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IN THE ARMY

4th QUARTERLY REPORT
SIGNAL CORPS CONTRACT NO. W-36-039-sc-36851
Period 15 March, 1949 to 15 June, 1949

ELECTROSTATIC ELECTROPHOTOGRAPHY

THE HALOID COMPANY
ROCHESTER, N. Y.

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Further information concerning this project may be obtained from Mr. Steven Levinos, Chief, Chemical and Methods Section, Photographic Branch, Squier Signal Laboratory, Fort Monmouth, N. J. Phone Eatontown 3-1060, extension 1612.

BATTELLE MEMORIAL INSTITUTE

INDUSTRIAL AND SCIENTIFIC RESEARCH

COLUMBUS 1, OHIO

June 23, 1949

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Dr. John Dessauer
The Haloid Company
Rochester 3, New York

Dear Dr. Dessauer:

We are enclosing 54 copies of Quarterly Progress Report No. 4 on Continuous-Tone Electrography. This report covers work for the three-month period from March 15, 1949, to June 15, 1949.

Major advances have been made in the achieving of high photographic speeds with selenium-coated electro-photographic plates and some advances have been made in the production of higher quality continuous-tone reproductions.

Work for the next quarter will be concentrated on further improvements in continuous-tone reproduction.

Very truly yours,

Lewis E. Walkup

Lewis E. Walkup
Assistant Supervisor
Graphic Arts Research Division

LEW:swr
Enc.

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QUARTERLY PROGRESS REPORT NO. 4
(March 15, 1949, to June 15, 1949)

on

CONTINUOUS-TONE ELECTROSTATIC ELECTROGRAPHY

to

THE HALOID COMPANY

(Subcontract Under Signal Corps Prime Contract
No. W36-039 sc-36851)

(Department of the Army Project: 3-99-04-052)

(Signal Corps Project: 195 B)

by

R. M. Schaffert, D. T. Williams, and L. E. Walkup

OBJECTIVE OF RESEARCH: To evolve an electrostatic electrographic
system capable of reproducing continuous-
tone photographs.

BATTELLE MEMORIAL INSTITUTE

June 15, 1949

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QUARTERLY PROGRESS REPORT NO. 4
(March 15, 1949, to June 15, 1949)

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(Subcontract Under Signal Corps Prime Contract
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(Department of the Army Project: 3-99-04-052)

(Signal Corps Project: 195 B)

from

BATTELLE MEMORIAL INSTITUTE

by

R. M. Schaffert, D. T. Williams, and L. E. Walkup

June 15, 1949

SUMMARY

This report covers experimental work on continuous-tone electro-
photography from March 15, 1949, to June 15, 1949.

Work for the past quarter was directed principally toward the two
objectives of the project which have been considered of greater importance,
namely, (1) photographic speed equivalent to 50 Weston, and (2) high-
quality continuous-tone reproduction. Notable advances have been made
toward both of these objectives during the quarter. In future work,

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emphasis will be concentrated primarily on improving the quality of continuous-tone prints. Consideration will also be given to the elements of the design of a camera embodying electrophotography.

Improved-quality continuous-tone pictures of outdoor scenes were produced at photographic speeds of A.S.A. 12 to A.S.A. 50 (A.S.A. rating approximately equivalent to Weston rating) as compared to a photographic speed of approximately A.S.A. 1 obtained with electrophotography in the past. These increases in photographic speed are attributable mainly to the introduction of powder-cloud developing methods using a development grid close to the plate during development and, to a lesser extent, to the use of plates having sensitivity to red light.

The powder-cloud development method does not produce consistent results in its present form due, probably, to nonuniformity in present equipment and methods. Work was initiated on the study of uniformity of powder-cloud development by setting up a fluorescent light source, equivalent to mean noon sunlight, to be used with a standard gray scale.

Experiments indicate that the electrophotographic process does not follow exactly the reciprocity law for light intensity and exposure time. Further work will be necessary to determine the practical importance of the observed departures from the reciprocity relationship.

Spectral transmission measurements indicate that selenium films absorb light in a manner qualitatively proportional to their spectral

sensitivities as electrophotographic plates. In the blue region of the spectrum, to which selenium electrophotographic plates are most sensitive, the light was found to penetrate the selenium layer only to about one-third of a micron or to about one-thirtieth of the thickness of the selenium film on a typical electrophotographic plate.

Other experiments demonstrated that the selenium layer on electrophotographic plates has its surface potential reduced approximately equally by light striking either the front or the back surface of the selenium layer. Selenium films deposited on electrically conductive glass were used for this experiment.

It was shown that selenium films on electrophotographic plates exhibit simple photoconductivity. An aluminum electrode was vacuum evaporated onto the selenium film of a plate. A battery and a galvanometer were then connected in series between the electrode and the metal backing plate. With this arrangement, a continuous electrical current was observed to flow through the selenium film as long as it was illuminated. The flow of current stopped when the illumination was removed. The electrophotographic plate with the auxiliary aluminum electrode would also accept a charge under the corona-discharge wires. This charge could be dissipated by an exposure to light in a manner similar to that of an ordinary selenium-coated plate.

Plates were treated with the vapors of the halides - bromide, iodine, and chlorine. All of these materials had detrimental effects on the plate, reducing the maximum potential which the plate would accept and increasing the dark-decay rate.

Exposure of selenium plates to infrared light produced inconsistent results. On some plates infrared irradiation increased the maximum potential which the plate would accept and decreased the dark-decay rate. On other plates, infrared irradiation appeared to have no effect or it decreased the potential which the plate would accept and increased the dark-decay rate slightly.

An apparatus was constructed for the addition of impurities to the selenium layer during vacuum evaporation. This apparatus involved a small screw-feed hopper into which could be introduced a mixture of selenium and the desired impurity. This hopper was intended to feed onto a hot evaporation source such as an electrically heated molybdenum boat. If the evaporation source was kept at a high enough temperature to evaporate either of the ingredients of the mixture then it was assumed that a uniform mixture of the two components would be deposited on the metal plate. The apparatus failed because of inability to overcome two defects, (1) the spattering of the materials from the evaporation source, and (2) the blocking of the screw feed with molten material.

Work on the simultaneous evaporation of selenium and an impurity was continued with a simple but less desirable method involving a separate evaporation source for the selenium and for the impurity. Successful plates were made with the first and only material tried, bismuth. Plates with bismuth added as an impurity appeared to accept higher surface potentials and to dark-decay less rapidly than plates coated with pure selenium. Work is to be continued with other impurities.

An extensive series of plates was made in which the aluminum backing plate was maintained at different temperatures during vacuum evaporation. Plates made at backing-plate temperatures between 60 and 70°C. were found to be more consistently acceptable and to exhibit better adhesion between the selenium and the aluminum than plates made outside of this temperature range. Red-light sensitivity was present only in plates made above 70 or 80°C.

Attempts were made to prepare selenium plates by methods involving vapor deposition of selenium at atmospheric pressures. Direct vapor deposition of selenium in atmospheres of nitrogen and helium did not produce acceptable plates. Acceptable plates were prepared by a method involving the heat vaporization of selenium into a stream of natural gas, followed by burning of this gas against a cool aluminum plate.

The suggestion was made by Dr. Helmut Kallmann of the United States Signal Corps that cadmium of zinc sulfide deposited onto a conducting plate might produce a satisfactory electrophotographic plate. Results from preliminary experiments indicate that plates made in this manner will function as electrophotographic plates, but no measurements have been made on photographic speed or on the quality of prints which can be produced from them.

The vibrating-probe electrometer was equipped with a device for automatically plotting the potential-decay curve on logarithmic paper as readings are being made. This device decreases the time required to make potential-decay measurements on plates.

An additional theoretical treatment of the selenium layer of an electrophotographic plate has been advanced. This theory involves the assumption that photoconductivity in vitreous selenium films is dependent on the presence of interstitial electrical charges. The results of this theoretical treatment are in qualitative agreement with experimental results obtained with selenium-coated plates.

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FUTURE WORK

Improvement of the quality of continuous-tone images produced by electrophotography will be the major objective for work during the next three months. Minor objectives will be the improvement in quality of electrophotographic plates and the development of the elements of a practical camera design.

The following more specific factors will receive attention:

1. Improvements in the powder-cloud developing technique by a study of the physical factors involved.

A. Establishment of test methods for measuring the quality of image, involving uniform exposure conditions, the use of a gray scale and the introduction of definite procedures in exposure and development process.

B. The testing of a variety of powders.

C. The investigation of alternative methods of producing a powder cloud.

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2. The investigation of development methods other than the use of a powder cloud. Further consideration will be given to development by cascading powder-carrier combinations across the plate, and to development by means of vapors.

3. Production of more uniform and more sensitive electrophotographic plates, involving selenium vacuum evaporated onto aluminum, by a study of the physical factors involved.

A. Effect of gas pressure on quality of selenium film.

B. Effect of nature of gas in vacuum chamber during evaporation.

C. Effect of electrical discharge during vacuum evaporation.

4. Addition of impurities to selenium in the attempt to increase the photographic speed and to alter the spectral sensitivity of selenium plates.

5. Determination of the effect of chemical treatment of selenium plates in an attempt to increase photographic speed and alter spectral sensitivity.

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6. Continuation of work on the use of cadmium sulfide and zinc sulfide in electrophotographic plates.
7. Continuation of the attempt to arrive at a theoretical explanation of electrophotography.
8. Development of the elements of a design for a practical camera involving electrophotography.

PUBLICATIONS AND REPORTS

(None)

EXPERIMENTAL WORK ON PLATE-DEVELOPING TECHNIQUES

(D. L. Fauser and J. P. Wronski)

Development of Plates with Electrically Charged Powder Cloud

The powder-cloud developing box, as described in Quarterly Progress Report No. 3, pages 285 to 390, was used to develop

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electrophotographic plates for an evaluation of their effective photographic exposure speeds.

It was found possible to produce improved-quality pictures of outdoor scenes at photographic speeds of A.S.A. 12 to A.S.A. 25 as compared to photographic speeds of approximately A.S.A. 1 obtained with electrophotography in the past. This improvement in photographic speed is primarily the result of an improved powder-cloud developing method and to a much lesser extent, to the use of electrophotographic plates having sensitivity to red light. (Red-sensitive plates are described in Quarterly Progress Report No. 3, pages 218 to 229.)

For these experiments, a camera was used to expose sensitized electrophotographic plates to an outdoor scene. The illumination of the scene was measured with a General Electric exposure meter. The practical plate speed was then determined from the readings on the exposure and lens opening which gave optimum results in the finished electrophotographic print.

In actual experiments, the plates were sensitized on a conventional unit in which the plate is carried under the corona-discharge wires by a motor-driven belt system. The time intervals involved were as follows:

1. One minute between sensitizing and exposing.
2. Five minutes between exposing and developing.
3. One-minute developing time.

Development involved the use of the development grid and the sorting grid as described in the first reference above. The developer consisted of approximately equal parts of A-1-K powder* and soybean meal**.

Figure 92 shows the electrical conditions used during the actual developing process. Approximately 800 volts potential difference was maintained between the sorting grid and the development grid. This potential was applied to attract negatively charged powder to the plate and to keep positively charged powder from reaching the plate. The potential difference between the conducting back of the electrophotographic plate and the development grid was adjusted to a value between zero and 500 volts to produce the best possible print.

Figure 93-a is a reproduction of the best electrophotograph produced at a photographic speed of A.S.A. 12 with a plate coated with selenium to the conventional thickness of 15 microns. The large clear areas on this print correspond to areas of the plate from which the selenium film had flaked off. As yet, there is no explanation for the presence of the small white spots on this print.

A preliminary investigation was undertaken to evaluate the advantages of plates having considerably thicker layers of selenium than those generally used. It has been noted that thick selenium-coated plates, when sensitized to their maximum potential, have light-decay rates which are more rapid than those of conventional thin selenium-coated plates. A plate with a selenium film approximately 60 microns in thickness (about

* 95.2 per cent (by weight) Amberol F-71 (Resinous Products and Chemical Co., Inc., 222 W. Washington Square, Philadelphia, Pa.) and 4.8 per cent raven bead carbon black (Binney and Smith Co., New York, New York (by weight).

** Obtained from Swift and Company, Fostoria, Ohio. Screen cut between 16 and 48 mesh.

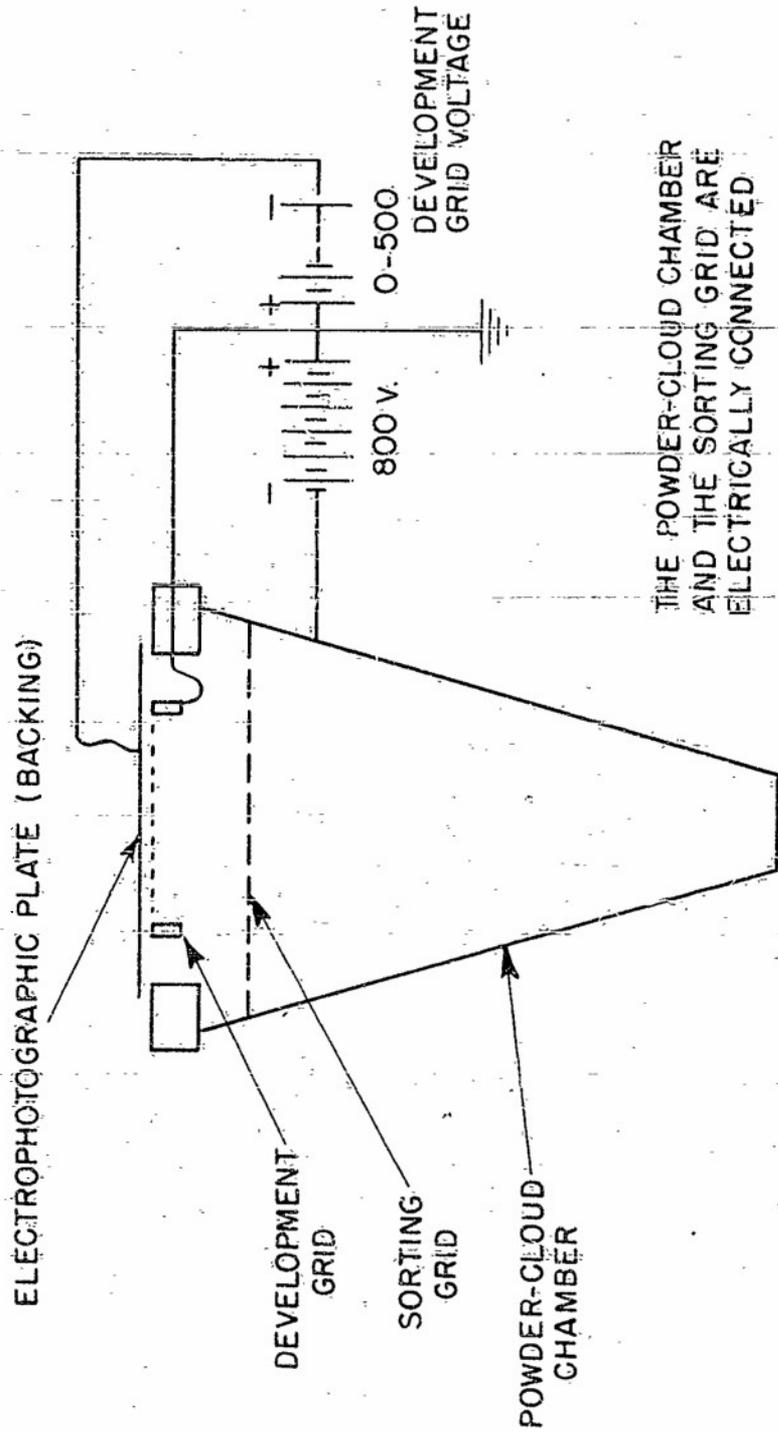
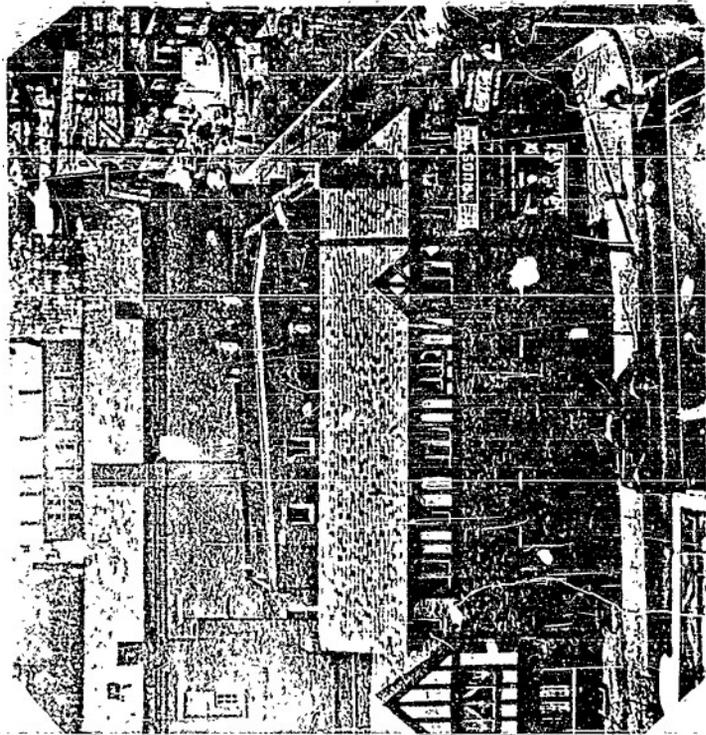
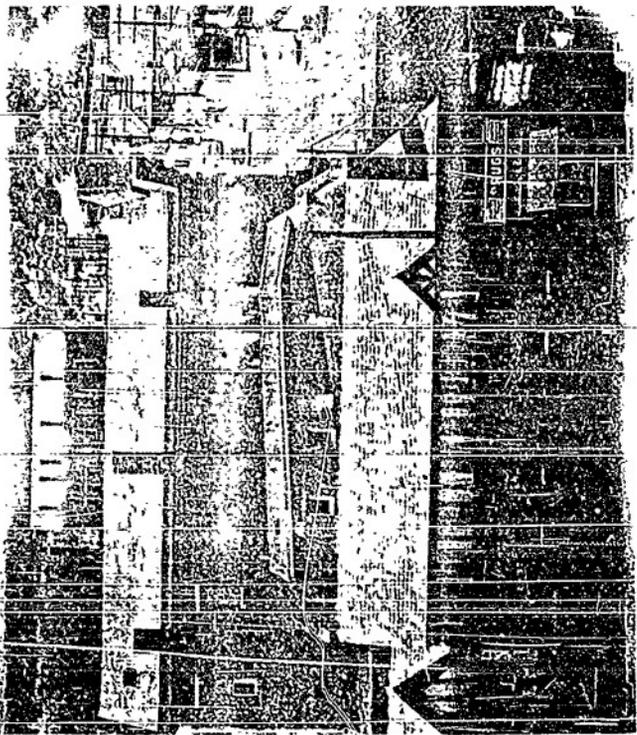


FIGURE 92. ELECTRICAL CONNECTIONS OF POWDER-CLOUD CHAMBER

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93a. PLATE, 2-29-49 A
SPEED, ASA 12
(F. 10, $\frac{1}{25}$ SECOND)
GRID POTENTIAL, 5 VOLTS



93b. PLATE, 5-5-49
SPEED, ASA 50
(F. 20, $\frac{1}{25}$ SECOND)
GRID POTENTIAL, 300 VOLTS

FIGURE 93. OUTDOOR ELECTROPHOTOGRAPHIC PICTURES DEVELOPED BY THE
POWDER-CLOUD TECHNIQUE.

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four times normal film thickness) was used in an attempt to attain higher photographic speeds. Figure 93-b is a reproduction of a print obtained from this plate at a speed of about A.S.A. 50. A picture of somewhat poorer quality was obtained at a photographic speed of A.S.A. 100.

Several light sources were evaluated for use in the illumination of a gray scale which is to be used in a further evaluation of electrophotographic continuous-tone photography. Combinations of daylight and blue fluorescent lamps were found most suitable for providing the intensity and color temperature required to simulate mean noon sunlight.

Reproducibility Studies Using Powder-Cloud Development

A combination color chart and gray scale was prepared and illuminated with light having approximately the same spectral characteristics as mean noon sunlight. A series of nine photographs of the chart was then taken using a selenium-coated electrophotographic plate at an A.S.A. speed of approximately three. Caution was exercised to maintain uniform photographic and development conditions throughout the series of tests. The densities of various parts of the photographs were then determined using a reflection densitometer. From this group of photographs, it was found that the densities of corresponding points varied by as much as 100 per cent.

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EXPERIMENTS ON VACUUM-EVAPORATED SELENIUM
ELECTROPHOTOGRAPHIC PLATES

(P. G. Andrus, D. C. Reynolds, J. L. Stockdale,
O. A. Ullrich, and J. P. Wronski)

Photographic Reciprocity in Selenium
Electrographic Plates

A series of tests were conducted on one electrographic plate to determine the relationship between the light intensity and the exposure time required to produce a given potential drop on the plate. The tests were directed toward finding a relationship similar to the reciprocity relationship for silver halide photography. However, instead of studying the photographic exposure necessary to produce a given image density, a given potential drop was chosen in order to eliminate variations caused by the development process. The data were taken in the form of potential drops resulting from various exposure times while the light intensity was held constant. These data were taken for several different light intensities at each of six different wavelengths of light.

From these data the exposure times and light intensities required to produce a given potential drop (in this case, 100 volts) were chosen. The relationship between these chosen exposure times and light intensities then provided the reciprocity information which was sought.

The tests were carried out using a white-light source filtered to produce the desired wavelength and light intensities. Wavelengths of 652, 606, 557, 459, 406, 353 millimicrons were used at intensities

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ranging from 0.33 to 185 microwatts per square centimeter. An "Alphax" camera shutter was used to produce exposure times of 0.04, 0.1, 0.2, and 0.5 seconds. Plate 12-21-48D was used.

The values for the plate potential drops for the various exposures at different wavelengths were determined in the following manner. The plate was charged using 7000 volts positive potential on the corona-discharge needle. The plate was regenerated between successive tests. The net potential drop for a given exposure consisted of the difference between the dark-decay curve and the decay curve after exposure, measured five minutes after charging the plate. (See Figure 94.) In every case, the plate was exposed at the time when its dark-decay curve passed through 600 volts. This always occurred within thirty seconds after the end of the charging cycle. This procedure was adopted in order to avoid difficulties arising from the dark-decay rate and also because the characteristics of selenium electrophotographic plates are somewhat stabilized after about five minutes' time.

Figure 95 illustrates the typical manner in which potential drop varies with exposure time for various light intensities at a wavelength of 406 millimicrons. From similar curves obtained at four other wavelengths, the exposure time necessary to produce a 100-volt drop of plate potential at each corresponding intensity was found. As shown in Figure 96, these values cluster rather closely about straight lines when plotted on log-log graph paper. From these curves, therefore, it may be inferred that the relationship between intensity and exposure time is expressed as follows:

$$(I) (t^m) = k$$

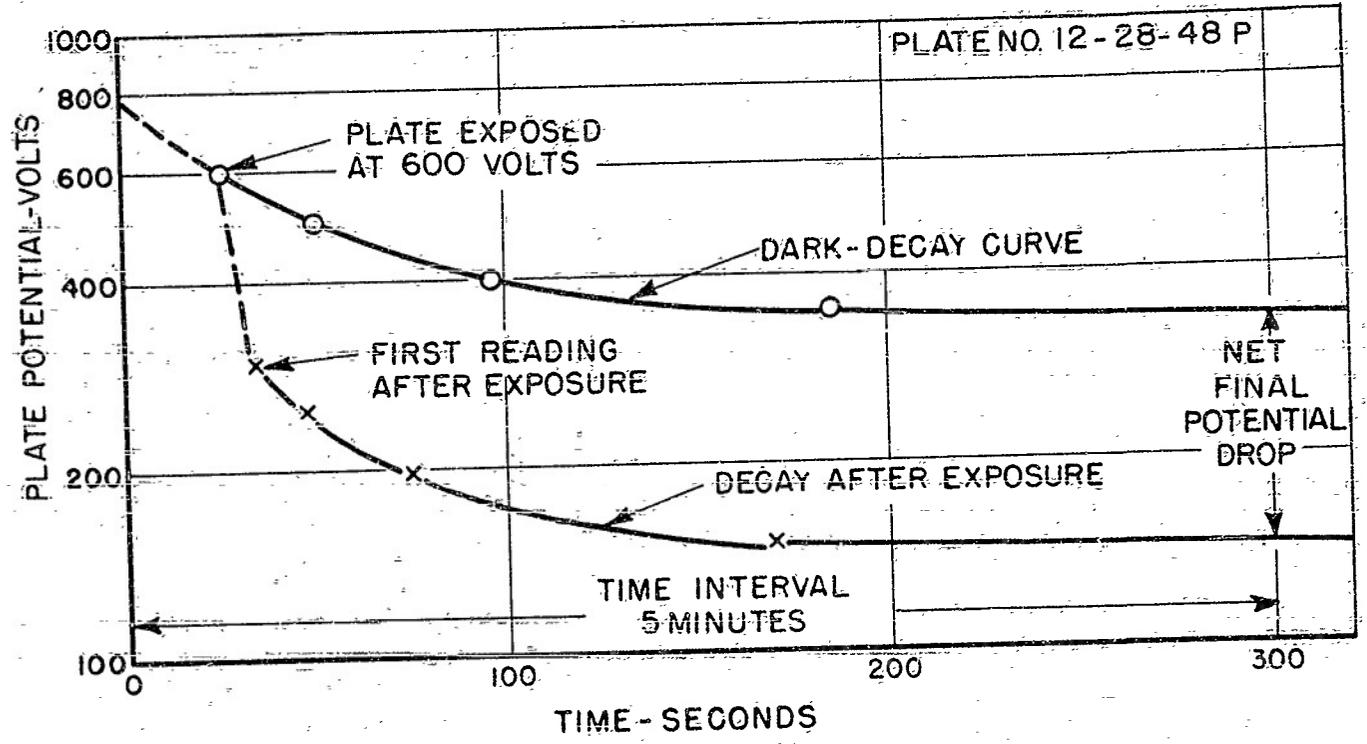
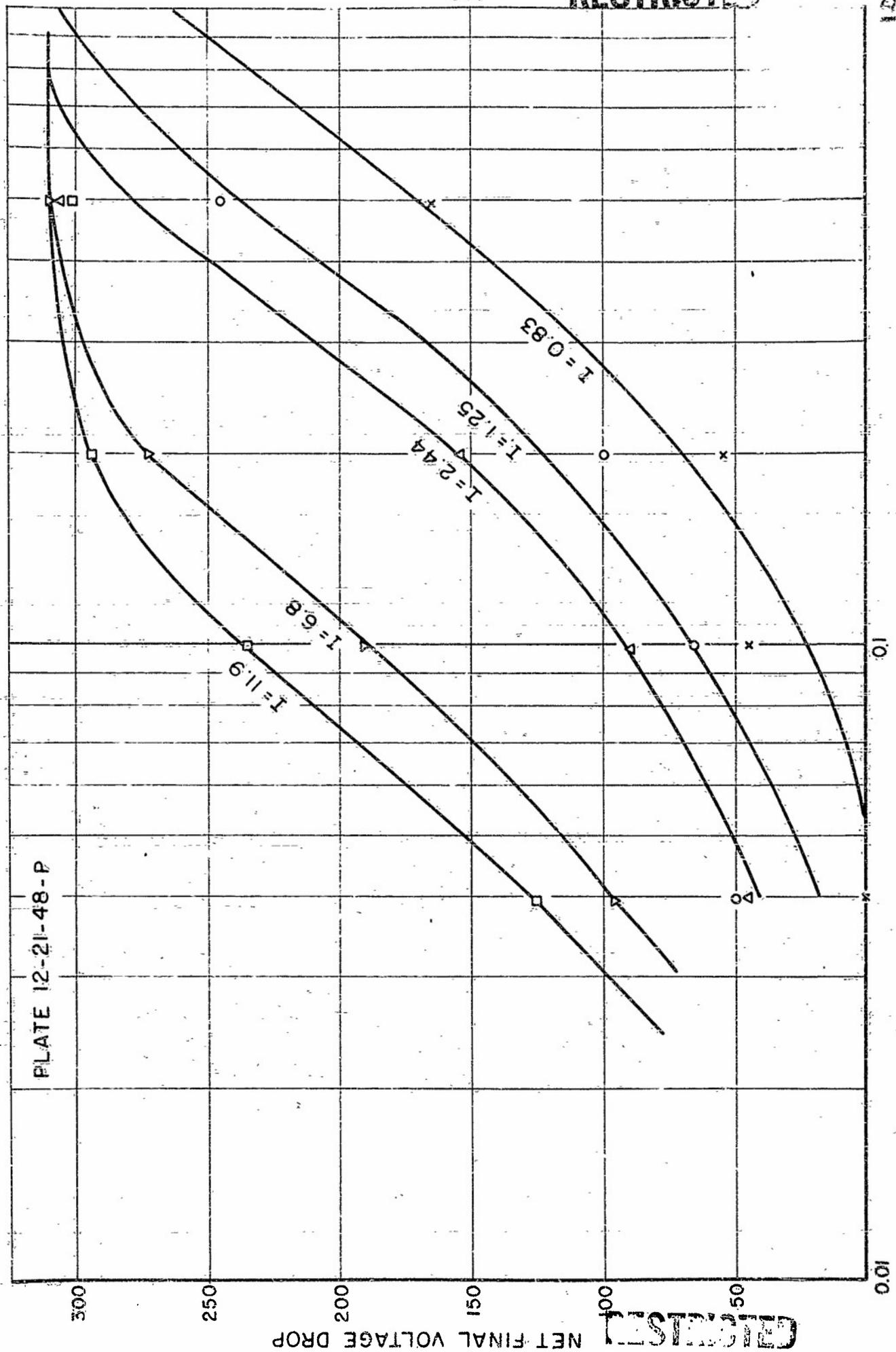


FIGURE 94. DEFINITION OF NET POTENTIAL DROP

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EXPOSURE TIME - SECONDS

FIGURE 95 - NET FINAL VOLTAGE DROP OF SELENIUM-COATED ELECTROPHOTOGRAPHIC PLATE VERSUS EXPOSURE TIME IN SECONDS FOR VARIOUS LIGHT INTENSITIES (I) IN MICROWATTS PER SQ. CM. AT WAVE LENGTH 406 MILLIMICRONS

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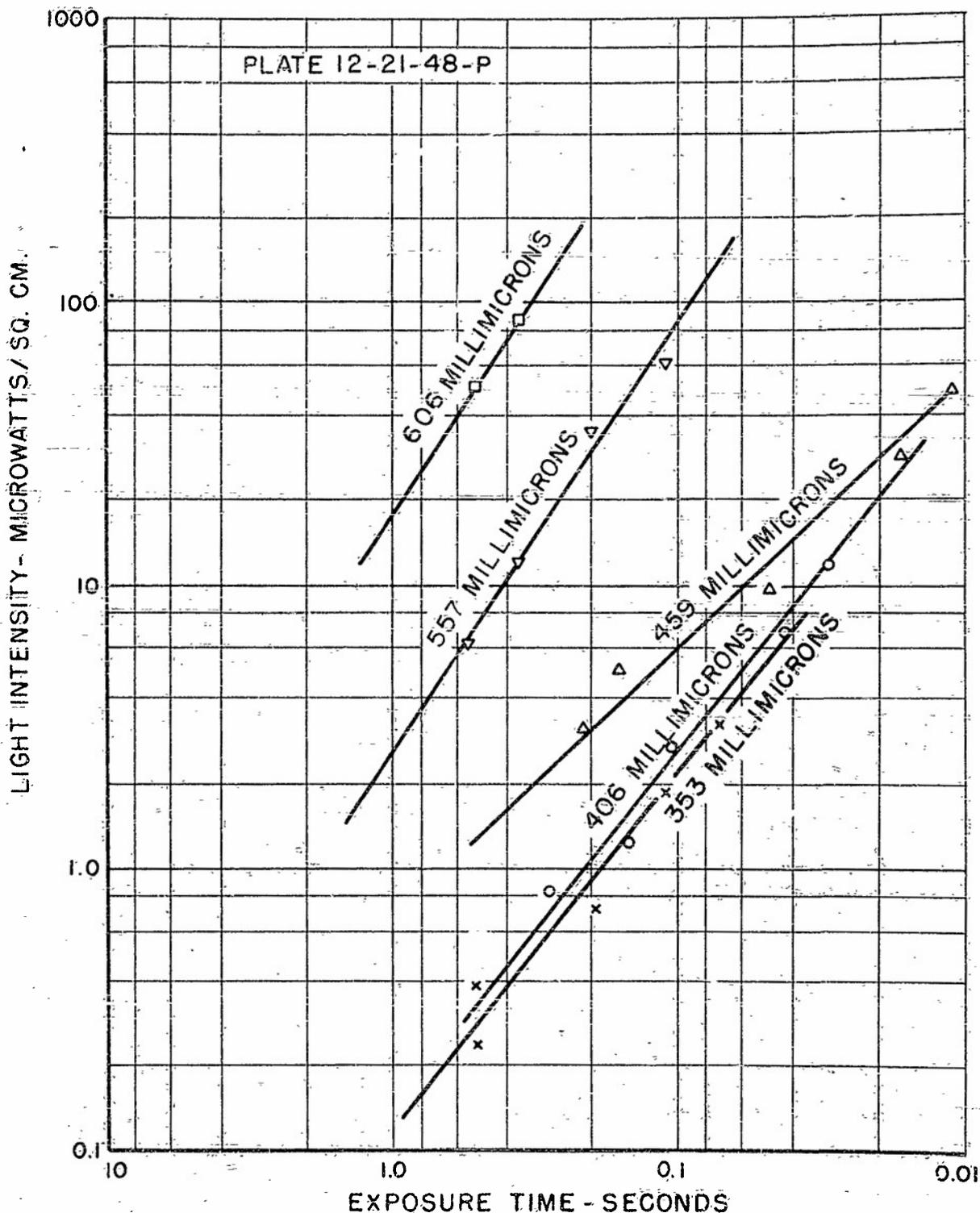


FIGURE 96. RELATIONSHIP BETWEEN LIGHT INTENSITY FALLING ON A SELENIUM-COATED ELECTROPHOTOGRAPHIC PLATE AND EXPOSURE TIME, FOR VARIOUS WAVELENGTHS, NECESSARY TO PRODUCE A NET VOLTAGE DROP OF 100 VOLTS.

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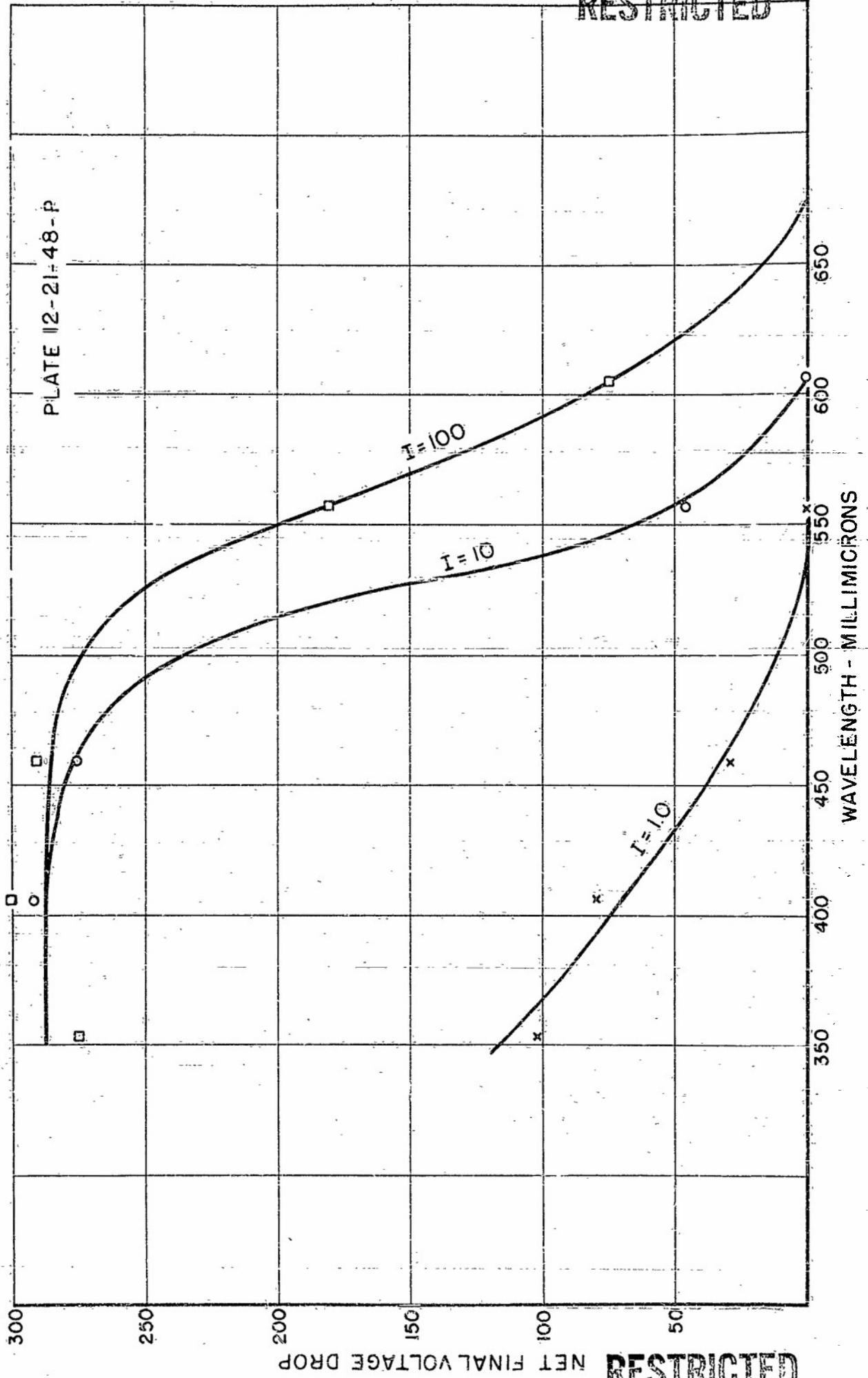
where I is the intensity of the light in microwatts per square centimeter, t is the exposure time in seconds, m is a quantity equal to the slope of the line on log-log graph paper, and k is a parameter which, in part at least, is a function of the wavelength of the radiation. From the curves, the following values for m were found:

<u>Wavelength,</u> <u>Millimicrons</u>	<u>m</u>
353	1.2
406	1.2
459	1.0
557	1.5
606	1.5

If the value of m is indeed greater than unity, as the above values indicate, then, in practical effect for exposure times less than one second, the above equation implies that higher light intensities are needed to produce a 100-volt drop than would be necessary if the normal relationship $(I)(t) = k$ were operative.

The curves in Figure 97 were derived from those in Figure 95 and from other similar curves, and indicate the manner in which voltage drop is dependent upon wavelength for various light intensities at an exposure time of 0.2 second. Here is illustrated, in a manner of presentation somewhat different from that used heretofore, the very strong sensitivity of selenium electrophotographic plates to blue light compared to its sensitivity to red light. Though this plate is not one which has been termed red-sensitive, the curves show that its sensitivity does not cut off sharply as previously suggested, but actually extends as far into the red as 650 millimicrons. The sensitivity in this red region, however, is extremely low.

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NET FINAL VOLTAGE DROP

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FIGURE 97. NET FINAL VOLTAGE DROP OF SELENIUM-COATED ELECTROPHOTOGRAPHIC PLATE VERSUS WAVELENGTH FOR VARIOUS INTENSITIES I (IN MICROWATTS PER SQ. CM.) AT AN EXPOSURE TIME OF 0.2 SECOND

Figure 98 shows another typical set of curves illustrating the manner in which the voltage drop varies with light intensity for various exposure times.

Inasmuch as the tests described here were conducted using a single electrophotographic plate, it must be assumed that the curves and equations for other plates will be similar in form but will probably differ in the values of the parameters involved.

Spectral Transmission and Reflection Characteristics of Selenium Films

Spectral transmission curves were obtained for five selenium films vacuum evaporated onto glass. The curves, Figure 100 in particular, show that blue light, to which the selenium plates are most sensitive, penetrates the selenium film to only a small fraction of its total thickness. This is of considerable significance in the formulation of a theory of the photoconductivity of selenium.

The thicknesses of the five selenium films tested were 0.05, 0.35, 0.88, 1.4, and 15 microns. These films were prepared by the vacuum evaporation of selenium from molybdenum boat-type sources. The different thicknesses of film were produced by placing the glass plates at different distances from the evaporation sources. The transmission curves are shown in Figures 99 through 103.

The maxima and minima which appear in the first four curves are not due to real differences in absorption, but arise from interference of the multiple reflections from the air-selenium and the selenium-glass interfaces. In Figure 103, the maxima and minima are so close together

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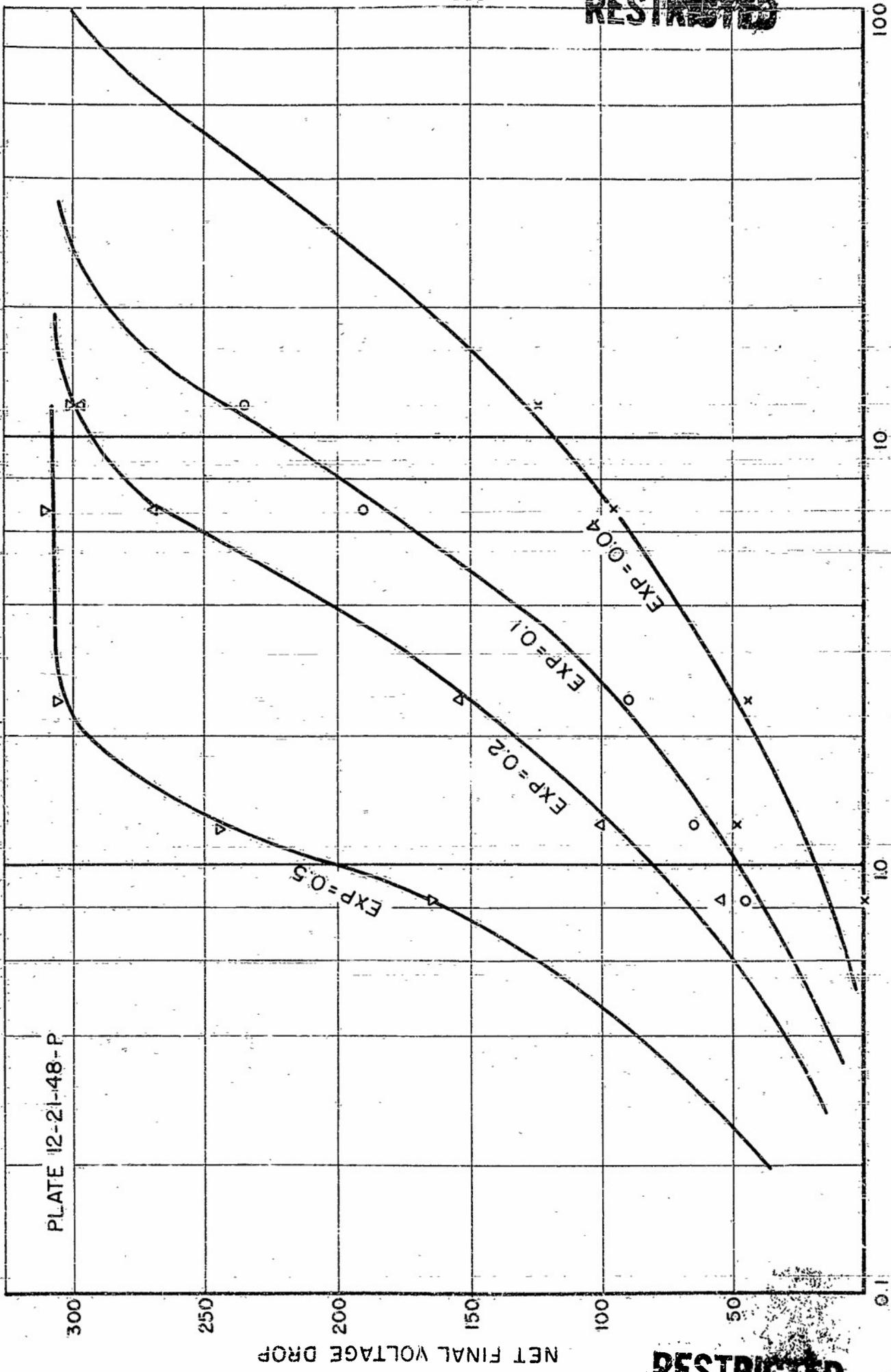


FIGURE 98. NET FINAL VOLTAGE DROP OF SELENIUM-COATED ELECTROPHOTOGRAPHIC PLATE VERSUS LIGHT INTENSITY FOR VARIOUS EXPOSURE TIMES (IN SECONDS) AT WAVELENGTH 406 MILLIMICRONS

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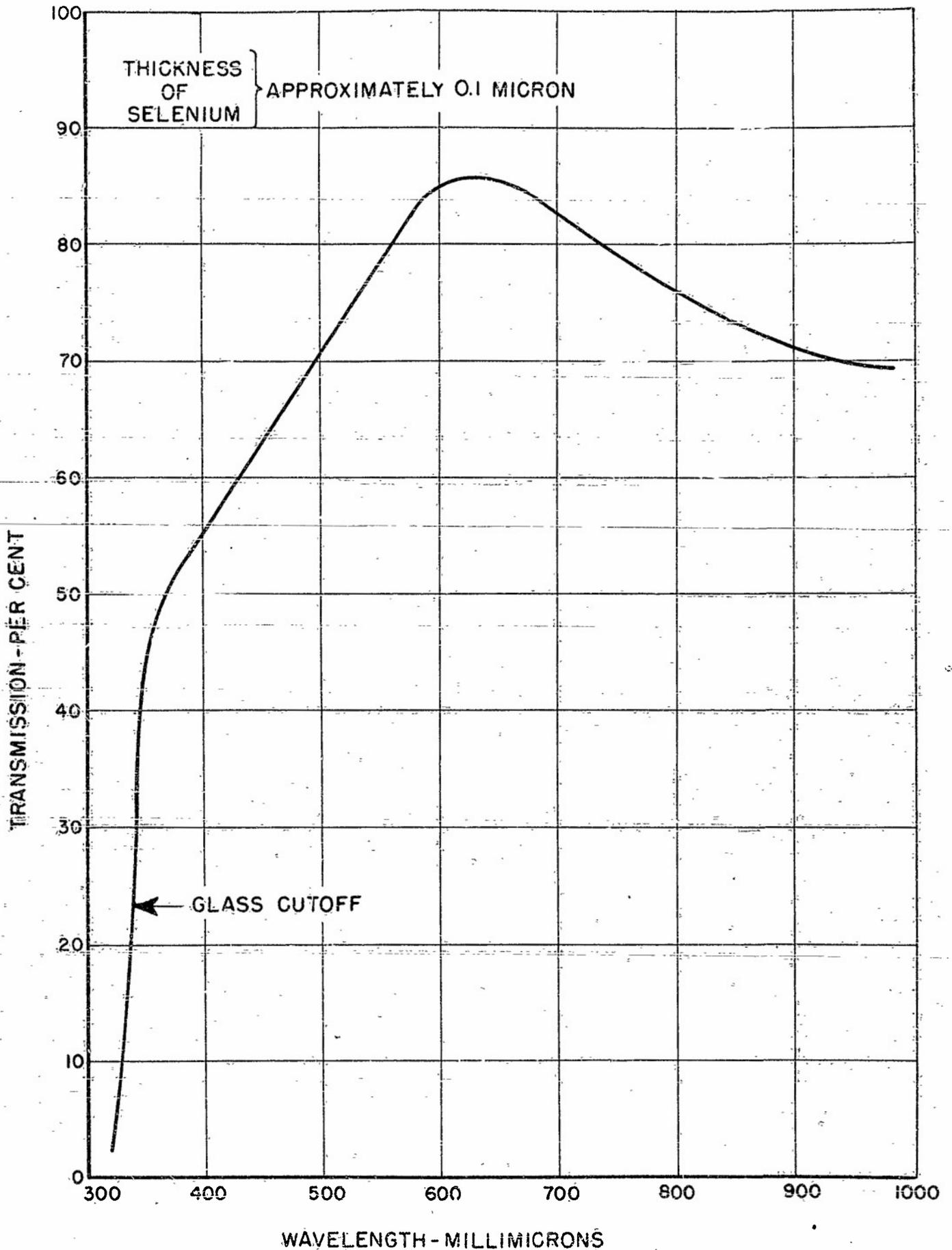


FIGURE 99. TRANSMISSION OF SELENIUM FILM ON GLASS

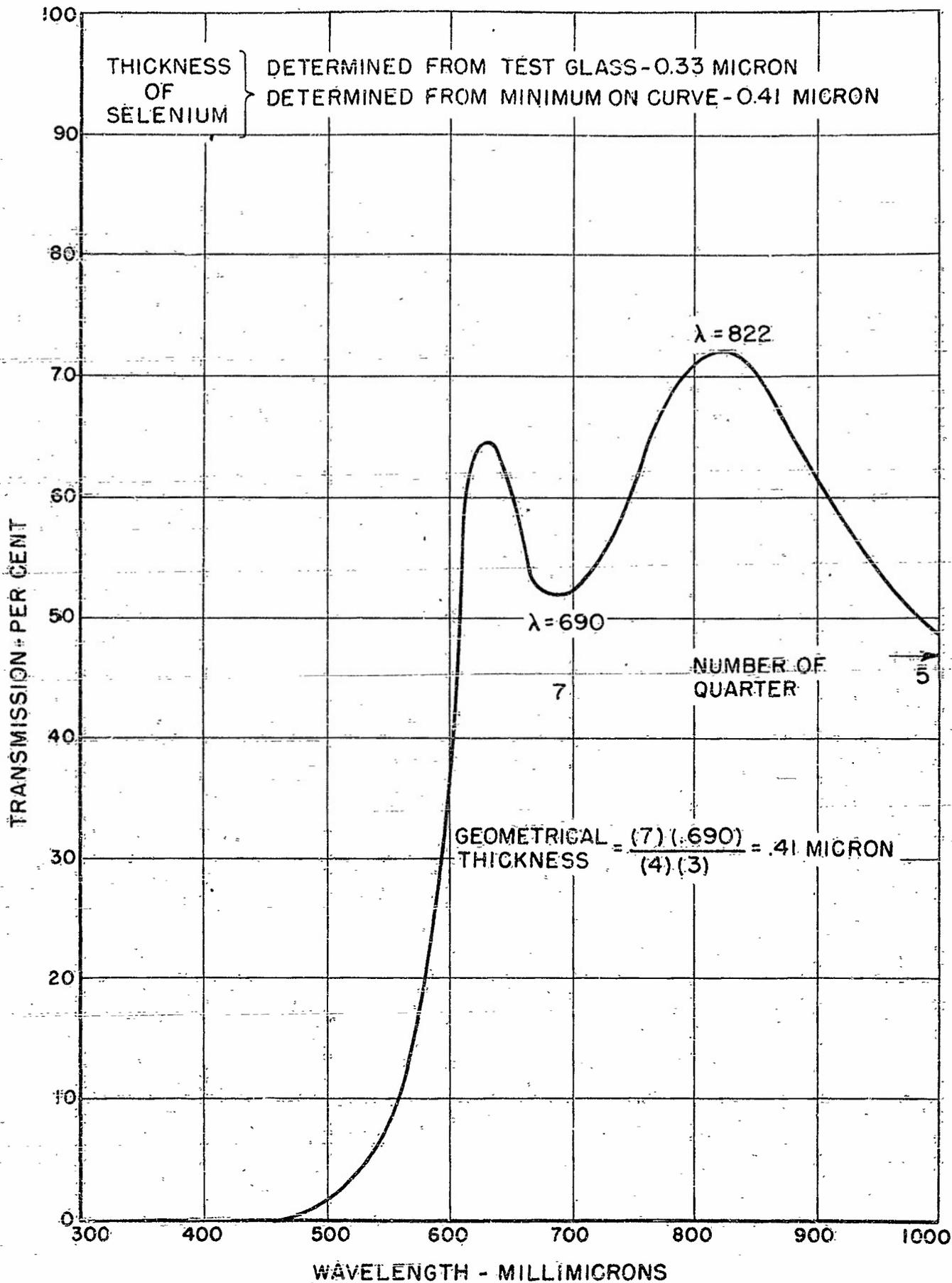


FIGURE 100. TRANSMISSION OF SELENIUM FILM ON GLASS

O-12057

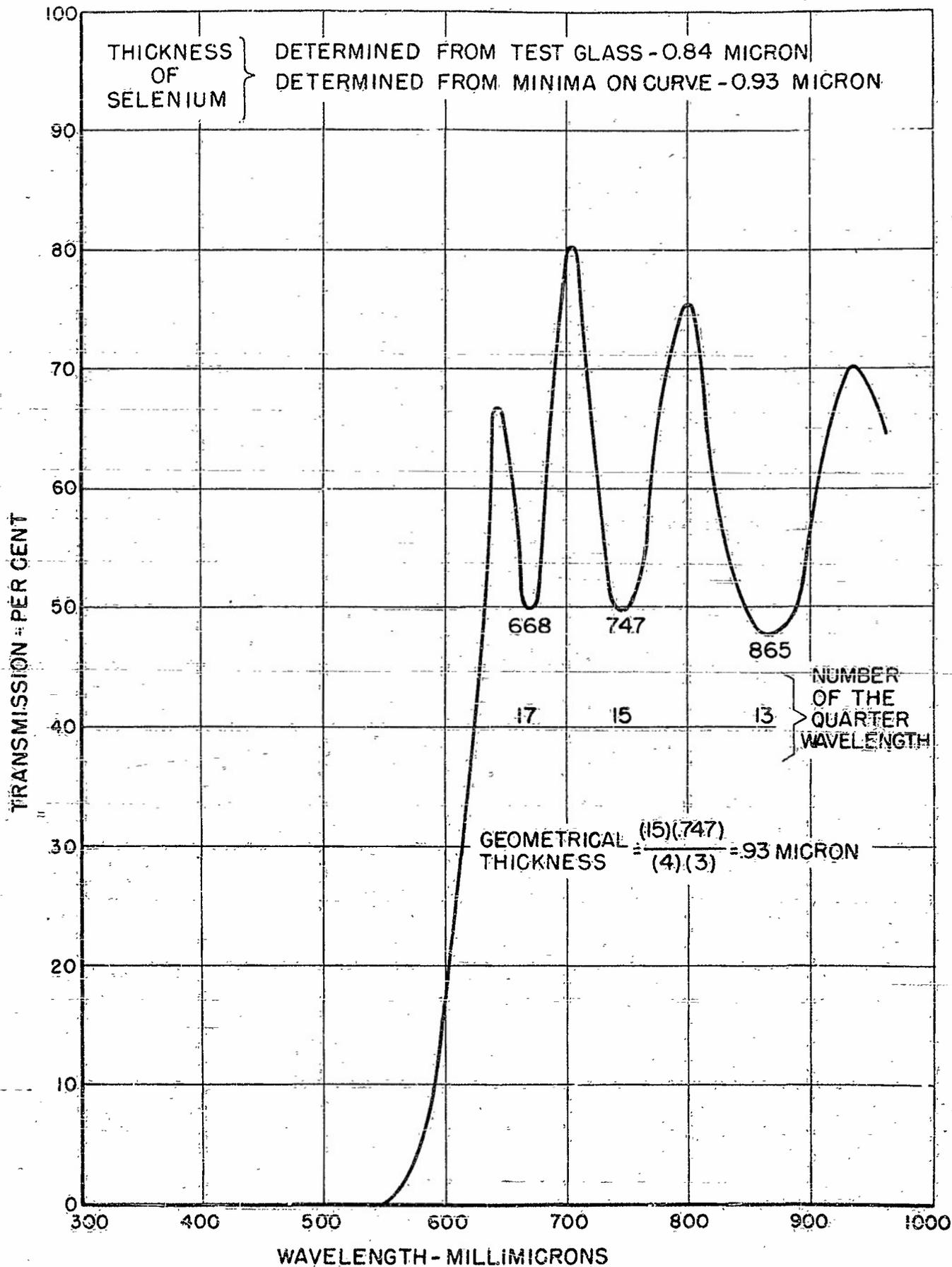


FIGURE 101. TRANSMISSION OF SELENIUM FILM ON GLASS

0-12058

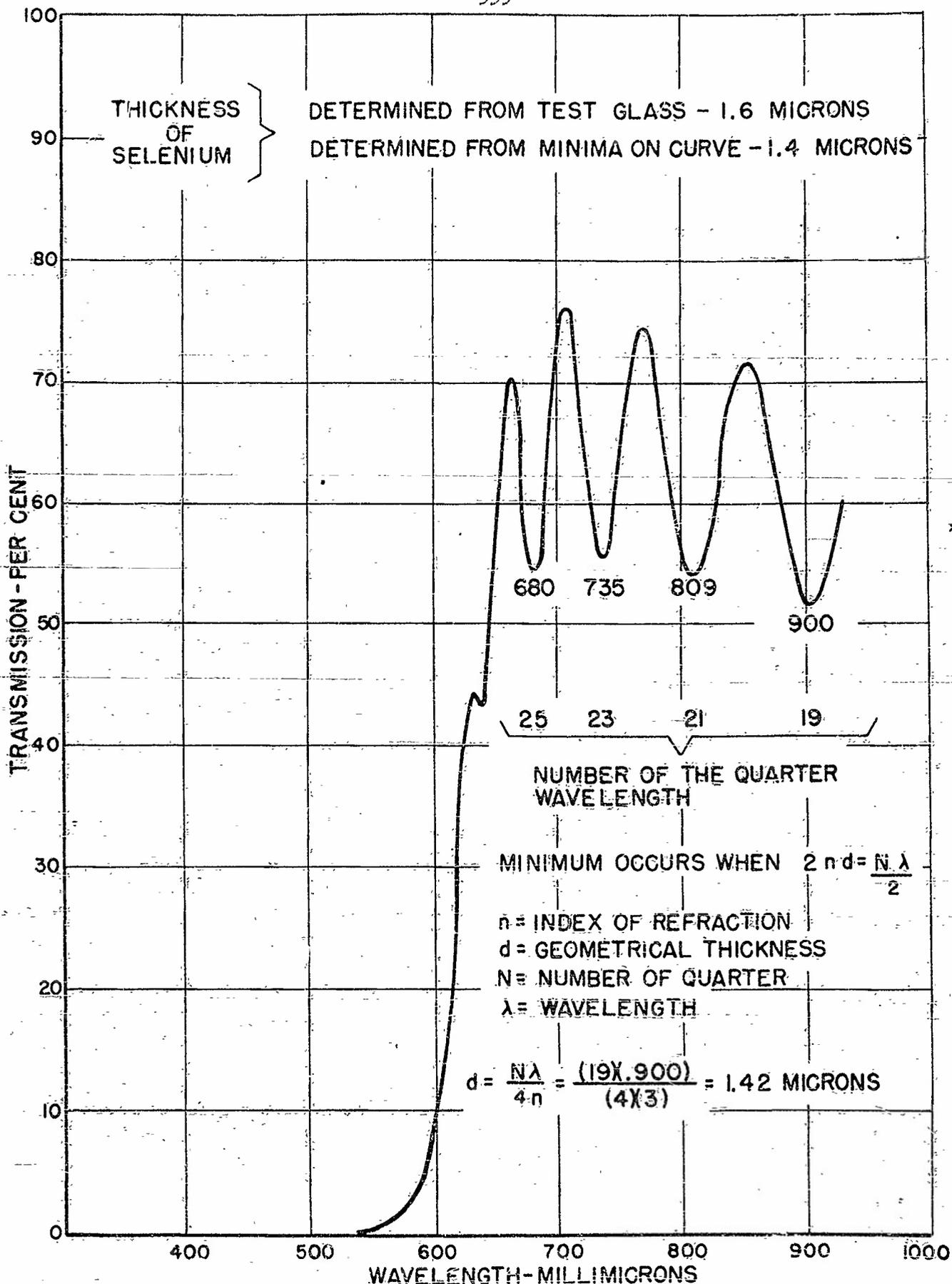


FIGURE 102. TRANSMISSION OF SELENIUM FILM ON GLASS

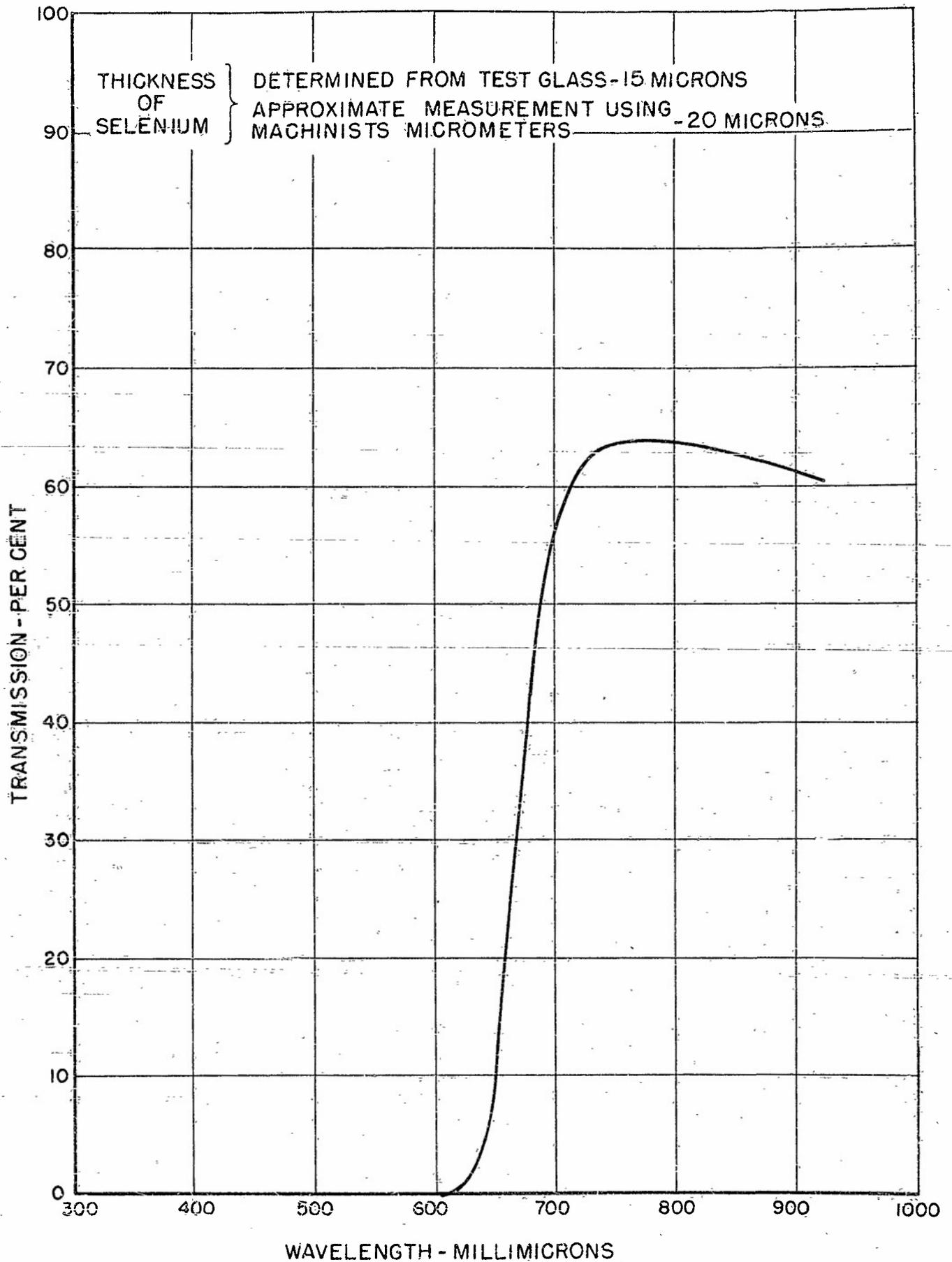


FIGURE 103. TRANSMISSION OF SELENIUM FILM ON GLASS

that they cannot be resolved by the Beckman spectrophotometer used in these experiments. The number of maxima and minima which appears in any wavelength range depend upon the distance between the reflecting interfaces - in this case, the thickness of the selenium film. If the index of refraction of the selenium in the spectral region in which the maxima and minima occur is known, the geometrical thickness of the film can be determined as follows. For a high index of refraction film, i.e. index of refraction higher than that of the medium on which it is placed, the minima in the transmission curve represent odd quarter wavelengths of optical film thickness. The formula which applies for normal incidence is,

$$2nd = \frac{NL}{2}$$

where n is the index of refraction of the selenium (approximately three), d is the geometrical thickness of the film, L is the wavelength at the minima, and N is the number of the quarter wavelength minimum involved. In the present case, both d and N are unknown, but since, in three of the curves, there are several N's, or minima, to use, N can be determined by assuming values of N until the product NL is the same for each minimum. In Figure 102, this is the case when the minimum at 900 millimicrons is taken as the nineteenth quarter wavelength. Then the geometrical thickness is found to be,

$$d = \frac{NL}{2n} = \frac{(19)(0.900)}{(4)(3)} = 1.4 \text{ microns.}$$

The selenium film thickness may also be determined as described in Quarterly Report Number 2, pages 156 to 158. This method consists in

counting the number of interference fringes between the surface supporting the selenium film and an optically flat surface placed as shown in Figure 104.

Of the two methods, the one involving the interference maxima and minima in the curves is the more accurate.

The spectral transmission curves show that the transmission of a film only 0.35 micron thick is less than one per cent for light of 450 millimicrons' wavelength. Normal electrophotographic plates have selenium films of at least ten-micron thickness. This means that, if the spectral transmission characteristics of the selenium film on an ordinary electrophotographic plate are the same as these selenium films on glass, the blue light to which the plates are most sensitive does not penetrate the photoconductive film to a depth of more than one-third of a micron. This is equivalent to penetrating the film a distance of about one-thirtieth of its total thickness. The evidence is strong, therefore, that the effect of the light itself is purely a surface phenomenon. The case is similar to the electron bombardment electrical conductivity of diamond. Here the bombarding electrons penetrate the diamond only a very small fraction of its total thickness. Nevertheless, this bombardment causes an electrical current to flow between the surfaces of the diamond across which a potential is applied. The predicted and measured rates of electrical current flow in diamond, after bombardment ceases, are similar to the voltage-decay rates obtained for selenium-coated electrophotographic plates when they are exposed to light.

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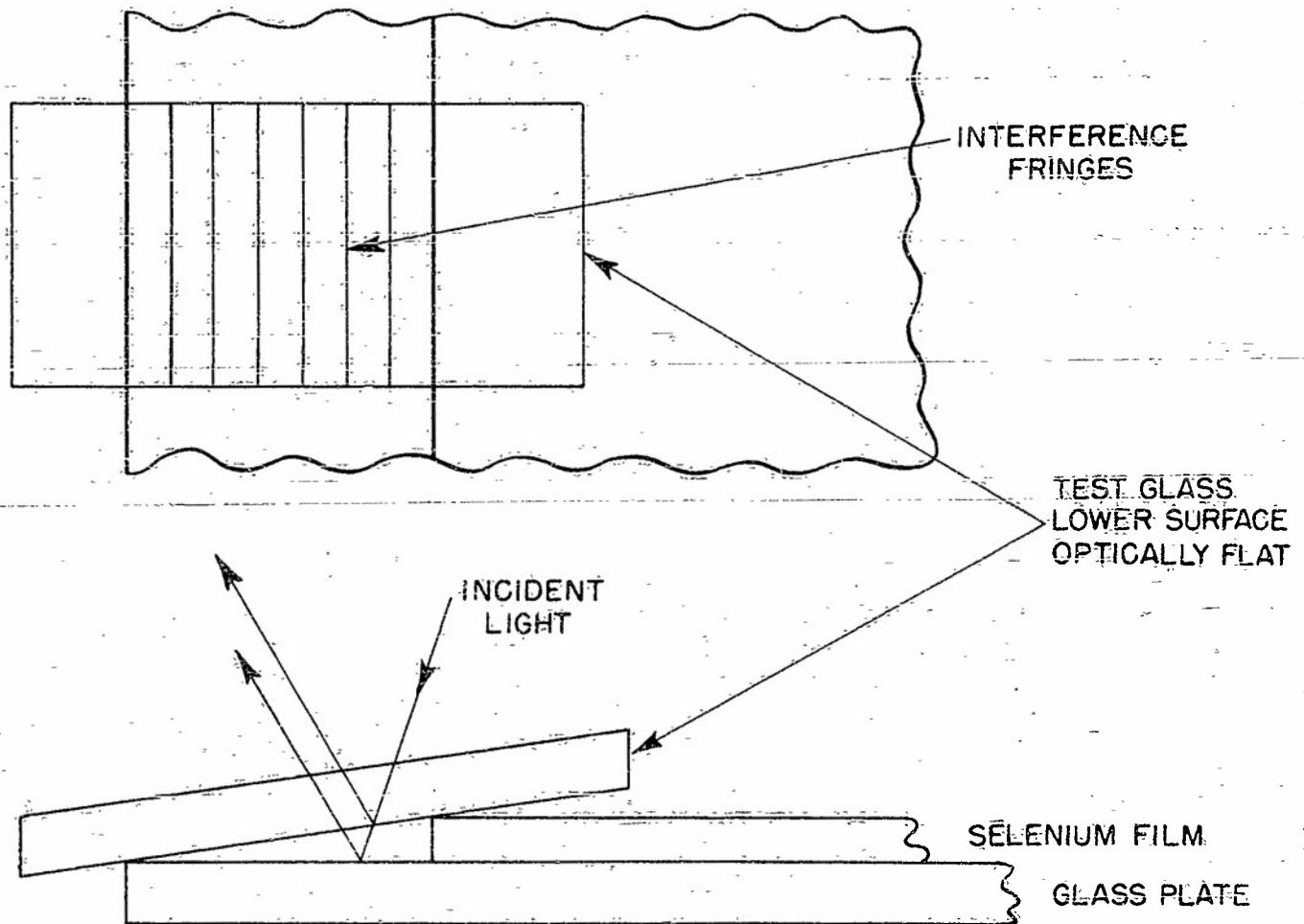


FIGURE 104. TEST-GLASS DETERMINATION OF SELENIUM FILM THICKNESS

0-12061

The spectral transmission curves also show a gradual shift of the blue-absorption cutoff toward the red end of the spectrum with increasing thickness of selenium film.

A comparison of the spectral transmission characteristics of red-sensitive and non-red-sensitive selenium films was also made. The purpose was to determine whether or not selenium films which exhibited red sensitivity, absorb light of those red wavelengths to which they are electrophotographically sensitive. The curves in Figure 105 are representative of several films tested. These results show that red-sensitive selenium films do have considerable absorption in the red spectral region. However, this red absorption is not nearly so complete as the blue absorption exhibited by the selenium films. The data for these curves were obtained by stripping the selenium from the aluminum backing plate with cellulose adhesive tape and measuring the transmission of the selenium-tape combination against plain cellulose tape as a reference.

The reflectivity of a non-red-sensitive selenium film approximately 15 microns in thickness on glass was also measured. It is shown in Figure 106. Also shown is the transmission of the same film-and-glass combination. Therefore, with a reflectivity of 25 per cent as shown in Figure 106, and a transmission of approximately 65 per cent as shown in Figure 103, it can be seen that, as expected, non-red-sensitive selenium films have relatively little absorption in the red end of the spectrum.

Simple Photoconductivity in Electrophotographic Plates

An electrophotographic selenium plate coated to a thickness of approximately sixty microns was prepared in the conventional manner.

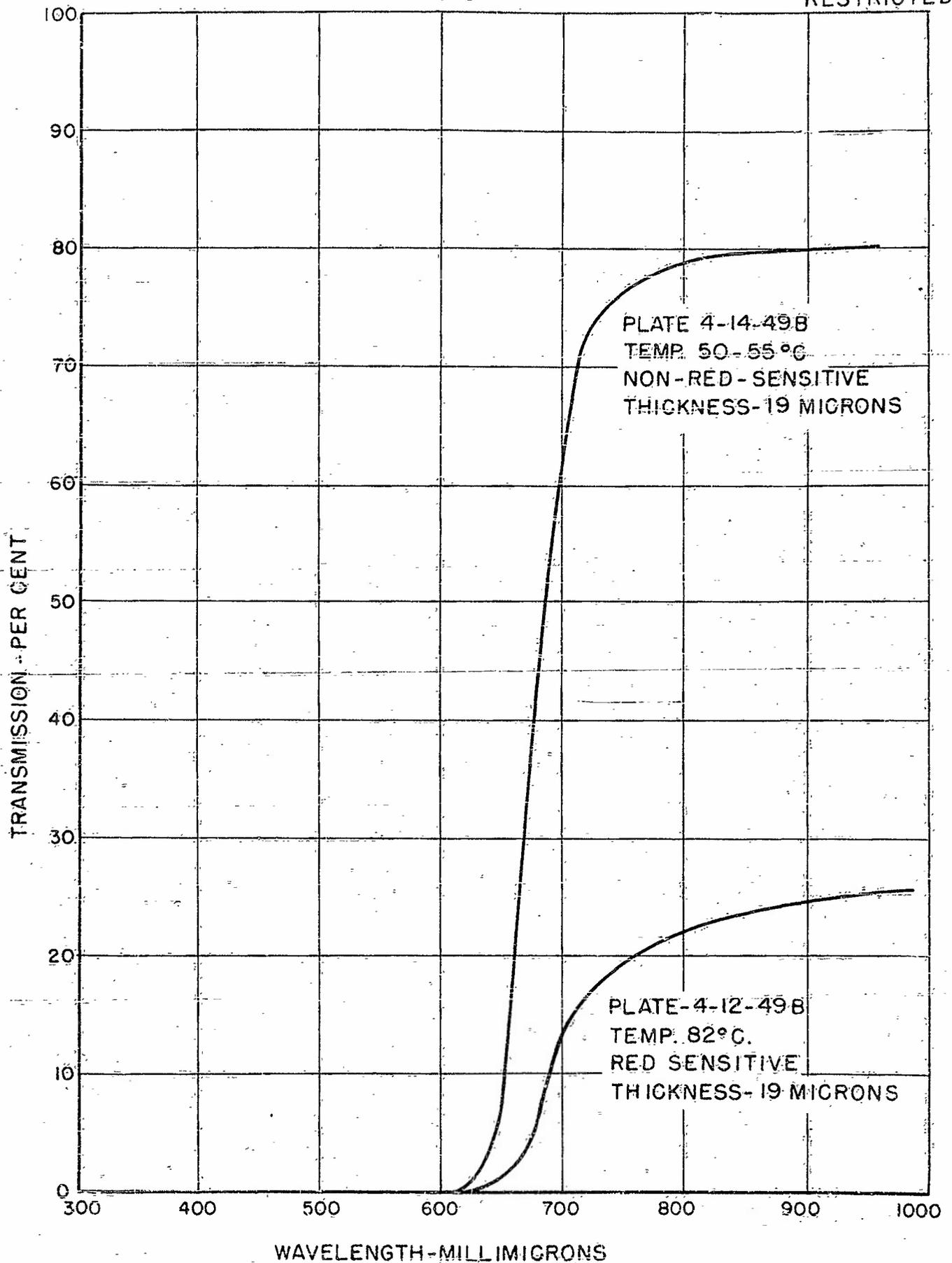


FIGURE 105. TRANSMISSION OF RED-SENSITIVE AND NON-RED-SENSITIVE SELENIUM FILMS-REPRESENTATIVE CURVES

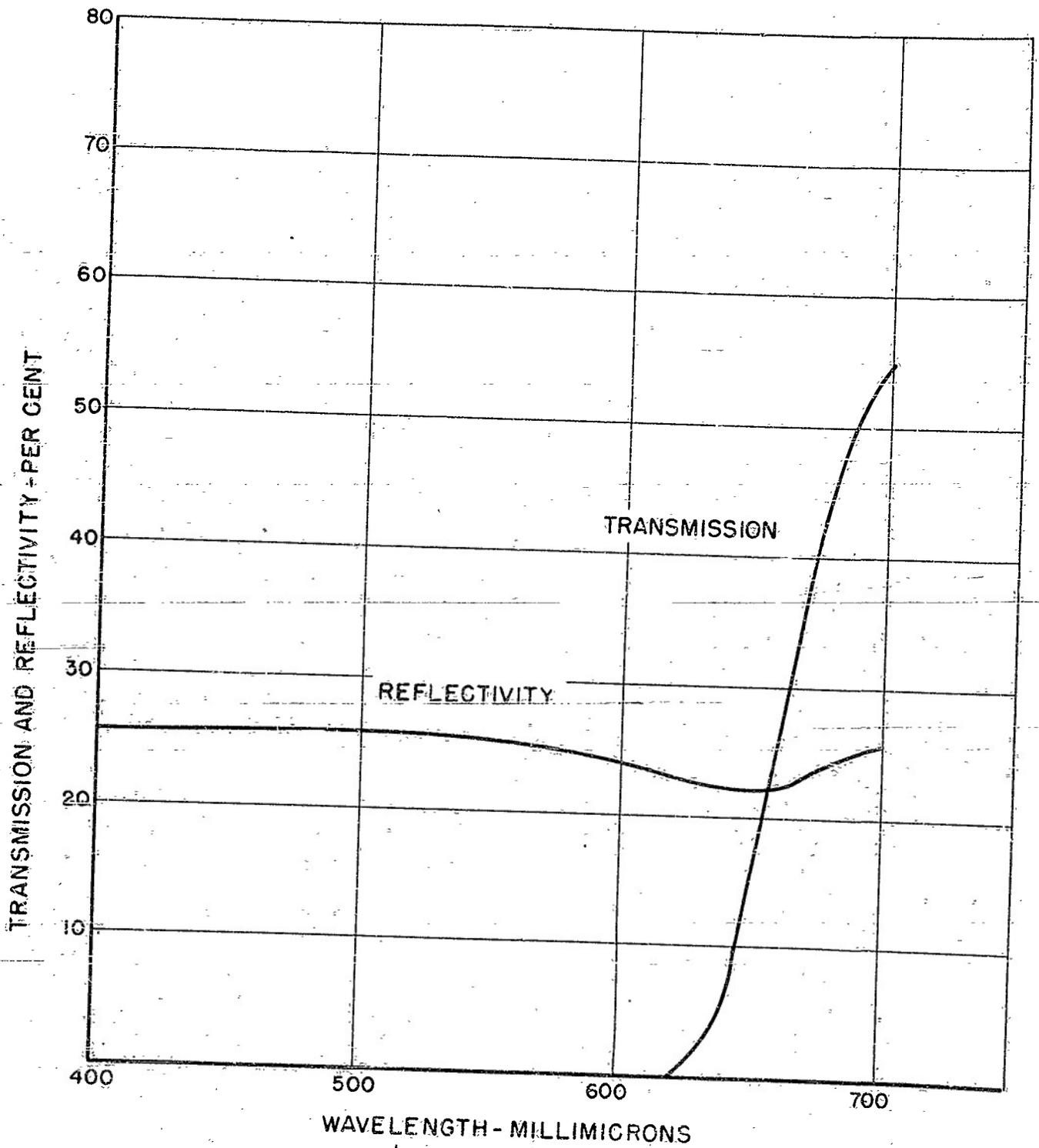


FIGURE 106. TRANSMISSION AND REFLECTIVITY OF SELENIUM FILM ON GLASS

0-12063

In order to establish electrical contact with the selenium surface, a thin aluminum film of from two to three per cent light transmission was evaporated onto the selenium. The coated plate was transferred to the vibrating-probe electrometer, and a potential of 500 volts was applied across the selenium layer. While the plate was still in the dark, the potential leads were removed and a reading of approximately 200 volts was observed on the electrometer. The film also had a normal dark-decay rate. Upon exposure of the plate to white light, the potential was observed to drop approximately 50 volts. Qualitatively, the same effect was observed when the plate was charged by corona discharge.

The same plate was used to determine the effect of visible radiation upon the electrical conductivity of the selenium layer. In this connection, a No. 2 Reflector photoflood bulb was placed directly above the aluminum-coated area of the plate at a distance of about ten inches, and a battery potential of 45 volts was applied across the selenium layer. Using a wall galvanometer having a sensitivity of about 0.001 microampere per millimeter to measure current flow, no current was observed to flow through the selenium layer when it was not illuminated. Upon illumination, a continuous current of approximately 0.02 microamperes was observed to flow through the selenium layer. The same order of current flowed when the polarity was reversed. Somewhat less current was observed with repeated tests, probably due to a fatigue effect.

Experiments on the Illumination of Selenium Films

The hypothesis basic to a theoretical model to be presented later in this report is that photoconductivity can be induced only near an interstitial electrical charge in the selenium layer. If this is true, and if the interstitial charges (a) exist only near the top of the selenium and (b) exert short-range forces, it follows that the decay rate of a selenium layer illuminated through a transparent backing plate should be quite different from that of one illuminated from the charged side.

To test this prediction, selenium layers of 12-micron and 60-micron thickness were deposited on an electrically conductive glass plate. These layers were charged and illuminated in turn from above and beneath with lights designed to be of equal intensity. The charge decay as a function of time is shown in Figure 107 as observed on the 60-micron film. It is seen that the decay rates are not significantly different. On the other hand, the residual voltage is apparently different by a factor of ten or so.

It is concluded that either interstitial charges have a force range greater than the film thickness, or else that they are themselves mobile in the film so as to be uniformly distributed in the selenium. No attempt has so far been made to explain the difference in residual potential.

The selenium layer used in this case would not hold a negative charge.

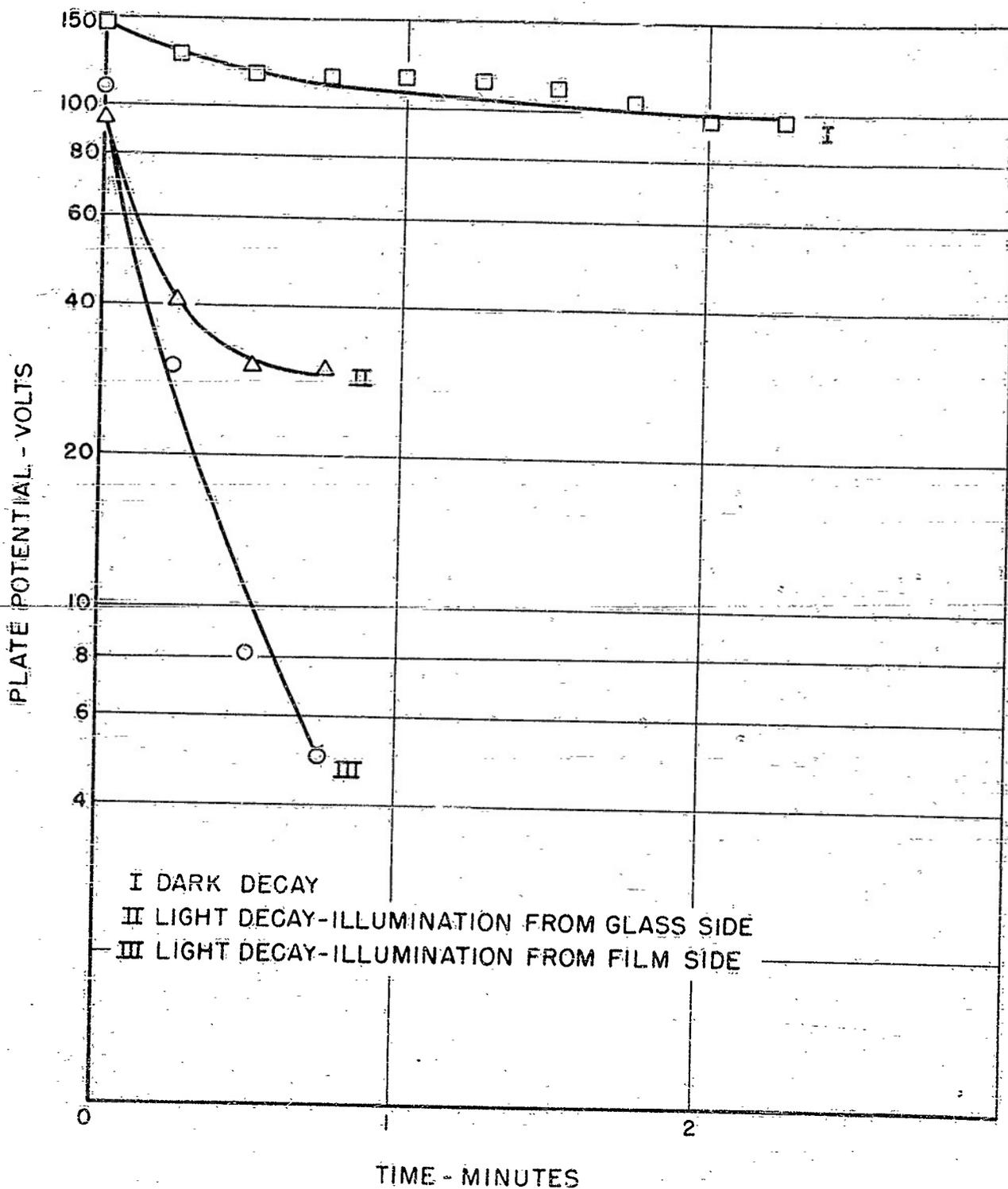


FIGURE 107. POSITIVE- POTENTIAL DECAY CURVES FOR SELENIUM FILM ON ELECTRICALLY CONDUCTIVE GLASS

0-12064

Chemical Treatment of Selenium
Electrophotographic Plates

An investigation was started of the possibility of increasing photographic plate speed by chemical treatment of selenium plates prepared by vacuum evaporation. Only preliminary experiments, in which selenium plates were exposed to halogen vapors, have been conducted to date.

In one experiment, a selenium plate was placed in a beaker containing saturated bromine vapor. After a few seconds, the selenium coating on the plate liquified and drained off of the plate (apparently as selenium monobromide, Se_2Br_2). Shorter exposure to the bromine vapor made the selenium coating sticky. When a very little bromine vapor was poured over the plate, interference fringes were observed to spread out over the surface of the selenium layer. The selenium remained hard but would not retain an electrical potential when attempts were made to charge it. Exposure to iodine vapor also affected the selenium-coated plates so that they would no longer retain an electrical charge.

A selenium-coated plate exposed to dilute chlorine vapors (obtained by moistening commercial calcium hypochlorite) exhibited the following changes in electrical characteristics (see Figure 108): The apparent maximum potential the plate would hold was reduced to approximately one-half of its original value. The dark and light potential decays of the plate were made more rapid.

Exposure of the plate which had been treated with chlorine vapors to high-intensity red and infrared light changed the electrical characteristics of the plate again so that the dark and light decays were

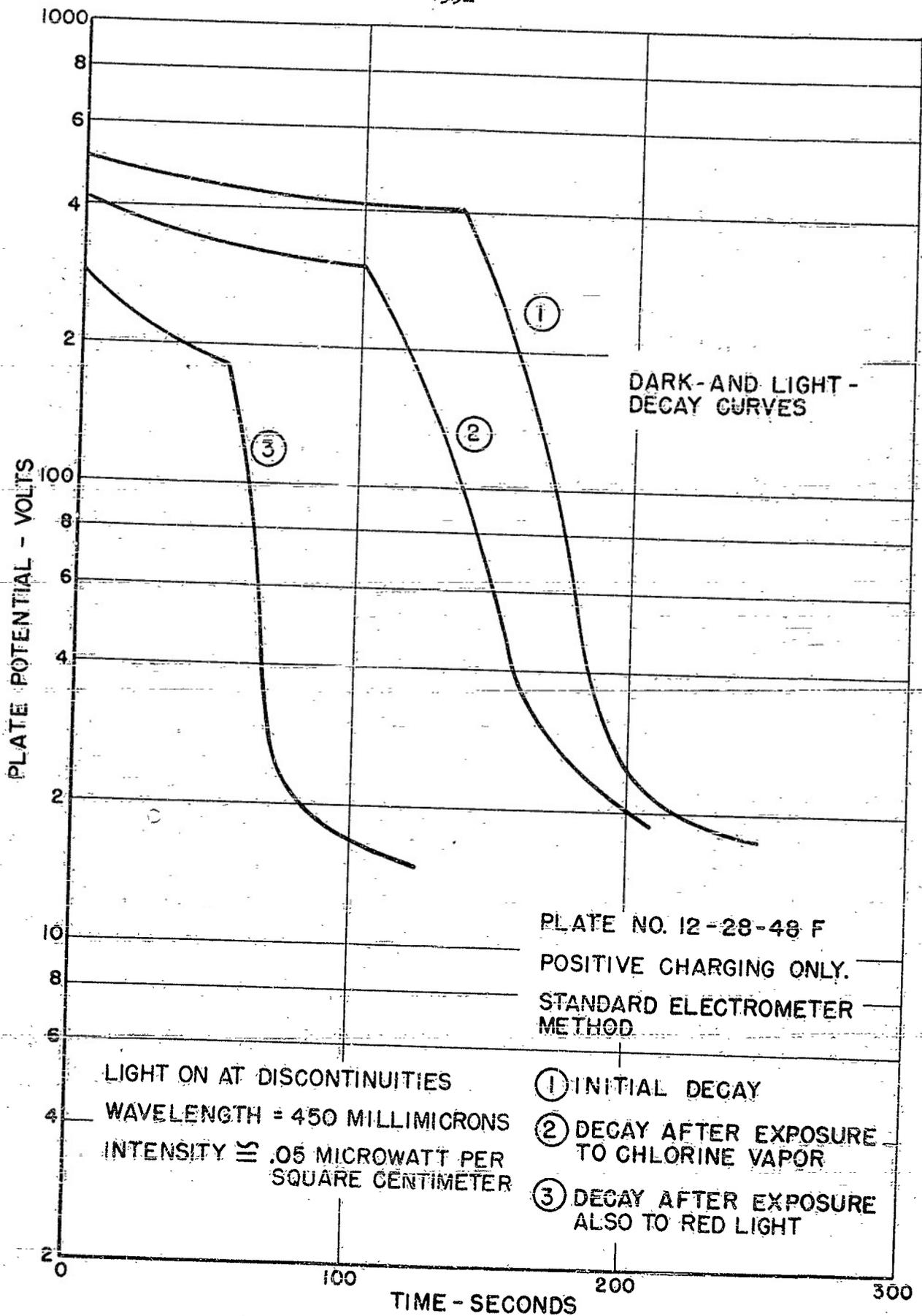


FIGURE 108. POTENTIAL-DECAY CURVES FOR A CHLORINATED SELENIUM PLATE.

more nearly equivalent to their original value before chlorine treatment. However, the maximum potential which the plate would retain remained much lower than that for the plate before chlorine treatment.

Effect of Infrared Radiation on Selenium
Electrographic Plates

It has been observed in the past that red light has some effect on the electrical characteristics of selenium-coated electrographic plates. Therefore, a series of observations was started to determine whether or not red or infrared irradiation improved plate characteristics, and whether or not infrared illumination can be used to stabilize the electrical characteristics of the plate.

The first observations on the effect of simultaneous illumination by red and infrared light were made in connection with electrical tests on a selenium plate that had been exposed to dilute chlorine vapor. The potential-decay curves for the plate (a portion of plate 12-28-48F) are shown in Figure 108. Curves 2 and 3 are reproducible dark- and light-decay curves for positive sensitization when the plate had previously been exposed to room light and red-infrared light, respectively. The room light was provided by two conventional 200-watt tungsten-filament light bulbs placed about fifteen feet from the plate. The red-infrared light was provided by strong sunlight filtered through selenium-coated plate glass. The exposure to both lights was for five-minute periods. These decay curves show a much slower dark-decay rate for the plate after it was exposed to the red-infrared light, but show a quite similar (though somewhat slower) light-decay rate after the red-infrared exposure.

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A number of other plates were examined for changes in electrical characteristics before and after exposure to red-infrared light. Included in the plates examined were several plates having a selenium layer which contained small quantities of bismuth. All of the plates tested showed some change in electrical characteristics with exposure to red-infrared light. In some cases, the irradiation was detrimental, and for several plates the change in characteristics of the plate due to red-infrared treatment was negligible compared to changes caused by consecutive charging, etc.

Figure 109 and 110 show potential-decay curves for two conventionally prepared selenium electrophotographic plates. The plates were kept in darkness except for the light exposure necessary to make the light-decay curves, and the red-infrared exposure. The decay curves shown in Figure 109 were taken after repeated alternate positive and negative charging of the plate. Curve 1 shows reproducible positive potential-decay characteristics which were slightly superior to the initial positive decay characteristics (slower dark-decay rate, same light-decay rate). Curve 2 shows the effect of red-infrared exposure. The maximum potential was almost doubled. The dark-decay rate was slower. The light-decay rate was also slower, but potentially more effective.

Figure 110 shows the effect of red-infrared illumination on the positive-potential dark-decay characteristics of another conventional plate. In this case, the plate was only charged positively and no light (other than the red-infrared irradiation) was allowed to reach the plate. Curve 1 is the initial dark decay. Curve 2 is the dark decay after

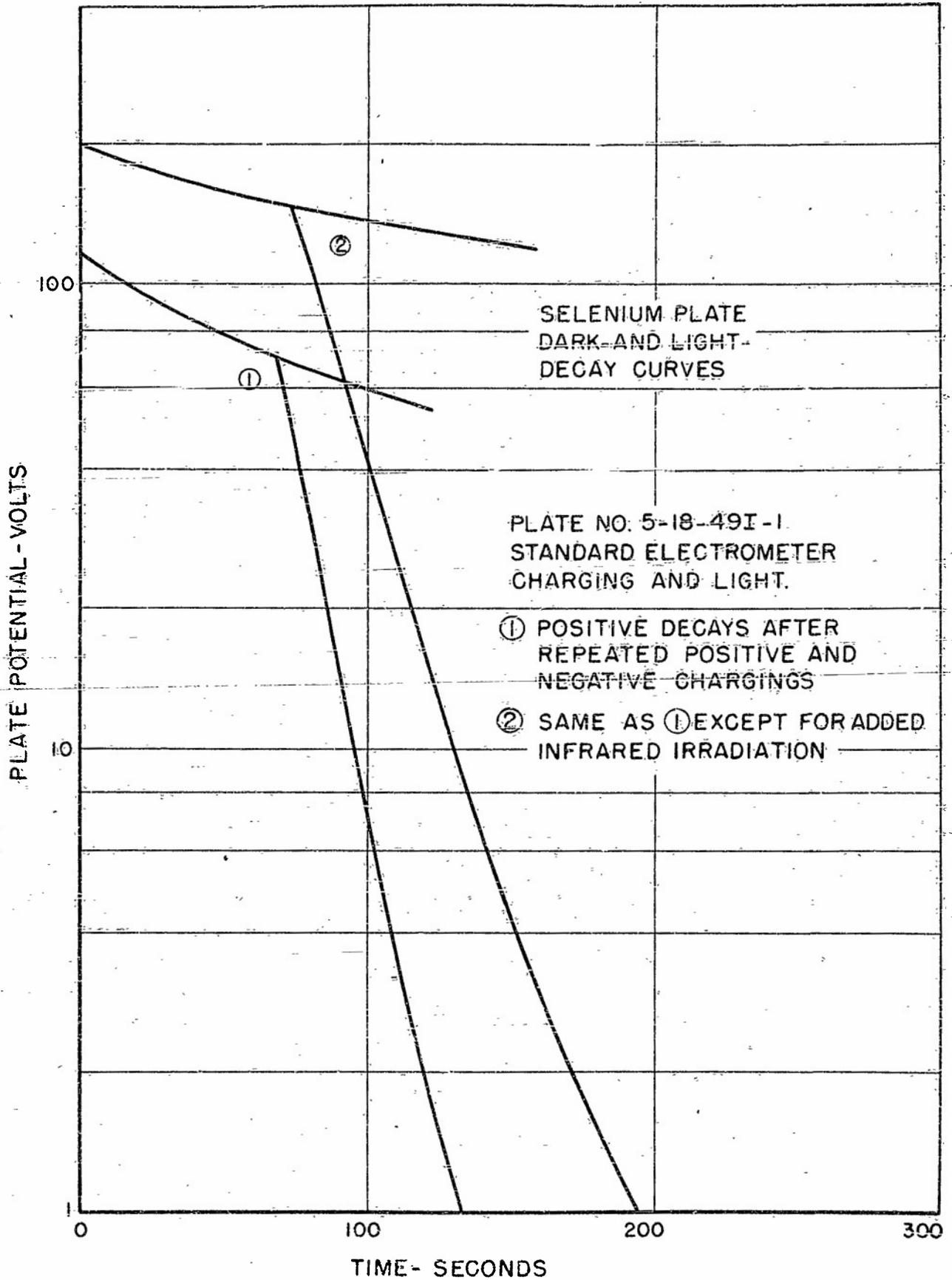


FIGURE 109. EFFECT OF INFRARED IRRADIATION ON A SELENIUM ELECTROPHOTOGRAPHIC PLATE

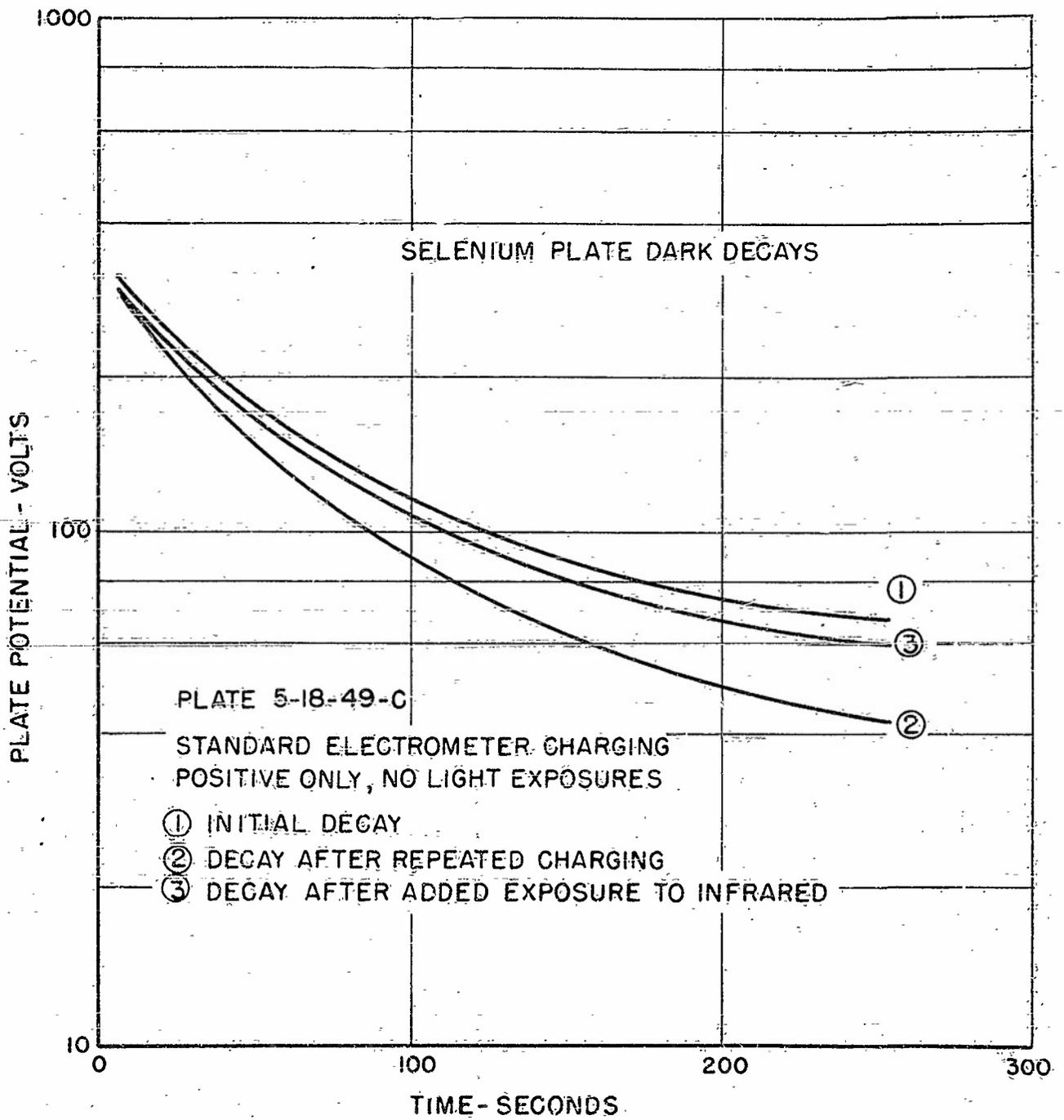


FIGURE 110. EFFECT OF INFRARED IRRADIATION ON A SELENIUM ELECTROPHOTOGRAPHIC PLATE

G-12067

repeated positive charging. Curve 3 is the dark decay after exposure to red-infrared light. In this case, the red-infrared exposure appeared to have only a small effect on the plate, seemingly of restorative nature.

These brief observations show that illumination of selenium electrophotographic plates by red-infrared light may be beneficial to some plates and detrimental to others.

Heat Treatment of Selenium Electrophotographic Plates.

Selenium plates which are maintained at temperatures below 40 to 50°C. during vacuum evaporation generally show a bluish, light-scattering haze on their surfaces. This light scattering is not completely random, as is the scattering from paper or a smoked-glass surface, but rather it has a sheen or halo which appears at certain angles of viewing. This type of effect arises when the particles of the scattering surface are oriented. The orientation may be in the form of small crystallites or of a dendritic type of structure which often forms in thick evaporated films.

Plates with this surface haze generally have inferior characteristics. Therefore, it was desired to determine whether these inferior characteristics were due to the haze itself, or to some other property of the selenium which generally appears with the haze. It was also desired to know whether or not the removal of this haze by heat treatment would improve the plate characteristics.

It was found that this haze could be removed by heating the entire plate to 65 or 75°C. for one or two minutes. With this treatment, the

plate took on the common, black, specular appearance. Measurements of electrical characteristics before and after heat treatment of the selenium film showed that this scattering in itself is not responsible for the inferior characteristics. In these studies, it was also observed that an additional selenium layer evaporated onto a scattering selenium surface will have a clean, specular surface if this second evaporation is carried out at temperatures of 60°C. or above.

Heating the plate above 85°C. for one or two minutes starts crystallization of the film, the same effect which is produced by maintaining the plate at these elevated temperatures during vacuum evaporation.

Impurity Materials Added to Selenium Layer on
Electrophotographic Plates

Preliminary experiments on the effect of adding metallic impurities (Quarterly Progress Report No. 3, pages 235 to 237) indicated that impurity materials modify the electrical properties of selenium-coated electrophotographic plates. Those experiments gave little information about the magnitude of the effect produced, and almost no information about the amount and position in the selenium film of the impurities involved.

It was undertaken, therefore, to prepare plates under closely controlled conditions so that the distribution of the impurity material in the selenium layer would be known. It was planned to prepare selenium films of uniform thickness, containing a gradually increasing amount of impurity in concentrations under one per cent from one end to

the other of the plate. The effect of the impurity would then be determined by observing the variation of the electrical characteristics of the plate from one end to the other.

To be certain that any variations in plate characteristics noted would be caused only by the presence of impurities, it was necessary to control certain other factors very carefully. The most important of these factors was the uniform distribution of the impurity through the thickness of the selenium film. Other variables to be controlled in the preparation of selenium plates were the temperature of the backing plate during plate preparation, and the total time of deposition.

Two methods were investigated for coveaporating selenium and an impurity to produce selenium layers containing a uniform distribution of impurity throughout the thickness of the selenium layer. In one method, selenium and the impurity were evaporated from separate sources. The sources each contained many times as much material as is required to produce the final film. The plate to be coated with the selenium and impurity was exposed to both sources only after they had reached equilibrium temperatures and were evaporating at substantially constant, but not necessarily equal, rates. The variation in the amount of impurity over the length of the plate was produced by placing the impurity source nearer to one end of the plate than to the other end.

In the second method of evaporating the impurity into the selenium layer, the selenium and the impurity were evaporated from a single source. The mixture of selenium and impurity were fed into the

evaporation source at a constant rate and the temperature of the source adjusted so that the materials would be evaporated as rapidly as they were fed to the source. To produce both a uniformly thick selenium film and the desired gradation of impurity from one end to the other of the plate, two evaporation sources were used. Pure selenium was evaporated from a source placed at one end of the plate and the selenium-impurity mixture was evaporated from a source placed at the other end of the plate.

The method involving the evaporation of a selenium-impurity mixture appeared to offer the advantage of allowing the preparation of a film containing a known percentage of impurity. This percentage would be fixed merely by adjusting the mixture of selenium and impurity fed to the source. For this reason, this method was selected for the first experiments.

A screw-type feed hopper was used to deliver the powdered selenium to the evaporation source. The feeder was constructed of a 1/8-inch screw (formed by twisting a 1/8-inch by 1/64-inch nichrome ribbon to approximately five turns per inch) turning in a stainless steel tube of 1/8-inch inside diameter. The tube formed the bottom of a conical hopper into which was fed the granular material to be evaporated. In actual use, it was found necessary to vibrate the hopper and screw to produce proper feeding.

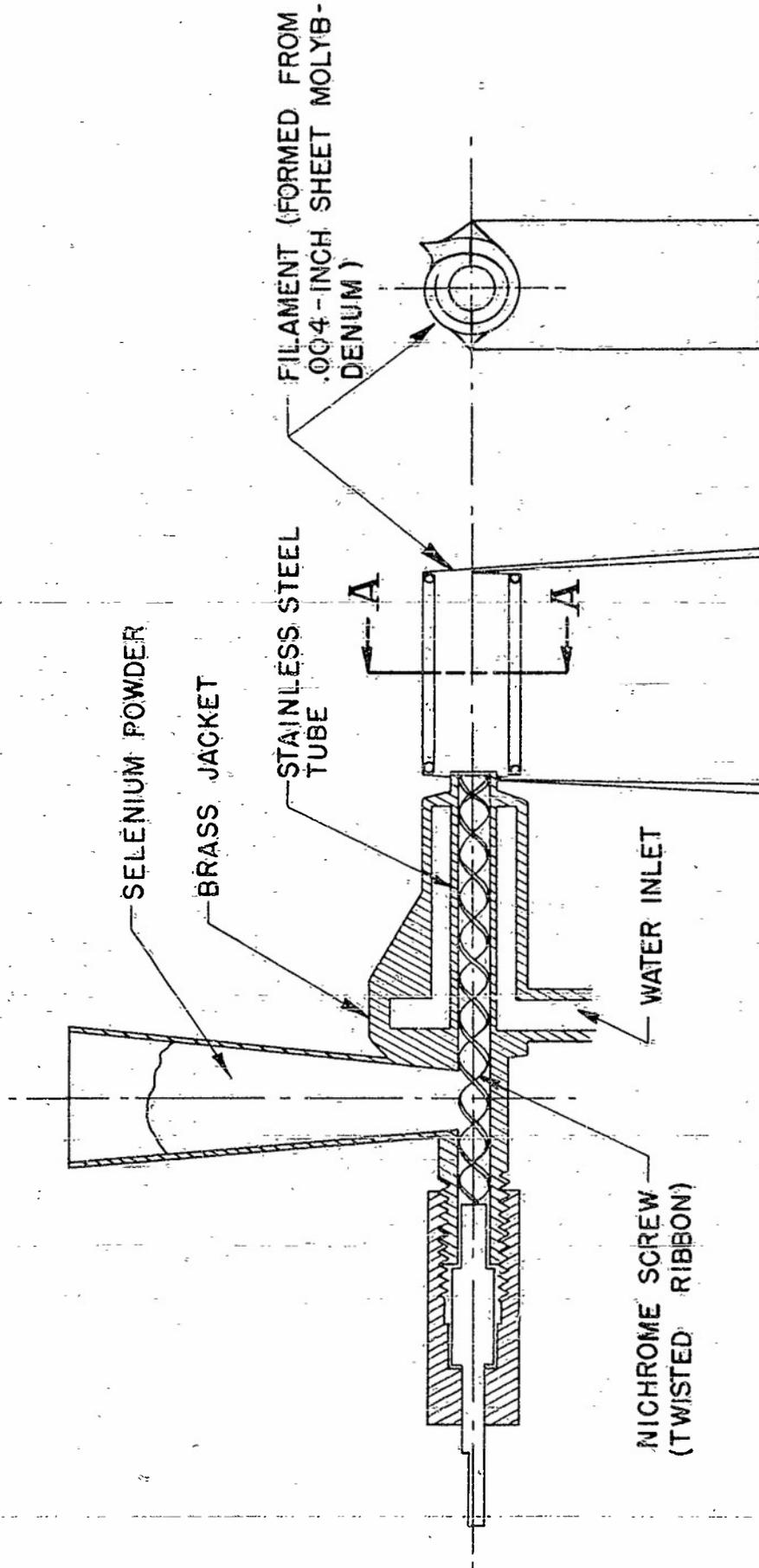
The first evaporation source tried with the feeder was a simple boat-type filament approximately 1/8 inch deep by 3/16 inch wide by 3/4 inch long formed from 0.004-inch molybdenum foil. When the source was heated to a dull red, it was

found that the selenium powder fed to it was bounced or blown out of the boat by the pressure of the vapor of the selenium being evaporated.

A number of other evaporation source designs were tried. The most successful design is shown in Figure 111. In this case, the evaporation source consisted of an essentially closed chamber into which the selenium was fed and attached to which was a circuitous chimney. Both the chamber and the chimney were formed from a strip of 0.004-inch molybdenum which could be heated by electrical currents.

The selenium powder, which was fed into this source, evaporated to give a smooth deposit on an aluminum plate held above it. However, the source heated the feeder to such an extent that the selenium melted in the feed tube and stopped the feeding action. Even a water-jacketed feeder (Figure 111) jammed, apparently because of molten selenium which had solidified in the cooled tube. Because of these difficulties, attention was turned to the separate-source method of impurity evaporation.

Figure 112 shows the apparatus used for evaporating selenium and impurity materials from separate sources. Two selenium sources, each consisting of a boat, approximately 3-1/2 inches by 1 inch by 5/8 inch, formed from 0.003-inch molybdenum, were placed twelve inches apart and six inches beneath the plate position. An impurity source, consisting of a v-shaped trough formed from 5-inch by 1/4-inch by 0.003-inch molybdenum, was placed about 4-1/4 inches below one end of the plate and so positioned that it did not shadow the selenium source. The backing

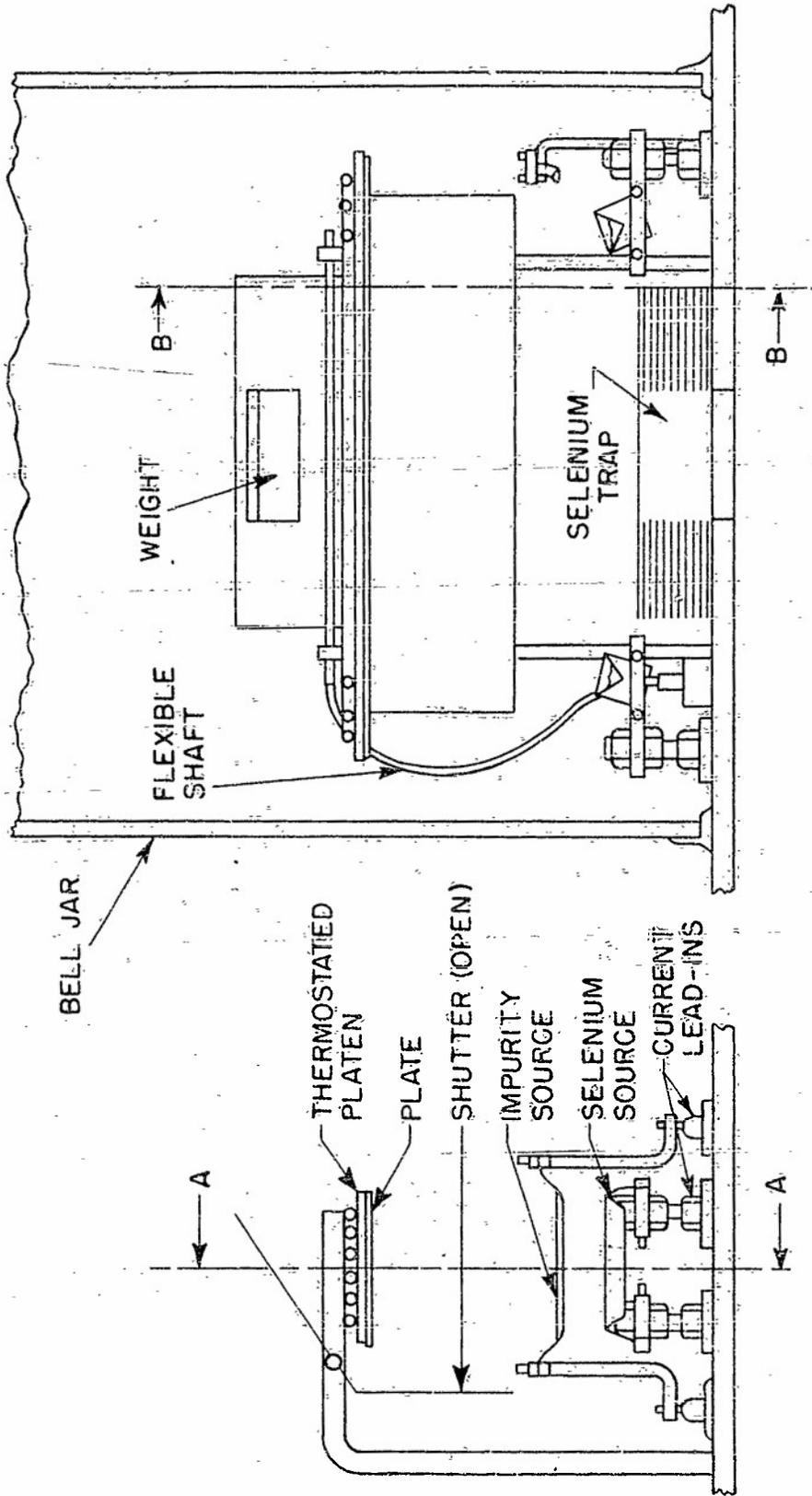


SECTION A-A
(FILAMENT ONLY SHOWN)

FEEDER TUBE, JACKET, AND FILAMENT IN SECTION

SCALE: DOUBLE SIZE

FIGURE III. SCREW - TYPE SELENIUM POWDER FEEDER AND FILAMENT



SECTION A-A
(SHOWING BELL JAR)

SECTION B-B

1/4 SCALE

FIGURE 112. SELENIUM-IMPURITY CO-EVAPORATION; ARRANGEMENT INSIDE BELL JAR

O-12069

plates used were 16-gauge aluminum, 1 1/4 inches by 3-1/4 inches in size. The plates were fastened to the surface of a slightly convex temperature-controlled platen the same size as the plates. A movable shutter was attached to the frame holding the platen so that it could be moved to cover or uncover the front surface of the plate.

The selenium and impurity evaporations were carried out in the following general manner: Each selenium filament was loaded with about five grams of G. P. ARQ shotted selenium. The impurity filament was loaded with the impurity desired (less than 0.1 grams). The plate, which had been washed with water and then degreased in isopropyl alcohol vapor, was clipped to the platen and covered with the shutter. The temperature of the platen was adjusted to 60°C. The system was then evacuated to a pressure of less than 1/2 micron of mercury. The current to the selenium sources was turned on and adjusted to 60 amperes. After two minutes, the current to the impurity source was turned on and adjusted to a predetermined value. After an additional one-minute interval, the shutter was opened and the plate was exposed to the selenium and impurity sources for one minute. The shutter was then closed and the charges remaining in the sources were completely evaporated by increasing the filament currents for a short time. Five minutes after the shutter had been first opened, the plate was removed from the bell jar.

Several plates were prepared in which bismuth was used as the impurity material. The highest concentration of bismuth anywhere on the

selenium layer was estimated to be less than 0.1 per cent. The selenium film thickness on the plate was from eight to ten microns. A summary of the potential-decay characteristics for different amounts of bismuth in the selenium layer is given in Figure 113. The data appear to indicate that the addition of a small amount of bismuth increases the maximum potential the film will accept. The addition of more bismuth appeared to raise the residual potential of the plate.

Dependence of Selenium Plate Characteristics on Backing-Plate Temperature During Preparation

A further study was made of the variations in the physical characteristics of selenium plates as a function of the temperature of the plate during vacuum evaporation of the selenium. Some progress has been made in correlating the quality of the print a selenium plate will make, for positive sensitization, with the electrical characteristics of the plate and the conditions under which the plate was made. In these tests, as in the temperature series reported in Quarterly Progress Report No. 3, pages 218 to 229, all conditions during plate preparation were kept constant except the temperature of the backing plate. This temperature was varied from 40°C. to 90°C. The previous temperature series showed the correlation between electrical characteristics of a plate and its temperature during evaporation. The work reported here extends this information to include the effect of temperature during evaporation on the quality of line-copy prints.

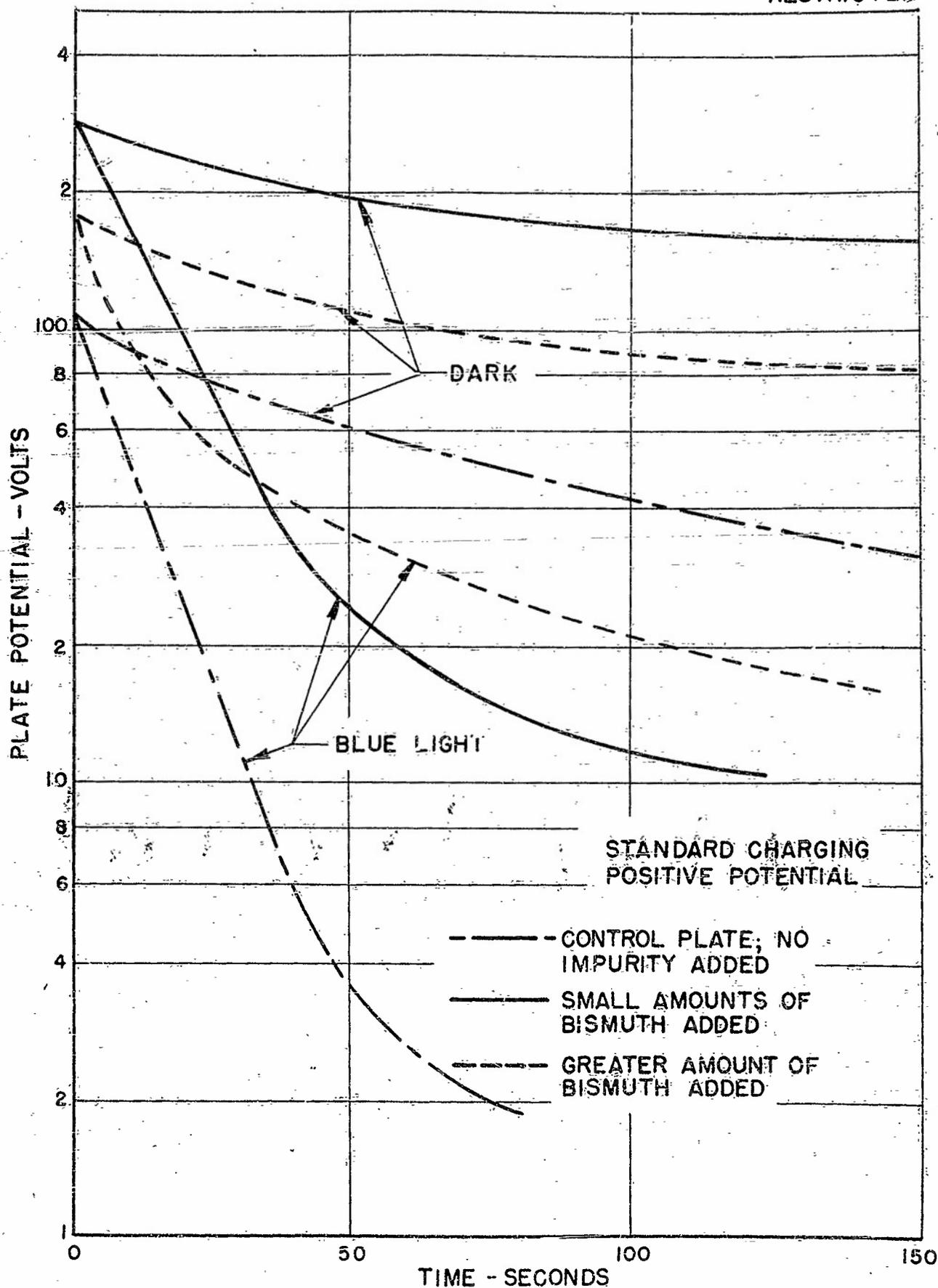


FIGURE 113. BISMUTH-SELENIUM ELECTROPHOTOGRAPHIC PLATES, POSITIVE POTENTIAL DECAY CURVES

The plates were prepared on 16-gauge, Number 1 reflector-finish, 11-inch by 14-inch aluminum. The aluminum plates were vapor degreased before coating in the National Research Corporation evaporator under the following conditions:

1. Pressure, between 0.1 and 0.3 micron.
2. Evaporation source, six molybdenum boats, each $3/4$ inch wide and two inches long. One boat placed under each corner of the plate, and one boat centered under each long edge of the plate.
3. Distance from source to plate, approximately eight inches.
4. Charge of selenium in each boat, five grams of ARQ brand, C.P. shot selenium.
5. Plates clamped securely to a platen through which water was circulated from a constant-temperature bath.
6. Total evaporation time, eleven minutes.

(These conditions are essentially those which prevailed during the temperature-control experiments described in Quarterly Progress Report No. 3, pages 218 to 229.)

The thickness of the selenium film was kept between 17 and 19 microns. From five to ten plates were made at each of the following temperature ranges: 40 to 48, 50 to 56, 61 to 65, 74 to 78, 80 to 85, and 85 to 91°C.

A tabulation of test data for representative plates is given in Table 30. Included also are several plates made by changing the platen temperature from 60 to 40°C. during their preparation.

TABLE 30. DEPENDENCE OF ELECTRICAL CHARACTERISTICS AND PRINT QUALITY ON TEMPERATURE OF SELENIUM ELECTROPHOTOGRAPHIC PLATES DURING THEIR PREPARATION

Temperature, Degree C.	Plate Number	Maximum Accepted Positive Potential, Volts	Residual Positive Potential, Volts*	Positive Dark Decay, Per Cent (See Text)	Red Sensitivity	Background	Contrast	Coverage of Large Dark Areas	Maximum Accepted Negative Potential, Volts	Residual Negative Potential, Volts	Negative Dark Decay, Per Cent (See Text)	Adhesion
40	4-19 A	1000	500	29	No	High	Low	Good	500	350	-	Very Poor
40-45	4-18 B	750	Dark Decay	27	"	Moderate	None	"	350	60	26	Good
40-47	4-16 C	500	Ditto	36	"	-	-	-	300	40	32	-
41-48	4-18 A	500	"	40	"	None	None	-	450	100	27	Poor
49-56	4-15 D	600	150	-	"	"	Low	Fair	600	50	0	"
49-56	4-14 E	1000	Dark Decay	25	"	-	-	-	300	45	45	-
50-55	4-14 D	900	300	26	"	Moderate	Low	Good	400	80	33	Poor
50-57	4-15 B	600	100	33	"	-	None	-	600	110	40	Spotty, poor, and fair
50-57	4-15 C	750	200	33	"	-	Low	-	450	50	20	Poor
58-53	4-19 B	1000	400	38	"	Moderate	"	Good	450	100	-	"
60-43	4-19 C	900	380	21	"	"	"	"	500	170	26	"
60-42	4-19 D	800	Dark Decay	34	"	-	"	"	450	120	25	"
61	3-21 A	450	0	36	"	None	Medium	Fair	400	20	15	Good
62	3-24 A	550	20	27	"	Slight	"	Good	-	-	-	Very Good
62	3-28 C	800	12	24	"	Moderate	"	Fair	500	80	14	Ditto
62	3-28 D	700	10	21	"	"	High	Good	500	60	16	Very Good
63	3-29 A	900	0	17	"	Slight	Medium	Fair	600	50	-	Very Good
64	3-28 A	900	40	20	"	Moderate	High	Poor	600	110	16	Ditto
64	3-25 B	900	10	20	"	"	"	Good	400	60	13	"

TABLE 30. CONTINUED

Temperature, Degrees C.	Plate Number	Maximum Accepted Positive Potential, Volts	Residual Positive Potential, Volts*	Positive Dark Decay, Per Cent (See Text)	Red Sensitivity	Background	Contrast	Coverage of Large Dark Areas	Maximum Accepted Negative Potential, Volts	Residual Negative Potential, Volts	Negative Dark Decay, Per Cent (See Text)	Adhesion
64	3-25 A	800	8	25	No	Slight	High	Good	350	40	17	Good
65	3-23 A	700	0	25	"	"	"	"	-	-	-	"
74	3-18 A	800	2	27	"	Moderate	Medium	"	350	20	33	Very Good
74	3-15 A	450	0	38	"	-	-	-	220	20	-	-
76	4-1 B	700	2	32	"	-	None	-	200	15	51	Very Good
76	3-31 B	900	2	28	-	None	Medium	Very Poor	20	1.5	71	Very Poor
80	4-11 B	700	14	32	Yes	-	-	-	20	-	-	-
80	4-12 A	900	16	29	-	-	-	-	20	-	-	-
82	4-12 B	700	10	32	Yes	Slight	High	Uniform but light	25	-	85	Poor
82	3-22 B	500	3	41	No	Moderate	Low	Poor	30	-	50	Good
84	4-6 B	810	50	28	Yes	Slight	High	"	40	2	-	"
91	4-4 A	800	9	34	"	"	Medium	Very Poor	25	2	32	Very Good
91	4-4 B	700	3	28	"	"	"	Ditto	20	2	-	Ditto

* Entry "Dark Decay" means no distinguishable difference between light or darkness on residual voltage.

Note: Potential measurements from vibrating-probe electrometer; residual voltages are for blue light (459 millimicrons);
Plate Thickness - 17 to 19 microns

For both positive and negative plate sensitization, data on the electrical properties of the plates include the approximate maximum potential accepted, the residual voltage after exposure to blue light (wavelength, 459 millimicrons), and per cent dark decay. The per cent dark decay was calculated as the difference between the plate potential measured 20 seconds and 200 seconds after charging, divided by the potential at 20 seconds. With regard to image-forming quality, information on background, contrast, large-area black coverage, and red sensitivity is given. Prints were made by the powder-carrier cascade method of developing. The large-area black coverage is a factor of great importance in making continuous-tone pictures by cascade-development methods.

The following general observations can be made concerning the electrical characteristics of the plates:

1. Plate potential. Temperature of the backing plate during evaporation had no marked effect on the maximum positive potential accepted by the selenium film. This is not in agreement with the results reported previously in which a gradual increase in accepted positive potential was noted with an increase in temperature of the plate during vacuum evaporation.

2. Residual potential. The same variations in residual potential reported previously appeared here. That is, plates prepared at temperatures below 50°C. had higher residual potentials than plates prepared at higher temperatures. The light-decay curves in some cases were identical to the dark-decay curves. In the 50 to 60°C. range, the

residual potential was lower, and in the 60 to 80°C. temperature range the plates showed a minimum in the value of the residual potential. Above 80°C. the residual potential tended to be somewhat higher than in the 60 to 80°C. range, though this difference was small. It is in this temperature range above 80°C. that sensitivity to red light appears.

3. Per cent dark decay. Though the dependence of the dark-decay rate of a plate on the temperature of the plate during its preparation is not so obvious as could be hoped, a definite trend does appear. The per cent dark decay can be seen, in all cases summarized in Table 30, to be above 25 per cent, and generally above 30 per cent, for temperatures below 60°C. In the 60 to 70°C. range, this value is consistently less than 25 per cent, and goes as low as 17 per cent in one case. Above 70°C. the dark-decay rate rises quite markedly.

For negative potential sensitization, plates made near 60°C. accept as much or more charge than plates made in other temperature ranges. Also, they generally have better light sensitivity and slower dark decays than do plates made in any other temperature range. A value of 16 per cent seems to be an average negative dark decay for a plate made in the 60 to 70°C. temperature range.

Turning from electrical characteristics to print quality, the results are considerably more subjective. Positive sensitization was used in all tests of image quality. Evaluation of prints from plates made below 60°C. was fairly simple, since very often it was impossible to obtain any images at all. These plates were also generally characterized by a complete lack of powder in nonimage areas, i.e., no background.

Plates made in the 60 to 75°C. range produced the most consistently good prints. Though it was necessary to balance the best contrast with acceptable background, all of the plates in this range made acceptable prints. In the temperature range above 75°C., some plates made acceptable prints and some plates did not. The selenium on the plates which did not make acceptable prints generally had a rough, grayish appearance, which characterizes the start of crystallization.

Another factor involved in the making of prints, particularly continuous-tone prints, by the powder-carrier method, is the ability of the plate to reproduce large gray or black areas faithfully. Often such areas are printed too dark around the edges with the central portion of the image, holding little or no powder. The temperature at which the plate was held during vacuum evaporation was found to affect this quality, which in Table 30 is called "coverage of large dark areas". Very uniform coverage was present for most of the cool plates (below 60°C.) that would form images at all. Coverage was fair to good in the 60 to 75°C. range of plates. For plates made above 75°C., however, this large-dark-area coverage was poor. Plates in this range almost always produced a very dark periphery with practically no powder in the central region of large dark areas.

As also reported from the previous temperature-control experiment, sensitivity to red light of 650 millimicrons wave length appeared only in plates prepared above 80°C.

The adhesion of the selenium to the aluminum backing plate was also examined. One test for adhesion is to determine whether or not the

selenium can be pulled away from the backing plate when cellulose adhesive tape is applied and stripped off. If the selenium is not removed from the aluminum even when the tape has been applied over a rough edge of selenium, as produced by nicking or chipping the selenium with a sharp instrument, it is considered to have very good adhesion. Plates prepared below 60°C. generally had poor adhesion. The temperature range between 60 and 80°C. consistently produced plates with extremely good adhesion. The temperature range above 80°C. often produced plates in which the selenium was easily removed. Indications are that this lack of adhesion is caused by a certain amount of crystallization of the selenium, as plates which show more pronounced crystallization have definitely poorer adhesion. The selenium on these plates is also considerably more brittle.

It may be concluded that, in general, the most consistently satisfactory electrophotographic plates can be made by holding the aluminum backing plate at a temperature between 60 and 70°C. during vacuum evaporation. In order to produce red-sensitive plates, the backing plate must be held at temperatures above 70°C. and probably above 80°C.

Alternative Methods of Preparing Selenium
Electrophotographic Plates

Possible alternative methods of preparing selenium electrophotographic plates were studied briefly. Two of the methods tried produced plates which were similar to vacuum-evaporated plates in electrical

electrical characteristics. In these preliminary experiments, neither of the methods produced plates whose surfaces were plain enough for good image formation.

In one of the methods investigated, plates were prepared by smoothing out a small quantity of molten selenium over the surface of a warmed aluminum plate. This was done by heating an aluminum plate, sprinkled with selenium shot, on a hot plate until the selenium melted. The selenium was then spread with a glass rod as the hot plate gradually cooled. The selenium-coated plate was removed from the hot plate as soon as the selenium became viscous enough to remain spread out over the plate. The plates produced by this method were glossy black in appearance and showed the marks of the spreading operation. Plates with areas having thicknesses of less than 100 microns were produced. These areas, less than 100 microns in thickness, accepted an electrical potential of more than 1000 volts, decayed slowly in darkness, and decayed in light similarly to plates produced by vacuum evaporation. It was possible to produce good-contrast line-copy reproductions on one of the plates, but the surface irregularities of the selenium film were visible in the finished print.

In another method of producing selenium electrophotographic plates, selenium vapor was condensed from a gas at atmospheric pressure upon an aluminum plate. The first unsuccessful attempts were carried out similarly to the vacuum-evaporation method. Selenium was boiled in an electrically heated steel boat above which was held an aluminum plate. The experiment was carried out in both nitrogen and helium atmospheres.

In both cases, during the heating of the boat, a red-brown vapor or smoke was seen to rise in the convection currents above the selenium. Some of this material deposited upon the plate as a red-brown powder that could be brushed off the plate.

In a third experiment, natural gas, heated above the melting point of selenium, was passed over heated selenium and into a heated chamber in which was mounted a water-cooled aluminum plate. A dark-brown deposit appeared on the plate. This plate would not take a charge and had an unpleasant odor. However, in part of the hot chamber, beads of a black, shiny material were formed which appeared to be vitreous selenium.

A more successful method of atmospheric deposition of selenium was carried out as follows: A quantity of selenium was placed in a stainless steel tube, $5/8$ inch in diameter. Natural gas was passed through the tube and ignited as it flowed out of the tube. The tube was then heated with gas burners. As the selenium became hot, red-black vapors were seen in the cone of the flame, and the color of the flame changed from yellow to an intense blue. An aluminum plate passed through the cone of the flame received a deposit of shiny, roddish-black, vitreous selenium.

It was possible to coat the surface of a 4-inch by 5-inch aluminum plate by playing the flame over the whole surface of the plate. The thickness of the selenium on such a plate was not very uniform. One plate accepted an electrical potential of about 800 volts in one area tested, decayed slowly in darkness, and decayed in light similarly to plates prepared by vacuum evaporation.

Phosphors as Photoconductors in Electrophotography

In the course of a visit to the U. S. Signal Corps' Squiers Signal Laboratories, Fort Monmouth, New Jersey, a suggestion was made by Dr. Helmut Kallmann, pointed toward a new way of producing an electrostatic image on a plate. This was to bind on a metal plate, by some means, a layer of very finely divided crystals of cadmium sulfide, or other similar material. These crystals were said by Dr. Kallmann to have resistivity of 10^{12} ohm-centimeters, which falls to 10^7 ohm-centimeters after a period of illumination by ultraviolet, and possibly by visible light. This resistivity reverts to the higher value upon illumination by infrared light of a wavelength of one micron. It was predicted that an image might be formed on an uncharged plate as an array of areas of different conductivity. Then a charge might be caused to form on the plate to make an image by imposing an external field for a suitable time. How this charge image might then be fixed and made visible is not quite certain, but possibly the use of infrared radiation of heat might immobilize the charge so that the plate could be developed in the usual fashion. Possibly a corona charge would distribute itself in some fashion due to the array of conductivities. At any rate, it was thought desirable to try to make a plate by the suggested means, and postpone the question of image development.

A sample of cadmium sulfide furnished by the Signal Corps was put into a binder of cellulose nitrate dissolved in amyl acetate, and flowed onto an aluminum plate. After the binder dried, the plate was tested

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for its ability to retain an electrical charge. The cadmium sulfide in the binder film did not retain an electrical charge, nor would it after the binder had been burned off. When the crystals were exposed to ultraviolet light, they failed to fluoresce to any perceptible degree.

The properties suggested to be present in silver-activated cadmium sulfide were investigated in a 2310 phosphor manufactured by the New Jersey Zinc Company. A plate was prepared from this phosphor in the same manner as described above for the cadmium sulfide plate. The plate was charged in the conventional manner and a potential of about 125 volts was found to be present on the surface of the plate. Subsequently the plate was illuminated with infrared light prior to charging. After this treatment, it was found to accept a larger charge and to exhibit a slower dark decay. Heating prior to charging produced a similar effect. The heating was carried out over a Bunsen flame until the phosphorescent glow was quenched. Figure 114 shows the electrical behavior of this material.

A charge was induced on the 2310-phosphor plate by applying a potential between the metal backing plate and an electrode placed on the top surface of the phosphor. The top electrode was a glass plate with an electrically conducting surface which was laid on top of the phosphor layer. The largest potential induced on the plate was 42 volts. This potential was produced when a potential of 2000 volts was applied across the electrode and the base plate. The induced potential, for a 2000-volt potential difference, varied from 8 volts to 42 volts. This variation is believed to be due to poor adhesion of the phosphor to the plate with the

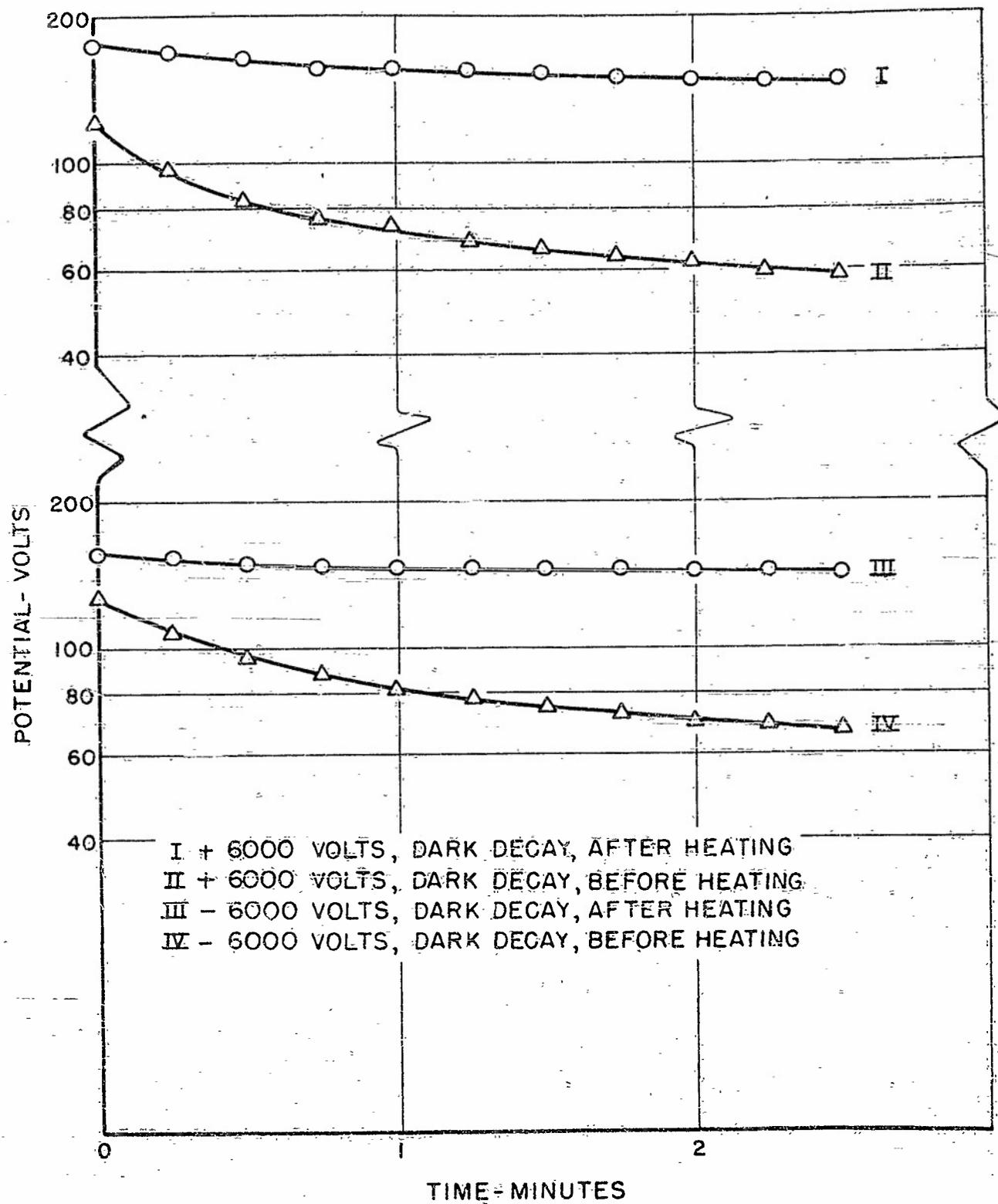


FIGURE 114. EFFECT OF HEATING ON DARK DECAY - PHOSPHOR 2310

O-12071

particular type of binder used. After charging, the top electrode was removed before the potential-decay curve was determined. In the process of removing the top electrode, the phosphor was disturbed and the degree of disturbance is believed to cause the variation in magnitude of the final induced charge.

The magnitude of the induced charge as a function of the time of charging was observed and is shown in Figure 115. These data were obtained without moving the top electrode; consequently, the variation mentioned in the previous paragraph does not appear here.

Two additional binders are being investigated as to their adhesion and abrasion resistance. One of the binders is sodium silicate, and the other a solution of potassium silicate and sodium sulfate.

INSTRUMENTATION

(P. G. Andrus, E. H. Runyan, and O. A. Ulrich)

Recording Mechanism for Vibrating-Probe Electrometer

A recorder was made for the vibrating-probe electrometer which makes it possible to plot the light- and dark-decay curves for electro-photographic plates directly onto graph paper. Previously, the decay data from the electrometer had to be recorded on a strip of paper and then later transcribed onto the graph paper.

The device described here is used in conjunction with the vibrating-probe electrometer described in Quarterly Progress Report No. 2,

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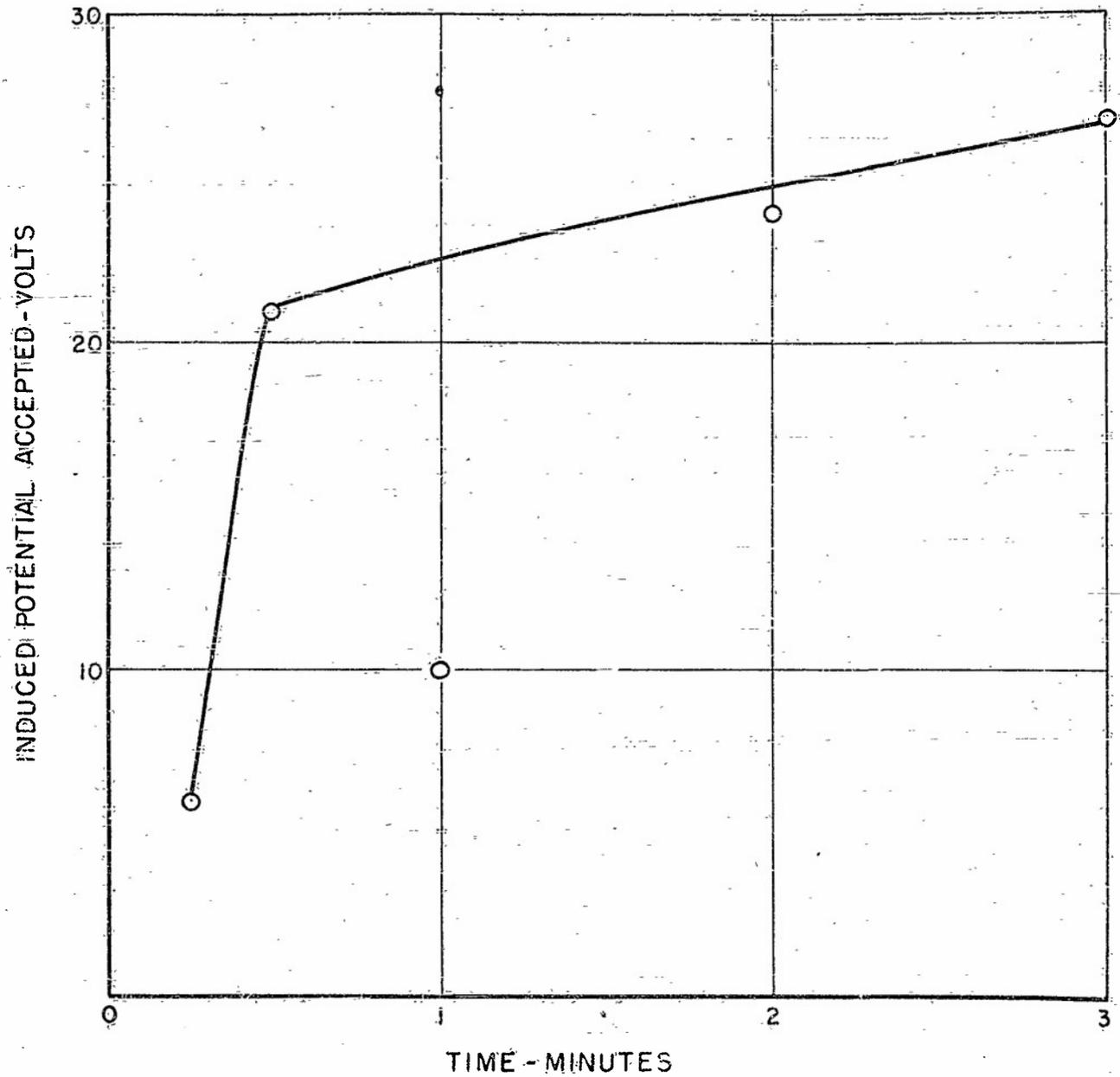


FIGURE 115. DEPENDENCE OF MAGNITUDE OF INDUCED CHARGE ON TIME OF CHARGING - PHOSPHOR 2310

0-12072

pages 193 to 200. It consists of three principal parts: (1) a pen carriage and solenoid-operated pen, (2) a voltage-changing switch, and (3) a motor-driven drum on which the semi-log graph paper is mounted. All of these parts are shown in Figure 116.

The pen carriage is mounted on two rods, one of which is cut with teeth to form a rack gear, and the other of which has a series of logarithmically spaced notches. The rack engages a pinion gear which is mounted on the carriage. The turning of the pinion gear by means of a hand knob serves to index the carriage to various points along the supporting rods.

The notches on the second rod correspond to the ordinates on the graph paper which represent the various balance voltages which can be applied to the back of the electrophotographic plate. A snap spring mounted on the carriage engages with the notches on this second rod and allows the indexing of the carriage progressively along the voltage axis of the graph paper as the various voltages are applied to the back of the electrophotographic plate.

The "pen" consists of a metal plunger which, when activated by a solenoid, marks the graph paper through carbon paper. The plunger makes an asymmetrical mark which allows the differentiation of several different curves on one sheet of graph paper.

As the carriage is moved along its supporting rods, an electrical spring-contact connects the back of the electrophotographic plate to a series of standard voltages which are selected lower and lower as the potential on the plate decays with time.

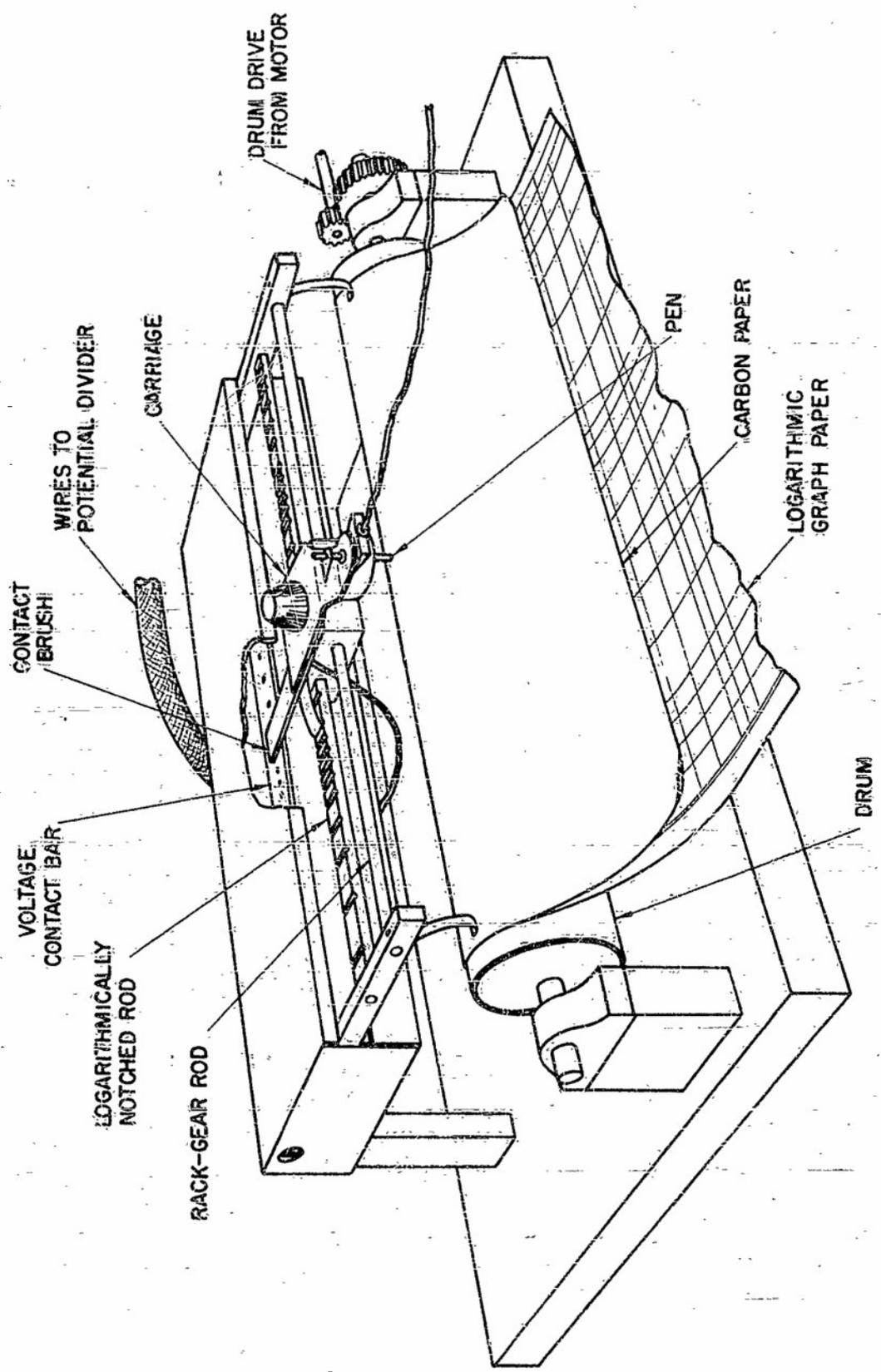


FIGURE 116: DIRECT- PLOTTING CURVE RECORDER FOR VIBRATING- PROBE ELECTROMETER

0-11853

The graph paper is mounted on a metal drum driven by a synchronous motor at a speed corresponding to the linear time scale on the graph paper.

In actual operation, the recording device is used by first mounting a sheet of semi-log graph paper and a sheet of ordinary carbon paper on the rotating drum. The electrophotographic plate is then mounted in the vibrating-probe electrometer, sensitized, and swung into position near the vibrating probe. The carriage of the recording device is then set at a potential tap somewhat lower than the potential on the plate surface, and the trace on the oscilloscope is observed as it approaches the straight line which indicates that the potential on the electrophotographic plate surface is just equal to the external potential applied to the conducting backing of the plate. At this point, a hand switch is closed and a point is marked on the graph paper. Next, the carriage of the recording device is moved to the next lower potential point and the process is repeated until the entire light- or dark-decay curve has been plotted.

The recording device has been found to reduce the time required to determine the potential-decay characteristics of electrophotographic plates and this measure of plate quality is being used increasingly in connection with various attempts to improve plate quality.

THEORY

(D. T. Williams)

Model Three as a Basis for a Theory of
Electrophotography

Introduction

As reported in the previous Quarterly Report, three models were briefly discussed as bases for a theory to describe the behavior of evaporated-selenium plates. Of these three models, the first two were discarded because they seemed to offer no likely simplification in understanding the phenomena involved. The third model was conceded to be interesting. It is proposed to look further into this model.

Review of Experimental Basis of Model Three

The experiment that was chosen as the principal basis for this model was based on the fact that the plate has a somewhat different behavior in the presence of light and an externally imposed electric field, as compared to light and a corona-induced surface charge. Experiments were described last quarter in which it was found impossible to induce a charge on selenium by use of light and an electrical field. Other experiments at the time, and some performed since then, appear to throw some doubt on the generality of this observation. However, the fact of essential irreversibility of the charging and discharging phenomena need not depend only on this type of experiment. All one needs to know is that a charge on the backing plate of an illuminated selenium layer results in

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no charge development on the selenium in the absence of a corona discharge.

The second generalization basic to the theory is that, in a corona-charged plate, with selenium deposited on a transparent conducting backing plate, photoconductivity is observed to be equally rapid for illumination on the charged side and on the backing-plate side of the selenium layer. This generalization is based on a series of experiments described elsewhere in this report.

The third generalization basic to the theory is that the light effective in discharging a corona-charged plate penetrates a relatively thin layer of the selenium. This generalization is based on experiments described elsewhere in this report.

A final fact that occupies a somewhat less crucial place in the theory is that the selenium film is considered to be vitreous. This fact has been well established; it makes certain assumptions of the theory more plausible than they would be otherwise.

The Proposed Model Three

The general nature of the proposed model will now be described.

The selenium layer is assumed to be composed of a random array of atoms. When a charge exists on the layer, it is assumed to be caused by interstitial charges in the layer. These charges are, for the moment, assumed to diffuse in such a way as to disperse more or less uniformly through the film; furthermore, they are assumed to be mobile with a mobility depending on the nature of the charge. (Supposedly, there

will be a limiting field such that the mobility vanishes at fields less than the limit.)

It is further assumed that the field due to an interstitial charge is necessary for the creation of a mobile charge by light absorption. Supposedly, these photocharges created near the interstitial charges have a mobility that may be different from that of the interstitial charges. Furthermore, both positive and negative charges may be created, with different mobilities.

The process of charge decay is now imagined to proceed as follows: After the corona charging process, light shines on the selenium surface and creates some photocharges. These diffuse rapidly in such a way as to destroy the local fields due to interstitial charges near the surface. Thereupon, interstitial charges diffuse out toward the illuminated surface, and the process is repeated.

Formal Analysis

We write V , the plate potential, as proportional to N , the interstitial charge density. That is

$$V \propto N. \tag{1}$$

Assume now that light of intensity I is incident on the surface, penetrating to the depth ξ . Within this layer, the interstitial charges have a density $N_\xi \ll N$. The number of mobile photocharges created per unit time will be proportional to I and N_ξ ; these photocharges cause the destruction of interstitial charges. That is

$$-\frac{dN}{dt} = \beta I N_\xi \xi \tag{2}$$

Here, in addition to the proportionality constant β , the factor $\frac{I\xi}{N_0}$ is introduced, equal to the ratio of thickness of illuminated layer to selenium thickness.

The equation describing N_ξ is

$$\frac{dN_\xi}{dt} = \frac{\beta I N_\xi}{N_0} - \delta [N - N_\xi - \gamma^A] \quad (3)$$

Here, the last term is the flow of interstitial charges from the selenium into the illuminated layer ξ , assumed proportional to the difference in density $N - N_\xi$, except for a threshold term γ^A .

The derivative of equation (3) by time is written

$$-\frac{d^2 N_\xi}{dt^2} = (\beta I + \delta) \frac{dN_\xi}{dt} + \beta \delta \frac{I N_\xi}{N_0} \quad (4)$$

The integral of this expression has the form

$$N_\xi = \frac{N_0}{a} e^{at}$$

where

$$a = -\left(\frac{\beta I + \delta}{2}\right) \pm \sqrt{\left(\frac{\beta I + \delta}{2}\right)^2 - \beta I \delta}$$

Hence, the general solution is, for $N_\xi = N_0$ at $t = 0$,

$$N_\xi = N_0 e^{-\beta I t} + B(e^{-\delta t} - e^{-\beta I t}) \quad (5)$$

We write the potential $V \propto N_\xi$ where ξ is the selenium film thickness.

It follows that V has the form

$$\begin{aligned}
 V &= -BI \int_0^t (N_0 e^{-BI t} + B(e^{-\delta t} - e^{-BI t})) dt \\
 &= \xi N_0 e^{-BI t} + B \xi \left(\frac{BI}{\delta} e^{-\delta t} - e^{-BI t} \right) + C \quad (6)
 \end{aligned}$$

If at $t \rightarrow \infty$, $V \rightarrow 0$, $C = 0$.

If furthermore at $t=0$ $V=V_0=N_0 \xi$,

$$B = N_0 \left(\frac{\xi}{\xi} - 1 \right) \div \left(\frac{BI}{\delta} - 1 \right) \quad (7)$$

Therefore, recalling that $V_0 = N_0 \xi$,

$$\begin{aligned}
 V &= V_0 \left[\frac{\xi}{\xi} e^{-BI t} + \left(1 - \frac{\xi}{\xi} \right) \frac{BI e^{-\delta t} - \delta e^{-BI t}}{BI - \delta} \right] \quad (8) \\
 &\approx V_0 \frac{BI e^{-\delta t} - \delta e^{-BI t}}{BI - \delta} = V_0 \frac{\delta e^{-BI t} - BI e^{-\delta t}}{\delta - BI}
 \end{aligned}$$

since $\frac{\xi}{\xi} \ll 1$.

This result predicts a decay rate proportional to I for small illumination intensity, and independent of I for large illumination intensity. This relation should be easily tested.

Discussion of Theoretical Predictions

In this analysis, certain assumptions were made that are not specifically required. Thus, the mobility of the photocharges was assumed large compared to that of the interstitial charges. Again, the range of the forces due to the interstitial charges was assumed small compared to the selenium-layer thickness. This particular hypothesis resulted in the introduction of the two periods of equation (8); the assumption of larger range may be equally plausible. Hence, the experiment suggested in which the decay rate is observed for various intensities

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I should serve to test whether the range of these forces is large or small.

If the mobility of the photocharges were assumed to be large, a different kind of dependence of V on time might be obtained.

In summarizing this model, it is to be noted that it rationalizes the irreversibility of electrophotographic processes, and the difference in decay rates for positive and negative charging. Although the detailed model is probably not correct, it would seem necessary to postulate two different charge types. In this sense, the theory is a necessary and useful step in understanding the phenomena of electrophotography.

Comparison With Experiment

A series of decay curves were run with varying intensities of yellow light on Plate 12-21-48-P, and decay rates were observed. The results are shown in Figure 117 as a plot of decay rates in reciprocal seconds as a function of the illumination intensity in ergs per square centimeter per second. Two sets of data are shown, obtained at different times. It is probably not proper to draw anything but a straight line through either set; but it is possible that an extension of the experiments would show a curvature in agreement with the prediction. For the present, it appears that the reciprocity law might be valid at low illumination, and that at higher intensity the relation is valid

$$V = V_0 e^{-BI^m t}$$

where $m = .728$.

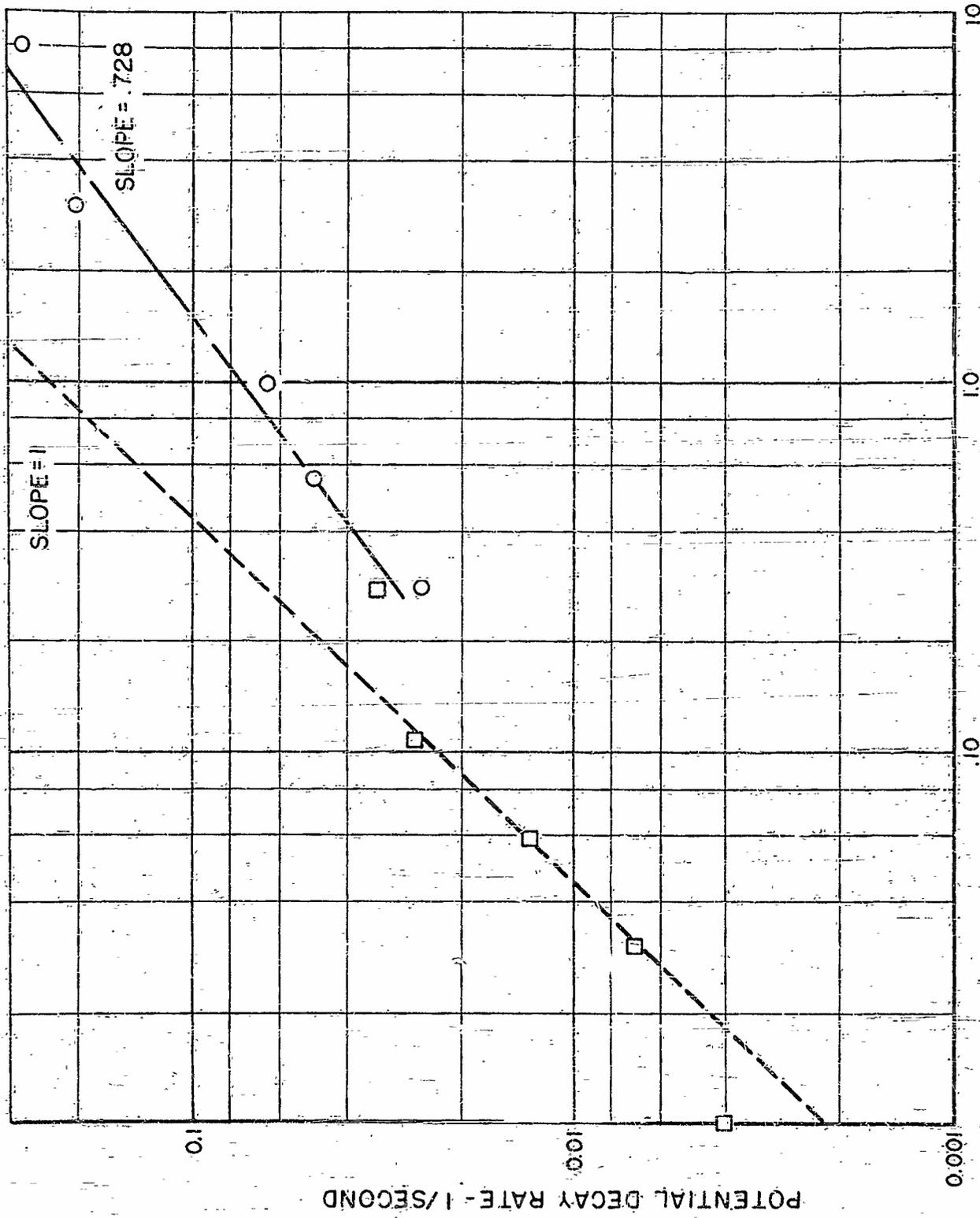


FIGURE 17. VARIATION OF CHARGE-DECAY CONSTANT WITH LIGHT INTENSITY

O-12073

Data for this report are recorded in Laboratory Record Books:

- No. 3903, pages 12 - 23, inclusive.
- No. 3931, pages 30 - 38, inclusive.
- No. 3956, pages 54 - 100, inclusive.
- No. 4241, pages 8 - 21, inclusive.
- No. 4388, pages 1 - 23, inclusive.
- No. 4536, pages 1 - 18, inclusive.
- No. 4574, page 1.

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