8.04			
7	18.42	9•91	8.51			
8	18.42	9.67	8.75	18.65	9.45	9.20
9	19.67	11.73	7.94	18.82	9.45	9.37
10	20.23	12.38	7.85	17.03	9.45	7.58
11		17.72		15.97	9.45	6.52
12	18.42	11.85	6.57	15.37	9.43	5.94
13	16.76	11.72	5.04	14.94	9.43	5.51
14	17.57	10.25	7.32	14.53	9.43	5.10
15	15.24	10.37	4.87	13.96	9.20	4.76
16	14.81	10.63	4.18	13.45	9.31	4.14
17	13.55	9.19	4.36	12.92	9.31	3.61
18	12.36	9.31	3.05			
19	11.49	9.06	2.43			
20	11.34	9.06	2.28			

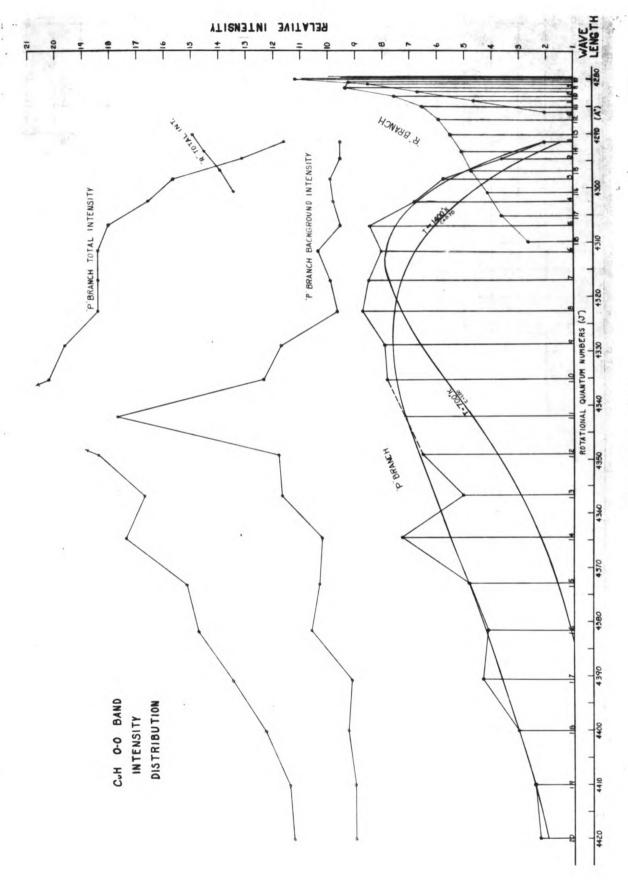


PLATE IV

TEMPERATURE HEASUREMENTS

The dependence of the intensity distribution upon temperature may be seen in Plate IV. There the theoretical intensity distribution for the P branch of the O-O band of CuH has been plotted for the two temperatures 700° and 1400° K. It is to be observed that as the temperature increases, the intensity distribution envelope flattens, its maximum shifts to higher J" values, and that the intersection between the envelopes for the P and R branches shifts to different J" values.

These effects, which become apparent when a change takes place in the temperature, may be used to calculate the temperature of the emitting molecules.

The temperature may be determined from a solution of the intensity distribution function for T. From:

$$Log_{e} \frac{I}{2J+2} = -\frac{B'J'(J'+1)hc}{hT} + Log_{e} C$$

This logarithmic equation has a linear form and may be solved graphically for the temperature. Values of $\log i_{J''}/2J' + 2$ are plotted against J'(J' + 1), and the temperature found from the measured value of the slope of the resulting straight line.

The temperature may be determined from the intensity

maximum by setting the first derivative of the distribution function equal to zero and solving the resulting expression for T. Such a procedure yields:

The temperature may be determined from the intersection of the intensity distribution envelopes for the P and R branches. Knauss and McCay (6) give:

$$T = \frac{(B'AC/R) \left[J_P'(J_P''-1) - (J_R''+1)(J_R''+2) \right]}{Log_e \left[(J_P''+1)/J_R'' \right]}$$

in which J_P^n and J_R^n are the J_R^n values for the intersection point. It is to be noted that J_P^n and J_R^n are not necessarily integral. The expression comes from a consideration of the fact that for the intersection point,

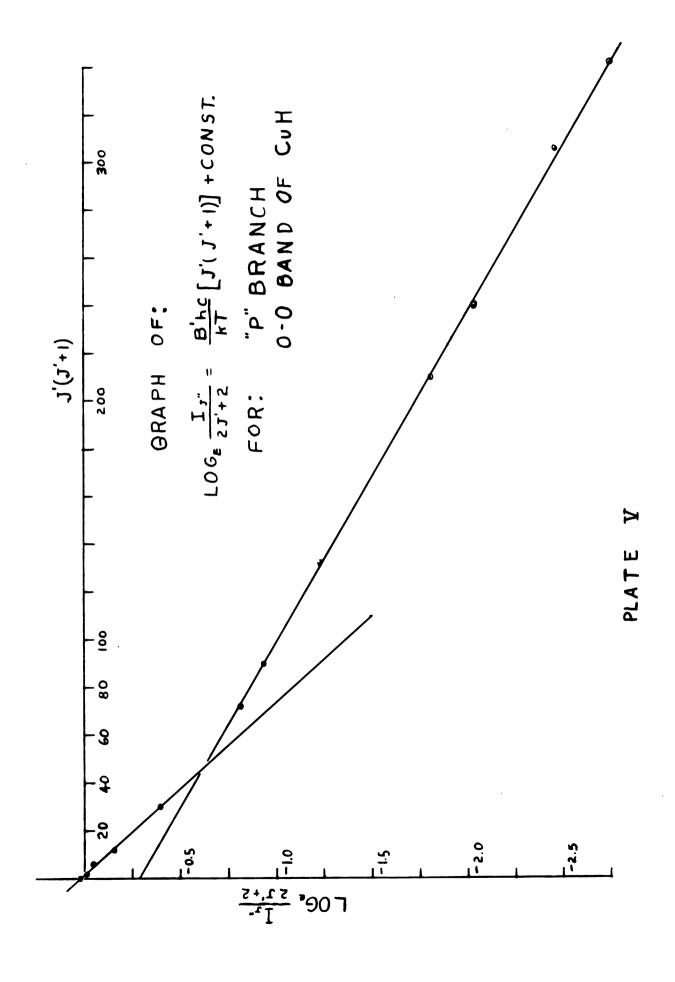
$$V_P = V_R$$
 $I_P = I_R$

Results

The data and graph for the temperature by the slope method may be found in Table III and Plate V. The calculations were made by using the measured intensities after discarding intensity values which because of error in the background correction (due to background being made up of diffuse lines as well as a continuous fog) are obviously out of place.

It will be noted that two separate and distinct straight lines result from the same branch. One line

					_	
						- AND J'(J'+1)
	J "	J'	J'(J'+1)	2 J*+2	I	$\log_{e} \frac{I}{2J'+2}$
	1	0	0	2	2.05	-0.0247
	2	1	2	4	3.61	-0.1026
	3	2	6	6	5.78	-0.0374
	4	3	12	8	6.80	-0.1625
	5	4	20	10		
	6	5	30	12	8.04	-0.4004
	7	6	42	14		
	8	7	56	16		
	9	8	72	18	7.94	-0.8186
	10	9	90	20	7.85	-0.9352
	11	10	110	55		
	12	11	132	24	6.57	-1.2266
	13	12	156	26		
	14	13	182	28		
	15	14	210	30	4.87	-1.8183
	16	15	240	32	4.18	-2.0357
	17	16	272	34		
	18	17	306	36	3.05	-2.4684
	19	13	342	39	2.43	-2.7497
·	20	19	380	40		



results from the low J" values and one from high values.

The temperature resulting from the slope of the line as drawn through the points representing low J" values is approximately 716° K and for the high J" values it is 1360° K. These values are quite critical. It was found that the calculated slope for 1360° + 50 = 1410° K could not be fitted to the points in Plate V for high J" values.

An explanation for the appearance of the two straight lines having different slopes any be found in the work of Lochte-Holtgreven and Maecker (7) who have considered the effects of temperature inhomogenosity, self absorption, self reversal and overlapping of lines upon the determination of temperature by this method. The effect observed above is a result of the temperature inhomogenosity of the That is, the intensity distribution in the branch results from a composite distribution function caused by the fact that some large proportion of the molecules in the arc are at some high temperature (in the core of the arc) and some small proportion are at some lower temperature (outer shell of the arc). The above workers have shown that such a condition for some two temperatures, say 1400° and 500, results in a curve such as is found in Plate V, the slope of which for higher J values gives a temperature of 1400° and for low J values a temperature considerably above 500°, say 700°. It may therefore be justifiably gaid that the temperature of the core of the copper arc is

(1360±30) K even though the temperature of the outer shell is not directly discernable. It should be noted however that by trial and error a choice of different proportions of molecules at different temperatures would give a composite intensity distribution which would fit the measured distribution.

and the corresponding composite intensity distribution, it is not to be expected that temperature determination by the intensity maximum or by the envelope intersection method will give valid results. Both of these methods depend upon the shape of the distribution envelope for low Jⁿ values, and it has been indicated above that when temperature inhomogenosity exists, the envelope for high J values only may be used. However the calculations were made for these methods with the following results:

A choice of J'' = 7 for the maximum J value gives 890° K and of J'' = 8 gives 1170° K. This difference in choice comes from the difficulty of determining the maximum of the measured intensity envelope.

The method of the envelope intersections for the P and R branches gives 1820° K for $J''_{P} = 2.6$ and $J''_{R} = 14.9$ as the J'' values of the intersection.

SUMMARY

A working model of a microdensitometer, along with accessory intensity calibration apparatus, has been designed, constructed, and applied to the problem of measuring the intensity distribution of the rotational structure of the O-O band of Copper Hydride.

In the process of intensity measurement, a method of making corrections for extreme background density of spectrograph plates has been tested and found to give reliable results.

Employment of the measured intensity distribution of the band has yielded information as to the temperature attained in a copper arc operated in an atmosphere of hydrogen.

The results obtained from the use of the working model indicate that it may well serve as the model for the construction of a microdensitemeter for routine density measurements in spectrographic intensity determinations.

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