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DEVELOPMENT

IN THE FIELD OF ELECTROSTATICS

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(A MONTHLY JOURNAL)

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WASTELFELD MEMORIAL INSTITUTE

1st Quarterly Report

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Period June 15, 1948 to Sept. 15, 1948

Research directed towards evolving a continuous tone electrophotographic process.

The Haloid Corp. (Battelle Memorial Institute)
Rochester, N. Y.

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September 17, 1948

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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. 1 on Continuous-Tone Electrostatic Electrophotography. This report covers work for the three-month period from June 15, to September 15, 1948.

Work has been concentrated on the organization of an efficient research program and the setting up and testing of equipment to be used on the project.

Very truly yours,

C. D. Oughton

C. D. Oughton
Assistant Supervisor
Graphic Arts Research Division

CDO:swr
Enc.

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QUARTERLY PROGRESS REPORT NO. 1

(June 15, 1948, to September 15, 1948)

on

CONTINUOUS-TONE ELECTROSTATIC ELECTROGRAPHY

to

THE HALOID COMPANY

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

by

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

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

BATTELLE MEMORIAL INSTITUTE

September 15, 1948

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QUARTERLY PROGRESS REPORT NO. 1

(June 15, 1948, to September 15, 1948)

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CONTINUOUS-TONE ELECTROSTATIC ELECTROGRAPHY

to

THE HALOID COMPANY

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

from

BATTELLE MEMORIAL INSTITUTE

by

R. H. Schaffert, D. T. Williams, L. E. Walkup,
and C. D. Oughton.

SUMMARY

This report covers the work on continuous-tone electrophotography from June 15, 1948 to September 15, 1948.

The research objectives for work on continuous-tone electrophotography are listed, and the relative importance of each is considered. Three of these objectives have been selected as being of greatest importance: (1) exposure speed equivalent to 50 Weston, (2) high-quality continuous-tone reproduction, and (3) spectral response equivalent to panchromatic film.

A research program based on the above three objectives has been outlined. The initial research problems are listed under five

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classifications: (1) theoretical considerations, (2) improvements in electrophotographic plate speed, (3) improvements in electrophotographic image development, (4) improvement of spectral sensitivity of electrophotographic plates, and (5) search for better photoconductive materials.

A detailed study of the equipment required for research in electrophotography and of the equipment now available has been made.

The electrical characteristics of two available Cenco-Dershem-Lindemann electrometers were measured. Two methods of measuring electrophotographic plate potential have been recommended. One method was used to measure electrophotographic plate areas as small as 1/4 inch in diameter.

Apparatus to measure the spectral sensitivity of electrophotographic plates was assembled and used in preliminary measurements of the spectral sensitivity of selenium-coated electrophotographic plates. Measurements using the visible portion of the electromagnetic spectrum disclosed that selenium-coated plates are sensitive below and relatively insensitive above about 5800 Å. (the yellow band).

A temperature-control platen, to be used in regulating the temperature of base plates during vacuum deposition of photoconductive materials, was designed and is being constructed.

Heat treatment of representative selenium-coated electrophotographic plates for a 10-minute period at either 80° Centigrade or 90° Centigrade resulted in a lowering of the potential to which the plates could be raised.

A study was made to determine the effect of the presence of volatile foreign material in the original selenium charge on the

photoconductive properties of selenium-coated plates. No effects were observed in preliminary experiments.

The available literature on photoconductivity was reviewed and a list of materials reported to be photoconductive was compiled. The investigation of the photoconductive characteristics of these materials was started. Selenium, anthracene, acenaphthene, benzidine, fluoranthene, chrysene, and zinc titanate exhibited photoconductivity.

An electrophotographic paper plate was prepared by evaporating selenium onto a sheet of aluminum-coated paper. The images were weak but showed all details clearly.

A method of calculating the photoelectric efficiency (the ratio of electrons released to the photons incident) that is required of an electrophotographic plate to give an exposure speed of any assumed value was devised. The method was based on a number of arbitrarily chosen assumptions and is subject to change as work progresses. It was used in its present form to calculate that a photoelectric efficiency of 940 per cent is required of a selenium-coated plate to give an exposure speed of 50 A.S.A.

A model of the electrophotographic plate has been proposed in which it is assumed that small crystallites imbedded in a vitreous matrix are essential to the electrophotographic process. This hypothesis was subjected to test, but results are inconclusive. Selenium-coated plates showing detectable crystallinity exhibited electrophotographic properties.

The manner in which charged selenium plates lose their charge, as a function of illumination time, was analyzed by use of painted plates. An exponential decay rate from the initial to a final residual voltage was concluded to be a fair description of the process. Probable deviation of exponential decay rate of 5 per cent was calculated by use of 4 plates. The decay rate is found to be about 20 per cent greater for plates with positive than with negative charge. Rationalizations of these properties of electrophotographic plates are shown as based on simple and plausible assumptions.

FUTURE WORK

Experimental work for the next three months will be concentrated on the following problems:

(1) Improvement of speed and spectral sensitivity of electrophotographic plates.

(a) Equipment will be assembled to measure conveniently and quickly the general properties of electrophotographic plates. This will include various light sources, a calibrated shutter, means of sensitizing the plate, means of connecting the plate to an electrometer and measuring its potential as a function of time of exposure. The various light sources will cover the spectrum from 2000 to 20,000 Å.

(b) Various methods will be used to measure the plate potentials with the several types of electrometers available.

(c) When the equipment in (a) above is in operating order, tests will be made on the best available electrophotographic plates to determine their electrophotographic properties and general characteristics.

(2) Collect and test other photoconductors.

(a) The equipment discussed in (1-a) above will be used to measure the electrical and photoconductive properties of the photoconductive materials.

(b) The compilation of a list of materials reported to be photoconductive will be continued.

(c) Additional materials appearing in the list discussed in (2-b) above will be collected and their photoconductive characteristics will be studied.

(3) Study developers and developing processes for use on continuous tone.

(a) Detailed consideration will be given to the determination of requirements for uniform development of large, dark areas.

(b) A charged powder cloud developing equipment will be set up and an attempt will be made to improve this system of development.

(c) An electrical conductor will be placed near the electrostatic image on the plate during the developing process. The effect of such an arrangement on the resulting image will be evaluated.

(4) Study selenium-coated plates. Experiments will be conducted on the preparation of selenium-coated plates containing no crystalline as well as various degrees of crystalline structure in the selenium coating. Measurements from the plates will be used in the development of the theoretical background of electrophotography which is necessary to the development of plates with better speed and spectral sensitivity.

(a) Temperature-controlled platen will be installed in the vacuum unit to hold temperature of plate constant during evaporation.

(b) The relationship between selenium film thickness and plate speed will be determined.

(c) The effect of plate temperature (during vacuum evaporation of selenium) on plate speed will be determined.

(d) Effect of impurities in, or addition to, selenium on the plate speed will be determined.

(5) Literature and general study of the problem.

(a) Collect and review literature on photoconductivity.

(b) Collect and review literature on fluorescent materials that exhibit photoconductivity.

(6) Theoretical considerations. The aim of this portion of the project is to develop a theoretical background for electrophotography in order to aid in the attainment of a rapid and practical means of dry photography and to explore the field for other possibilities.

(a) Work will be concentrated on the evaluation of a theory to explain electrostatic and photoconductive mechanisms of electrophotography.

PUBLICATIONS AND/OR TALKS

(None)

RESEARCH PROGRAM FOR CONTINUOUS-TONE ELECTROPHOTOGRAPHY

Research Objectives

The following is a list of the objectives called for in the research proposal on this project for developing continuous-tone

electrophotography. Each item is discussed briefly as to its relative importance and relative difficulty in the research plan:

(1) Rapid Development by Dry Process

Rapid and dry development is now possible by methods evolved in past research efforts. Further improvements may be possible but such work will be in later stages of the project, after more definite information is available on improved plates and developing powders.

(2) Resolving Power of 20 to 30 Lines per Millimeter

The photographic resolving power using present materials is approximately seven to ten lines per millimeter. No work is planned on this specification until after plates of greater photographic speed have been produced and other techniques of the process have been established.

(3) Exposure Speed Equivalent to 50 Weston

This is considered the most important of all of the specifications listed. A major portion of effort will be concentrated on this problem in the early stages of the project. The fundamental photoconductive phenomena involved in electrophotography will be investigated and searches will be made for more sensitive photoconductors.

(4) Spectral Response Equivalent to Panchromatic Film

This specification will be considered along with exposure speed, but it will be considered of secondary importance until plates of relatively high exposure speeds have been developed.

(5) High-Quality Continuous-Tone Reproduction

This specification will be considered in the early stages of the project as it is relatively independent of the exposure speed of the electrophotographic plates.

(6) Retention of Sensitivity for Reasonable Periods

(7) A Minimum of Photoregression for a Period of Twelve Hours

No work is contemplated on these two specifications until an electrophotographic plate of adequate exposure speed has been developed.

(8) Adherence to Reciprocity Law Equivalent to Silver Photography over a Wide Range of Light Intensities

Tests will be made on the reciprocity of selenium electrophotographic plates as they are developed.

(9) Permanence of Prints

Present prints are considered to be sufficiently permanent, but this specification will be considered if changes are proposed in the developing or the fixing techniques.

(10) Uniformity of Quality of Plates or Film from Batch to Batch

This cannot be considered until specifications are available on plates to be manufactured.

(11) Methods for Sensitizing Plates or Film to be of Light-Weight Construction and Simple to Operate

(12) Adaptability to a Continuous Process of Photography, Developing, and Printing in a Single Unit

These specifications concern the design of the final camera and it will be considered after the more fundamental problems involved have been solved.

(13) Satisfactory Operation of the Process and Materials in
Extreme Humidity

This specification will be considered after more definite information is available on the materials and process to be proposed.

(14) Satisfactory Operation of the Process in the Temperature
Range of minus 65°F. to plus 165°F.

Present selenium plates will deteriorate at plus 165°F., but plates involving other photoconductors may meet these specifications.

(15) Evolution of an Electrophotographic Film Flexible Enough to
Use in Roll Form

This specification will not be attempted until more is known about the fundamentals of the process and the materials involved.

Major Initial Research Problems

A consideration of the specifications for this project led to the selection of certain items as being more important than others in the early stages of the project. Other specifications will become more important as work progresses.

The following outline includes the major initial problems and a tabulation of a number of specific items to be considered under each problem.

Theoretical Considerations

- (1) Evolve theory to explain electrostatic and photoconductive mechanism of electrophotography.
- (2) Determine from theoretical considerations the role of primary and secondary photoconductivity in dissipation of electrical charges on electrophotographic plates.
- (3) Evolve theory to explain role of triboelectricity in electrophotographic image development.

Improvements in Electrophotographic Plate Speed

- (1) Calculate the rate of charge decay (constant light intensity) corresponding to 50 A.S.A. (photographic speed approximately equal to the older Weston rating) and calculate the approximate photoelectric efficiency required of electrophotographic plates.
- (2) Assemble apparatus capable of measuring decay rates necessary for the exposure of electrophotographic plates.
- (3) Determine relationship between selenium thickness and light decay rate (plate speed).
- (4) Determine effect of temperature (during vacuum evaporation of selenium) on plate speed.
- (5) Determine effect of impurities in, or additions to, selenium on the plate speed.

(6) Determine effect of dye sensitizers on electrophotographic plate speed.

(7) Collect and review literature on photoconductivity.

(8) Develop technique for detecting forms of photoconductivity and of measuring amounts under different electrical fields, different lights, and in different forms of matter.

(9) Determine the photoconductive properties of a number of new materials both as crude materials and as coatings on plates.

(10) Collect and review literature on fluorescent materials that exhibit photoconductivity and test promising materials in the electrophotographic process.

(11) Test the photoemission of insulators or of metals on insulating surfaces as a possible basis for a system of electrophotography.

(12) Determine how the electrical charges reside on an electrophotographic plate and how it is limited in potential.

(13) Use combinations of long and short wave length light on electrophotographic plates to enhance sensitivity.

(14) Develop methods for using small test probes with electrometers or of connecting the electrically conductive backing of electrophotographic plates to electrometers as a means of measuring photoconductivity.

(15) Determine the dependence of photoconductivity in selenium on the physical state and structure of the material.

(16) Form photoconductive layers of lacquers or other binders containing photoconductive particles.

(17) Determine the nature of the electrical charged latent image on electrophotographic plates and the nature and magnitude of the electrical fields just above such images.

(18) Measure the charge and force on single particles of developer powder to use in connection with the amount of potential needed on practical plates.

(19) Improve the efficiency of development process because it may improve the effective speed of the plates.

(20) Construct synthetic plates with small areas which can be charged to known potentials to explore the functioning of electrophotographic developers.

(21) Determine the value of a "keeper" on charged electrophotographic plates.

(22) Construct two humidity-control cabinets for carrying out experiments under various conditions.

Improvements in Electrophotographic Image Development

(1) Develop apparatus for quantitative measurement of triboelectricity.

(2) Determine requirements for linear relationship between powder deposition and plate potential (continuous-tone development).

(3) Determine effect of particle size of powder on continuous-tone development.

(4) Arrange electrometers for measuring charge and potential on small areas of electrophotographic plates.

- (5) Determine the forces on and the motion of charged particles in the electrical field near the latent electrostatic image.
- (6) Measure electrical charge on developer powder particles.
- (7) Determine the reason for some combinations of plates, plate charge, and developer giving better continuous-tone reproduction than other combinations.
- (8) Determine the efficiency of developer powders in producing black images on paper.
- (9) Design plates which have a mottled image both by light mottling and by mottling the photoconductive layer. Use such plates in the reproduction of continuous tones.
- (10) Devise means of developing plates by clouds of electrically charged dust.
- (11) Place an electrical conductor near the electrostatic image on the plate during the developing process.

Improvement of Spectral Sensitivity of Electrophotographic Plates

- (1) Assemble apparatus for routine determinations of spectral sensitivities of electrophotographic plates.
- (2) Measure spectral sensitivity of selenium plates prepared under varied conditions and also measure spectral sensitivity of other plate materials as developers.
- (3) Determine effect of additions and impurities on spectral sensitivity of selenium plates. Also include effect of such additions and impurities with other photoconductive materials.

(4) Determine effect of dye sensitizers on spectral sensitivity of electrophotographic plates.

Search for Better Photoconductive Materials

(1) Survey the literature to compile list of promising photoconductive materials.

(2) Develop experimental technique for preparing sample electrophotographic plates or test plates with coatings of various photoconductive materials.

(3) Develop apparatus for routine analysis and measurement of plate characteristics with various photoconductive materials as coatings.

Research Facilities and Equipment

In the early stages of research work, equipment will be acquired and assembled for performing a number of specialized measurements connected with the electrophotographic process. A brief statement with respect to these items of equipment will be recorded here.

Optical Measurements

Equipment is being assembled to provide monochromatic and polychromatic light of calibrated intensities for tests on plate sensitivities and spectral characteristics. It is necessary to the understanding of the process of photoconductivity that such light sources extend into the infrared and ultraviolet regions of the spectrum. In later stages of research

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work it will be necessary to measure such properties as the resolving power, reciprocity, contrast, and latitude of electrophotographic plates.

Electrical Measurements

Electrometer systems will be required for a number of tests associated with the measurement of photoconductivity of materials, and the electrical fields near electrophotographic plates. Such electrometers will also serve to measure the resistivities of materials.

Physical Measurements

Equipment is available for the determination of a number of physical properties of materials. This includes X-ray and electron-diffraction equipment, petrographic and metallographic microscopes, spectrographic analysis, chemical analysis, classifiers for fine powders, and equipment for determining the thickness of thin films.

Equipment is available for the vacuum deposition of thin films and for the production of fine powders.

EXPERIMENTAL WORK

by

J. J. Rheinfrank, W. E. Bixby, D. L. Fauser,
P. G. Andrus, and E. C. Rickor.

Electrometers for Testing Plate Surface Potential

In the study of electrophotographic plates, it is desirable to measure the potential of the outer surface of the photoconductive layer

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with respect to the conductive backing plate and to measure the total quantity of electrical charge collected on the surface of the photoconductive layer.

No practical methods have been devised for measuring the quantity of electrical charge on the plate, but two methods have been proposed and tested for measuring plate surface potential. These methods are shown schematically in Figure 1. In the first method (1a), a small test probe is brought near the charged surface of the plate and the deflection on the electrometer is taken as a measure of the potential on the surface of the plate. This deflection is calibrated by replacing the electro-photographic plate with a metal plate and adjusting the potential of the plate to various known values. The equivalent electrical circuit for this arrangement is shown in Figure (1b). Here C_1 represents the electrical capacity through the layer of photoconductive material, C_2 the capacity between the plate surface and the test probe, and C_3 the leakage capacity from the test probe and the electrometer to ground. The electrometer is exposed to a fraction of the plate potential dependent on the relative values of C_2 and C_3 .

In the second method of testing plate surface potential, Figure (1c), the electrometer is connected directly to the electrically conductive backing of the plate. This backing is grounded after the plate surface has been charged under corona discharge wires. The electrometer reads zero when first connected, but increases in readings as the plate is discharged under a light. The equivalent circuit for this test is shown in Figure (1d). Here the leakage capacities to ground of the front and

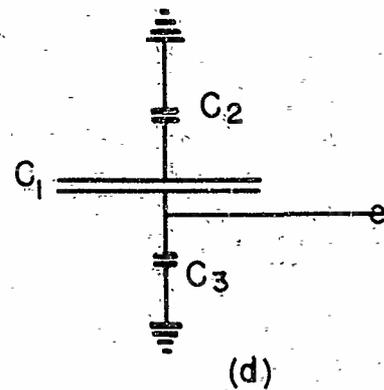
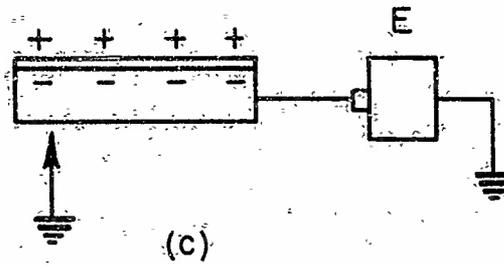
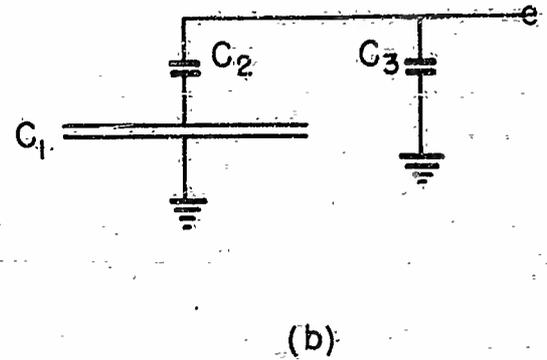
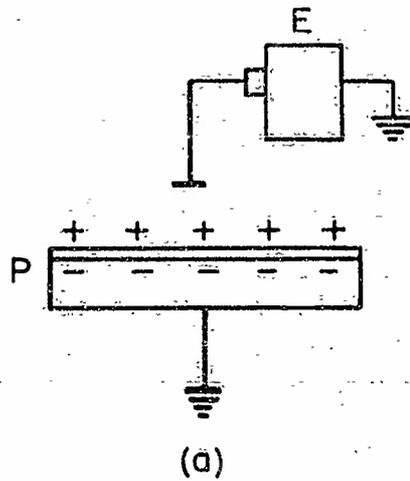


FIGURE 1. ELECTROMETER TESTS FOR PLATE POTENTIAL

back of the plate are of primary importance. During the charging of the plate, C_3 is shorted out but C_1 and C_2 are charged to the same potential. The short is then removed from C_3 , but the electrometer still reads zero because C_3 is not charged. When the plate is exposed to light, the equivalent capacity C_1 is shorted out and its charge is effectively neutralized. At this point the charge on C_2 is shared with C_3 and this potential appears across the electrometer.

Both of these methods have been tested in the laboratory and found to function satisfactorily with Cenco-Dershem electrometers. The second method (1b) was used to measure the decay characteristics of plate areas as small as 1/4-inch diameter by (a) placing the charged plate behind a metal masking form having a circular window of 1/4-inch diameter, and (b) using, as a test probe, a circular loop of wire slightly larger than 1/4 inch.

Electrometers will be important tools for experimental work on this project. Two Cenco-Dershem instruments (Central Scientific Company, No. 71007) are available and two other types of electrometers have been ordered (Central Scientific Company electronic electrometer, No. 71010, and a Cambridge Instrument Company, Ryerson-Lindemann electrometer).

Tests on the Cenco-Dershem electrometers have determined their sensitivities and have revealed a serious drift in readings with time. Sensitivities up to 500 millimeters per volt (scale at one meter) can be obtained with usable stability. However, under these conditions the scale reading on the electrometer increased 35 per cent over its initial

reading in ten minutes of exposure. Lower sensitivity settings show increases of 10 to 15 per cent in ten minutes. Both of the available instruments are subject to the same type of drift.

The observed drifts in the Cenco-Dershem electrometers are attributed to imperfect silvering of the armature and they will introduce serious errors in any quantitative readings taken with them.

Apparatus to Measure the Photographic Characteristics
of Electrophotographic Plates

A Beckman Photoelectric Quartz Spectrophotometer and thirteen Baird Interference Filters were procured, and a focal plane shutter was constructed, to aid in the measurement of the spectral sensitivity, the speed, and the reciprocity characteristics of electrophotographic plates.

The spectrophotometer and the interference filters will make available monochromatic light, the spectrophotometer supplying light in the 2000 Angstrom to 20,000 Angstrom range and the interference filters transmitting bands about 100 Angstroms wide in approximately equal steps from 4000 Angstroms to 7000 Angstroms. The focal plane shutter will enable exposures between 0.01 second and 30 seconds to be made.

The Beckman Spectrophotometer, in addition to being used as a source of monochromatic light, can be used in determining (1) spectral-transmission and spectral-absorption curves of filters and (2) spectral-radiation curves of luminous sources.

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Spectral Sensitivity of Selenium-Coated
Electrophotographic Plates

Preliminary measurements on the spectral sensitivity of selenium-coated electrophotographic plates were made using the apparatus mentioned in the above section.

Measurements at wave lengths of 4075 Å, 4640 Å, 4875 Å, 5530 Å, 5760 Å, 6050 Å, 6430 Å, 6745 Å, and 7045 Å disclosed: (1) that selenium-coated electrophotographic plates were sensitive in the violet (4000 Å - 4460 Å) and the indigo (4460 Å - 4640 Å) portions of the electromagnetic spectrum; (2) that selenium-coated electrophotographic plates were probably most sensitive in the blue portion (4640 Å - 5000 Å); (3) that the sensitivity of selenium-coated plates was high in the green up to at least 5530 Angstroms; (4) that the sensitivity of selenium-coated plates decreased rapidly near the end of the green (5760 Å); and (5) that the sensitivity of selenium-coated plates was extremely low in the red (6200 Å - 7200 Å).

Design and Construction of a Temperature-Control
Platen for Vacuum Evaporation

Work was started on the assembling of equipment necessary to control and measure the temperature of the plate during vacuum deposition of the selenium. Temperature control will be achieved through a temperature-controlled platen in contact with the plate. Temperature measurement of the plate will be made with thermocouples.

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Heat Treatment of Vacuum-Evaporated Selenium-Coated
Electrographic Plates

Two vacuum-evaporated selenium-coated electrographic plates were cut into quarters. Each quarter was then checked for its initial potential. The initial potential is defined as that potential which a given plate will hold in the dark, ten seconds after it has been sensitized under standard conditions. The plates were tested after both positive and negative sensitization.

Each quarter of plate was then heated in an oven at atmospheric pressure at a known temperature for a known time. The initial potential was determined again after the heat treatment. It was found that heating a plate for 10 minutes at 80°C. caused a definite decrease in the initial potential when the plate was charged positively, while not affecting the negative characteristics to a great degree. A 10-minute heat treatment at 90°C. caused a plate to lose its ability to hold a positive charge and a definite decrease in its negative initial potential.

Effect of Volatile Foreign Material on Vacuum-Evaporated
Selenium Electrographic Plates

An attempt was made to determine if there were characteristics present in selenium electrographic layers that were peculiar to plates prepared in each of the two following groups: (1) plates on which the selenium layer was deposited during the first portion of the evaporation process, and (2) plates on which the selenium layer was deposited during the last portion of the evaporation process.

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It was assumed that the foreign volatile materials in the selenium charge, e.g., water or gas, would have a greater concentration in the plates formed during the first part of the evaporation cycle.

For this work, a charge of shotted, chemically pure selenium was evaporated onto two weighed five by six-inch mirror-finished aluminum plates. An area of four by five inches was coated on one side of each plate. A plate for each of the two above groups was prepared during one evaporation cycle. This was made possible by a device which exposed only a four by five-inch area of one plate at a time and which could be activated to replace the first plate by the second plate after the first portion of the selenium charge had been evaporated.

The weighed selenium charge was placed in the vacuum chamber on a flat plate 6 inches from the surface to be coated. The charge was spread uniformly over a 4 by 5-inch area of the plate which was heated by tungsten wire filaments during the evaporation cycle.

Ten plates were prepared as follows:

1. The plates to be coated and the selenium charge were placed into the bell jar.
2. The bell jar was pumped to a vacuum of approximately 0.38 microns.
3. The selenium charge was heated to evaporation temperature and selenium was deposited on the first plate.
4. The filaments were turned off and the remaining selenium charge and the coated plate were allowed to cool.
5. The first plate was replaced by the second plate without breaking the vacuum.

6. The remaining portion of the selenium charge was evaporated onto the second plate.

7. The evaporating chamber was allowed to cool.

8. The vacuum was released.

9. The coated plates were removed from the bell jar.

Table 1 gives the known data for each evaporation cycle.

The ten plates prepared as discussed above were tested for their maximum potential and their potential decay characteristics by means of a calibrated Cenco-Dershem-Lindomann electrometer. The plates were charged to a maximum potential (both negative and positive polarity) and both the light and dark decay (potential decrease per unit time) were recorded.

There was found to be no characteristics peculiar to the electrophotographic plates of group one as compared to those of group two (i.e. plates coated by the first and second portions, respectively, of the selenium charge). Hence, it is indicated that volatile foreign materials in the selenium charge have no effect upon the properties of selenium electrophotographic plates within the limits of the measurements employed in this test.

The data obtained in the above work have not been fully analyzed. The results of further analysis will be presented in the next report.

Compilation of a List of Materials
Reported to be Photoconductive

The literature records a number of materials as being photoconductive. Not all of these would be suitable for electrophotographic plates because of their lack of insulating properties in the dark or because of other unsuitable physical characteristics.

TABLE 1. DATA ON THE PRODUCTION OF SELENIUM ELECTROPHOTOGRAPHIC PLATES

Properties of Process	Cycle				
	1	2	3	4	5
Plate Number	1 and 2	3 and 4	5 and 6	7 and 8	9 and 10
Weight of Selenium Charge (grams)	7	7	4	5	5
Amount of Selenium Deposited on First Plate (grams)	.0728	.6873	.1934	.5242	.2058
Intermediate Cooling Time (minutes)	5	105	15	15	15
Amount of Selenium Deposited on Second Plate (grams)	.9843	.3093	.3266	.1680	.3318
Final Cooling Time (minutes)	8	0	0	0	5

Table 2 on the following page has been prepared from literature references available at the present time.

Investigation of Materials Reported
to be Photoconductive

A program to investigate the photoconductive characteristics of the materials listed in Table 2 was begun. Five techniques of observing photoconductivity were selected for use in preliminary work: (1) Evaporate the material onto a metal plate; sensitize the coated plate by passing it under a corona discharge; place the charged plate near an electrostatic electrometer; record the rate of decay of the charge when the plate is in darkness and when the plate is exposed to a white light. (2) Break up the material using mortar and pestle; paint material onto a metal plate using a suitable binder; sensitize and measure rates of charge decay as set forth in (1). (3) Break up the material using a mortar and pestle; spread a thin layer of the material onto a metal plate; sensitize and measure rates of charge decay as set forth in (1). (4) Break up the material using a mortar and pestle; place material into a special conductivity cell; record the current through the material in darkness and when exposed to a white light. (5) Compress the material into thin discs; fire; evaporate or spray a conductive coating on one side of the disc; sensitize and measure rates of charge decay as set forth in (1).

Using methods (3) and (5) above, materials were checked for photoconductivity. A No. 2 photoflood at a distance of approximately 6 inches was used as the source of white light. A Cenco-Dershaw electrometer (Central Scientific Company, No. 71007) in Battelle Mount (I-15552),

TABLE 2. MATERIALS KNOWN OR SUSPECTED
TO BE PHOTOCONDUCTIVE

Inorganic Compounds		
Chemical Name	Chemical Formula	Other Name
Aluminum oxide	Al_2O_3	Corundum
Antimony	Sb	-
Antimony trisulphide	Sb_2S_3	Stibnite
Bismuth	Bi	-
Bismuth trisulphide	Bi_2S_3	Bismuthinite
Boron	B	-
Cadmium	Cd	-
Cadmium selenide	CdSe	-
Cadmium sulphide	CdS	Greenockite
Cadmium telluride	CdTe	-
Calcium fluoride	CaF_2	Fluorite
Cerium tungstate	$Co(WO_4)_2$ $Co_2(WO_4)_3$	-
Cuprous iodide	CuI	Marshite
Cuprous oxide	Cu_2O	Cuprite
Cupric oxide	CuO	Paramelaconite Tenorite
Germanium	Ge	-
-	$Pb_2Sb_2S_5$	Jamesonite
-	$Pb_3Sb_2S_6$	Boulangorite
-	$PbCuSbS_3$	Bournonite
Lead chromate	$PbCrO_4$	Crocoite

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TABLE 2. Continued

Inorganic Compounds		
Chemical Name	Chemical Formula	Other Name
-	-	Lead halides
Lead selenide	PbSe	Clausthalite
Lead sulphide	PbS	Galena
Lead telluride	-	-
-	-	Leonard Phosphors
Mercuric iodide	HgI ₂	-
Molybdenum disulphide	MoS ₂	Molybdenite
Potassium bromide	KBr	-
Potassium chloride	KCl	Sylvite
Selenium	Se	-
Selenium sulphur	Se·S	-
Silicon	Si	Adamantine
Silicon dioxide	SiO ₂	Yellow citrine
-	AgSbS ₂	Miargyrite
Silver thioantimonite	Ag ₃ SbS ₃ (3Ag ₂ SSb ₂ S ₃)	Pyrargyrite
Silver thiocarbonate	Ag ₃ AsS ₃ (3Ag ₂ SAs ₂ S ₃)	Proustite
-	Ag ₅ SbS ₄	Stephanite
-	Ag ₉ SbS ₆	Polybasite
-	Ag ₉ AsS ₆	Plarcite
Silver bromide	AgBr	-

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TABLE 2. Continued

Inorganic Compounds		
Chemical Name	Chemical Formula	Other Name
Silver chloride	AgCl	Cerargyrite
Silver iodide	AgI	Iodyrite
Silver oxide	Ag ₂ O	-
Silver sulphide	Ag ₂ S	Argentite, acanthite
Sodium chloride	NaCl	Rock salt
Strontium selenide	SrSe	-
Sulphur	S	-
Sulphur-anthracene	-	-
-	-	Thallium halides
Thallos sulphide	Tl ₂ S	-
-	Tl.S.O	Thalofide
Tin (stannous) sulphide	SnS	-
Titanium oxide + barium titanate	TiO ₂ + BaTiO ₃	-
Titanium oxide + magnesium titanate	TiO ₂ + MgTiO ₃	-
Titanium oxide + zinc titanate	TiO ₂ + ZnTiO ₃	-
Tungsten trioxide	WO ₃	-
Vanadium pentoxide	V ₂ O ₅	-
Zinc	Zn	-
-	-	Zinc cadmium sulphide
Zinc orthosilicate	Zn ₂ SiO ₄ (2ZnOSiO ₂)	Willemite

TABLE 2. Continued

Inorganic Compounds		
Chemical Name	Chemical Formula	Other Name
Zinc oxide	ZnO	Zincite
Zinc silicate	$2\text{ZnOSiO}_2\text{H}_2\text{O}$	Calamine
Zinc sulphide	ZnS	Wurzite, sphalerite
Zinc titanate	ZnTiO_3	-
Zirconium dioxide	ZrO_2	Baddoloyite
(Organic Compounds)		
Acenaphthene	$\text{C}_{10}\text{H}_6(\text{CH}_2)_2$	Naphthylene othylene
Anthracene	$\text{C}_6\text{H}_4:(\text{CH})_2:\text{C}_6\text{H}_4$	-
Anthraquinone	$\text{C}_6\text{H}_4:(\text{CO})_2:\text{C}_6\text{H}_4$	-
Benzidine	$(\text{NH}_2\text{C}_6\text{H}_4)_2$	-
Chrysene	$\text{C}_{18}\text{H}_{12}$	-
Fluoranthene	$\text{C}_{15}\text{H}_{10}$	Idryl
Naphthalene	C_{10}H_8	-
Phenanthrene	$\text{C}_{14}\text{H}_{10}$	-
Pyrogallol	$\text{C}_6\text{H}_3(\text{OH})_3$	Pyrogallic acid
Quinone	$\text{O}:\text{C}_6\text{H}_4:\text{O}$	-
Tartaric acid	$\text{C}_4\text{H}_6\text{O}_6$	-

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TABLE 2. Continued

Inorganic Compounds		
Chemical Name	Chemical Formula	Other Name
-	-	Halogenated, alkylated, hydroxylated, amino, and quinone derivatives of benzol and polycyclic aromatic hydrocarbons.
-	-	Dyes such as: Fuchsine, crystal violet, methyl violet, brilliant green, malachite green, Auramine O, Rhodamine B, Rhodamine 6G, Pinaveridol, Pinacyanol, Phenosafranin, Erythrosin B.

at a sensitivity setting of 6-50, was used to record the rates of charge decay. Selenium, anthracene (Kodak), anthracene (Reilly), acenaphthene, benzidine, fluoranthene, chrysene, and zinc titanate exhibited photoconductivity. Pyrene, naphthalene, barium titanate, magnesium titanate, Rhodamine B, Erythrosin B, diphenylene oxide, potassium nickel edithiooxalate, diphenylene oxide, and s-diphenylcarbazine did not exhibit photoconductivity under the test conditions used. (See Table 3.)

Preparation of Electrophotographic Plate
on Aluminum-Coated Paper

A sheet of aluminum-coated paper, obtained from The Haloid Company, Rochester, New York, was selenium-coated by vacuum evaporation (Plate 9-9-4P-E). Images were obtained when the plate was sensitized, exposed, and developed under standard conditions. The images were weak, but showed all details clearly. Tests with an ohmmeter revealed the aluminum-coated paper to have a resistance greater than 250,000 ohms per inch; hence, it cannot be considered a good conductor. The paper was probably prepared by spreading a stearic acid coated aluminum powder on paper coated with an adhesive. The stearic acid is a good electrical insulator. The large amount of charge held by the paper itself was revealed by the dust patterns which formed in the nonimage areas of the plate. Such patterns have been noted previously on Lucite plates.

TABLE 3. MATERIALS TESTED FOR PHOTOCONDUCTIVITY

Test Material	Test Method*	Decay in Darkness**	Photoconductive
Selenium	(3)	Rapid	Yes
Anthracene (Kodak)	(3)	Rapid	Yes
Anthracene (Reilly)	(3)	Moderate	Yes
Acenaphthene	(3)	Moderate	Yes
Rhodamine B	(3)	-	No
Fluoranthrene	(3)	Moderate	Yes
Erythrosin B	(3)	-	No
Zinc titanate	(5)	Rapid	Yes
Pyrene	(3)	-	No
Naphthalene	(3)	-	No
Benzidine	(3)	Slow	Yes
Barium titanate	(5)	-	No
Magnesium titanate	(5)	-	No
Chrysene	(3)	Slow	Yes
Diphenylene oxide	(3)	-	No
Potassium nickel odithiooxalate	(3)	-	No
A-Diphenylcarbazine	(3)	-	No
Diphenylene oxide	(3)	-	No

* Test Method (3): Break up the material using a mortar and pestle; spread a thin layer of the material onto a metal plate; sensitize the coated plate; place the charged plate near an electrostatic electrometer; record the rate of decay of the charge when the plate is in darkness and when the plate is exposed to white light (No. 2 photoflood at 6 inches). Test Method (5): Compress the material into thin discs; fire; evaporate or spray a conductive coating on one side; sensitize and measure rates of decay as set forth in Method (3).

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Footnotes to Table 3 continued

** Slow: 0 to 2.5 centimeters in first minute. Battelle Instrument I-15552 at sensitivity of 6-50. Moderate: 2.5 to 7.5 centimeters in first minute. Rapid: 7.5 to 10.0 centimeters in first minute.

Preliminary Calculations of Electrophotographic
Plate Sensitivity

The electrophotographic plate is given a latent electrostatic image by having the electrostatic charge on portions of its area discharged by the action of light. At present this discharging action is assumed to be a manifestation of photoconductivity, i.e., individual photons of light are believed to liberate individual electrons. In some photoconductors, these electrons travel only a short distance; in others, they travel effectively from one electrode to the other. In still other cases, they may move at sufficient speeds to liberate other electrons in a cascade effect so that the original single photon effectively liberates many electrons by "triggering" the cascade discharge.

It is not known which of the several photoelectric effects are actually present in selenium plates or which of them can be utilized ultimately in improved plates. However, it is possible to make calculations of importance to future planning of research by making a series of arbitrary assumptions about the nature of the electrical system involved. The following are pure assumptions and are subject to change as research progresses:

1. The electrical charge involved in electrophotographic plates resides on the surface of the photoconductive layer.
2. Potentials measured by a substitution method involving an electrometer (method discussed elsewhere in this report) are the true potentials of the surface of the photoconductive layer.

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3. Electrons liberated by the action of light in the photoconductive layer carry one electronic charge of electricity from the surface of the photoconductive layer to the conductive backing plate.

On the basis of these assumptions the following equations may be set down as applying to electrophotographic plates:

$$Q = \frac{1.407 VKA}{d} \times 10^6$$

Q = Electrons liberated by light.

V = Volts drop in potential of surface of photoconductive layer.

K = Dielectric constant of photoconductor.

A = Area in square inches of the photoconductive layer.

d = Thickness in inches of the photoconductive layer.

If A and d are expressed in centimeters, the equation becomes:

$$Q = \frac{0.554 VKA}{d} \times 10^6$$

Using the above expression, Q may be calculated for present selenium plates. The following numerical assumptions are involved. These values are approximately correct for an average, usable plate.

0.0003 inch = Thickness of selenium layer.

6.0* = Dielectric constant of selenium.

50 = Voltage drop of selenium surface potential necessary to produce image.

1.0 square inch = Area of plate.

$$\begin{aligned} Q &= \frac{(1.407) (50) (6.0) (1.0)}{0.0003} \times 10^6 \\ &= 1.40 \times 10^{12} \text{ electrons / in.}^2 \\ &= 0.218 \times 10^{12} \text{ electrons / cm.}^2 \end{aligned}$$

Knowing the number of electrons which must be liberated by the action of light in exposing an electrophotographic plate provides one

* Handbook of Chemistry and Physics, 29th Edition.

portion of the data necessary to calculate the photoelectric efficiency that is required of a plate to give an exposure speed of an assumed value. The remaining necessary data must be calculated from the quantity of light available to make the exposure.

In modern photographic literature, the A.S.A. (American Standards Association) exposure index is employed to designate film exposure speeds. A strict definition of the number may be found in the literature*, but it is sufficient for the present calculation to take the definition as:

$$\text{A.S.A. Exposure Index} = \frac{1}{E}$$

where E is the minimum average exposure (in meter-candle-seconds) necessary to produce negatives which will result in excellent prints of an object having a wide brightness range. This assumption is justified only for use in estimations like the present one and more work will have to be done before an adequate expression of electrophotographic plate exposure sensitivity is devised. The inherent differences between electrophotography and silver halide photography are responsible for this lack of applicability of A.S.A. speed ratings to this process.

Other assumptions are necessary to the calculations. As in the case of the calculations of the electrical properties of the photoconductive layer these assumptions are made arbitrarily. They may have to be changed in later work; and the final results must be interpreted only in the light of the limitations imposed by these assumptions. These assumptions are:

1. An A.S.A. exposure index of 50 is assumed from the research proposal on this project.

* American Standard Method for Determining Photographic Speed and Speed Number (Z38.2.1 - 1946) (originally published in Jour. Opt. Soc. American 33: 479-86, No. 8, (August, 1943)).

2. An amount of light equivalent to $1/50 = 0.02$ meter-candle-seconds is assumed to be necessary to produce the 50-volt drop employed in the calculations of the electrical characteristics of the plate.

3. The photoconductive layer on the plate is assumed to be uniformly sensitive to radiations between 400 and 700 millimicrons wave length and to be completely insensitive to other radiations.

4. The light involved in the exposure of the plate is assumed to be in accord with the light used to test silver halide photographic films*. This light corresponds closely in spectral distribution to noon sunlight. (On Figure 2, A represents the assumed spectral distribution and B average noon sunlight.**)

5. A spectral visibility curve is assumed.***

6. A conversion factor of 688 lumens per watt at maximum visibility is assumed****.

Figure 2, curve A, gives the spectral distribution of energy in the assumed light source in terms of microwatts per square centimeter per millimicron. Values taken from this curve are given in Table 4, Column 4. Column 1 of this table represents a numerical integration of the area under this curve 400 to 700 millimicrons in 10 millimicron steps. The total area represents 27,900 microwatts of radiant energy.

Figure 3 shows a plot of the spectral sensitivity of the average human eye. This function is multiplied by the energy distribution curve A

* Jour. Opt. Soc. Amer., 23, 359 (1933).

** Handbook of Colorimetry, A. C. Hardy, 19-22, 1936.

*** IES Lighting Handbook, Illuminating Engineering Society, 4-5, 1947.

**** Handbooks of Chemistry and Physics, Chemical Rubber Publishing Co.

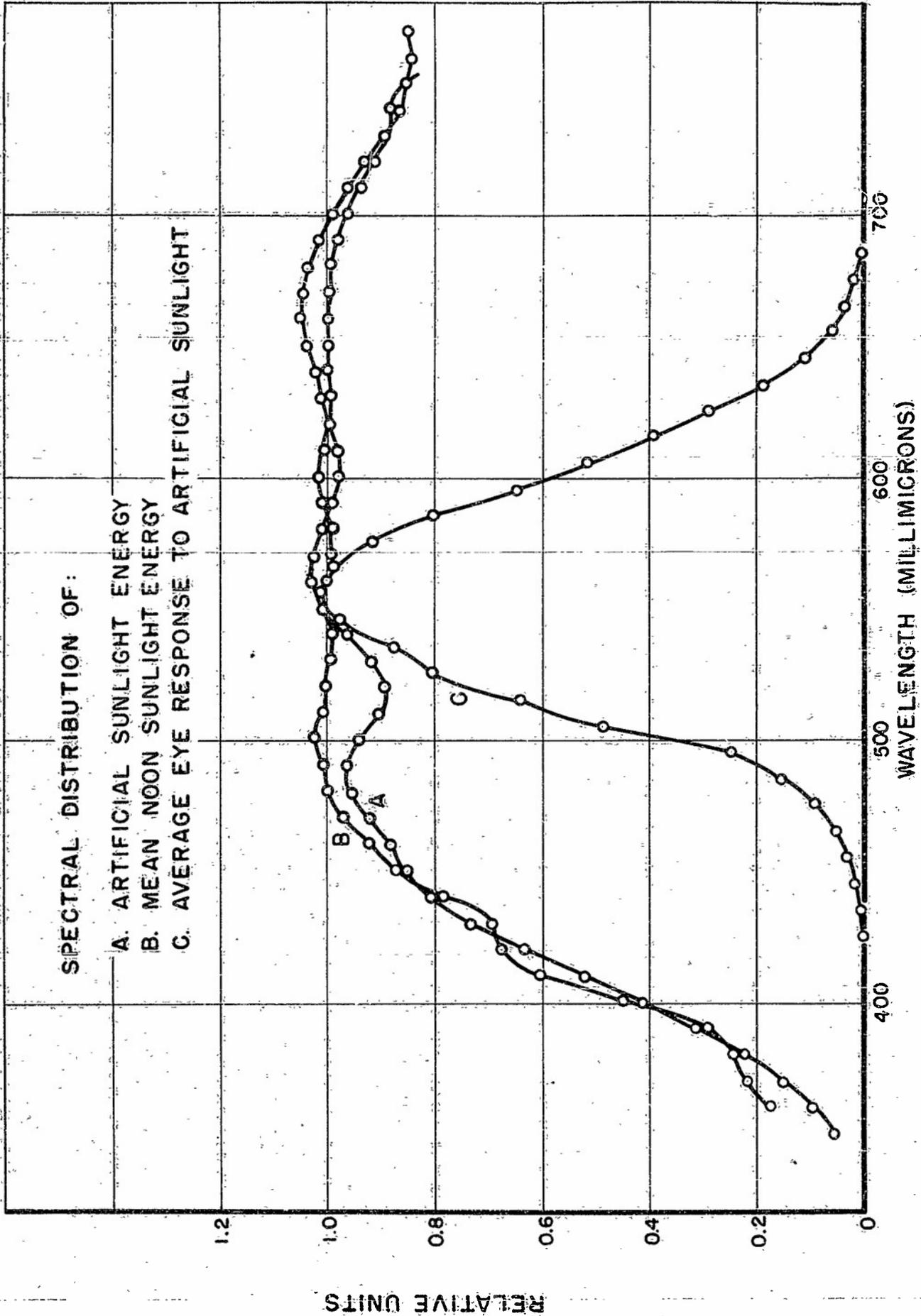


FIGURE 2. SPECTRAL - RADIATION CURVES

TABLE 4. TABULATION OF VALUES TO ACCOMPANY CALCULATION OF PHOTOELECTRIC EFFICIENCY OF SELENIUM ELECTROPHOTOGRAPHIC PLATES

Microwatts Per 10 Mu.* Zone	Relative Sensitivity of Human Eye	Microwatts Effective to Human Eye	Microwatts Per Mu.*	Wave Length of Light, Mu.*	Photons per Second
470	0.000	0	47.0	405	959×10^{11}
570	0.000	0	57.0	415	1190
680	0.006	4	68.0	425	1457
770	0.012	9	77.0	435	1690
830	0.025	21	83.0	445	1860
870	0.040	35	87.0	455	1990
905	0.065	59	90.5	465	2120
940	0.100	94	94.0	475	2250
960	0.160	154	96.0	485	2340
960	0.260	250	96.0	495	2390
925	0.420	388	92.5	505	2350
900	0.600	540	90.0	515	2330
900	0.780	702	90.0	525	2380
940	0.930	875	94.0	535	2530
990	0.990	980	99.0	545	2720
1024	1.000	1024	102.4	555	2860
1024	0.970	994	102.4	565	2920
1020	0.900	918	102.0	575	2950
1000	0.800	800	100.0	585	2940
982	0.660	648	98.2	595	2940
980	0.530	520	98.0	605	2980
986	0.400	394	98.6	615	3060

TABLE 4 - Continued

Microwatts Per 10 Mu.* Zone	Relative Sensitivity of Human Eye	Microwatts Effective to Human Eye	Microwatts Per Mu.*	Wave Length of Light, Mu.*	Photons per Second
1005	0.29	292	100.5	625	3160
1020	0.19	194	102.0	635	3260
1030	0.11	113	103.0	645	3350
1048	0.06	63	104.8	655	3450
1050	0.04	42	105.0	665	3520
1043	0.02	21	104.3	675	3550
1030	0.01	10	103.0	685	3550
1006	0.00	0	100.6	695	3520

* Millimicron

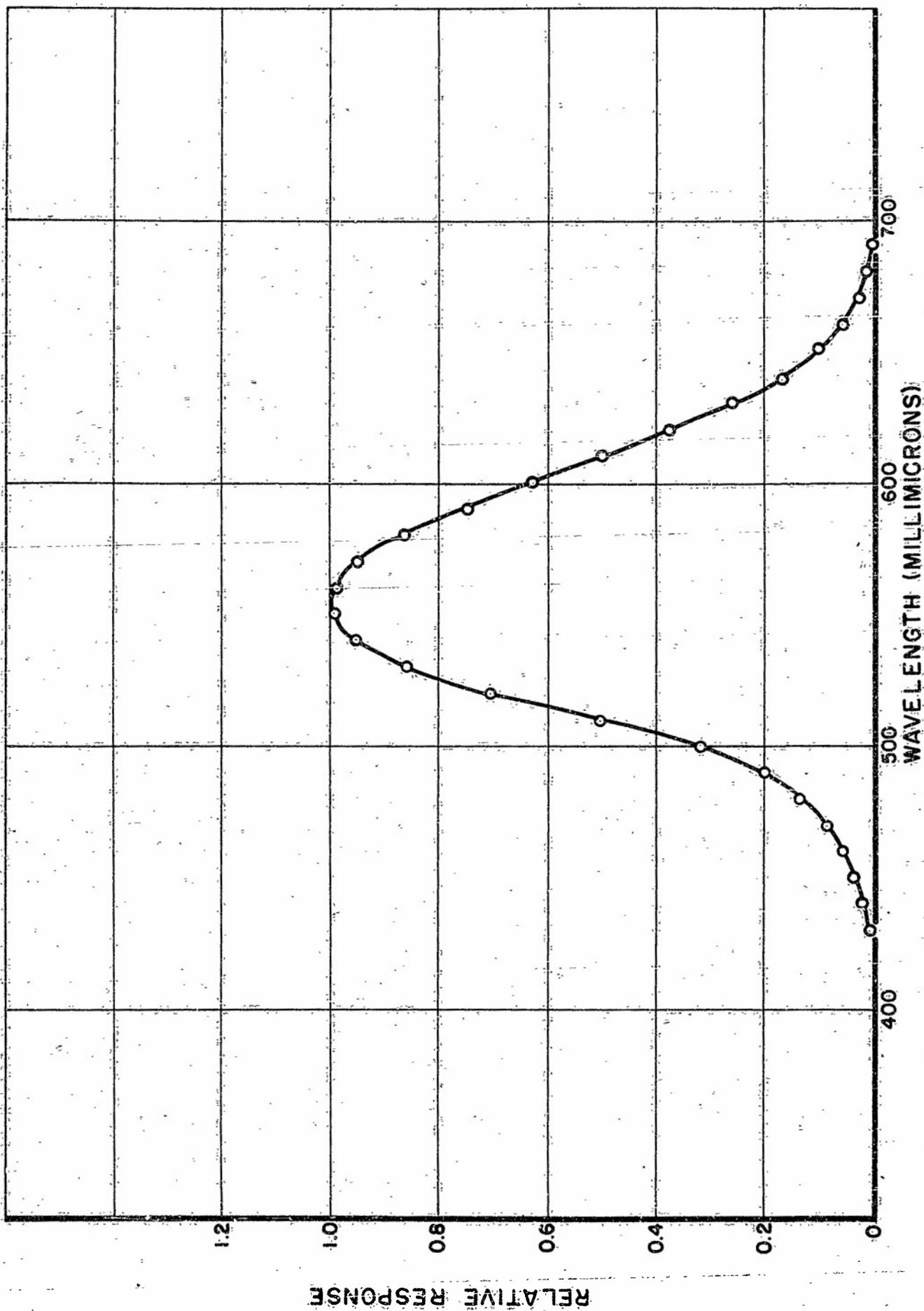


FIGURE 3. RESPONSE OF AVERAGE EYE TO EQUAL ENERGY SOURCE.

in Figure 2 to give curve C in Figure 2. This derived curve represents the amount of energy from the original source of radiation that would be effective in producing the sensation of light in the eye (the luminous intensity of the radiation). In Table 4, columns 2 and 3 represent a numerical integration of this function which results in the value of 10100 microwatts of energy available in visible radiation. The ratio $27900./10100. = 2.745$ represents the ratio of total to visible radiant energy.

The 0.02 M.C.S. assumed above for exposure of the plate is equivalent to 0.02 lumen per square meter or $(0.02) (0.1496) = 0.00295$ microwatt second per square centimeter of visible radiation, or to $(0.00295) (2.745) = 0.00822$ microwatt second per square centimeter of total radiation.

Figure 4 shows a conversion of the total radiation curve into photons per square centimeter per millimicron. This conversion involved the relationship:

$$e = h \nu$$

Where e = the energy per photon in ergs.

h = Planck's constant, 6.62×10^{-27} erg-seconds.

ν = The frequency of the light or the velocity of light divided by the wave length of the particular radiation being considered.

Table 4, column 6, shows this calculation which leads to the figure of $786,000 \times 10^{11}$ photons per second between 400 and 700 millimicrons as being equivalent to the 27,900 microwatts for the same wave band. The 0.02 M.C.S. assumed above for the exposure of the plate was found to be equivalent to 0.00822 microwatt second per square centimeter

PHOTONS PER SECOND PER SQUARE CENTIMETER PER MILLIMICRON

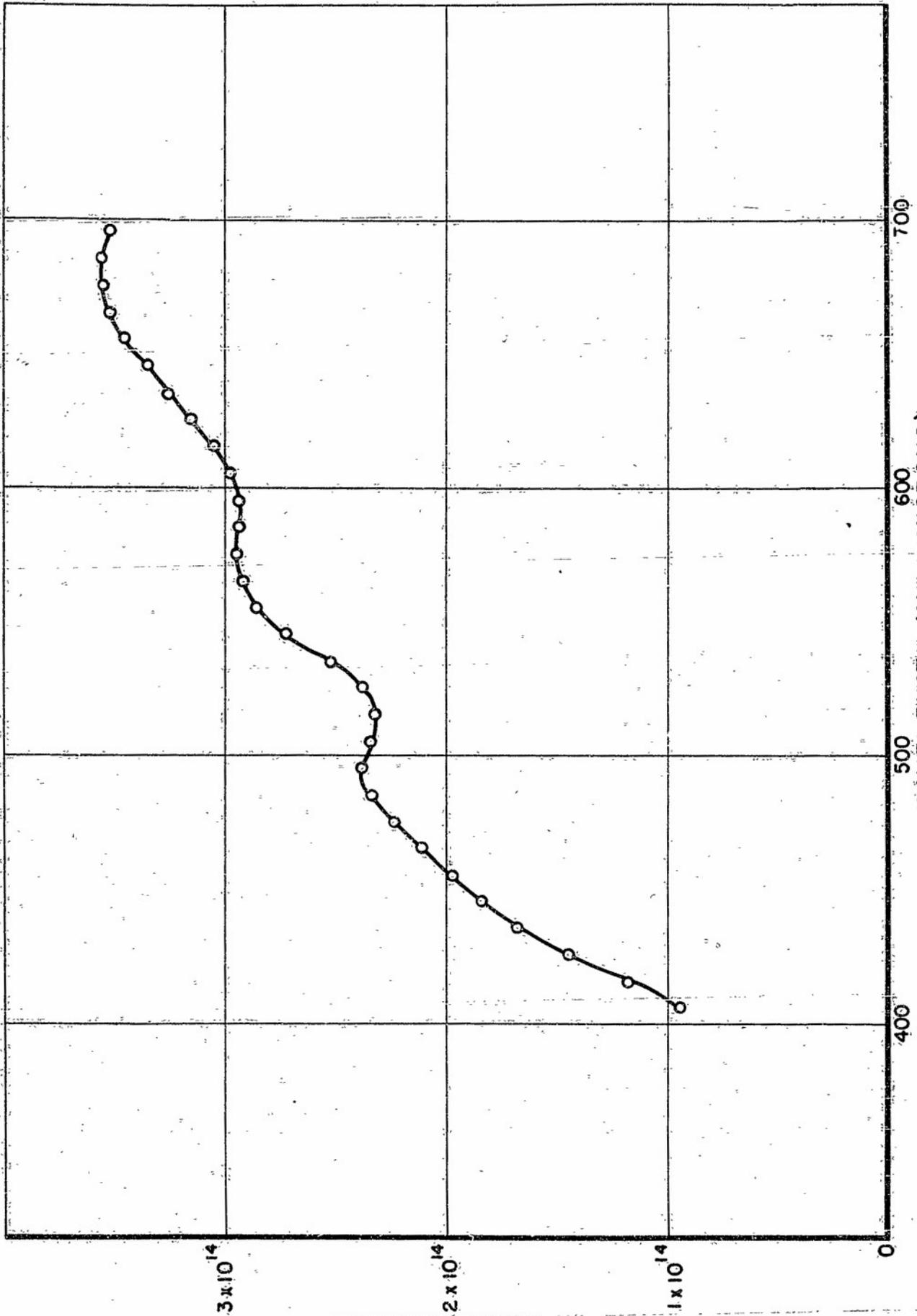


FIGURE 4. SPECTRAL DISTRIBUTION OF PHOTONS FOR ARTIFICIAL SUNLIGHT SOURCE OF AN ARBITRARY INTENSITY.

0-9394

of total radiation. The number of photons should then be proportional to the ratio of these two energies, and equal to $\frac{0.00822}{27,900} \times 785,000 \times 10^{11}$, or 0.0231×10^{12} .

It can now be seen that for this particular set of assumptions, the 0.02 meter-candle-second is equivalent to 0.00295 microwatt second per square centimeter of visible radiation, or to 0.00822 microwatt second per square centimeter of total radiation between 400 and 700 millimicrons, or to 0.0231×10^{12} photons per square centimeter. This represents, in various ways, the total energy or the total number of photons available to affect the electrophotographic plate.

On the basis of all the above assumptions, it may be calculated that the photoelectric efficiency of the electrophotographic plate (the ratio of the electrons released to the photons incident on the photoconducting layer) must be $(0.218 \times 10^{12} / 0.0231 \times 10^{12}) (100) = 940$ per cent, (9.4 electrons released per photon).

It has been estimated by very inaccurate methods that exposure speed of current selenium plates is 0.3 A.S.A. On the basis of the above calculation, such a speed would represent a photoelectric efficiency of about $940 \times 0.3/150 = 6.2$ per cent or that about 16 photons are required to liberate one electron.

If the above calculation is approximately correct, then it appears that photoconductivity effects involving cascade or secondary photoconductivity of electrons will be required to produce the desired plate sensitivities. It must be realized, however, that the present calculation is based on a number of assumptions, many of which will be subject to

change as research work proceeds on this project. The thickness and the dielectric constant of the photoconductive coating may be changed, and the potential drop required of the plate surface during exposure may be decreased by more effective developing techniques.

The present calculation was made to establish a method for later similar calculations and to provide preliminary information on the order of magnitude of the photoconductive effects that must be sought in later research work.

Preliminary Analysis of Photoconductivity
and a Crucial Experiment

by

A. E. Middleton, O. J. Mengali, K. Cochran,
K. Jackson, and B. Brand.

The Proposed Hypothesis

Data have been taken upon selenium plates prepared by evaporation at different temperatures and tested by use in making pictures, by measurement of maximum charge held on the surface, and by X-ray diffraction. In this series, plates prepared at lower temperatures showed no sign of crystal structure; but these made at intermediate temperatures showed maximum charge retention and hence superiority in making pictures, along with some evidence of crystal line structure.

A theory was proposed in conformity with these observations, in which it was postulated that crystals held rigidly in a vitreous insulating matrix were responsible for the electrophotographic phenomena. These

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crystals were assumed to be strained by imposition of an electric field, and to exhibit a piezoelectric charge.

Two quick checks on this proposal appeared to throw some doubt on its validity. In the first place, it seemed strange that plates showing no crystal structure should fail to hold as much charge as plates showing some crystalline structure, since the vitreous form of selenium is an excellent insulator and if the data should be found spurious, the single claim of the theory to validity would be case in doubt. In the second place, the sign of the charge imposed on the plate by charging needles is experimentally the same as that on the needles, in disagreement with the prediction of the theory.

In order to obtain a further test, it was proposed to imbed selenium, crystalline and again vitreous forms, in a matrix possessing itself no electrophotographic properties. If then crystals in the matrix should be found necessary to give a suitable plate, the theory would be strengthened.

An account of the experiment follows.

Preparation of Selenium

High-purity, Canadian Copper Refinery selenium, cast in sticks (6 x 1/2 x 1/2 inches) in the vitreous state, was (a) powdered as vitreous selenium, and (b) powdered after annealing 1-1/4 hours at 205°C. to completely crystallize the stick.

In both cases, powdering was accomplished by first crushing with a mortar and pestle and next milling in a one-gallon ball mill for a

continuous period of four hours, using one-inch-diameter porcelain balls. In addition, the powder was classified by means of a Federal Dust Classifier.*

Twenty-five-micron-diameter selenium particles were arbitrarily selected as (a) a reasonable upper limit on the size of particles for this application, and (b) a size which could be readily separated by the classifier. After the milling operation, four passes of the ground selenium through the classifier resulted in selecting particles with sizes less than 25 microns. Petrographic examination revealed the information on size. Specifically the vitreous selenium powder contained 92 per cent of less than 25-micron-diameter particles and the crystalline powder contained 100 per cent of less than 5-micron-diameter particles.

The two batches, one of vitreous and the other of crystalline powder, which contained less than 25-micron-diameter selenium particles were used in preparing selenium pigmented lacquers as described in the next section.

Preparation of Selenium-Pigmented Lacquers

Except for knowing that vitreous selenium has a very high resistance, the properties of vitreous selenium which should be matched by a substitute binder material are unknown. Therefore, the selection of lacquers to be tried was somewhat arbitrary. In the present work, the

* See Bulletin No. 25 distributed by Federal Pneumatic Systems, Inc., 127 N. Dearborn Street, Chicago, Illinois, for a description of the Federal Dust Classifier.

lacquers were selected on the basis of their dielectric strength* properties.

Three types of resins were chosen, representing high, medium, and low dielectric strengths. The resins and their dielectric strengths** are listed in Table 5.

Vitreous and crystalline selenium powders were dispersed in each of these resins. The pigment binder ratio was prescribed by the oil-absorption requirements of the selenium metal powders.*** Because it appeared this criterion should closely approximate the requirements for obtaining a separation of the selenium particles in the resulting paint films, the ratio of binder to selenium used was that which allowed the binder to just wet the selenium.

Table 6 lists the 6 types of selenium-pigmented lacquers which were made for application on metal bases.

Preparation of Selenium-Pigmented
Lacquer Films on Aluminum Bases

Films made from the paints in Table 6 were produced on freshly cleaned 4 x 7-inch aluminum panels, with a 0.008-inch draw-down blade.

To provide ample data for analysis purposes, several types of films were prepared as shown in Table 7.

* Dielectric strength, short time, 1/8-inch thickness, volts per mil.

** Published by Plastics Catalogue Corp., 122 E. 42nd Street, New York 17, New York.

*** Crystalline selenium metal powder had an oil-absorption value of 19.6, vitreous powder 13.95. These values were arrived at by calculating the volume of linseed oil required to just wet the selenium as determined by the Gardner-Coleman rub-out method.

TABLE 5. PROPERTIES OF RESINS USED AS BINDER MATERIAL

Resin	Dielectric Strength*
Shellac	200-600
Ethyl Cellulose (Ethocel)	400-600
Chlorinated Rubber (Parlon)	2300

* Dielectric Strength, short time, 1/8-inch thickness, volts per mil.

TABLE 6. INGREDIENTS OF SELENIUM-PIGMENTED LACQUERS

Ingredients	Quantity of Ingredients
Paint A	
Amorphous selenium	168.6 grams
Dry orange shellac	38.6 grams
Paco solvent	174.7 grams
Paint B	
Amorphous selenium	168.6 grams
Ethocel (10 centipoises standard)	39.6 grams
Toluene	214.8 grams
Paco solvent	63.2 grams
Paint C	
Amorphous selenium	161.7 grams
Parlon (10 centipoises)	53.6 grams
Toluene	168.2 grams
Solid Rezyl 859	23.1 grams
Paint D	
Crystalline selenium	224.7 grams
Orange shellac (dry)	54.0 grams
Paco solvent	237.3 grams
Paint E	
Crystalline selenium	224.7 grams
Ethocel (10 centipoises standard)	54.3 grams
Toluene	198.0 grams
Ethyl alcohol	49.9 grams
Paint F	
Crystalline selenium	224.7 grams
Parlon (10 centipoises)	77.4 grams
Toluene	165.0 grams
Solid Rezyl 869	26.7 grams

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TABLE 7. ANALYSIS OF FILMS PREPARED FROM
SELENIUM-PIGMENTED LACQUERS

Panel Number	Resin Used as Primer	Type of Binder	Paint Number	Type of Selenium
1	Shellac	Shellac	A	Amorphous
2	Ethocel	Ethocel	B	Amorphous
3	Parlon	Ethocel	B	Amorphous
4	Parlon	Parlon	C	Amorphous
5	Ethocel	Parlon	C	Amorphous
6	None	Shellac	A	Amorphous
7	None	Ethocel	B	Amorphous
8	None	Parlon	C	Amorphous
9	None	Shellac	D	Crystalline
10	None	Ethocel	E	Crystalline
11	None	Parlon	F	Crystalline
12	Shellac	None	-	-
13	Ethocel	None	-	-
14	Parlon	None	-	-

Control plates consisting of films of each of the resins in Table 5 were made. Plates painted with vitreous selenium powder in each of three binders were also made. In addition, a similar series was made for the crystalline selenium powders. Besides these types of plates, several combination plates painted with primers and selenium-pigmented lacquers were also made (in some of these plates primer and binder material were alike and in others they were unlike).

These plates were subjected to two types of tests as discussed in the next section.

Preliminary Testing of Selenium-Pigmented Lacquer Film

The tests used to roughly evaluate the above plates included a combined photoeffect and charging test and a physical composition test.

The techniques of testing for photoeffects and ability to accept a charge were those developed at Battelle in previous work on electrophotography. Each plate was charged in the dark by a moving row of needles, carrying a potential of 4500 volts. At this point in the test, the charge accepted by the plate was measured by a probe attached to an electrometer. The movement of the electrometer as a function of time gave a relative indication of the dark decay rate. On exposure of the plate to light the change in the rate of movement of the electrometer needle indicated whether a photoeffect had occurred. If the photoeffect was complete, subsequent removal of the plate resulted in no further shift of the electrometer needle. If, however, the photoeffect did not occur or was incomplete, subsequent removal of the plate caused the electrometer needle to shift owing to the residual plate charge.

Table 8 illustrates some types of qualitative data on the properties of each of the plates listed in Table 7.

It will be particularly noted that:

1. All plates with vitreous selenium in a parlon binder, regardless of whether a primer was applied to the base plate or not, showed a photoeffect; all other plates showed no observable photoeffect.

2. The vitreous selenium-pigmented ethocel did not take a good charge, but when a parlon binder and an ethocel base primer were used, photoeffects were more pronounced than when a parlon binder and a parlon primer were used.

3. The crystalline selenium-pigmented lacquers did not accept a significant amount of charge. It appeared that they were not acting as insulating films.

Because it was suspected, from the crystalline selenium data, that the binders were not providing adequate separation of the selenium particles, specimens of crystalline and vitreous selenium plates were cross-sectionally polished by metallographic methods. The patterns revealed in Figure 5 were observed. Note that the selenium, especially in the crystalline case, is not well separated.

Observations of Electrophotographic Properties of Painted Plates

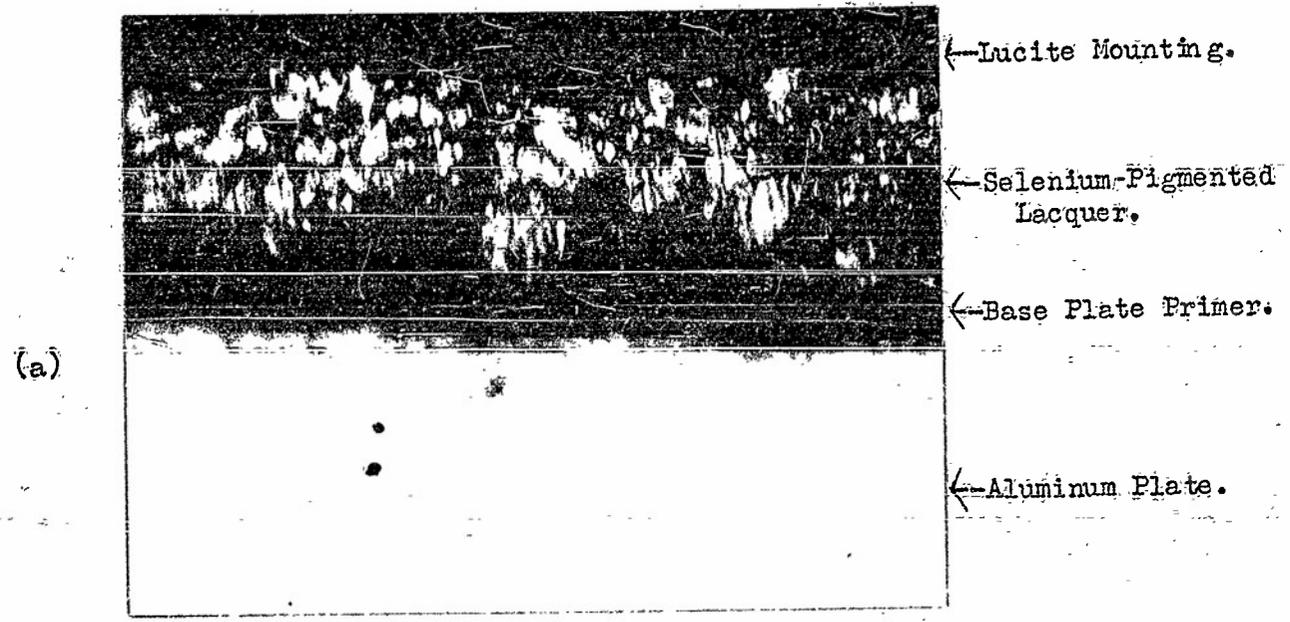
Beside the ability to retain a charge in the dark, a suitable electrophotographic plate must lose it in the light. Accordingly, plates of type C with vehicle parlon and primer ethocel were subjected to systematic tests as detailed later. Besides this, they were used to

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TABLE 8. PRELIMINARY DATA ON SELENIUM PIGMENTED
LACQUER COATED PLATES

Plate Number	Primer	Type of Paint	Initial Charge	Photoconductivity	Residual Charge (After Approx. 30-Sec. Light)	Dark Decay	Remarks
No. 1	Shellac	A	None	-	-	-	-
No. 2	Ethocel	B	None	-	-	-	-
No. 3	Parlon	B	None	-	-	-	-
No. 4	Parlon	C	Approx. 470 volts	Yes (slow)	Approx. 80 volts	Slow	-
No. 5	Ethocel	C	Approx. 240 volts	Yes (fast)	Approx. 140 volts	Slow	Chosen as best plate for photo work.
No. 6	None	A	Low	None	-	-	-
No. 7	None	B	Very low	None	-	-	-
No. 8	None	C	Approx. 230 volts	Yes	Approx. 130 volts	Slow	-
No. 9	None	D	Very low	None	-	-	-
No. 10	None	E	Very low	None	-	-	-
No. 11	None	F	Very low	None	-	-	-
No. 12	Shellac	None	Low	None	-	-	-
No. 13	Ethocel	None	Very low	None	-	-	-
No. 14	Parlon	None	High	None	-	-	-
No. 5	Ethocel	No. 3	Approx. 180 volts	Yes (quite slow)	Approx. 90 volts	Slow	Surface of Se. scraped with razor blade.

NOTE: All charges are positive.



Neg. No. 57529
 Crystalline Selenium Plate Cross Section (X1000).



Neg. No. 57528
 Vitreous Selenium Plate Cross Section (X1000).

Figure 5. Cross section of vitreous and crystalline pigmented parlon coatings on ethocel primers applied to aluminum bases. (White particles are selenium and the dark spaces are the binder.)

make electrophotographic prints on paper. By both tests, these painted plates were found to behave similarly to electrophotographic plates made by evaporating selenium on aluminum.

Conclusion

As indicated, a more detailed examination of the electrophotographic properties of the painted plates is reported below. A conclusion may be stated at once, however, regarding the subject of investigation. It was concluded that vitreous selenium is capable of exhibiting electrophotographic phenomena. It follows that the proposed theory requiring the presence of crystals is not valid in this case, and that any future theory must be adequate to describe photoconductivity in vitreous selenium.

It may be remarked at this point that although this conclusion is still valid, as far as experimental proof is concerned, it may require amendment as further data are obtained. Further examination of the selenium paint for reasons not involved above have recently revealed evidence of crystals in the paint. Whether these crystals are directly involved in the electrophotographic properties of the paint films has yet to be established, however.

Analysis of Decay-Rate Curves of
Selenium-Painted Plates

by

D. T. Williams, K. Cochran, and B. A. Kulp

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Summary

The best selenium-painted plates were compared as to charge decay under illumination with a vacuum-coated plate. It is tentatively concluded that whereas the latter showed an exponential decay curve, the former appear to decay exponentially only to a constant residual voltage. Sufficient uncertainty exists as to the precise manner of decay, to warrant some further thought.

Experimental Procedure

A Genco electrometer has been set up for routine examination of electrophotographic plates as they are prepared by evaporation. The plate is put into a grounded, darkened, light-tight box, and charged by an automatic traversing charging wire. It is then illuminated by a reasonably dim illumination, and its discharge is observed by use of an electrometer with probe fixed above the plate. The electrometer needle is initially grounded; its deflection proceeds linearly with the plate decay, and at length reaches a limiting value when the plate is finally removed. A calibration is made by replacing the electrophotographic plate by a similar metal plate so charged as to bring the electrometer needle back to 0.

The accuracy of the apparatus is not altogether above reproach; no careful measurement of the drift rate was made. However, no drift was observed as long as the maximum deflection of the light spot was less than 2 cm. With care, the reading could be made to $\pm .01$ cm.

Figures 6 to 11 are semi-log plots showing the data as obtained. The ordinates are the deflections of the electrometer needle, and the abscissae are times of illumination in minutes.

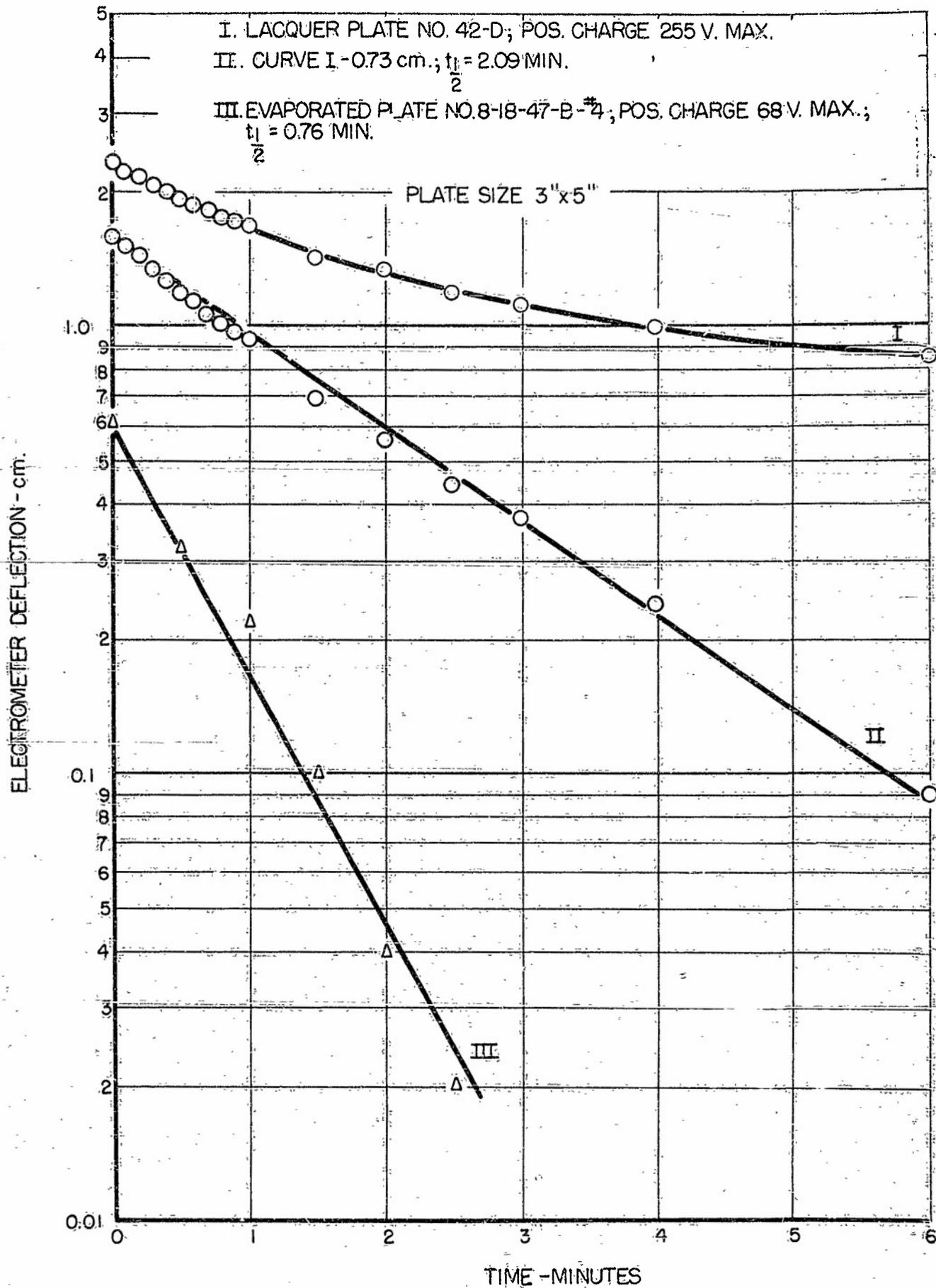


FIGURE 6. DISCHARGE WITH LIGHT SOURCE AS USED FOR STANDARD TESTS ON EVAPORATED PLATES

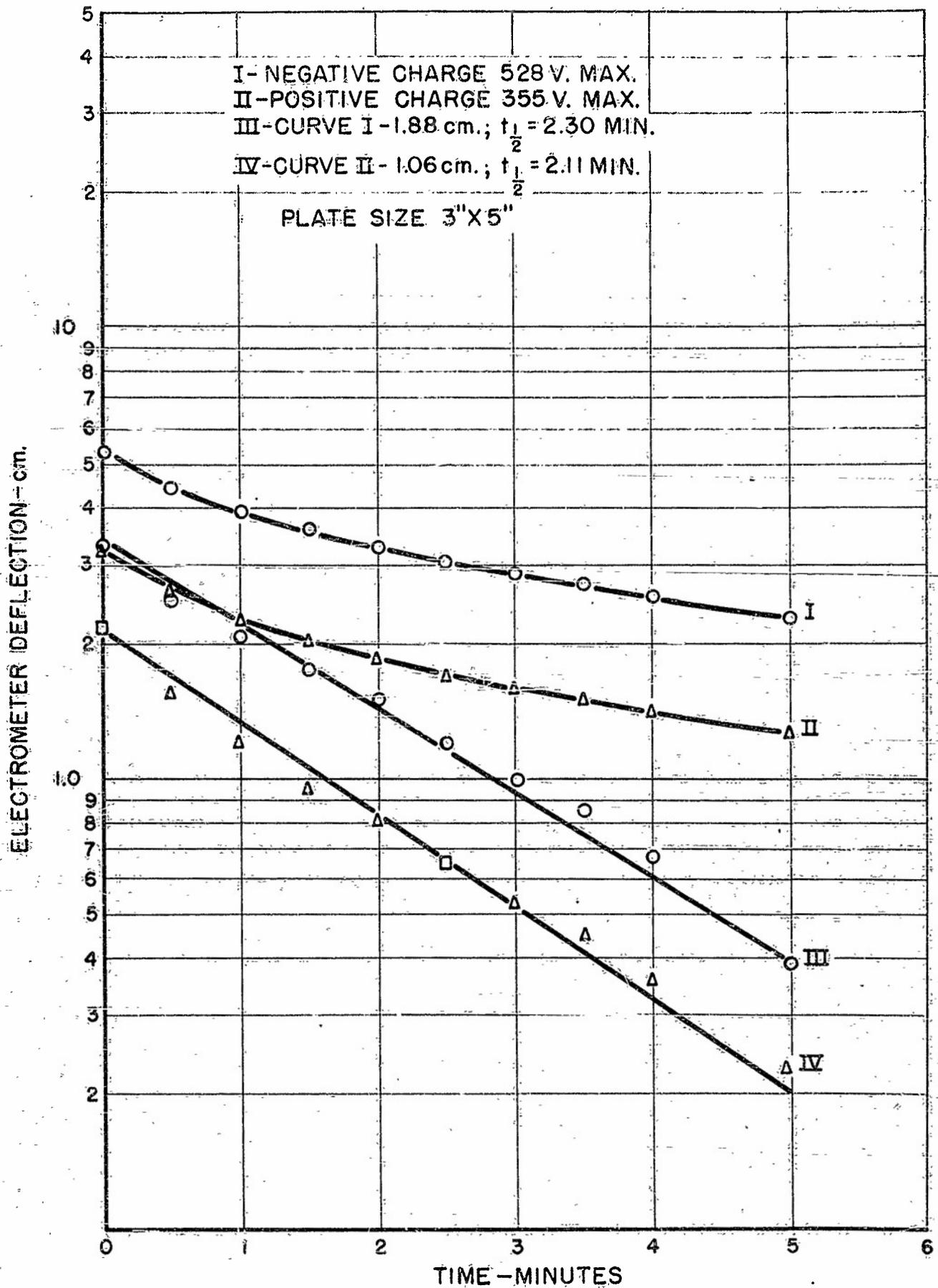


FIGURE 7. DISCHARGE WITH LIGHT SOURCE, AS USED FOR STANDARD TESTS ON EVAPORATED PLATES. LACQUER PLATE NO. 42-G; NO. 3A PAINT WITH LAB. SE.

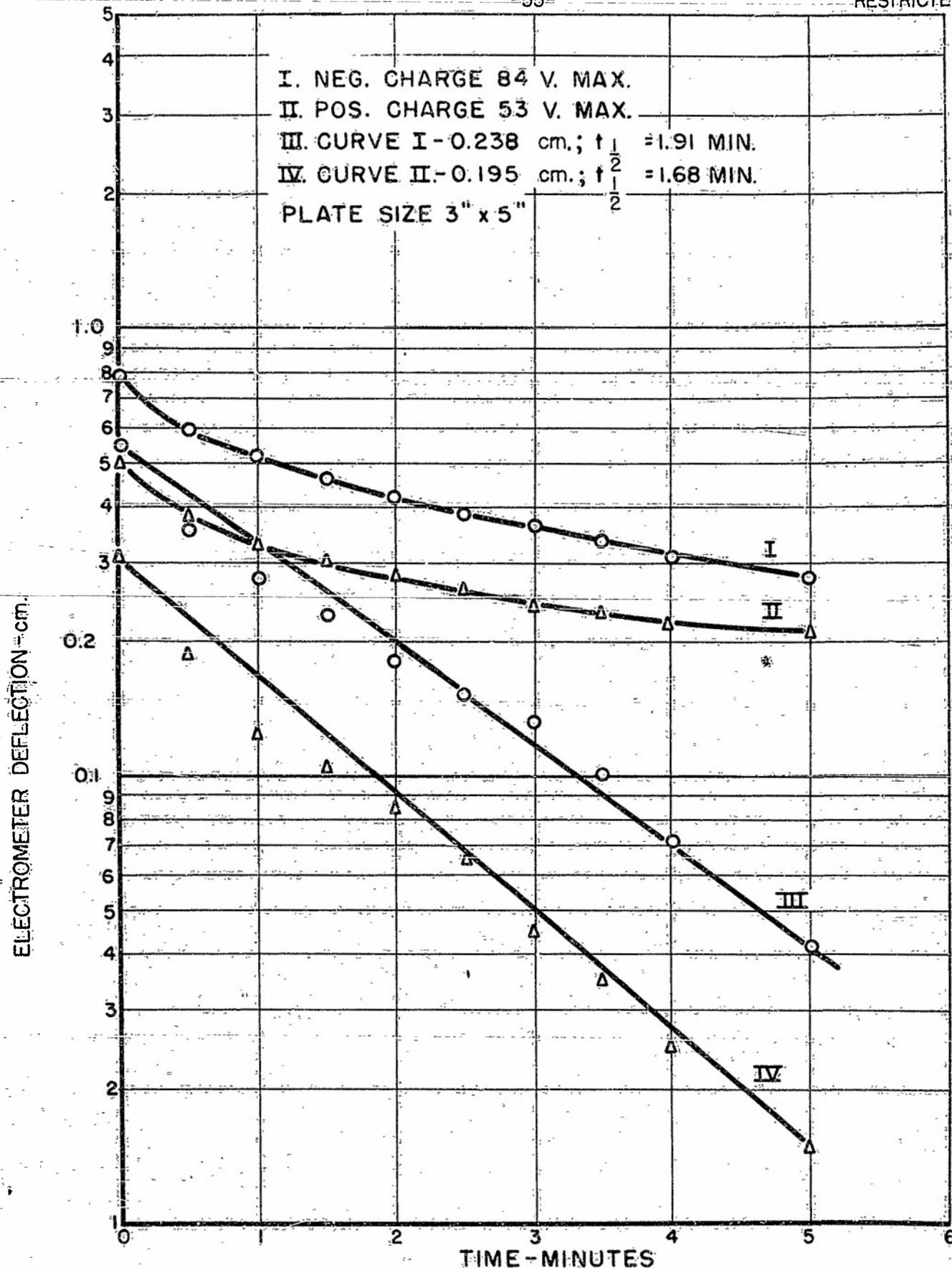


FIGURE 8. DISCHARGE WITH LIGHT SOURCE AS USED FOR STANDARD TESTS ON EVAPORATED PLATES. LACQUER PLATE NO. 3997-3A; NO. 3A PAINT WITH CGR POWDERED SE AND 120% EXTRA REZYL 869

0-9413

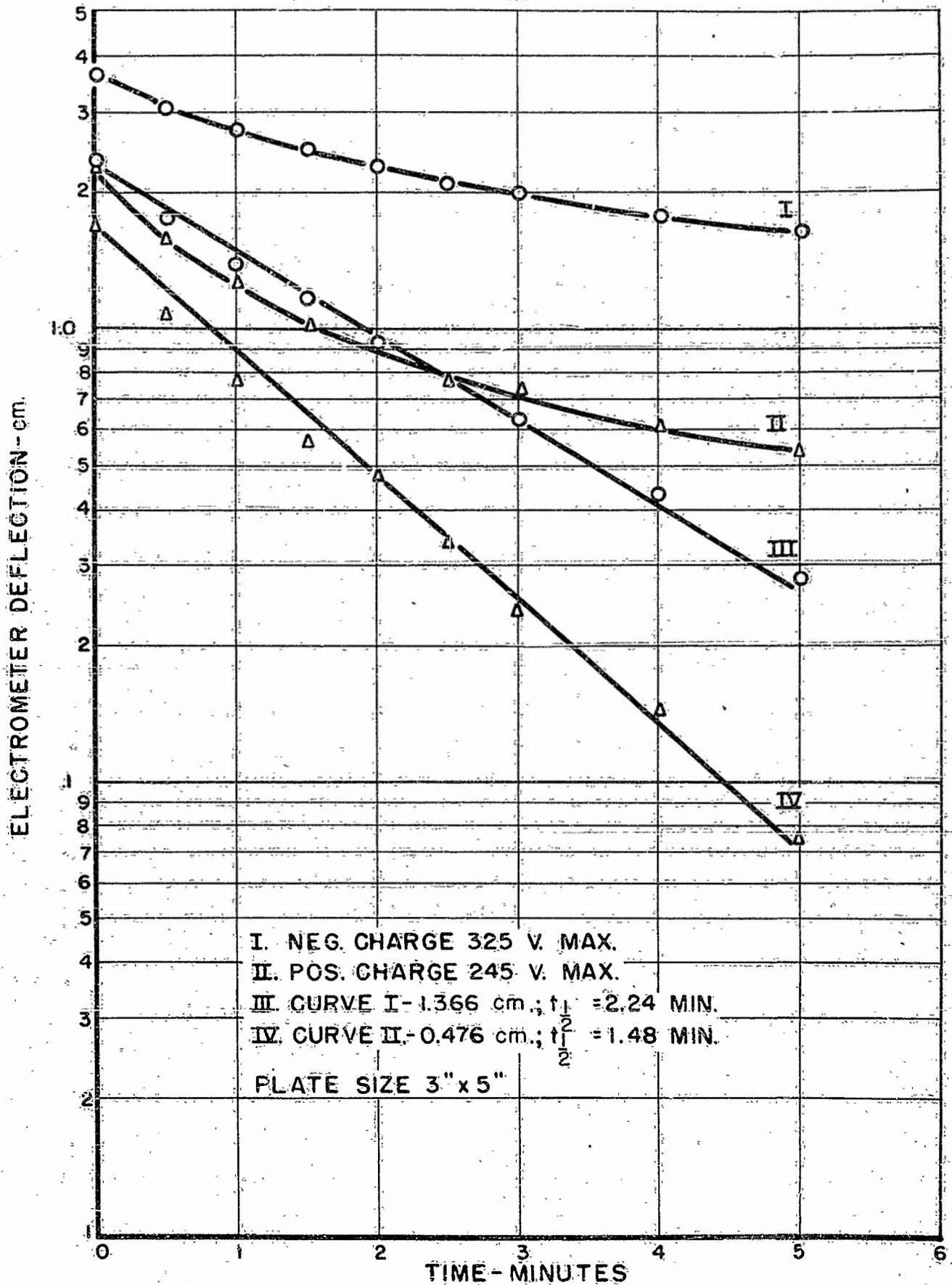


FIGURE 9. DISCHARGE WITH LIGHT SOURCE AS USED FOR STANDARD TESTS ON EVAPORATED PLATES. LACQUER PLATE NO. 3997-46; NO. 3A PAINT WITH CCR POWDERED SE.

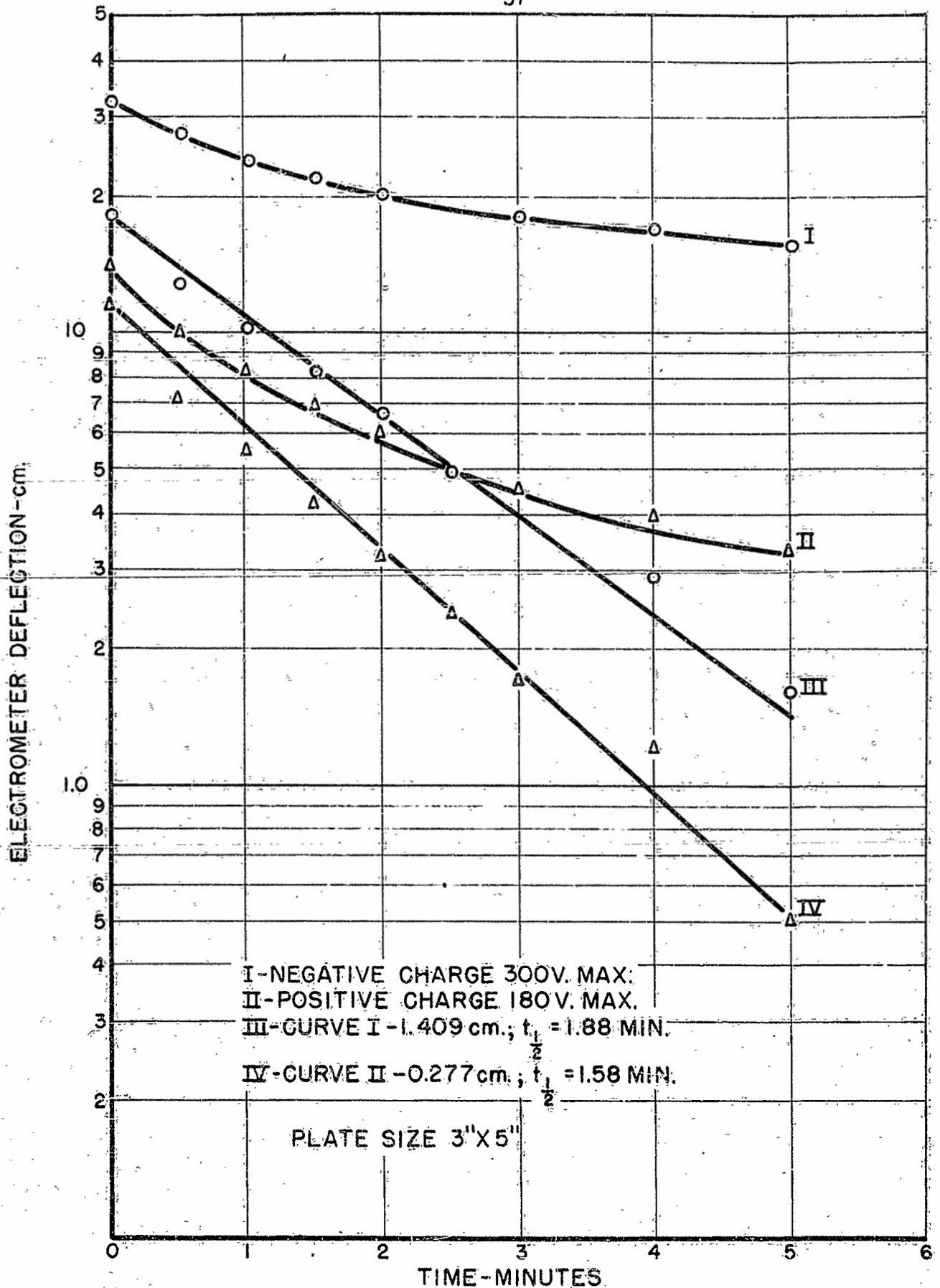


FIGURE 10. DISCHARGE WITH LIGHT SOURCE AS USED FOR STANDARD TESTS ON EVAPORATED PLATES. LACQUER PLATE NO. 3997-4H; NO. 3A PAINT WITH COR POWDERED SE.

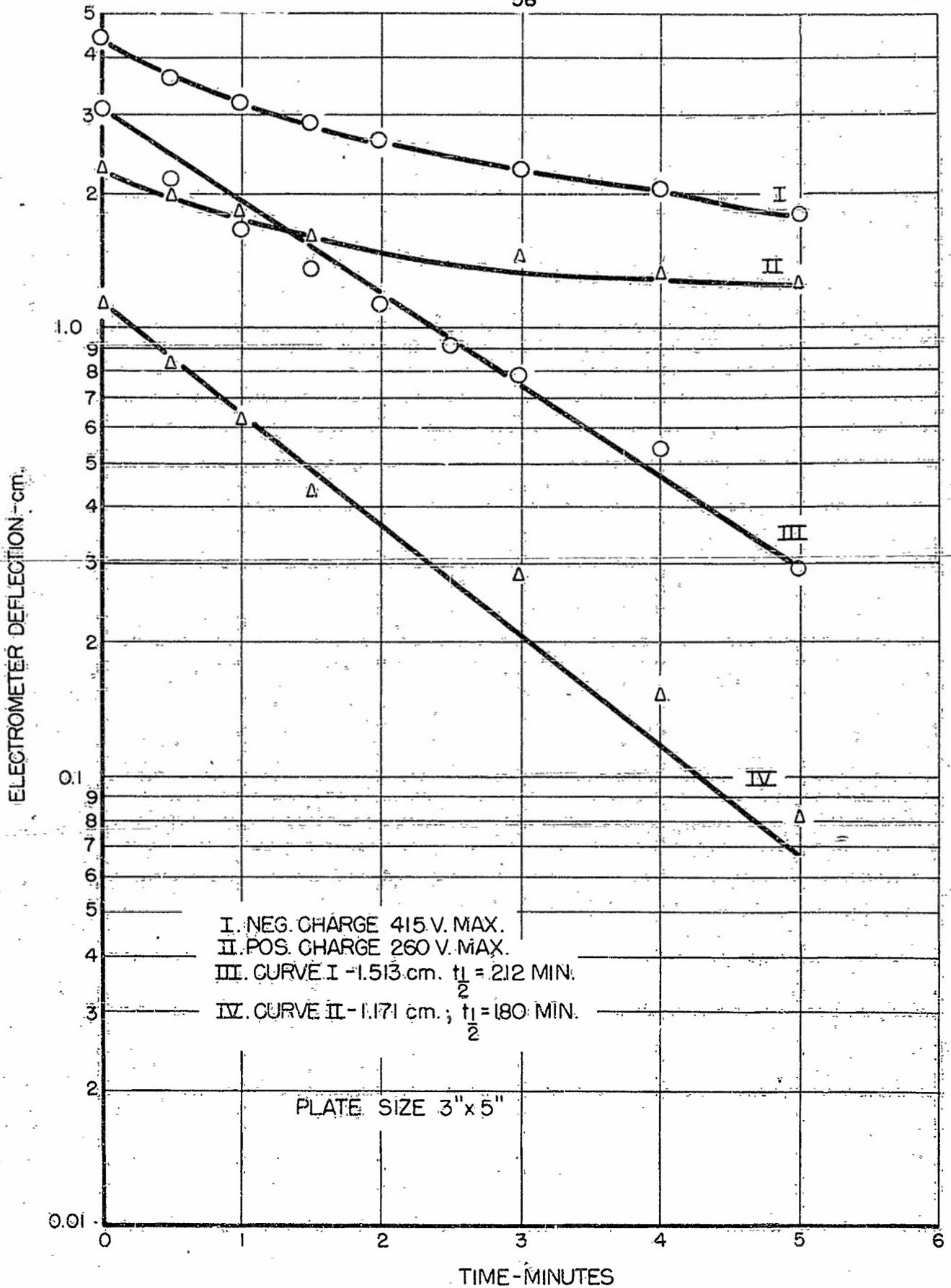


FIGURE 11. DISCHARGE WITH LIGHT SOURCE AS USED FOR STANDARD TESTS ON EVAPORATED PLATES. LACQUER PLATE NO. 3997-4I, NO. 3A PAINT WITH CCR, POWDERED SE

In Figure 6, curve III was obtained by use of an evaporated plate. Its straight-line shape indicates an exponential variation with time. Specifically, the plate had a voltage V given in volts by

$$V = 68 e^{-.481 t}$$

The time for decay to half the initial value is shown as $t_{1/2} = .76$ minutes, under the conditions of illumination of the experiment.

The form of decay curve here shown is not always obtained with such plates, and other types have been observed. However, it does have a fairly plausible explanation in terms of other facts known about electro-photography. It is proposed to show a theoretical rationalization of the exponential decay rate derived on the basis of assumptions that have been found convenient in previous research on photoconductivity.

Theory

Assume the charges on the surface of the selenium layer cause a potential V to exist across the selenium. This potential is related to the charge density q by the equation

$$VC = q \tag{1}$$

Here C is the capacitance per unit area of the selenium layer considered as the dielectric in a condenser. Then if the dielectric constant of the selenium is ϵ ,

$$C = \frac{\epsilon}{4\pi d} \tag{2}$$

or

$$V = \frac{4\pi q d}{\epsilon} \tag{3}$$

It is now assumed that proportional to illumination I of the selenium layer, a number n of charges are made mobile each second. That is

$$n = k_1 I \tag{4}$$

where k_1 is a constant. During the lifetime of each mobile charge, it is assumed to move a distance x toward the grounded backing plate. Then the voltage will decrease during illumination at a rate

$$-\frac{dV}{dt} = \frac{4\pi x k_1 I}{\epsilon} \tag{5}$$

In Hughes and Dubridge "Photoelectric Phenomena" (McGraw-Hill, New York, 1932), page 386, an assumed manner of variation of x is proposed as describing the photoconductivity of other materials that have been investigated.

$$x = \kappa F = \frac{hV}{d} \tag{6}$$

Here F is the force on a charge, equal to the ratio of potential to thickness of selenium.

With x as given by equation 6, equation 5 becomes

$$\frac{dV}{dt} = - \frac{4\pi k k_1 I}{\epsilon d} \tag{7}$$

or, if $x \equiv \frac{4\pi k k_1 I}{\epsilon d}$, by integration

$$V = V_0 e^{-xt} \tag{8}$$

This is the exponential form of decay as observed on the evaporated plate. A refinement of this type of derivation is described in the reference cited, in which the effect is analyzed of a plate thickness not large compared to x. This refinement appears not to be important in this case.

The Theory as Applied to Painted Plates

In Figures 6 toll it is apparent that the selenium-painted plates do not have an exponential form of decay curve like the evaporated plate. It seemed possible that the potential could, however, be resolved into a constant and exponential component. This is done very handily by use of the following analysis:

Assume

$$V = V_r + V_{10} e^{-\alpha_1 t} \quad (9)$$

where V_r is the constant residual voltage, α_1 and V_{10} are constants.

We choose three points with $t = t_0$, $t_0 + \Delta t$ and $t_0 + 2 \Delta t$, respectively.

We indicate ordinates corresponding to the three points by subscripts /a, /b, and /c. Then it is evident that

$$\log_n (V_a - V_r) = -\alpha_1 t_0 \quad (10)$$

$$\log_n (V_b - V_r) = -\alpha_1 (t_0 + \Delta t) \quad (11)$$

$$\log_n (V_c - V_r) = -\alpha_1 (t_0 + 2 \Delta t) \quad (12)$$

Then

$$\alpha_1 \Delta t \cdot \log_n \left(\frac{V_a - V_r}{V_b - V_r} \right) = \log_n \left(\frac{V_b - V_r}{V_c - V_r} \right) \quad (13)$$

It follows that

$$(V_b - V_r)^2 = (V_a - V_r)(V_c - V_r) \quad (14)$$

whence

$$V_r = \frac{V_a V_c - V_b^2}{V_a + V_c - 2V_b} \quad (15)$$

In this fashion the constant component V_r was determined as a constant residual deflection. For each plate, the assumption that the deflection is linear with potential being true to an estimated 10 per cent or less. As a test of the plausibility of this procedure, the data are replotted in the form $(V - V_r)$ as a function of t on semi-log paper. It is evident that the resulting best curves are not far from a straight line. It is concluded that the procedure indicates a true property of the plates, and that the decay is exponential to a constant residual voltage within present limits of accuracy.

Analysis of Trends on Painted Plates

There is a plausible rationalization of the conclusion reached just above, as far as painted plates are concerned. Suppose the photoconductive material be assumed to be pigment grains imbedded in an insulating vehicle matrix. Then if a charge is imposed on a layer of the paint, and it is exposed to light, there will be a redistribution of charge in the photoconductive pigment and possibly even a leakage of the charge to the backing plate if the vehicle is not present in too high concentration. This charge redistribution will presumably take place exponentially with time, but a residual charge will be left on the vehicle and this will not normally be entirely dissipated by the action of light.

If this analysis is correct, one would expect the slope of the semi-log plot of $(V - V_r)$ to be determined by the size and composition of the pigment particles, whereas the magnitudes of initial and final voltage would be fixed by the amount and nature of the vehicle, initial charging conditions, etc.

In the curves 5 to 8 are data to check this prediction. Among the plates tested there were two, 42D, and 42G made from a paint with a pigment made of selenium especially purified at Battelle, shown in Figures 6 and 7. There are two curves in all, taken with positive charge and one with negative charge. The time for decay to half initial potential - that is, half the variable component - is signified by $t_{\frac{1}{2}}$. The values are 2.09 and 2.11, and 2.30 ^{minutes,} respectively. These half times are the same within experimental error, and are equal for negative and positive plate charging.

Figures 9, 10, and 11 show results with plates all made with the same paint, in this case with pigment of selenium obtained from the Canadian Copper Refinery and used without further purification. The curves for negatively charged plates have half times of 2.24, 1.88, and 2.12 minutes; for positively charged plates, the half times are 1.48, 1.58, and 1.80 minutes. In Figure 8 is shown data from a plate with the same paint except for an increment of the material in the vehicle that is added for improving adhesion to metal. This paint shows half times of 1.91 and 1.68 for the negative and positive charges respectively. These figures support the conclusion that the decay rate, as expressed by α , depends on the pigment independent of the vehicle, since with the vehicle altered in such a manner that the maximum charge is reduced to a quarter of its original value, the decay rate is the same within observational error. To summarize the conclusions, the mean half times are $2.04 \pm .07$ minutes and $1.64 \pm .05$ minutes. The probable errors are calculated as the average deviation divided by the square root of the number

of plates. The values are such that in a new observation there is about an equal chance of the error being greater than the probable error shown, and less than that value. As a rough estimate, one anticipates about a 5 per cent deviation in successive observations of decay rate in identical painted plates.

The difference between the decay rate for positive and negative charge with CCR selenium appears to be greater than the probable experimental error, since the difference between the means is about 20% of the larger. It is interesting to note that the decay rate is smaller for the negatively charged plate, which also bears the higher maximum potential. A possible explanation is that the negative electrons are likely to penetrate further into the selenium than are positive ions. Hence, the potential will be higher; and since the light penetrates equally in both cases, but is supposedly less intense in the inner layers, the decay rate ought to be less with negative charge, as it appears to be. Accordingly, one predicts a higher decay rate for lower charging voltage.

It is noted that the two rates are equal for the single plate with lab-selenium pigment.

Conclusions

This experiment leads to the following conclusions:

1. The decay of charge on an electrophotographic plate follows the same physical laws as have been studied in previous experiments on photoconductivity. In this case, such decay is exponential with time.

2. The characteristics of a photoconductive material can be studied by use of a paint in which that material is the pigment, and in which the vehicle is an insulator.

3. Successive observations of the half time of decay for a given paint are expected to agree within about 5 per cent.

4. A plate prepared by evaporation and chosen at random had more than twice as high a decay rate as the painted plate.

Data for this work are recorded in Laboratory Record Books:

No. 3864, pages 1 - 28, inclusive.

No. 3871, pages 1 - 15, inclusive.

No. 3931, pages 1 - 20, inclusive.

No. 3956, pages 1 - 5, inclusive.

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