### UNCLASSIFIED

### AD NUMBER

### AD014092

### CLASSIFICATION CHANGES

TO:

unclassified

FROM:

restricted

### LIMITATION CHANGES

TO:

Approved for public release, distribution unlimited

### FROM:

Distribution authorized to U.S. Gov't. agencies and their contractors; Administrative/Operational Use; 31 DEC 1952. Other requests shall be referred to Army Signal Corps, Washington, DC.

### AUTHORITY

E.O. 10501 5 Nov 1953; USAEC ltr, 7 Jul 1972

### THIS PAGE IS UNCLASSIFIED

# Armed Services Technical Information Agency A'RLINGTON HALL STATION ARLINGTON 12 VIRGINIA

Rentu

FOR

MICRO-CARD

CONTROL ONLY

Classification cancelled in accordance with Executive Order 10501 issued 5 November 1953 8/28/3-9

> Document Service Center Armed Services Tech. Info Agency

SIFI

NOTICE: WHEN GOVERNMENT OR OTHER DRAWINGS, SPECIFICATIONS OR OTHER DATA ARE USED FOR ANY PURPOSE OTHER THAN IN CONNECTION WITH A DEFINITELY RELATED GOVERNMENT PROCUREMENT OPERATION, THE U.S. GOVERNMENT THEREBY INCURS NO RESPONSIBILITY, NOR ANY OBLIGATION WHATSOEVER; AND THE FACT THAT THE GOVERNMENT MAY HAVE FORMULATED, FURNISHED, OR IN ANY WAY SUPPLIED THE SAID DRAWINGS, SPECIFICATIONS, OR OTHER DATA IS NOT TO BE REGARDED BY IMPLICATION OR OTHERWISE AS IN ANY MANNER LICENSING THE HOLDER OR ANY OTHER PERSON OR CORPORATION, OR CONVEYING ANY RIGHTS OR PERMISSION TO MANUFACTURE, USE OR SELL ANY PATENTED INVENTION THAT MAY IN ANY WAY BE RELATED THERETO.

c = b

ū

RESTRICTED SECURITY INFORMATION

(Subcontract Under Signal Corps Prime Contract No. DA 36-039 sc-123)

(Department of the Army Project No. 3-99-04-052)

(Signal Corps Project No. 36-195B)

Tenth and Final Progress Report (January 1, 1952, through December 31, 1952)

to

#### THE HALOID COMPANY

on

CONTINUOUS-TONE ELECTROSTATIC ELECTROPHOTOGRAPHY

December 31, 1952

#### bу

W. E. Bixby, P. G. Andrus, O. A. Ullrich, L. E. Walkup, R. M. Schaffert, and W. T. Reid

OBJECTIVE OF RESEARCH: To evolve an electrostatic electrophotographic process capable of producing continuous-tone photographs of high quality.

This document contains information affecting the national defense of the United States within the meaning of the Espionage Act, 50 U. S. C., 31 and 32, as amended. Its transmission or the revelation of its contents in any manner to an unauthorized person is prohibited by law.

> BATTELLE MEMORIAL INSTITUTE 505 King Avenue Columbus 1, Ohio

# Best Available Copy

#### TABLE OF CONTENTS

	Page
	10-1
PURPOSE	10-2
PUBLICATIONS AND REPORTS	10-2
SUMMARY	10-2
Results	10-2
Illtrapure-Selenium Plate	10-3
Single-Laver Selenium-Tellurium Plates	10-3
Two-Laver Selenium-Tellurium Plates	10-3
Microscopic Examination of Electrophotographic Films	10-4
Sensitization of Selenium Tellutium Plates	10-4
Image Development	10-5
Radioactive-Sensitizing Device	10-5
Transfer Material	10-5
Panid-Processing Device	10-5
Report Frocessing Device	10-5
	100
EXPERIMENTAL WORK	10-6
Plates	10-6
Illtranure Selenium	10-7
Selenium-Tellurium Plates	10-10
One-Laver Selenium •Tellurium Plates	10-11
Two-Layer Selenium Tellurium Plates	10-18
Microscopic Examination of Electrophotographic Plates	10-94
Effect of Sensitization on Electrical Characteristics of Selenium-Tellurium Dister	10-28
Detecting Dister Drepared in 1959	10-23
Detecting Plates Prepared in 1051	10 33
Recessing rides richated in 1301,	10-31
Electropholographis Made with Single Layer Selemani Terrarian Plate Using	10-09
Standard Darkoom Procedure	10-38
Rapid Processing Electropholographic Camera,	10-40
Designs Considered for Rapid-Processing Camera	10-40
Description of Rapid-Processing Camera	10-44
Operation of the Rapid Processing Camera	10-44
Results of Tests With the Rapid-Processing Camera	10-46
Sensitization of Electrophotographic Plates,	10-46
Conventional Corona Unit ,	10-46
Radioactive Sensitizing Unit	, 10-49
Radium	10-49
Beta Source,	10-52
<b>Radium-D</b>	10-52
Polonium	10-52
Image Development on Electrophotographic Plates	10-58
Sensitometry	10-60
Special Gray Scale	. 10-61
Schedule for Processing Electrophotographs	10-61
First Results	10-65
Nonuniformity of Development	10-67
Triboelectric Charging of Powder Clouds	10-71
BATTELLE MEMORIAL INSTITUTE	

## RESTRICTED

### LIST OF TABLES (Continued)

9

		rage
Table 10-4.	Effect of Powder-Cloud Density and Specific Electrical Charge on Time Required to Develop an Electrophotographic Image	10-113
	LIST OF FIGURES	
Figure 10-1.	Electrical Characteristics of Ultrapure $^{-}$ and ARQ-Scienium Plates $.$	10-9
Figure 10-2.	Characteristics of Good Sclenium Place and a Single-Layer Sclenium-Tellurium Plate	10-12
Figure 10-3.	Electrical Characteristics of Four Plates Made of 93 Per Cent Selenium and 7 Per Cent Tellurium	10-14
Figure 10-4.	Spectral Sensitivity of Two-Layer Selenium-Tellurium Plates Having Various Concentrations of Tellurium in Top Layer	10-20
Figure 10-5.	Variation of Panchromaticity With Tellurium Concentration in Top Layer of Two- Layer Plates	10-22
Figure 10-6.	Photomicrographs of Selenium Chip Removed From Electrophotographic Plate	10-29
Figure 10-7.	Change in Dark-Decay Characteristics of One-Layer Selenium-Tellurium Plate R-4-25-52-A With Successive Chargings	10-31
Figure 10-8.	Effect of Different Grid Potentials and Charging Times on Dark-Decay Characteristics of One-Layer Selenium-Tellurium Plate 7-11-52-A	10 <b>-32</b>
Figure 10-9,	Characteristics of One-Layer Selenium-Tellurium Plate R-4-25-52-A for Different Charging Conditions	10 <b>-3</b> 5
Figure 10-10.	Characteristics of One-Layer Selenium-Tellurium Plate R-4-16-52-A for Different Charging Conditions	10-36
<b>Fig</b> ure 10-11.	Prints Made Using Single-Layer Selenium-Tellurium Plate R-5-16-52-A and a Selenium Plate	10-41
Figure 10-12.	Designs Considered for Rapid-Processing Electrophotographic Camera.	10-43
Figure 10-13.	Rapid-Processing Electrophotographic Camera	10-45
Figure 10-14.	Electrophotograph Made on Two-Layer Selenium-Tellurium Plate 11-4-52-C at Approximately ASA 30, Daylight	10-47
Figure 10-15.	Sectional View of Plate-Sensitizing Unit Using a Radioactive Ion Source	10-51
Figure 10-16.	Ion Current Flowing Between Polonium Source and Metal Plate at Various Spacings Between Source and Plate	10-54
Figure 10-17.	Potential Acquired by Selenium Plate Exposed for Various Times to Polonium Source With 300 Volts Potential Difference Between Source and Plate	10-55
	RESTRICTED	•

### LIST OF FIGURES

### (Continued)

		Page
Figure 10-39.	Multitube Electrophotographic Powder-Cloud Development Device	10-109
Figure 10-40,	Photograph of Electrophotograph Produced Using the Eight-Tube Powder-Cloud Development Device	10-111
Figure 10-41.	Modified Powder-Cloud Mixing Chamber Used on High-Speed Development Unit	10-115
Figure 10-42.	Device for Measuring Force Required to Transfer Powder Images to Adhesive ~ Coated Paper	10-120
Figure 10-43.	Comparative Adhesive Transfers of Electrophotographic Images to Dye-Transfer Paper and to a Coated Kromekote Paper	10-121

S

f

10-2

#### PURPOSE

The general objective of this project has been to evolve a process of electrophotography suitable for producing high-quality continuous-tone photographs. A detailed outline of the initial objectives of this investigation is given on page 1-2 of this series of reports. These objectives specify an electrophotographic process to produce photographs equivalent in quality and appearance to photographs made by more conventional processes, but without the limitations of such processes.

The chief aims of the work on this project for the calendar year 1952 were: (1) to work out the steps of a process of electrophotography in which the plate can be carried from sensitization through development in a total elapsed time of from five to ten seconds; (2) to consolidate the process for making selenium electrophotographic plates containing seven per cent tellurium; and (3) to study the sensitometry of electrophotography so as to make the most of the process in faithful rendition of the densities found in the original scene being photographed.

#### PUBLICATIONS AND REPORTS

On July 30, 1952, a demonstration of the current status of electrophotography was given at the Signal Corps Engineering Laboratories, Fort Monmouth, New Jersey, for interested personnel of the Squier Signal Laboratory. The regular informal monthly letter reports, minutes of the meetings held with the Sponsor's representatives, quarterly progress reports, and this final progress report constitute the remainder of the publications and reports made by the Battelle staff on continuous-tone electrophotography during the calendar year 1952.

#### SUMMARY

#### Results

Major advances have been made on several of the problems considered this year, and some progress has been made on all of them. Electrophotographic plates developed this year have greater sensitivities than ever before, having speeds by daylight approximating ASA 20 to 30. In addition,

ESTRICTED

INSTITUTE

### **ESTRESTR**

10-

plates are more panchromatic than he stofored the response of some plates extending into the red end of the visible spectrum. The time required to develop a plate is nowilled than one second, as opposed to the 10 to 15 seconds required at the beginning of this year. The large and bulky power supply required with prevalence has been eliminated entirely by using a radioactive sensitizing of the second of these and other advances is given below.

the phatesens:

Cristics When the count

Ultrapure-Seleniura Plate.

A large number of plates have been made by vacuum deposition of mixtures of selenium and tellurium as single layers on brass plates. These selenium-tellurium plates have shown a wide, and so far unexplained, variation in electrical characteristics. Some plates had a panchromatic response and the high photographic speed desired in electrophotographic plates; the majority did not have these properties, or had such high residual potentials or high rates of dark decay that they would not be practical in a camera.

Tellurium Plates

Considerable effort was expended in attempting to identify the cause of this variability in electrical characteristics from one plate to the next. Attempts were made to improve the control of plate-preparation conditions in the vacuum system; many of the preparation conditions were varied in an effort to map the effects of these variables and to determine which must be closely controlled to get reproducible results; and studies were made of various plate-sensitization techniques in order to reduce or eliminate variations due to different sensitization techniques. None of these provided an answer to variations in the electrical characteristics of single-layer selenium-tellurium plates.

#### **Two-Layer Selenium-Tellurium Plates**

One of the important results obtained on this project has been the development of two-layer selenium-tellurium electrophotographic plates having essentially panchromatic response, at a photographic speed as great as 15 times that of the selenium plates used previously. These twolayer plates are superior to single-layer selenium-tellurium plates in that

RESTRICTED

INSTITUTE

BATTELLE MEMORIAL

10 - 4

they have lower rates of dark decay, lower residual potentials, and can be reproduced more consistently than one-layer plates.

Plates made with a foundation layer of about 40 microns of selenium, and covered with two to four microns of a mixture of 80 per cent selenium and 20 per cent tellurium, show photographic speeds up to 15 times that of selenium plates; in addition, their response extends well into the red portion of the spectrum. These two-layer plates are less affected by small changes in preparation conditions than are single-layer selenium-tellurium plates. Actually, they can be produced almost as consistently as selenium plates, but still greater reproducibility is needed to assure commercial success. Two-layer plates generally show lower dark-decay rates and lower residual potentials than one-layer selenium-tellurium plates of comparable speeds and spectral response.

Good images have been produced on these two-layer seleniumtellurium plates, the plates being significantly more sensitive to daylight than is the case with selenium plates. Equally important, the quality of the images produced on the selenium-tellurium plates appears to be inherently as good as those obtained on selenium plates.

### Microscopic Examination of Electrophotographic Films

Microscopic examination of selenium films taken from electrophotographic plates showed crystals in the brass-selenium interface of red-sensitive selenium plates, but no apparent crystals in non-redsensitive plates. However, crystallinity did not always accompany panchromatic response in selenium-tellurium plates as it did red sensitivity in selenium plates.

#### Sensitization of Selenium-Tellurium Plates

The electrical characteristics of selenium-tellurium plates are more susceptible to variations in sensitization methods than they are in selenium plates. In general, the electrical characteristics of seleniumtellurium plates improve with repeated charging or when high-current sensitizing conditions are used. This effect is particularly noticeable in the dark decay of plates which, when charged with low corona current, may be rapid enough to obscure the light sensitivity of the plate but which may be reduced by charging with high corona current so that accurate measurements of light sensitivity can be made.

#### 10 - 5

#### Image Development

Another important accomplishment has been a notable reduction in the time required to develop an electrophotographic plate. Images of high quality can be produced now with a development time of less than one second. Results are reproducible and can be obtained on portable equipment. In addition, a device was built which, by logical extension, can be used to develop much larger plates than could be handled with previous equipment.

#### Radioactive-Sensitizing Device

Still another important result of this project is the development of a new type of sensitizing apparatus well suited to both laboratory and field use. A small radioactive source is used to create ions in the air above the electrophotographic plate. These ions are then driven onto the plate by an electrical field, producing the necessary electrical potential on the plate. No heavy, high-voltage power supply is necessary, the electrical field being supplied by a few hundred volts of miniature "B" batteries. Plates can be sensitized in approximately 15 seconds.

#### Transfer Material

A material currently undergoing test appears to be a suitable medium to which electrophotographic images can be transferred. In the laboratory, transfers of high quality have been made to this coated paper which requires no moistening prior to use, and, although slightly tacky, does not require interleaving. This paper material is relatively inexpensive, and probably can be supplied in several different weights and types.

#### Rapid-Processing Device

BATTELLE

A device has been constructed which can process an electrophotographic plate from the sensitization step through exposure and development in less than three seconds. This device has permitted taking photographs on selenium-tellurium plates having fast dark-decay rates.

#### Recommendations for Future Work

Further work on electrophotographic plates should be directed mainly to consolidating the position these plates have now attained. Although electrophotographic plates have not reached either the speed or the spectral response of the best photographic films, so that they are not entirely

RESTRICTED

MEMORIAL INSTITUTE

10-6

suitable as yet for aerial photography, they could be used successfully for most other applications where silver halide emulsions are commonly used. For the immediate future, work on electrophotographic plates probably should be limited to learning how to produce such plates consistently. When it has been demonstrated that this is possible, then further consideration might be given to a plate program leading to the development of electrophotographic plates having a speed and a spectral response appreciably better than the best of those available today. Such a program could be expected to be both expensive and time consuming.

It is recommended that further work be done with radioactivesensitizing units to determine how they can best be used, and to establish precautions for handling them safely.

Attention should be given to devices which might simplify the processing of selenium-tellurium plates. Also, further work is needed to gain an understanding of some of the many problems involved in development of electrophotographic images using powder-cloud techniques. Investigation of other developer materials may result in images of better quality or of more pleasing appearance.

That full advantage may be taken of some of the unique characteristics of the electrophotographic process, the sensitometry of electrophotography needs to be studied in greater detail. Such factors as being able to choose the gamma of the plate for any given exposure condition might give electrophotography a considerable advantage over other photographic processes.

The transfer material developed during 1952 gives good results, but it is recommended that further attention be given to the problem of transfer in the belief that further improvement can be made.

#### EXPERIMENTAL WORK

#### Plates

The aim of work during the past year on electrophotographic plates has been to devise a method for consistently producing highly sensitive, panchromatic electrophotographic plates capable of yielding good quality pictures in a rapid-processing electrophotographic camera. This aim has been reached to the extent that, by following a prescribed vacuumdeposition procedure, it is now possible to produce, almost every time, plates that show the desired characteristics. Such plates, made from mixtures of selenium and tellurium, have 10 to 15 times the photographic

MEMORIAL

RESTRICTED

INSTITUTE

BATTELLE

10-7

speed of selenium plates in daylight, are essentially panchromatic in their response to light, and have dark-decay rates and residual potentials low enough to permit use in a rapid-processing camera devised as a part of this project.

These plates reproduce fine detail and as wide a range of tones as do selenium plates. However, they are more sensitive than selenium plates to variations in sensitization procedure, and to repeated use and other fatigue factors. Accordingly, they are, at present, more difficult to use; additional work will be required to eliminate these effects.

Most of the data on plates in this section were obtained after the plate had been sensitized in a "scorona" unit designed to give relatively rapid charging of the plate. This arrangement consisted of a single coronadischarge wire, 0.0035 inch in diameter and four inches long, located 11/16 inch above the surface of the plate. The corona wire was raised to a positive potential high enough that the corona current was 100 microamperes at about 7000 volts. One-half inch above the corona wire was located a metal shield 1.5 inches wide and held at the potential of the potential-control grid. The potential-control grid consisted of 16 wires, 0.10 inch in diameter, spaced 0.1 inch apart, located 3/16 inch above the plate, and connected to a positive potential between zero and 500 volts, depending on the value of initial potential required on the plate. Sensitization took five seconds, during which the entire unit was moved by hand back and forth across the plate.

#### Ultrapure Selenium

The study of the effect of impurity materials on the characteristics of selenium plates began in 1951 with determinations of the effect of: (1) removing as many as possible of the impurities present in the ARQ(1)selenium used at that time for preparing plates, and (2) adding controlled amounts of impurities to the selenium. The results of work on the second approach were reported in the Final Progress Report dated December 31, 1951, but at the time that report was written, the preparation of the ultrapure selenium had been completed but no plate had been prepared from it.

A plate was made from the ultrapure selenium late in 1951. The electrical properties of this plate were practically identical to those of plates made with ARQ-brand selenium.

Ultrapure selenium used in the above tests was prepared by forming selenium hydride from ARQ selenium, vacuum distilling the hydride

(1) ARQ-brand, C. P., Shotted Selenium, Canadian Copper Refiners Limited, Montreal East, Canada.

BATTELLE MEMORIAL INSTITUTE

#### 10-8

several times, and then thermally decomposing the hydride into selenium and hydrogen, the hydrogen being removed from the evacuated apparatus through a palladium wall. Spectrographic analysis of the resulting selenium showed no lines for any other elements. About ten grams--enough for preparing one plate--was obtained.

Precautions were taken to avoid contaminating the selenium during preparation of the place. The sample was handled in an evacuated, sealed Pyrex tube which was nicked for easy opening inside an evacuated bell jar. A special heater was prepared to fit the Pyrex tube, and the selenium was evaporated directly from it, thus avoiding contact with any other material. The proper electrical input to the heater was established by making several evaporations of ARQ selenium from tubes similar to the one that contained the ultrapure selenium. After the operating conditions were established for evaporating from this source at the desired rate, these conditions also were used to prepare plates from ARQ selenium for direct comparison with the plate made from the ultrapure selenium.

Evaporation of the ultrapure selenium, and of the ARQ selenium in the trial runs which preceded it, extended over a 90-minute deposition period. An additional one-hour period was used to cool the plates to room temperature. During evaporation, the brass backing plates were mounted against a temperature-controlled platen maintained at 79 C plus or minus one degree. Under these conditions, selenium layers approximately 42 microns thick were obtained.

Figure 10-1 shows the characteristics of Plate R-12-31-51-A made from the ultrapure selenium, and the characteristics of Plate R-10-24-51-A made from ARQ selenium under the same conditions. These plates were measured under the low-current sensitizing conditions used in earlier work. In <u>A</u>, Figure 10-1, the sensitivity is given as the reciprocal of the time in seconds for the potential on the plate to decay from 200 to 100 volts when the plate is exposed to light of specific wavelengths at an intensity of about 0.030 microwatt per square centimeter, or the equivalent. (1)

<sup>(1)</sup> Measurements were not made by finding the time for the potential actually to fall from 200 volts to 100 volts while the plate was illuminated with the given intensity of light. When this procedure was attempted, the times involved varied greatly for different colors of light, and the relatively rapid decay of the potential in darkness introduced an unknown error in the results. Therefore, the procedure was changed by using a constant rate of decay for the potential so that the effects of decay in darkness would be more nearly equal in all cases. In this procedure, the intensity of the light was adjusted to decay the plate from 200 to 100 volts in exactly five seconds. Then, assuming the reciprocity law holds, the five seconds was multiplied by the ratio of the intensity found in the above experiment to the intensity in terms of which the results are given, 0.030 microwatt per square centimeter. The reciprocal of this number was taken as the sensitivity of the plate.



FIGURE ID-1. ELECTRICAL CHARACTERISTICS OF ULTRAPURE-

in the

A-4330

#### 10 - 10

<u>B</u>, Figure 10-1, shows potential-decay curves for these two plates. These two figures show measurable differences in the potential-decay characteristics of plates made from the two samples of selenium; however, these differences do not alter the spectral characteristics of plates to an extent important to the practical objectives of achieving panchromatic response in plates.

Potential-decay studies for the plate made from ultrapure selenium revealed that the residual potential remaining after exposure of the plate to red light is lower than the residual potential remaining after exposure to blue or to green light. This is contrary to the usual characteristics shown by plates made either with ARQ selenium or with ARQ selenium containing various impurities. Though of little practical importance, this difference may indicate a different type of trapping or space-charge build-up in the two materials -- possibly a trapping of negative carriers in the ARQ selenium and positive carriers in the ultrapure selenium.

It is concluded that removing impurities present in ARQ selenium does not produce any changes of practical significance in the electrical characteristics of plates made from this material.

#### Selenium-Tellurium Plates

BATTELLE

Electrophotographic plates made with a film of vitreous selenium on a metal backing plate ordinarily are sensitive only to blue light. Some o sensitivity to red light can be introduced in such plates by forming crystals of metallic selenium in the vitreous matrix, but the red sensitivity obtained in this way has never exceeded about 20 per cent of the blue-light sensitivity.

Adding tellurium to the vitreous selenium produces profound changes in the characteristics of the plates. Depending on the amount of tellurium added, and on other plate-preparation conditions, the absolute value of the sensitivity to blue light may be increased three- or fourfold, and sensitivity to green, yellow, and red light may be increased from near zero for selenium to values as great as or greater than the sensitivity of selenium plates to blue light.

That the presence of tellurium in selenium electrophotographic plates could enhance their sensitivity and spectral response was demonstrated by Dr. Paul Keck of the Signal Corps Engineering Laboratories in 1951. Since then, most of the work at Battelle on electrophotographic plates has been aimed at determining how to make the best possible plate using mixtures of selenium and tellurium, and how to produce such a plate consistently.

MEMORIAL

TRICTED

INSTITUTE

### 10-11

Two major difficulties have been encountered in this study: (1) slight, or at least unobserved, variations in the techniques of preparing the plate cause large changes in plate characteristics, and (2) the way in which the plates are used or tested greatly affects the outcome of the test. Thus, techniques that would produce consistently good scienium plates will not produce consistently good scienium-tellurium plates, and sensitization -procedures satisfactory for use with scienium plates may not produce the high light-sensitivity obtainable with scienium-tellurium plates when special high-current sensitization procedures are used.

<u>One-Layer Selenium-Tellurium Plates</u>. At the beginning of this year, many selenium-tellurium plates had been made under a variety of plate-preparation conditions. These conditions included various concentrations of tellurium in the selenium film, various temperatures of the backing plate during deposition of the photosensitive film, and various deposition rates. In addition, several types of laminated or layered plates had been prepared. Of all the plates made, the one showing the highest photographic speed and substantially better spectral response than any other was a plate made from a mixture of 93 per cent selenium and 7 per cent tellurium deposited in 90 minutes on a brass plate maintained at 79 C.

Figure 10-2 shows the characteristics of that single-layer seleniumtellurium plate compared with a selenium plate. The selenium-tellurium plate is about ten times faster photographically than the selenium plate; it has a speed of about ASA 25. Although the dark decay of the seleniumtellurium plate is high, and its residual potential is appreciably greater than for the selenium plate, neither is particularly objectionable.

Because this plate was far superior to other plates made in 1951, work in 1952 was started with attempts to prepare additional panchromatic plates using preparation conditions similar to those that produced this superior plate. The first work was an attempt to reproduce the desirable characteristics of the one plate in four additional plates prepared under the same conditions.

1. 1.

> Table 10-1 lists the preparation conditions for the four plates. The apparatus was the same as that used to prepare the earlier panchromatic plate. In it, the brass plate to be coated with the mixture of selenium and tellurium was fastened against a copper platen backed with copper tubing through which water at a constant temperature was passed. The temperature of the brass backing plate, clamped to the platen with spring clips, was measured with an iron-constantan thermocouple connected to a Leeds and Northrup portable potentiometer.

The source used for evaporating the selenium-tellurium mixture consisted of a porcelain crucible placed six inches below the plate. The

BATTELLE MEMORIAL INSTITUTE

ù

( . .



FIGURE 10-2. CHARACTERISTICS OF GOOD SELENIUM PLATE AND A SINGLE-LAYER SELENIUM-TELLURIUM PLATE

#### 10-13

TABLE 10-1.	PREPARATION CONDITIONS FOR FOUR PLATES M	ADE
	WITH 93 PER CENT SELENIUM AND 7 PER CENT	_
	TELLURIUM	0

Plate	Deposition period, minutes	Backing- plate temperatures, C	Coating thickness, microns
R-12-12-51-A	80	80.0	38
R-12-14-51-A	80	80.0	42
R-12-17-51-A	83	79.0	37
R-12-18-51-A	84	80.5	38

crucible was filled with nine grams of the premelted mixture of 93 per cent ARQ selenium and 7 per cent chemically pure tellurium. The rate of evaporation was controlled by adjusting the power input to the ceramiccovered heating element in which the crucible was placed. The source temperature was measured with a thermocouple placed between the crucible and the heater. Under these conditions, a film about 40 microns thick was obtained.

The selenium-tellurium mixture, designated as Batch 4, was premelted in a Pyrex distillation flask which had been sealed from the atmosphere but had not been evacuated. In this flask, a 200-gram charge was heated and thoroughly mixed for one hour at approximately 500 C.

Figure 10-3 shows the spectral sensitivities of these plates, and their potential-decay curves when exposed to light of 400-millimicron wavelength. Only a part of the spectral sensitivity curve for Plate R-12-18-15-A is shown because of the obscuring effect of high residual potential. Sensitivity measurements could not be made on Plate R-12-17-51-A because of its high dark-decay rate. Dark-decay halftimes for the four plates were as follows: R-12-12-51-A, 29 seconds; R-12-14-51-A, 92 seconds; R-12-17-51-A, 5 seconds; and R-12-18-51-A, 31 seconds. These plates were tested using the older, low-current sensitizing conditions.

The variation in the dark-decay halftimes and the residual potentials was evidence that the conditions of plate preparation were not being well controlled. Although this conclusion is probably correct, work during the rest of the year, directed in large measure in attempting to isolate and control the variables involved, has not greatly improved the reproducibility of the one-layer selenium-tellurium plates. This work is described in detail in the Seventh, Eight, and Ninth Quarterly Progress Reports on this project. The results are summarized here.

BATTELLE MEMORIAL INSTITUTE



**B** Potential Decay at Wavelength of 400 Millimicrons

FIGURE 10-3. ELECTRICAL CHARACTERISTICS OF FOUR PLATES MADE OF 93 PER CENT SELENIUM AND 7 PER CENT TELLURIUM

7. N.

A-4332

water and the second second and the

5.

#### 10-15

One of the variables that might be controlled more closely is the degree of uniformity of the selenium-tellurium mixture used in preparing the plates. Originally, this mixture was prepared simply by mixing the selenium and tellurium in an open crucible heated over a hot plate. This procedure was refined by carrying out the mixing in a sealed, and later in an evacuated flask, and by extending the mixing time to as much as eight hours, during which time the temperature of the flask was maintained at a temperature higher than the melting points of both selenium and tellurium. These precautions did not yield a noticeable improvement in the uniformity of the plates produced.

The control and measurement of the temperature of the brass backing plate and of the evaporator were improved by making the following changes:

- In one vacuum system, a water-contact platen was installed, in which the temperature-controlled water flowed directly against the back of the plate, so that thermal contact was good. The temperature of the plate was considered to be the temperature of the water in the platen chamber.
- (2) In another vacuum system, use was made of the conventional platen in which temperature-controlled water circulated through a copper block to which the backing plate was clamped. The temperature of the plate was measured carefully by pressing a thermocouple against the back of the plate. The hot junction of this thermocouple was immersed in vacuum grease that touched only the plate and not the platen.
- (3) The constant-temperature water supply was improved by using immersion heaters with a low thermal capacity, so connected to a thermostat that only a small fraction of the heater current was controlled by the thermostat.
- (4) The temperatures were measured using calibrated copperconstantan thermocouples and a Leeds and Northrup semiprecision potentiometer, an arrangement easily capable of reading temperatures to closer than 0.1 C.
- (5) The evaporator was changed from the porcelain crucible used originally to a sheet-molybdenum boat shaped to have about the same form as the porcelain crucible. The molybdenum boat was heated simply by passing current through it. Its temperature could be controlled much more easily and rapidly than that of the porcelain crucible heated partly by contact and partly by radiation. The temperature of the boat was measured using a Chromel-Alumel thermocouple either

STRICI

INSTITUTE

### 10 - 16

welded to the boat or immersed in the melt in a thin walled, Pyrex capillary tube. The thermocouple was connected to a potentiometer recorder.

These improved methods of controlling and measuring temperatures of the backing plate and the evaporation sources did not appreciably improve uniformity of the electrical characteristics of the seleniumtellurium plates produced.

Q.

As with any vacuum-deposition process, there are a number of platepreparation conditions that are more difficult to control than simple variables like temperature. For instance, the true gas pressure in the vacuum chamber, or the presence of condensable vapors in the chamber, cannot be determined easily. Other conditions which may be difficult to control are the treatment given the surface of the backing plate before it is placed in the vacuum system, and the time the plate is in the vacuum system before a sufficiently low pressure is reached to begin evaporation. The possibility that such variables might be the cause of uncontrolled electrical characteristics has been checked in some cases by changing a part of the treatment radically and then observing changes in electrical characteristics. No correlation of this type was observed.

Finally, variables such as deposition rate, postevaporation heat treatment, and backing-plate temperature, all of which had been investigated previously, were rechecked. Although not enough work was done to establish definitely the importance of these variables in determining the electrical characteristics of selenium-tellurium plates, they now appear to be of less import than the concentration of tellurium in the plate, or, more particularly, the tellurium concentration in the portion of the selenium film near the exposed surface of the film.

Work on deposition rate involved two approaches; first, establishing a means for measuring and controlling the rate of deposition; and second, determining the effect on panchromaticity of high rates of deposition of the selenium-tellurium mixture.

To measure and control the rate of deposition, an auxiliary device was installed in the vacuum system to indicate the rate at which the selenium-tellurium mixture evaporated. This device consists of an arrangement for advancing a special metal plate past an opening in a shield exposed to the selenium vapor. Mounted to one side of the regular metal plate being coated, and advanced once every ten minutes during evaporation, the special plate becomes covered with spots of selenium. The thicknesses of these spots are a measure of the amount of selenium-tellurium mixture deposited during the successive ten-minute intervals, providing a means for calculating the rate of deposition.

BATTELLE MEMORJAL INSTITUTE

### 10-17

Twelve plates were made with the temperature of the boat maintained at 260 C. At this temperature, nine grams of the selenium-tellurium mixture evaporated in about 80 minutes. The backing plate was held at 80 C, and one hour was used in cooling the plate to room temperature. The resultant films were about 47 microns thick.

Twelve plates produced using this arrangement show that maintaining the crucible at a constant temperature during successive runs will not insure a constant rate of deposition of the selenium-tellurium mixture. The deposition rate varied as much as 20 per cent from one plate to the next.

Although most of the electrical characteristics of the plates made during this test varied considerably, the residual potentials of all of the plates were 30 volts or less. Dark-decay rates tended to increase with increasing panchromatic response, as expected. The long-wavelength cutoffs varied from 600 to 700 millimicrons, and the maximum sensitivities varied from 0.3 to 0.9.

For investigating the effect of high rates of deposition on the panchromaticity of selenium-tellurium plates, the backing plates were maintained at temperatures between 82 C and 83 C and all of the seleniumtellurium mixture was evaporated from the molybdenum boat in 15 to 20 minutes. Plates removed soon after the completion of evaporation had long-wavelength cutoffs at about 600 millimicrons. Plates given a heat treatment by leaving them on the platen maintained at 82 C to 83 C for as long as 30 minutes after completion of the evaporation cycle, then cooling them to room temperature within two minutes, showed a slight increase in panchromaticity; the cutoffs of these plates were about 650 millimicrons.

Greatest variations from plate to plate were observed in the darkdecay rates and in the residual potentials. Variations in dark-decay rates seemed to be completely random, but variation in residual potentials seemed to occur in cycles. Thus, residual potentials would be low for a week followed by several weeks when all plates made would have high residual potentials.

During part of the last work on single-layer selenium-tellurium plates, residual potentials were as high as 800 volts. These plates had a correspondingly low sensitivity to light. However, when these plates were charged to a negative potential, they did not exhibit this same high residual, but they did show a relatively high panchromatic response to light. Both dark-decay rates and resid<sup>a</sup> potentials changed over a period of time, the more usual effect being for the dark-decay to become slower, and the residual potential lower. Such changes were most noticeable in the first few weeks after the plate was made.

BATTELLE MEMORIAL INSTITUTE

#### 10-18

In general, as long as variation occurs in the electrical characteristics of plates made under presumably identical conditions, the true role of the plate-preparation variables can be determined only after much work. In a number of past exper: nents, all preparation conditions but one were held as constant as pose  $d_{ij}$  and the variation in electrical characteristics of the plate were attributed to that one varied condition. The significance of the conclusions drawn from such experiments is questionable, because the observed variation in plate characteristics could have occurred even if no preparation condition were intentionally varied. For this reason, much of the work in the past has been empirical, in hopes of establishing practical conditions for preparing plates whose electrical characteristics would be closely reproduced from one plate to the next, rather than following a more systematic study of the dependence of electrical characteristics on each recognized variable in making plates. For the same reason, only the following conclusions are tenable at the present time:

- 1. Plates made by evaporating mixtures of 90 to 95 per cent selenium and 10 to 5 per cent tellurium onto brass plates maintained at temperatures in the range of 70 C to 80 C, generally show an increased blue sensitivity and an extended spectral response compared with ARQ-selenium plates.
- 2. Single-layer selenium-tellurium plates may have a photographic speed ranging from two to 15 times faster than plates made with ARQ-grade selenium alone. Figure 10-2 compared a typical single-layer plate containing tellurium with a plate made with ARQ selenium. In this case, the selenium-tellurium plate was ten times faster photographically than the selenium plate.
- 3. The dependence of the electrical characteristics of singlelayer selenium-tellurium plates on plate-preparation conditions is not clear. A yet unidentified and inadequately controlled variable has obscured the results of experiments designed to determine this dependence. There are indications that the concentration of the tellurium is the most important variable, with temperature of the backing plate, rate of deposition of the mixture, and postevaporation heat treatment being less significant.

<u>Two-Layer Selenium-Tellurium Plates</u>. Work on two-layer seleniumtellurium plates has been considerably more encouraging than work on onelayer plates. Two-layer plates have been made having 17 times the sensitivity of an ARQ-selenium plate, being roughly equivalent to an ASA speed of 40 to 50, daylight. Two-layer plates have consistently lower

BATTELLE MEMORIAL INSTITUTE

e 👟 i tan in the state of the

1

#### 10-19

residual potentials than one-layer plates, and can be made more consistently with desirable characteristics.

Two-layer plates are formed by first depositing a layer of selenium on a brass backing plate, and then depositing a layer of a mixture of selenium and tellurium. The two layers may be deposited at different temperatures of the backing plate and at different deposition rates. Usually, the under layer of selenium accounts for the bulk of the film, the top layer of the selenium-tellurium mixture accounting for 20 per cent or less of the total film thickness.

Two-layer plates have a number of possible advantages. For example, if residual potentials indicate space charge resulting from a short range of carriers in selenium-tellurium films, then substituting selenium, in which carriers have long ranges, for the bulk of the seleniumtellurium layer should reduce the residual potential. Or, if the dark decay is the result of thermally produced carriers throughout the bulk of the selenium-tellurium layer, then reducing that bulk by substituting selenium should reduce dark-decay rates. Recent work on two-layer plates indicates that these advantages can be realized, at least in part.

Work carried out recently on the preparation of two-layer plates used the same molybdenum boat as the source for both the selenium and the selenium-tellurium mixture. The selenium was placed in the boat before the system was closed, and then, as soon as it was evaporated completely, and without breaking the vacuum, the selenium-tellurium mixture was dumped into the same boat. The backing plate to be coated was clamped to a copper platen about six inches above the boat, and the under layer of selenium was deposited in about ten minutes.

One series of experiments was carried out to learn how the concentration of the tellurium in the outer layer affects the electrical characteristics of the two-layer plates. The fixed conditions chosen for this experiment were: (1) total plate thickness, 60 microns; (2) thickness of outer layer, ten per cent of the total thickness, or six microns; (3) temperature of backing plate, 81 C; (4) deposition time for selenium layer, 10 minutes; (5) deposition time for the top layer, six minutes; and (6) heattreatment time after deposition is completed, two minutes to cool plate to room temperature. The variable under study was the tellurium concentration in the outer layer.

Figure 10-4 shows the spectral sensitivities of the plates made in this series. The plate made with 20 per cent tellurium in the outer layer has a very high sensitivity, and plates made with lesser amounts of tellurium have less sensitivity. The plate made with 20 per cent tellurium has a photographic speed 17 times that of a selenium plate.

BATTELLE MENORIAL INFELTUTE



FIGURE 10-4. SPECTRAL SENSITIVITY OF TWO-LAYER SELENIUM-TELLURIUM PLATES HAVING VARIOUS CONCENTRATIONS OF TELLURIUM IN TOP LAYER

朝空市

A-4333

 $\mathbb{Z}_{2,2}$ 

1. Mary Mary

#### 10-21

Table 10-2 lists the dark-decay halftimes and residual potentials for these seven two-layer plates. It is evident that the dark-decay halftime decreases as the tellurium content is greater, but the relationship is not a simple one.

Ç 6	Tellurium concentration in top layer,	Dark-decay halftime,	Residual potential,
Plate	per cent	seconds	volts
R-10-10-52-D	7	630	15
R-10-16-52-A	8	480	12
R-10-16-52-B	10	138	12
R-10-21-52-C	12.5	94	45
R-10-17-52-A	15	85	15
R-10-30-52-A	17.5	23	90
R-10-13-52-A	20	12	12

### TABLE 10-2.DARK-DECAY HALFTIMES AND RESIDUALPOTENTIALS FOR TWO-LAYER PLATES

The residual potentials are between 12 and 15 volts, except for two plates. These two plates are ones that fall "out of line" by having unexpectedly low absolute sensitivity. From the spectral-sensitivity curves plotted in Figure 10-4, the plate containing 17.5 per cent tellurium in the top layer should have a considerably higher sensitivity than is shown. The plate with 12.5 per cent tellurium also should have somewhat higher sensitivity.

Figure 10-5 shows how the panchromaticity of this series of plates varies with the concentration of tellurium in the top layer of the plate. In this graph, panchromaticity is measured by the wavelength at which the sensitivity drops to one fifth its maximum value. This measure of panchromaticity was chosen instead of, say, the long-wavelength cutoff, to minimize the effect of varying dark-decay rates or varying residual potentials. In addition, this measure of panchromaticity is a more practical measure of the usefulness of a plate in photographing colored objects.

Another series of plates was made in which the tellurium concentration was held at ten per cent, and other plate-preparation conditions were held constant at the same values as before, except for the length of time to deposit the outer layer. This time was varied from 1.5 minutes to 75 minutes. No correlation was observed between any of the electrical characteristics and the deposition time, although electrical characteristics varied considerably, the maximum sensitivity varying from 0, 38 to 0, 92,



FIGURE 10-5. VARIATION OF PANCHROMATICITY WITH TELLURIUM CONCENTRATION IN TOP LAYER OF TWO-LAYER PLATES

A-4334

#### 10-23

and the wavelength for a sensitivity equal to one fifth the maximum sensitivity varying from 585 and 640 millimicrons. No conclusions were drawn on the basis of this series of plates.

Another series of 27 plates was prepared in which both the temperature of the backing plate and the rate of deposition of the outer layer were varied. A mixture containing 20 per cent tellurium was used, but the thickness of the outer layer was reduced to about five per cent of the total thickness, or to about three microns. Backing-plate temperatures of 60 C, 70 C, and 80 C, and evaporation rates for the outer layer of 1/6, 1/20, and 1/60 gram per minute were used at each backing-plate temperature. Three plates were made at each of the nine conditions. These plates were made in a random order.

The results of this experiment were:

1. Maximum sensitivity of the plates fell within 15 per cent of a constant value of approximately 1.0, with the exception of three plates that had roughly half that value. No correlation appeared between these maximum-sensitivity values and either the temperature or the rate of deposition.

Panchromaticity, as measured by the wavelength at which the sensitivity dropped to one-fifth its maximum value, showed essentially no variation with backing-plate temperature, but was a little greater for plates prepared using an average evaporation rate of about 1/20 gram per minute than for those using a rate of 1/6 gram or 1/60 gram per minute. For instance, the average cutoff wavelength for the plates prepared using an average evaporation rate of 1/20 gram per minute was about 685 millimicrons, while those for evaporation rates of 1/6 gram and of 1/60 gram per minute were 640 and 660 millimicrons, respectively.

3. Dark-decay rates varied widely and did not correlate either with backing-plate temperature or deposition rate.

4. All residual potentials, except for one plate, were below 50 volts. The residual potential of plates made at 70 C averaged a few volts lower than for plates made at 60 or 80 C, regard-less of the evaporation rate.

1 U T E

These data are now being analysed statistically to see if the trends noted above are significant. However, it appears that the temperature of

7 . . . .

#### 10-24

the backing plate may vary widely without greatly affecting the characteristics of the plates. In addition, variations in the deposition rate have comparatively little effect on plate characteristics.

Since more reproducible results are obtainable with two-layer selenium-tellurium plates than with the one-layer plates, a series of twolayer plates was prepared for use in the rapid-processing camera described later in this report. The preparation conditions used were the same as those described earlier in this section, except that the vacuum system was modified for preparing a photoconductive surface with an area of four inches by five inches. The modifications involved the installation of a water-contact platen to accommodate backing plates five inches by seven inches, increasing the distance between the boat source and the platen to nine inches, and increasing the amount of selenium and seleniumtellurium mixture evaporated from the boat to a total of 25 grams. The plates prepared had photoconductive films about 50 microns thick, with the outer selenium-tellurium layer being about two per cent of the total thickness. Temperature of the base plate during evaporation was 80 C. The outer layer contained 20 per cent tellurium.

Table 10-3 is a summary of the electrical characteristics of the eleven plates made under these conditions. Variations in the maximum sensitivity are greater in this series than in the series of plates made to investigate the effect of deposition rate and of the temperature of the backing plate. On the other hand, variations in panchromaticity are less for this series, although one of the plates, 11-12-52-C, shows a particularly poor response. Variations in photographic speed are considerable, the speeds for various plates ranging from 3.6 to 8.9 times that of ARQselenium plates, comparable to a range of from ASA 8 or 10 to ASA 20 to 25.

An interesting variation occurred in the dark-decay rates. All plates measured within two days after preparation showed more rapid decay rates than plates measured ten days later.

#### Microscopic Examination of Electrophotographic Plates

Most testing of electrophotographic plates has consisted of measuring the electrical potential on plates under conditions similar to those under which the plate is used. Such procedures have the practical advantage that the results can be translated directly into a practical evaluation of the plate. However, other testing techniques that appear particularly useful without involving great experimental difficulty are: microscopic examination of the structure of the selenium films; measurement of the optical characteristics of the films; and measurement of the electrical current

BATTELLE MEMORIAL INSTITUTE

10-25

इ.स.च. १९४१ व

ŗ

õ

8 A T TE LLE

5

TAI	BLE 10-3. CH/ FOI	ARACTERISTIC R USE IN RAPI	S OF TWO-LAY D-PROCESSING	ER SELENIUM-TI CAMERA	ELLURIUM PLA	TES
		Maximum	Relative	Wavelength	Dark-décay	Residual
	Date	sensitivity	<b>phot</b> ographic	for $S_{max}/5$ ,	halftimes,	potential,
Plate	measured	(S <sub>max</sub> )	speed	millimicrons	seconds	volts
()) ())		90 90	-	E DE	0001 <	
1-18-26-09-0	1	<b>0</b> 0	1• O	COC	1000	<b>1</b> 1 ∨
11-11-52-A	11-13-52(2)	0.92	7.2	647	43	30
11-11-52-B	11-13-52(2)	0.64	5, 8	632	80	15
: 11-12-52-A	11-13-52(2)	0.75	5.7	638	46	60
11-12-52-B	11-13-52(2)	1.06	7.9	638	50	20
11-12-52-C	11-13-52(2)	0.64	3.6	578	06	。 30
11-13-52-A	11-13-52(2)	1.07	8.9	641	27	20
	11-25-52(3)	0.82	<b>6.</b> 5	665	108	25
11-13-52-C	11-25-52(3)	0.47	3.7	655	228	30
11-14-52-A	11-25-52(3)	0.73	5.9	648	133	25
11-14-52-B	11-25-52(3)	0.57	4.7	633	320	25
11-17-52-A	11-25-52(3)	0. 75	6.0	645	205	25
- - - -	nated with 50 micros	of ABO celenium				
· versions (r)		WINTING AND IN				
(2) Plate measured w	ithin two days after pr	e paration.				

(3) Plate measured eight or more days after preparation.

### 10-26

passing through the films as a function of potential, time, and intensities of illumination at various wavelengths.

During the past year, selenium films have been examined under a microscope. These examinations were of an exploratory nature to find out what differences the microscope could show between various types of selenium films. For microscopic examination, a small flake of selenium was chipped from the plate and examined both by transmitted light and by vertical illumination, using, for the most part, a 97-power, 1.25-numericalaperture oil-immersion achromat and a ten-power ocular. Since selenium films generally have low transmittance in the far red, a carbon-arc lamp was used with a variable-focus substage con enser to provide intense illumination when examining the films by transmitted light.

When viewed by transmitted light, plates prepared as long as a year ago from ARQ selenium at backing-plate temperatures below 70 C showed no apparent structure and had a clear red color. Similar plates prepared at about 80 C, and having a secondary response peak in the red, showed circular dark spots about four microns in diameter, presumably crystallites of hexagonal selenium. Because the matrix surrounding these crystallites was also relatively clear, it was possible to locate the position of the crystallites approximately by focusing through the film. All crystallites occurred near the interface of the brass and the selenium.

Plates prepared from selenium containing various amounts of tellurium had matrices of higher optical density than that of pure selenium. In this case, the circular crystallites could be detected<sup>o</sup> only where the film had fractured fortuitously in a tapered edge. At such edges, the film was transparent enough to see crystallites near the interface between the brass and the selenium-tellurium film, but not elsewhere in the film.

Plates made with a layer of selenium-tellurium mixture over a layer of selenium generally were too opaque to be studied by transmitted light through the entire thickness of the films. However, at tapered edges, the selenium layers were relatively transparent as compared with layers containing tellurium. Layered plates showed crystallites at the interface between the selenium film and the brass backing plate, as well as some other crystallites apparently located near the interface of the selenium and the selenium-tellurium mixture.

Examination of selenium chips under vertical illumination showed practically no structure at the top surfaces of the films. Lower surfaces, which were good replicas of the brass backing plates, showed many fine polishing scratches.

A number of solvents for selenium were tested as etchants to reveal structure. Two solvents, carbon disulfide, and a dilute solution of less

BATTELLE MEMORIAL INSTITUTE

#### 10-27

then one per cent of potassium sulfite in water, were found useful. Selenium chips treated in these etchants for about five minutes showed a definite, irregular, rough top surface. At the lower surface, the etchants attacked part of the surface but left unchanged circular areas which corresponded to the circular dark spots, or crystallites, observed by transmitted light. The unetched circular areas were all of approximately the same size on a given specimen. On chips from a film of ARQ selenium, etching revealed no more dark spots than were revealed by transmitted light and vertical illumination.

Figure 10-6.shows two photomicrographs of an interface surface of a chip of ARQ selenium removed from a plate. One was viewed by transmitted light; the other is the same area etched with carbon disulfide and viewed with vertical illumination.

As a different technique for examining electrophotographic plates using vertical illumination, chips of selenium were mounted edge up, and the fractured surface was examined. Vitreous selenium showed the same conchoidal fracture pattern that characterizes the fracture of glass. However, any crystals present in the film will show up as interruptions in the smooth lines of the conchoidal fracture pattern or as small circular dark areas. In most samples examined to date, most of the crystals appear at the interface between the selenium and the brass. In a few cases, notably the two-layer plates, some dark spots--presumably crystallites--have been observed distributed throughout the film. In some multilayer plates, layers containing high concentrations of tellurium can be distinquished from adjacent selenium layers because of slight differences in reflectivity.

Some high-sensitivity panchromatic plates show no crystallinity anywhere in the film. Here, the development of crystals apparently is not a factor in producing a high-sensitivity panchromatic response. A different mechanism must be active here than is the case with red-sensitive selenium plates, where, in every case examined, red sensitivity was accompanied by a definite crystallinity at the interface of the selenium and the brass. As mentioned previously, the crystallites observed in most selenium or selenium-tellurium films are located very near the interface between the film and the brass. This suggests that a foreign material or discontinuity of some type is necessary for the formation of the nuclei about which the crystallites grow. This idea is supported by the observation that the crystallites sometimes appear to be concentrated along the more prominent scratches in the brass plate, where there might be a greater amount of foreign material, or a more pronounced discontinuity.

The fact that the crystallites of a given plate are all about the same size suggests that all of the nuclei were formed at about the same time. The only likely time for such nucleation is at the beginning of deposition of

BATTELLESSMENORPALS INSTITUTE

#### 10-27

then one per cent of potassium sulfite in water, were found useful. Selenium chips treated in these etchants for about five minutes showed a definite, irregular, rough top surface. At the lower surface, the etchants attacked part of the surface but left unchanged circular areas which corresponded to the circular dark spots, or crystallites, observed by transmitted light. The unetched circular areas were all of approximately the same size on a given specimen. On chips from a film of ARQ selenium, etching revealed no more dark spots than were revealed by transmitted light and vertical illumination.

Figure 10-6.shows two photomicrographs of an interface surface of a chip of ARQ selenium removed from a plate. One was viewed by transmitted light; the other is the same area etched with carbon disulfide and viewed with vertical illumination.

As a different technique for examining electrophotographic plates using vertical illumination, chips of selenium were mounted edge up, and the fractured surface was examined. Vitreous selenium showed the same conchoidal fracture pattern that characterizes the fracture of glass. However, any crystals present in the film will show up as interruptions in the smooth lines of the conchoidal fracture pattern or as small circular dark areas. In most samples examined to date, most of the crystals appear at the interface between the selenium and the brass. In a few cases, notably the two-layer plates, some dark spots--presumably crystallites--have been observed distributed throughout the film. In some multilayer plates, layers containing high concentrations of tellurium can be distinguished from adjacent selenium layers because of slight differences in reflectivity.

Some high-sensitivity panchromatic plates show no crystallinity anywhere in the film. Here, the development of crystals apparently is not a factor in producing a high-sensitivity panchromatic response. A different mechanism must be active here than is the case with red-sensitive selenium plates, where, in every case examined, red sensitivity was accompanied by a definite crystallinity at the interface of the selenium and the brass. As mentioned previously, the crystallites observed in most selenium or selenium-tellurium films are located very near the interface between the film and the brass. This suggests that a foreign material or discontinuity of some type is necessary for the formation of the nuclei about which the crystallites grow. This idea is supported by the observation that the crystallites sometimes appear to be concentrated along the more prominent scratches in the brass plate, where there might be a greater amount of foreign material, or a more pronounced discontinuity.

The fact that the crystallites of a given plate are all about the same size suggests that all of the nuclei were formed at about the same time. The only likely time for such nucleation is at the beginning of deposition of

BATTELLE MEMORIAL INSTITUTE

#### 10-28

the selenium. Several mechanisms could account for a large amount of nucleaction at this time and little or no nucleation later. A volatile impurity present with the original selenium might be required for nucleation, or nuclei might form only when the rate of deposition of the selenium is relatively low and the depositing molecules of selenium may have a mobility which is absent in molecules depositing at a faster rate. However, no matter what the mechanism for the formation of nuclei, it appears that a critical part of the deposition of selenium is the laying down of the first thin film on the metal backing plate.

### Effect of Sensitization on Electrical Characteristics of Selenium-Tellurium Plates

ļ

The electrical characteristics of selenium-tellurium plates depend more on the way the plates are charged than do the characteristics of plates coated only with selenium. In addition, the effect of different charging conditions varies from plate to plate.

Electrical measurements are made on all experimental plates by recording, as a function of time, the positive potential on the surface of • the plate in darkness, and as the plate is exposed to light of various wavelengths. Such sensitivity measurements are made at eight or ten wavelengths throughout the visible spectrum.

The electrical potential on the surface of selenium plates decreases slowly in darkness, indicating that the selenium film exhibits an apparent resistivity as high as  $10^{16}$  ohm-centimeters(1). Dark-decay rates for typical one-layer and two-layer selenium-tellurium plates are rapid, showing apparent resistivities as low as  $10^{14}$  ohm-centimeters. These dark-decay curves frequently appear to consist of two distinct parts: an initial, relatively rapid decay, and a slower, residual decay.

Figure 10-7 shows a series of dark-decay curves made on the onelayer selenium-tellurium plate identified as R-4-24-52-A. These curves show the two-slope form characteristic of many dark-decay curves. These curves also show the effect of repeated charging on the plate. In this case, the plate was charged several times at five-minute intervals. During this treatment, the dark-decay curves changed progressively until they reached a final, stable form. This treatment reduced the decay rates in both the early and the later portion of the curve. This same general effect appears 0

 $p = 2.58 t \times 10^{12}$ 

where  $\underline{p}$  is the resistivity in ohm-centimeters, and  $\underline{t}$  is the time in seconds for the potential to fall to one-half its original value. This formula assumes a dielectric constant for selenium of 6.31.

BATTELLE MEMORIAL INSTITUTE

<sup>(1)</sup> The average apparent resistivity of selenium films during the measuring period may be obtained from potential-decay measurements by applying the following formula:

10-29 and 10-30





1000X

1

92698

1000X

92699

0

A Transmitted Light

<u>B</u> Vertical Illumination

Plate: R-4-22-52-A Backing Plate Temperature: 80.1 C Thickness: 43 microns Etchant: Carbon disulfide Etching Time: One hour

### FIGURE 10-6. PHOTOMICROGRAPHS OF SELENIUM CHIP RE-MOVED FROM ELECTROPHOTOGRAPHIC PLATE


10-31

with most selenium-tellurium plates, although it is usually not so pronounced as for this particular plate.





Varying the amount of electrical current flowing to the plate during charging has an effect somewhat similar to varying the time of charging. Larger currents generally produce higher plate potentials and slower initial dark-decay rates, without much effect on residual dark-decay rates. However, in many cases the potential at which this residual decay becomes predominant seems to vary with the charging current.

Figure 10-8 shows the effect of various charging currents on the dark-decay characteristics of the one-layer selenium-tellurium plate 7-11-52-A. In this case, the charging current was varied by adjusting the potential applied to the grid of the scorona charging unit while maintaining the current in the corona discharge at a constant value of 100 microamperes. The characteristics of this plate are not so representative of the average panchromatic plate as might be desired, as it decays in the dark at a slower rate than average. Plates with faster decay rates show similar

BATTELLE MEMORIAL INSTITUTE



FIGURE 10-8. EFFECT OF DIFFERENT GRID POTENTIALS AND CHARGING TIMES ON DARK-DECAY CHARACTERISTICS OF ONE-LAYER SELENIUM-TELLURIUM PLATE 7-11-52-A

RESTRICTED

A - 3097

TUTE

#### 10-33

characteristics, except that, with lower charging currents, the initial dark decay may be so rapid that it cannot be recorded.

The effect of various charging conditions on the light sensitivity of panchromatic selenium-tellurium plates, both one layer and two layer, is not well understood. However, the following tentative conclusions appear to hold for these plates:

- 1. Light-decay curves have residual portions similar to those of dark-decay curves, and located at about the same potential. Repeated or continued charging does not raise this potential, as had been believed previously.
- 2. Initial light-decay rate is independent of charging conditions, providing the potential is greater than the potentials of the residual decay.
- 3. A sensitive selenium-tellurium plate may appear insensitive if its potential has decayed to the residual portion before the plate is exposed to light, or if its initial dark-decay rate is comparable with the lightdecay rate, thus obscuring the effect of light. This is more specifically applicable for one-layer seleniumtellurium plates.
- 4. In some cases, exposing a plate to light increases the residual-decay level, sometimes enough to interfere with light-decay measurements.

<u>Retesting Plates Prepared in 1952.</u> Plates made during the first six months of 1952 were tested after being sensitized with a charging unit using a corona current of about one microampere. Plates charged in this way show characteristics that can be divided roughly into four categories:

- 1. About one-third of the plates had characteristics not greatly different from those of selenium plates. This group was made up mostly of selenium control plates, plates having less than four per cent tellurium, and some two-layer plates in which the greater percentage of the total film thickness was selenium.
- 2. About one-sixth of the plates showed a much higher sensitivity to light of all wavelengths than is found with selenium plates. In a number of cases, their response was nearly panchromatic.

BATTELLE MEMORIAL INSTITUTE

#### 10-34

- 3. About one-third of the plates accepted a potential of at least 200 volts but showed no sensitivity to light of any wavelength.
- 4. About one-sixth of the plates had such fast dark decays or accepted such low potentials that they could not be used practically.

About 50 of these plates were retested, using higher corona currents in sensitizing them. Most of the plates previously found insensitive showed the high, nearly panchromatic response to light observed previously for only a few plates.

In this retesting, plates of the first and second categories showed very little change from the original tests. Plates in the third category, those which accepted a potential of about 200 volts but were insensitive to light, improved in two ways. First, it became apparent that the potential accepted by the plate in the original testing was really only a high residual potential. Using the higher, 100-microampere corona current, it was possible to charge the plates to potentials considerably higher than these residuals and, hence, to obtain a high sensitivity in decays to this residual level. Secondly, many of these high residual potentials apparently decreased during the two- to three-month period between tests. With these plates, a return to the original charging conditions produced a lower residual potential than determined in the original measurements, and the plates might have well been reclassified into the fourth category as a result of this apparent effect of aging.

Plates in the fourth category, those which originally had such fast dark-decay rates that they were not usable, were improved by a retardation of the dark-decay rate to the point that sensitivity measurements could be made. A few plates in this category, even when charged with the higher corona current, still had dark-decay rates too fast to permit meaningful measurements of sensitivity.

Figures 10-9 and 10-10 show decay curves and spectral-sensitivity curves for typical plates having a film containing 93 per cent selenium and 7 per cent tellurium, previously believed insensitive but later found to have high sensitivities when charged with the higher corona currents.

Figure 10-9 shows the characteristics of the one-layer plate R-4-25-52-A, which originally showed no sensitivity because of a high residual potential. The original decay curves for this plate were recorded on April 25, 1952, using a corona current of about one microampere. The later characteristics were recorded in July 14, 1952, using a corona current of 100 microamperes. Light-decay curves were made by exposing the plate to

BATTELLE MEMORIAL INSTITUTE



FIGURE 10-9. CHARACTERISTICS OF ONE-LAYER SELENIUM-TELLURIUM PLATE R-4-25-52-A FOR DIFFERENT CHARGING CONDITIONS

2

ß

c

A-4335

:/" :/"



8.2

FIGURE 10-10. CHARACTERISTICS OF ONE-LAYER SELENIUM-TELLURIUM PLATE R-4-16-52-A FOR DIFFERENT CHARGING CONDITIONS

A-4336

10-37

0

light of 400 millimicrons wavelength at an intensity of 0.030 microwatt per square centimeter.

Figure 10-10 shows the characteristics of the one-layer plate R-4-16-52-A, which originally could not be charged to a potential high enough to show sensitivity. Charging conditions were the same as those descilled in the paragraph above. Original measurements were made on Ap 11 16, 1952, and the later measurements were made on July 14, 1952.

Retesting Plates Prepared in 1951. About 50 plates made in 1951 also were rechecked using higher corona currents in sensitizing them. Most of these plates were selenium-tellurium plates, but many of them contained lower percentages of tellurium than were used in 1952. In many cases, the spectral response of the plates was not measured because these fast dark-decay rates were assumed to make the plate useless.

In retesting these plates, several were found to have an extended spectral response. Some of these plates were those made near the end of the year, when plates were being prepared with long deposition times from mixtures of 93 per cent selenium and seven per cent tellurium. However, two plates, R-8-27-51-B and R-8-28-51-B, that showed an extended spectral response, that is, long-wavelength cutoff at about 650 millimicrons, were made with tellurium concentrations of only 2.2 per cent and 3.0 per cent. Another plate, R-8-20-51-B, had the most nearly uniform response to light of any plate tested thus far. This plate was made from a seleniumtellurium mixture having a tellurium concentration of 4.3 per cent. The absolute value of its sensitivity to blue light was only a little greater than that of an ARQ-selenium plate, but its sensitivity remained within ten per cent of that value from 400 to beyond 600 millimicrons, and only dropped to about half that value at 750 millimicrons.

A possible explanation for the suprisingly good charateristics of aplate made with such low concentrations of tellurium lies in the preparation of the selenium-tellurium mixture used. In all three cases, the selenium and tellurium were heated in an open crucible in air for only a few minutes before being placed in the vacuum system. If the mixing of the two materials was not complete, and particularly if some particles of tellurium remained undissolved, then the last material to be vaporized from the crucible might be very rich in tellurium. Thus, the tellurium concentration may have varied from little or none to a rather high concentration in the top layer of the film.

Another plate retested after sensitizing at a corona current of 100 microamperes, Plate R-7-23-51-A, was made in July, 1951, by evaporating selenium and tellurium from separate sources in such a way as to produce a variation in tellurium content from one end of the selenium film to the

BATTELLE MEMORIAL INSTITUTE

#### 10-38

other. At the point on this plate where the tellurium concentration is greatest, the plate showed considerable panchromaticity, the longwavelength cutoff being about 750 millimicrons. The response dropped rapidly for areas containing lower concentrations of tellurium.

On the basis of the characteristics of this plate, the following important conclusions were drawn:

- 1. Panchromaticity in a plate does not necessarily depend on an interaction between selenium and tellurium when they are melted together before evaporation.
- 2. It is not necessary to evaporate all of the material in the boat in order to obtain a panchromatic response. A shutter may be used to end a deposition process at a predetermined time, without concern over whether the desired high sensitivity is due to a possible high concentration of impurities in the last fraction of melt to leave the source.

!.

Using a two-source technique, it is possible to control the concentration of tellurium throughout the film by controlling the relative rates of evaporation from the two sources. Also, the time for deposition and any heat treatment after deposition may be determined accurately using the shutter. However, these advantages are offset by the practical difficulty of controlling the rates of evaporation of different materials in a vacuum system.

### Electrophotographs Made With Single-Layer Selenium-Tellurium Plate Using Standard Darkroom Procedure

Work described in this section was done to determine the picture quality obtainable using single-layer selenium-tellurium plates, and to learn something about the empirical photographic speeds of the plates from actual exposure data.

Plate R-5-16-52-A, used for these tests, was made using a 90minute deposition time, a backing-plate temperature of 80.5 C, and a tellurium content of seven per cent. The film thickness on this plate was about 34 microns. The characteristics sought in this plate were high initial potential, a dark-decay rate of about 30 seconds or longer, and panchromatic sensitivity.

### BATTELLE MEMORIAL INSTITUTE

#### 10-39

This plate had the following characteristics when originally tested using the low-current charging device:

Initial potential	-	500 volts
Dark decay to half voltage	-	27 seconds
Residual potential	-	40 to 80 volts
Sensitivity(1) at:		
400 millimicrons	-	1.02
500 millimicrons	-	0.89
600 millimicrons	-	0.67
700 millimicrons	-	0.22

0

The plate was charged to a positive potential of slightly over 300 volts, using high corona current, and exposed through a positive transparency to light from a 100-watt tungsten lamp. This exposure unit, described on pages 1-118 of the Final Progress Report, dated December 18, 1951, was used to assure the same exposure for each test. The plate was developed for ten seconds in the high-speed development unit, as described on pages 1-74 through 1-82 of the Final Progress Report dated December 31, 1951. A negative potential of 40 volts applied to the plate during development was sufficient to prevent deposition of powder in white areas.

Figure 10-11 shows photographic reproduction of the pictures developed on selenium-tellurium plate R-5-16-52-A and on a selenium plate. Exposure times of 3/8 second and 22 seconds, respectively, were used with all other exposure conditions being held constant. Many of the blemishes on the picture made on the selenium-tellurium plate<sup>°</sup> are due to scratches and finger prints on the surface of the plate; others resulted from incomplete cleaning of the backing plate prior to coating. The texture and contrast of the print are not quite so good as those in the picture made on the selenium plate. However, the 50-fold increase in apparent photographic speed is real and significant.

An outdoor exposure was also made using the selenium-tellurium plate, R-5-16-52-A. With the sun shining through a partial overcast, a photographic speed of ASA 12 was determined from readings on an exposure meter. This is a five-fold increase over the nominal speed of ASA 2.5 for selenium plates. However, this speed of ASA 12 may be in error by as much as 50 per cent, due to uncertainty of actual shutter speed. Also, the rapid dark-decay rate on this plate made it difficult to establish the best conditions for exposure and development.

MEMORIAL

RESTRICTED

BATTELLE

INSTITUTE

<sup>(1)</sup> Sensitivity used here is the reciprocal of the time for the plate voltage to decay from 200 to 100 volts for light of the wavelength stated and a light intensity of 0, 030 microwatt per square centimeter.

### 10 - 40

### Rapid-Processing Electrophotographic Camera

The high-sensitivity characteristic of the two-layer streatum tellurium plates, as evidenced by electrometer tests, surgested that they be tested for picture-making characteristics. However, their high darkdecay rates made such photographic testing impossible will, the usual darkroom procedure, too much time being required in handling the plate

In usual darkroom procedure, the plate is charged if passing it under a corona discharge and placing it in a plate holder. The plate is then carried to and placed in a camera, exposed, and retuined to the darkroom where it is removed from the plate holder, placed on the development unit, and the image is developed. The shortest time in which this procedure can be carried out is about one minute. However, in appreciably less than one minute, the potential on a high-sensitivity selenium-tellurium plate will drop too far, even in darkness, for the plate to be useful in making a picture.

In order to use these plates, a "rapid-processing electrophotographic camera" was constructed, whereby the plate could be handled rapidly in & the interval between charging and development. For the purpose of demonstrating the type of image that can be developed using the eleniumtellurium plate, a simple, manually operated camera was constructed. It incorporated only the steps which require rapid sequencing, the steps of transfer and cleaning being carried out independently.

#### Designs Considered for Rapid-Processing Camera

A number of different designs were considered for the rapid processing camera. Points considered in evaluating the designs were speed and convenience of operation, trouble-free operation simplicity in construction, smallness in size, and lightness in weight.

Figure 10-12 shows five designs considered. Several preared equivalent when judged on the above points, but Design <u>B</u> wes selected finally as fulfilling the requirements best.

If the two-layer selenium-tellurium plates(1) used in these tests free exposed to room light prior to charging, their decay of potential in subsequent darkness will be too rapid to permit use, even in a humera which takes the plate from charging through development in two to three seconds

MEMORIAL

RESTRICTED

INSTIT

(1) Preparation and characteristics of these plates are described elsewhere in this report?

BATTELLE



ò

ŗ



FIGURE 10-12. DESIGNS CONSIDERED FOR RAPID-PROCESSING ELECTROPHOTOGRAPHIC CAMERA

MEMORIAL

REST

A-4337

INSTITUTE

n

Consequently, the plates must be handled in total darkness exceptor the camera exposure itself. For simplicity in camera design the complished by load ng and unloading the camera in the darkroup rather than loading the plate into the camera from a cassette.

### Description of Rapid-Processing Camera

Figure 10-3 shows a butaway view of the hinge-type rapid-processing camera constructed or testing selenium-tellurium plates. In this cameral the back opens to permit placing the plate in the holder. When the back is closed, the holder can be positioned so that the plate is in the image plane of the lens. A polonium radioactive charging unit(1) used in this camera swings from the top of the camera to a position directly in front of the plate. In this position, electrical contact is made, voltage is applied between the plate and the charging unit, and charging begins.

Development is accomplished by swinging the plate to the bottom of the camera, against shims which space the plate 0.015 inch above the electrode of a powder-cloud development unit. Pressing the plate against an "O-ring" gasket seals the plate over the electrode and previous develop ment powder from entering the main camera chamber. The camera ais mounted over the belt-type powder-cloud generator and the whole assembly mounted on a wheeled cart to facilitate moving from the darkroom to the window for outdoor photographs.

The camera is equipped with a 12-inch, f/4.5, Wollensak, Series II, Velostigmat lens.

### Operation of the Rapid-Processing Camera

The operations of charging, exposing, and developing are carried out in the rapid-processing camera.

In charging, the polonium source is spaced 5/8 inch from the fiveinch by seven-inch electrophotographic plate, and a potential of 300 volts is applied for one to two minutes, charging the plate electrically positive.

Immediately at the end of charging, the charging unit is swung away from the plate, the exposure is made, the plate is swung down, sealed against the electrode, and developed. Development requires approximately one second. The total time from the end of charging to the end of development is approximately three seconds.

ß

BATTELLE MEMORIAL INSTITUTE

<sup>(1)</sup> Described later in this report.



#### 10-46

# sesults of issts With the Rapid-Processing Camera

Using a selenium plate of known photographic characteristics, tests where camera showed it to be operating satisfactorily in all respects. Where camera showed it to be operating satisfactorily in all respects. We ages were then developed using a two-layer selenium-tellurium plate. We hough the resulting prints were not so good as those made using the effective plate, they were of inherently good quality. They were free of wein, streaks, and other blemishes, indicating that there were no gross where is in the plates. Absence of a "snow-storm" appearance indicated eredom from over harging and localized electrical breakdown. The streak is in the plates in these pictures were in density and contrast. Greater the setting of the from improper development conditions, such is development time, and lectrode potential. The tast dark-decay rates to these plater and the high esidual potentials make the handling of the Plate and the setting of development conditions much more critical than in is case of lain selenium plates.

Figure 10-14 shows a picture the suing a two-layer scleniumrium plate, 11-4+52-C. This plate we coated with 46 microns of prium over the base plate and with a first control layer of 80 per cent is overdeveloped, but is interwise of good quality. It was exposed at a structure speed of about 4.4 30, daylight, verifying the photographic speed of dicted from electriitests on this type of plate and representing a map

When this plate was used to make pictures at approximate ir intervals, the residual potential gradually increased from a star ats to about 120 volts. In this same interval, the photographic ared to drop slightly. After storage in schedules of the att, the original perties of the plate were restored.

Sensitization Sectrop Iterraphic Plan 8

One of the off the state of its or lect was sinvestighte, fernat ie tods of sensiliting element of noto carly plates. From thes invest method well suited to use in the 1

Vention Corona Unit

BATPELLE

81-1-1

Ŷ

The sensitizing technique used in the plate by means of a sensitizing technique used in the plate by means of a sensitive plate, and then driving these ions onto

MEMORIAL

\_ 10=17-ind 10-10-10



98734

### FIGURE 10-14. ELECTROPHOTOGRAPH MADE ON TWO-LAYER SELENIUM-TELLURIUM PLATE 11-4-52-C AT APPROXIMATELY ASA 30, DAYLIGHT

#### UTE £3

10-49

electrical field. In the conventional technique in use at the beginning of this contract, the ions were created in air by raising the potential of a fine wire to approximately 7000 volts, causing corona discharge around the wire. The ions created by this discharge are then driven onto the plate surface by the electrical field which exists between the fine wire and the electrophotographic plate. To prevent excessive charging of the plate -which is limited only by the 7000 volts applied to the corona wires -- a control grid of wires is placed between the corona wire and the plate. By maintaining the potential on the control grid at approximately the potential desired on the plate, overcharging of the plate is avoided.

This technique places a uniform electrical charge on the plate. In addition, results can be reproduced consistently. However, it does not lend itself well to use in portable cameras because of the weight of the required high-voltage source and the need for electrical energy.

As these needs have been eliminated in the development apparatus by using triboelectric charging of the powder, doing away with the bulky power supply for sensitizing plates would make the entire electrophotographic equipment much more portable.

#### Radioactive-Sensitizing Unit

17 P

One method of eliminating the power supply would be to use a radioactive material to create the necessary ions in the air. Any radioactive material creates ions in the air surrounding it, because of collisions between the molecules of the air and the alpha, beta, and gamma rays emitted by the source. Suggestions as to material to use were made by a representative of the Signal Corps carly in this development.

Two radioactive sources were tried. The first was a relatively weak source used because it was available at the time this idea was proposed. A second, stronger source, was purchased later.

Radium. The first radioactive material tried as a suitable source of ions was radium and its decay products as contained in an Ionotron(1). By placing the Ionotron and the electrophotographic plate to be sensitized in an electric field, the ions created by the radioactive source were driven onto the electrophotographic plate giving it the required electrical potential.

MEMORIAL

PFSTRICTE

INSTITUTE

(1) United States Radium Corporation, 535 Pearl Street, New York, New York.

BATTELLE

10-50

The particular Ionotron used consists of a metal housing containing a thin metal foil impregnated with radium. This foil, measuring one-half inch wide and six inches long, has an activity of 0.2 millicurie per square inch, giving a total activity of approximately 0.6 millicurie. This material, with a half life of 1600 years, emits alpha, beta, and gamma rays, with the major portion of the ionization produced in the immediate vicinity of this source being due to collisions between the alpha particles and molecules in the air near the source.

Figure 10-15 is a sectional view of the sensitizing unit incorporating this radioactive source. The source is mounted in a metal housing which fits directly onto a five-inch by seven-inch conventional photographic plate holder. The housing supports the radioactive material at a fixed distance from the plate, serves as a light shield, provides additional protection against radiation from the back of the source, and, when connected to a source of potential, directs to the electrophotographic plate the ions created by the radioactive material. The plate may be sensitized to either polarity by choosing the proper polarity of the field between the housing and the plate; when the housing is positive with respect to the plate, the plate will be sensitized positively. Miniature "B" batteries serve as the source of potential. They need to supply a current of only about one microampere, giving them a service life approximately equal to their shelf life.

The potential acquired by the plate is controlled by the voltage applied between the metal housing and the plate and by the length of time this potential is applied. The potential on the plate will never exceed that applied between the housing and the plate. Leaving the radioactive source over the plate for an extended period does not overcharge the plate, nor does it appear to have any other deleterious effects with sensitizing times as long as 24 hours.

Radioactive units can be mounted directly in a camera so that a plate can be maintained in a sensitized condition until it is exposed. With this particular source and arrangement, approximately two and one-half minutes are required to charge a selenium plate to 175 volts when approximately 300 volts is applied between the metal housing and the plate. However, by increasing the area of the source and using a source of greater activity, the time required for sensitization can be reduced appreciably. Images produced on plates sensitized with this unit are similar to those produced from plates sensitized by corona means at the same charging currents.

One of the disadvantages of this radium source is that one of the decay products of radium, radium-C, emits energetic gamma rays. This limits the concentration of radium that can be used safely without heavy shielding when persons are near the source, as they would be in this particular

BATTELLE MEMORIAL INSTITUTE



ţ

· - {

FIGURE 10-15. SECTIONAL VIEW RADIOACTIVE ION

TE

Ģ

## RESTRICT

10-52

application. Unfortunately, all substances emitting alpha particles also emit gamma ray. Polonium-210 emits alphas and only very weak gammas, but initially it was considered to have too short a half life to be useful and other solutions to the problem were investigated.

Beta Source. Beta particle emitters were considered as possible ionizing sources, as some radioactive materials emit no radiation except beta rays, against which shielding is relatively simple. Such a source is strontium-90, a relatively nexpensive isotope produced artificially with high specific activity, and having a useful half life of approximately 25 years. However, the ionizing power of a beta particle per unit length of path is only one one-hur dredth that of an alpha particle of equal energy, so that very high intensities of beta activity would be required to create enough ions to sensitize a plate in a reasonable length of time. While the use of such a material is not precluded, a more promising approach was found in the use of certain alpha emitters.

Radium-D. Mr. S. Levinos of the Siginal Corps suggested, as an icnizing source, adjum-D in equilibrium with its decer products. By chose h a material in the radium-decay series below radium-C, the energetic tam na rays from ridium-C are avoided, yet the material will decay into polones, and give of the alpha particles most useful in ionizing air. Radium-D is such interial. Its half life of 22 years makes it more practical than polonium-210 with its half life of only 140 days.

The Canadian Radium and Uranium Corporation<sup>(1)</sup> reported that radium-D could be plated onto a foil and that the price would be approximately \$100.00 per millicurie. This is approximately the same price as for radium, the parent product. Although a radium-D source has not been obtained nor used for sensitizing plates, there appears to be no reason why it would not be a satisfactory material.

<u>Polonium.</u> Because of the relatively low initial cost of polonium and the exploratory nature of the work, preliminary work was done with a polonium source rather than the longer lived radium-D source. A polonium source was available at a cost of approximately \$6.00 per millicurie.

The polonium source obtained consists of a metal plate four inches by five inches coated with polonium of an intensity of one millicurie per square inch, or a total of 20 millicuries. This size was chosen to match the selenium-coated area of the standard five-inch by seven-inch Signal Corps electrophotographic plate. For safety purposes, the polonium is overcoated with a thin layer of gold and thin layer of lacquer. These coatings

STRICTED

NSTITUTE

(1) 630 Fifth Avenue, New York 20, New York.

BATTELLE

ふり-53

help prevent chemical reaction of the polonium with foreign substances and prevent dult and air currents from contacting the polonium itself.

Several variables must be investigated before precise recommendations can be made on using polonium as the radioactive source in a sensitizing unit. Some where variables have been investigated to some degree as described below, but more work remains to be done.

The spacing between the polonium surface and the electrophotographic plate affects the rate of charging. The alpha particles emitted from a polonium source have a natural range of about 3.6 cm. These particles are most effective in ionizing the molecules in the air near the end of this range. If the polonium and electrophotographic plate are separated by less than 3.6 cm, the alpha particles are stopped by the selenium before they have created the maximum number of air ions. From this standpoint, the optimum spacing would be at least 3.6 cm.

When an alpha particle strikes a molecule of a gas, both positive and negative ions are formed. If these oppositely charged ions are permitted to remain near each other, they will recombine to form neutral molecules and hence detract from the ions available to charge the electrophotographic plate. However, if an electrical field is applied to the region containing the ions, the positive and negative charges will be separated and the chance of recombination will be reduced. If the electrical-field gradient is sm. 1, the tendency for separation will be small. Increasing the gradient will reduce the chance of recombination. For a given voltage between the source and plate, by definition, an increase in the spacing between source and plate will decrease the voltage gradient, and hence the spacing again indirectly affects the rate at which the plate will be charged. Another factor to be considered in the use of this source is that, as the electrophotographic plate is being charged, its potential increases and hence the gradient between it and the source decreases. Allowance must be made for this effect.

Although the investigation of these problems is not complete, preliminary results show, when the distance between the source and the plate is approximately equal to the distance an alpha particle will travel, that the gradient necessary to reduce recombination to a reasonable value requires an electrical potential difference between the source and plate much greater than the initial potentials normally desired for continuous-tone work. Therefore, the conditions to be used must be some compromise of spacing, potential difference, and sensitizing time.

A number of experiments were run to determine the magnitude of the ion current to be expected from the polonium source. A potential of 300 volts was applied between the four-inch by five-inch polonium source and an eight-inch by ten-inch sheet of bare aluminum. These plates were then

BATTELLE ... MEMORIAL INSTITUTE

PESTRICTED

10 - 54

spaced 0.5 inch apart and the current flowing between them was measured. Similar readings were taken for spacings of 0.75 inch and 1.0 inch.

Figure 10-16 shows the results obtained from these measurements.



FIGURE 10-16. ION CURRENT FLOWING BETWEEN POLONIUM SOURCE AND METAL PLATE AT VARIOUS SPACINGS BETWEEN SOURCE AND PLATE

į,

A-4339

This curve indicates that the additional collisions of the alpha particles with molecules in the air, which occurred in the space between 0.25 inch and 0.5 inch from the polonium source, contributed many additional ions to increase the current flowing between the polonium and the metal plate. However, increasing the spacing from 0.5 inch to 0.75 inch does not produce a similar increase in current. This is probably due to the fact that, although more ions have been generated in the additional space, the voltage gradient has been reduced from 600 volts to 400 volts per inch. This is apparently enough of a reduction that considerable recombination of ions can occur. The net result is that approximately the same number of ions reaches the metal plate at a spacing of 0.75 inch as at 0.5 inch. Increasing the spacing to 1.0 inch decreases the gradient to only 300 volts per inch. At this gradient, recombination is greater and the current is reduced still further.

Using the spacing which gave the highest current in the previous experiment, 0.75 inch, and again using 300 volts potential difference, a selenium-coated plate was substituted for the bare metal plate, and the

BATTELLE MEMORIAL INSTITUTE

RESTRIC

#### 10-55

potential which the plate acquired was measured after exposure to the polonium source for various lengths of time.

Figure 10-17 shows the results obtained from these measurements.



FIGURE 10-17. POTENTIAL ACQUIRED BY SELENIUM PLATE EXPOSED FOR VARIOUS TIMES TO POLONIUM SOURCE WITH 300 VOLTS POTENTIAL DIFFERENCE BETWEEN SOURCE AND PLATE

#### A-4340

æ

As the plate becomes charged, the rate of charging drops rapidly because the increase in potential on the plate decreases the potential gradient in the ionized air. For example, after 20 seconds, the potential difference between the source and the electrophotographic plate is only 125 volts, or a gradient of less than 95 volts per inch as opposed to 400 volts per inch initially. The effect of decreased gradient was observed in the experiment preceeding this one.

As a further check on the effect of voltage gradient on the ion current flowing from the polonium source to an electrophotographic plate, the previous experiment was repeated with the potential difference between the source and plate changed from 300 volts to 600 volts.

Figure 10-18 shows the results obtained when this potential gradient was doubled.

By comparing the absolute values obtained in the last two experiments, the importance of voltage gradient is easily seen. Although plates can be sensitized more rapidly using larger voltage gradients, it becomes

BATTELLE MEMORIAL INSTITUTE

RESTRIC

10-56



=-7).

FIGURE 10-18. POTENTIAL ACQUIRED BY PLATE EXPOSED FOR VARIOUS TIMES TO POLONIUM SOURCE WITH 600 VOLTS POTENTIAL DIFFERENCE BETWEEN SOURCE AND PLATE

increasingly important to time the process accurately if the initial potential desired on the plate is considerably below the absolute potential difference between the radioactive source and the electrophotographic plate.

To determine whether an electrophotographic plate sensitized with the radioactive unit would have the same electrical and optical characteristics as the same plate when sensitized with the conventional coronadischarge unit, light decays and dark decays were determined for selenium-coated plate 7-15-52-A after sensitization with each unit. Sensitizing conditions for the corona method were: standard electrometer unit; corona voltage, 7000; grid voltage, 100; total corona current, 50 microamperes; time, 5 seconds. Conditions for the radioactive source were: spacing of source to plate, 0.75 inch; voltage, 300; time, 30 seconds. These conditions were chosen so that, in each case, the initial potential on the plate would be the same.

Figure 10-19 shows the light-decay and dark-decay rates obtained from measurements on this standard continuous-tone plate coated with 50 microns of selenium. Light-decay readings were taken using 400millimitron light. Within experimental error, no difference could be detected between the action of the plate when sensitized with the corona

BATTERLE MEMORIAL INSTITUTE

10-57



ŗ,

FIGURE 10-19. CHARACTERISTICS OF SELENIUM PLATE 7-15-52-A SENSITIZED WITH CORONA SOURCE AND WITH RADIOACTIVE-CHARGING UNIT

**RESTRICTED** 

A-4342

TUTE

#### 10-58

unit or with the radioactive source. In addition, images made on this plate after sensitizing with each source were indistinguishable.

As reported elsewhere in this progress report, both one-layer and two-layer selenium-tellurium plates have sensitizing characteristics different from those of selenium plates. To check the possibility that radioactive sensitization and corona sensitization might not give similar results on these plates, the electrometer measurements described above were repeated on a two-layer selenium-tellurium plate, R-5-2-52-A. This plate is coated with 20 microns of selenium on the base plate followed by another 20-micron layer, consisting of 93 per cent selenium and 7 per cent tellurium, on top of the selenium layer. Sensitizing conditions using the corona were: standard electrometer unit; corona voltage, 7000; grid voltage, 200; total current, 100 microamperes; time, 5 seconds. Conditions for the radioactive unit were: spacing of plate to source, 0.75 inch; voltage, 500; time, 30 seconds. Again, these conditions were chosen so that, in each case, the same initial potential would be applied to each plate.

Figure 10-20 shows the light-decay rates of the plate when sensitized with the corona-charging unit and when sensitized with the radioactive source. These light-decay curves have different shapes but the average rates of decay are similar. Further checks will be made of the relative actions of the two sensitizing methods. For the present, it is assumed that sensitization by the radioactive source is equivalent to sensitization by the corona unit.

Work still must be done to determine optimum conditions for use of the radioactive-sensitizing unit. Some advantage may be found in using a specially shaped housing near the source. Also, more information is needed concerning voltage gradients, and the spacing between the radioactive unit and the electrophotographic plate.

This radioactive-sensitizing unit offers a compact, light-weight, rugged, portable, self-contained apparatus which is well suited to field use. There are no moving parts, no delicate fine wires, and no necessity for heavy power supplies.

### Image Development on Electrophotographic Plates

During the period covered by this report, several major advances were made in the understanding and practical use of powder-cloud development of continuous-tone electrophotographs.

RESTRICTED

BATTELLE

MEMORIAL INSTITUTE



!

FIGURE 10-20. LIGHT-DECAY RATES OF TWO-LAYER SELENIUM-TELLURIUM PLATE R-5-2-52-A SENSITIZED WITH CORONA SOURCE AND WITH RADIO-ACTIVE-CHARGING UNIT

A- 4343

-1

10-60

Images of good quality can now be developed in less than one second. Apparatus has been built which, by logical extension, could be used to develop an electrophotographic plate of any width desired. Images of improved quality and blacker tones have been produced.

In addition, a study was begun of the sensitometry of the electrophotographic process, and further attention was given to triboelectric charging of powders in capillary tubes, agglomeration in powder clouds, and of undesirable deposition of powder in the development apparatus. A srief attempt was made to create a new type of powder-cloud generator. Each of these items is discussed in more detail below.

#### Sensitometry

In the past, several techniques for developing continuous-tone electrophotographs produced pictures having the appearance of silver halide photographs. However, in this past work, pictures were evaluated only in terms of subjective estimates of their photographic appearance, rather than in terms of any quantitative sensitometric units. Sensitometric studies would have had limited value in the past because the electrophotographic techniques used gave such variable results that measurements on any one picture would have been of little value in determining the actual or potential characteristics of the technique. Futhermore, there were always more serious and more immediate problems requiring attention.

To a considerable extent, the situation is different at the present time. The development technique embodied in the high-speed development unit produces successive pictures that are nearly alike, and, though many problems still await solution, some information about tone reproduction with the present technique is needed to determine which problems should receive first attention. Further, a careful statement of the capabilities of the present electrophotographic process is needed to evaluate its usefulness in comparison with other photographic processes.

Accordingly, a sensitometric study of the present electrophotographic process has been started. This study should be a continuing one, becoming more refined as the control of the electrophotographic process is improved. It will include a number of considerations not important to silver halide photography, such as the effect on reproduction characteristics of repeated use of the electrophotographic plate, or the effect of the times between sensitizing and exposure, and between exposure and development.

The first objective of the sensitometric study was to determine the tone-reproduction curve for prints prepared under conditions that have been found to produce better-quality electrophotographs. For reasonable control

BATTELLE MEMORIAL INSTITUTE

11.0 - 61

of the exposure of electrophotographic plates, the subject chosen is a transparency of a gray-scale step wwwedge illuminated by a tungsten lamp. The final curve of tone reproductions was plotted as a graph of reflection densities of the electrophotograph and a function of the transmission density of the original gray-scale transparemency.

Special Gray Scale. The special gray scale used has the following characteristics:

 The area of each step of t the gray scale is large enough that several density meassurements can be made on it, so that the uniformity of • development over small areas can be determined.

£

• • •

- (2) The various density steps.s-are distributed at random over the plate to disclose syst matic errors on the tonereproduction curves. Usesing a wedge-type gray scale, a nonuniformity of development from one end of the plate to the other might be inteserpreted as a compressed exposure scale. However, . with steps of the gray scale arranged in random ordeer, this nonuniformity would show up as scatter of points about the tone-reproduction curve.
- (3) A large number of steps are provided, especially in the low-density portion of these gray scale, so that a reasonably accurate tone-reproductition curve can be obtained in spite of undesired variations in the development process.

Figure 10-21 is a reproductio on of the gray-scale transparency used as the photographic original in the sensitometric study. The actual transparency used is twice the size of the reproduction, the standard exposure device giving a reduction of two to one in size. To form the composite subject, rectangles cut from variou us densities of sheet film were taped onto a glass plate.

Schedule for Processing Elecotrophotographs. The electrical characteristics of selenium plates depend H on the conditions to which the plate has been subjected just prior to use. TTo reduce the effect of these factors, a short study was made of the effect of previous use on dark-decay rates and on light-decay rates. The two plat tes used, 9-17-51-D and 9-18-51-B, were of the type supplied with the Signal . Corps electrophotographic camera. After charging on a scorona-type conharging unit, using low corona current, the initial potentials and the potenticial-decay rates were measured with the vibrating-probe electrometer, within the plate in darkness and with the plate

BATTELLE MEMMORIAL INSTITUTE

#### 10-62

exposed to white light. The measurements were repeated at about tenminute intervals, between which the plates were exposed to room light.

The first readings were made after the plates had been kept in darkness for more than 50 hours. For the test, the plates were charged to an initial potential of about 90 volts. Under these conditions, the dark-decay rates were very low. After five successive runs, the initial potentials accepted by both plates under the charging conditions used dropped to about 70 volts; the dark-decay rates had increased about five times, the lightdecay rates remained essentially unchanged, and the residual potential remained below two volts. The light decay and the dark decay of the plates tended to level off at these values. However, the dark-decay rate of the fifth run was somewhat greater than desired.

Previously, it had been shown that exposure of places to intrared radiation sometimes decreases the rate of potential deca ' in darkness. Therefore, the series of tests was altered to include irradiation to infrared for five minutes after each complete cycle of charging, measuring darkdecay and light-decay rates, and exposure to room light. Under these conditions, the dark-decay rate only increased to about twice its original rate. The infrared energy was provided by a 350-watt, reflector-type heat lamp covered with a red filter having a short-wavelength sutoff at about 650 millimicrons, the lamp being located about six inches above the plate. Heat from the lamp increased the temperature of the plate and it may have been this, rather than the infrared radiation itself, that produced the improvement in plate characteristics. Such treatment may be detrimental to the plate, so that it cannot be recommended as a usual procedure. However, as it actually improved the reproducibility of plate characteristics, it was incorporated into the schedule for processing electrophotographs for the sensitometric study.

The final scheduling established for sensitometric tests is:

- (1) The plate is irradiated with infrared energy for five minutes.
- (2) Immediately after irradiation, the plate is charged to about 80 volts with a scorona-type charging unit, using low corona current.
- (3) About 15 seconds after charging, the plate is exposed to the gray-scale image for from 12 to 15 seconds.
- (4) About 15 seconds after exposure, the image is developed with the development unit which normally requires ten

BATTELLE MEMORIAL INSTITUTE

10-63 and 10-64

*/*•

()

ſ



98797



HATTELLE MEMORIAL INSTITUTE

#### 10-65

seconds to develop an average image., A 30-second development period was used to insure complete development in these tests.

- (5) The image is transferred to dye-transfer paper and the plate washed with Duponol and water, rinsed and dried, all in such time that the complete cycle requires ten minutes.
- (6) The cycle is repeated at least twice before pictures for the study are made.

<u>First Results</u>. In spite of the precautions taken to insure plates being treated the same way for each picture produced, the tone-reproduction curves obtained from different exposures were not so uniform as had been hoped. The shapes of the curves were similar, but the maximum reflection densities varied from 1.1 to 1.5.

Figure 10-22 is a typical tone-reproduction curve for the electrophotographic process using the powder-cloud development unit. On this graph, the points marked with circles are for the reflection density of the final picture versus the transmission density of the gray-scale transparency. The points marked with squares form the plot of final picture densities versus the negative logarithm to the base 10 of the relative illuminance incident on the plate, these units being directly comparable to density units. The latter plot includes a correction for nonuniformity of illumination of the gray scale and for the effect of flare light, as is described in detail in the following section.

As seen from Figure 10-22, the acceptance range of the present electrophotographic process is about 1.2 in units of the logarithm of subject brightness. This range is too short to reproduce detail both in shadow and highlight areas for most subjects found in nature.

The maximum contrast of the process, as represented in Figure 10-22, is equivalent to a gamma of 1.4. This value can be altered by changing the initial potential of the plate. However, the usefulness of this technique for changing gamma is limited because the maximum density of the picture is also determined by the initial potential of the plate. Thus, changing the plate potential to give a gamma of about 1.0 would result in the maximum density of the picture being less than 1.0. Because of this, electrophotographs frequently are too low in contrast and too short in brightness range. At the present time, electrophotographs judged to be best are those involving a compromise between contrast and the density range of the final image,

BATTELLE MEMORIAL INSTITUTE



CURVE FOR POWDER-CLOUD DEVELOPMENT UNIT A - 1300 FIGURE 10-22. TYPICAL TONE-REPRODUCTION

#### 10-67

1

ţ

<u>Nonuniformity of Development.</u> As seen in Figure 10-22, the points defining the tone-reproduction curves do not lie on a smooth curve. Scatter of the points is an indication of nonuniformity in the characteristics of some portion of the electrophotographic process--sensitization, exposure, development, or perhaps others. Some work has been done to determine which of these characteristics is most responsible for the nonuniformity observed.

First, more precise determinations were made of the actual amount of light reaching the plate during its exposure to the gray-scale image. A silver halide film was exposed to the image and then to an auxiliary step wedge under known light intensities. A plot of the densities produced by the known light intensities constitutes a characteristic curve of the film. From this characteristic curve, the light intensities given by the grayscale image can be read.

Measurements were made only of the relative intensities of the light in different density areas of the image produced by the standard exposure device. Therefore, it was not necessary to know the absolute values of light intensities in the auxiliary step wedge, a contact exposure from a gray-scale step wedge of known densities giving a sufficiently accurate measure of the relative light intensities.

In the first attempt, photographic enlarging paper was used, but it had an inadequate density range. Kodak Commercial film was satisfactory and was used for further tests.

Figure 10-23 shows the departure of the light intensities, as measured from the values that would have been predicted if the gray-scale illumination were uniform and the lens system ideal. Figure 10-22 includes a plot of a typical tone-reproduction curve using values corrected by the curve shown in Figure 10-23. As shown there, the correction of the lightintensity values did not greatly reduce the scatter of the data.

Uniformity of development can be determined simply by charging a plate uniformly to a potential somewhat lower than the usual potential for making pictures and then developing the plate without exposing it.

Figure 10-24 is a reproduction of a developed image showing nonuniformity of development. Selenium plate 9-18-51-B was charged to approximately 50 volts by passing the plate diagonally under the scorona unit, so that any nonuniformity of sensitization would show up as diagonal streaking. The plate was then developed with the powder-cloud development unit for 30 seconds and the image transferred to moist dye-transfer paper. The particular image reproduced in Figure 10-24, with the grid work added to facilitate measuring densities, shows greater nonuniformity than is usual

BATTELLE MEMORIAL INSTITUTE

د. م Actual performance FIGURE 10-23. CHARACTERISTICS OF STANDARD-EXPOSURE DEVICE 2.0 Ideal performance <u>.</u> 0 0 <u>9</u>. 0 a 1.0 1.2 1.4 Transparency Density 0 O **8**0 Ó Շ **0**0 40 0.2 0 0 4. 2 08 0.6 1.6 04 CI O 0 0.1

**RESTRICTED** 

- Log<sub>10</sub> Relative Illuminance

Ċ,

10-69 and 10-70



98795

FIGURE 10-24. IMAGE DEVELOPED ON UNIFORMLY CHARGED PLATE WITH POWDER-CLOUD DEVELOPMENT UNIT

RESTRICTED

HATTELLE

MEMORIAL INSTITUTE
#### 10-71

after this procedure. This image was selected because it exaggerates the type of nonuniformity of development produced in the powder-cloud development unit.

Figure 10-25 is a contour plc of the reflection densities of the image shown in Figure 10-24. This plot shows the characteristic heavy deposition of powder in two bands at the sides of the image, the relatively uniform development at the trailing edge, and the agglomerated deposit near the point where the cloud flows from the capillary tube. The dark bands at the sides of the image obtained powder from the portion of the flow that had become laminar before reaching the selenium-coated area; the lighter portion obtained powder from the flow that was still somewhat turbulent, that is, it was still producing a heavy deposit of powder on the brass backing plate when it reached the edge of the selenium. This effect can be seen clearly at the bottom edge of the reproduction in Figure 10-25.

#### Triboelectric Charging of Powder Clouds

The present technique for developing continuous-tone electrophotographs involves passing a powder cloud through a length of metal capillary tubing. This procedure was conceived originally as a way of electrically charging the particles in the powder cloud by means of the triboelectric effect. More recently, the role of the capillary tube appears to include other important functions, those of breaking up agglomerates in the powder cloud and providing a short transit time from the generator to the development region. Nevertheless, an understanding of the effect of the capillary tube is desirable.

A number of experiments were performed to obtain more information about how powder clouds are charged electrically while flowing through capillary tubes. These experiments were designed to show how the electrical charge produced on the powder particles in the cloud depends on tube length, tube diameter, and rate of flow.

The experimental arrangement used in these experiments consisted of the disposable-cloth-belt type of powder-cloud generator, fitted with an adapter so that different sizes of stainless steel capillary tubes could be attached. The powdered charcoal flowing from the capillary tubes was collected in a device for measuring the electrical charge on the particles in the cloud of powder. Capillary tubes tested had inside diameters of: 0.023, 0.020, 0.016, and 0.010 inch. For each diameter, tubes were tested in lengths of: 1.45, 2.55, 4.50, and 7.85 inches. The flow rates through the tube specimens were varied by applying gas pressures of 2.05, 3.6, 6.3, 11.1, 19.5, 34.2, and 60.0 psi to the powder-cloud generator. Not all these pressures were used with all the specimens, as, with some of the short

BATTELLE MEMOBIAL INSTITUTE

그렇지, 것이 아파가 가지 않는 것이 있는 것이 가지 않는 것이 같이 있다.

# RESTRICTED



FIGURE 10-25. REFLECTION DENSITIES ON UNIFORMLY CHARGED PLATE DEVELOPED WITH POWDER-CLOUD UNIT

A-1302

#### 10-11

tubes, the higher pressures gave inconveniently high flow rates, and because some of the lower pressures did not produce sufficient electrical charge on the powder cloud to be measured accurately. Between three and eight readings were made for each set of conditions.

Figure 10-26 is a plot of most of the data obtained in this series of experiments. In this plot, the specific electrical charge of  $t^{h}$  powder, that is, the amount of electrical charge per unit weight of powder in the cloud, is plotted against the Reynolds number for the flow through the different tubes.

The most obvious information obtainable from Figure 10-20 is that the data are not consistent. The variations in the electrical charge are undesirable in themselves, and they may obscure trends that otherwise might have been uncovered in this work.

The electrical charge produced on the powder cloud did not depend on the length of the tube within the limits of this test. The electrical charge obtained with the tube 1.45 inches long, for a given diameter and a given rate of flow, was about the same as the electrical charge obtained from the tube 7.85 inches long for the same rate of flow. This was true for tubes of all four diameters tested.

For a constant Reynolds number for the flow through the tubes, tubes having smaller diameters produced more electrical charge on the particles of powder than did the large tubes. For example, the amount of electrical charge produced for the same Reynolds number with flow through the tube 0.010 inch in diameter was roughly twice that produced by flow through the tube 0.020 inch in diameter. The mean velocity of the flow through the smaller tube was twice that through the larger tube. This observation suggests that the electrical charging may be chiefly a function of the force with which a particle strikes the wall of the tube, which, for highly turbulent flow with a Reynolds number much greater than 2200, would be proportional to the mean velocity of flow through the tube.

The results of these experiments suggested several modifications to the high-speed development technique. One such modification, shortening the capillary tube used in the development unit, has been tried. The capillary tube having a diameter of 0.023 inch, which was usually six to eight inches long, was shortened to 1.6 inches. The pressure of the Freon gas was then reduced until the flow rate through the shorter tube was about the same as with the longer tube. In all other respects, the development technique was left unchanged.

Figure 10-27 is a reproduction of an electrophotograph produced using the capillary tube only 1.6 inches long. The original of this picture

HATTELLE MEMORIAL INSTITUTE

10--74



#### 10-75 and 10-76



98796

FIGURE 10-27. ELECTROPHOTOGRAPH DEVELOPED WITH POWDER-CLOUD UNIT USING CAPILLARY TUBE 1.6 INCHES LONG



10-77

and all of the electrophotographs produced using the short capillary tube are apparently similar in quality to those obtained with the longer tube of the same internal diameter. As described later in this report, images of very high quality have been prepared using capillary tubes as short as onesixteenth inch. The need is evident for more fundamental information on the mechanism of charging particles of powder electrically while they are suspended in a gas.

#### Study of Characteristics of Development Process

<u>The Formation of Agglomerates in Aerosols.</u> Deposition of large particles of powder on the plate during powder-cloud development constitutes one of the difficulties yet to be overcome in electrophotography. These large particles, appearing on the final prints, may be large chunks of charcoal that existed in the original powder, or they may be agglomerates that either were not broken up in the capillary tubes, or that formed between emission of the cloud from the capillary tubes and deposition of the powder on the electrophotographic plate. The probability that agglomeration may be important led to the investigation of this general problem in handling aerosols. This study is still in progress and conclusive results cannot be given.

In an aerosol, the particles of material are in perpetual "Brownian" motion due to continual molecular bombardment. Whether the aerosol is at rest or moving, the Brownian motion provides ample opportunity for collisions between particles. In the study of aerosols, it has been generally assumed that all or most of such collisions result in a combination of the two particles to form an agglomerate. This almost universal coalescing of particles on contact may be due to electrical forces, Van der Waal forces, or mechanical adhesion.

The density of the powder cloud, the size of the particles of material, and the time interval between the forming and using of the cloud, are only a few of the factors known to determine the degree of agglomeration in an aerosol. A number of other factors are of importance.

When any two particles collide and remain in contact, a primary agglomerate is formed. This new particle increases the heterogeneity of the aerosol, and, since the heavier particle responds less readily to Brownian forces than does a single particle, there will exist a difference of velocity between the lighter and the heavier particles. In this fashion, additional particles strike the primary agglomerate and adhere to it. If the time interval is long enough and the electrical forces negligible, the larger particles may settle out, leaving a less densely populated volume in which new collisions can occur.

BATTELLE MEMORIAL INSTITUTE

#### 10-78

In 1917, M. Von Smoluckowski(10-1) made a theoretical study of the process of agglomeration in aerosols. Assuming a homogeneous distribution of uncharged particles and using only the diffusion caused by Brownian movements, he arrived at an equation for the rate at which agglomeration would reduce the number of particles originally present in the suspension. If short periods of time are considered, then this equation, with minor changes, can be used to determine the rate of formation of primary twoparticle agglomerates in the aerosol. In this form, the equation becomes:

$$\frac{\mathrm{dn}}{\mathrm{dt}} = \frac{2 \mathrm{RT} \mathrm{n}_{\mathrm{O}}^2}{3 \mathrm{N}_{i'}} \quad (10-1)$$

where <u>R</u> is the universal gas constant, <u>n</u> is the number of agglomerates, <u>T</u> is the absolute temperature,  $a_0$  is the original number of particles per cubic centimeter, <u>N</u> is Avagadro<sup>+</sup>, number, and <u>r</u> is the viscosity of the gas.

This equation gives the minimum rate at which agglomerates would form in an aerosol. It is interesting to note that the rate is independent of the size of the particles but is proportional to the square of the number of particles. The rate is proportional to the first power of the absolution perature, which makes it relatively independent of normal temperature changes for usual ambient conditions.

If approximate values are substituted in Equation 10-1, an estimate can be made of a lower limit of agglomeration. Using 8.31 ergs per degree centigrade per gram molecular weight for R, an absolute temperature of 300 K, 186 x 10<sup>-6</sup> poise for the viscosity of the gas,  $\mu$ , and 6.023 x 10<sup>23</sup> for the number of molecules per gram molecular weight, Equation 10-1 becomes:

 $\frac{dn}{dt} = 1.49 n_0^2 \times 10^{-10} . \qquad (10-2)$ 

A value for  $\underline{n}_0$  can be arrived at by logical assumptions concerning the powder used. The particles were assumed to be 2.5 microns in diameter, and the density of the particles was estimated at 1.45 by noting that the particles float in a liquid having a density of 1.50, but sink in one having a density of 1.40. Using these data, and the observed fact that in the cloth-belt powder-cloud generator, about 0.2 milligram of powder is blown into every 60 cubic centimeters of gas, there are about 2.8 x 10<sup>5</sup> particles per cubic centimeter of aerosol. Using this figure in Equation 10-2 gives a value of only 12 agglomerates formed per second for each

(10-1), Von Smoluckowski, M., Versuch einer mathematischen Theorie der Koagulationkinetik Kolloider Lösungen: 3, physik, Chem., Vol 92, 1917, pp 129-168.

BATTELLE MEMORIAL INSTITUTE

10-79

cubic centimeter of aerosol. This is negligibly small, which means only that there is a possibility that agglomeration is not a serious problem in the practical case; it certainly does not prove the absence of agglomeration from causes not accounted for in this calculation. For example, this consideration takes no account of the electrical charges known to be present on particles in powder-cloud development.

The attempted calculation of agglomeration in powder clouds was considerably more difficult than anticipated originally. Important factors such as turbulence in the cloud and the electrical charge on particles could not be taken into account. A number of tests were outlined to determine how the density of the powder cloud affects the amount of agglomeration. Images were developed with the high-speed, continuous-tone powder-cloud development device and examined under a microscope. A question arose immediately as to whether the large areas of powder observed were large single particles or agglomerates of smaller ones. To settle this question, the size of the original powder particles was determined by making a series of electron photomicrographs of particles of charcoal which had been ball milled for 48 hours.

Figure 10-28 is a typical example of one of these photomicrographs. The particles seem to be of all sizes, ranging from barely perceptible ones even at this magnification of 10,820X, to particles so large that a single one fills the entire field. A particle count and a particle-size determination were made on ten different fields containing a total of 993 countable particles. An analysis of this count showed that over 90 per cent of the powder particles had diameters of less than one micron. This indicated that most of the powder comes from the ball mill as small discrete particles, but it did not rule out the possibility of an occasional large particle. However, not enough large particles were observed to account for the many large particles seen on developed electrophotographic plates.

Following ball milling, the powder was put into a powder-cloud generator which metered and deposited it on a moving cloth belt. To determine if agglomeration is caused by the powder-cloud generator itself, photomicrographs were made of the output from the generator.

Figure 10-29 is a photomicrograph of the powder as it comes from the original powder-cloud generator. Agglomerates as large as 20 to 30 microns appear in this picture, showing that some agglomeration occurs in generating the powder cloud. These agglomerates are approximately the size of some of the "pepper" observed on developed electrophotographs.

Another experiment raised doubts as to whether the original powdercloud generator is the main source of agglomerates. In this experiment, the output of the powder-cloud generator was passed through a settling

BATTELLE MEMORIAL INSTITUTE

10 - 80

Ū.

chamber and then to the device used to coat the cloth belts for the developing apparatus. Photomicrographs were then made of the particles not removed by the settling chamber.

Figure 10-30 shows the arrangement used to separate the large and small particles. Applying Stokes' in for a body falling in a viscous medium, it was calculated that a volume of 12 liters of air per minute passing through this particular chamber. carrying powdered charcoal having a density of approximately 1.45, would cause settling of particles larger than five microns in diameter. Particles smaller than this would pass through the chamber to the device for impregnating the developer belt.

Figure 10-31 is a photomic regraph of the powder as it came from the settling drum. Comparing this figure with Figure 10-23, it is obvious that most of the larger particles had been removed from the cloud of passing through the settling chamber.

Particle counts and particle-size determinations on the samples used for Figures 10-29 and 10-31 revealed that 10 per cent of the powder particles entering the settling chamber had diameters of five microns or larger, while only 0.7 per cent of the particles leaving the chamber had a diameter larger than five microns.

Using the device just described, two belts were impregnated with charcoal powder, one with powder directly from the powder-cloud gene itor, the other with powder from the settling drum. Distinct differences were observed between the images developed with these two different belts.

Pictures developed with unseparated powder appeared "normal" as expected. Pictures developed with the smaller particles showed excessive contrast and some halo around dark objects. Both of these characteristics have been associated with low electrical charges on the powder particles. A possible explanation for this low charge is given later.

More important to the study at hand was the observation that the number of large agglomerates in the pictures developed with the fine particles was about the same as in pictures developed with the powder containing many larger particles. It was concluded that agglomeration of powder occurs somewhere in the development apparatus or as a result of the powder being deposited on the cloth belt. Further study must be made to locate the exact source of agglomerates. But it has now been established that the large particles appearing on the print are agglomerates, and that they are formed either during the coating of the belts or later in the development procedure.

#### BATTELLE MEMORIAL INSTITUTE

10-81 and 10-82



1.0μ 10820X

E-3208-A 95439

INSTITUTI

an réélé s to étable alles

FIGURE 10-28. ELECTRON PHOTOMICROGRAPH OF CHARCOAL BALL MILLED FOR 48 HOURS



10-83 and 10-84



450X

1

1

10.0μ

95440

and the second second

FIGURE 10-29. PHOTOMICROGRAPH OF POWDER PARTICLES PRODUCED BY POWDER-CLOUD GENERATOR

#### BATTELE MEMORIAL INSTITUTE

10-85



FIGURE 10-30. POWDER-SETTLING DEVICE USED TO REMOVE LARGE POWDER PARTICLES FROM OUTPUT OF POWDER-CLOUD GENERATOR

**RESTRICTED** 

A-3101

10-86

An explanation of the supercents caller charges on the finer particles may be found in a recent article  $L_2$  W. B. Kunkel(10-2). According to Kunkel, small particles are cushioned by the nolecules of air and may not strike the capillary charging tobe in the same manner or as frequently as larger particles, and, hence, bouid acquire a lower electrical charge In general, the charge on a particle superconduct rease almost as the square of the diameter of compared compared by

The Movement of Powder Particles in the Development Region. In powder-cloud development, a powder image is produced on the electrophotographic plate because electrically charged particles are drawn out of the powder cloud by the electrical field created between the plate and the development electrode. If the cloud were stationary during development of an image, the motion of powder particles would be simple, and probably would not be a factor in determining the quality of the images produced. However, since the cloud must move during development, the particles must describe curved trajectories in moving to the plate, and the final distribution of powder on the image must be the result of a complicated dynamic situation.

In performing the work reported in this section, it was assumed that an understanding of the behavior of in lividual particles of powder in the development zone would provide a better understanding of the whole process of powder-cloud development, and, hence, would lead to practical improvements in powder-cloud development. Representing an initial attack on the problem of the dynamics of powder-cloud development, the following serves to open the field for future investigation, ir addition to giving several interesting and valuable results.

In a first approach to the problem of was assumed that: (1) the powder cloud moves in simple laminar flow between the plate and the development electrode, (2) the particles of powder are uniform in size and electrical charge, (3) the motion of the particles does not disturb the movement of gas, and (4) the movements of the particles at right angles to the flow of gas are completely independent of their movement with the laminar flow of gas. More-sophisticated ass imptions may be possible later.

Figure 10-32 shows the region between the electrophotographic plate and the development electrode with the parabolic distribution of gas velocity characteristic of laminar flow. The origin of the coordinate system is placed midway between the plates for convenience. For this situation, where  $v_x$  is the velocity parallel to the flow of the gas between the plates,

(10 2). Kunkel, W. B., The Static Electrification of Dust Particles on Dispersion Into a Cloud: J. Applied Phys., Vol 21, August, 1950, pp 820-832.

BATTELLE MEMORIAL INSTITUTE

10-87 and 10-88



450X

;

10.0μ ⊨--- 95441

FIGURE 10-31. PHOTOMICROGRAPH OF POWDER PARTICLES LEAVING CYLINDRICAL SETTLING DRUM



HAFTFLLE MEMORIAL INSTITUTE

#### 10-89

 $\underline{\mathbf{y}}$  is the distance from the center of the plates and at right angles to the plates,

$$\mathbf{v}_{\mathbf{x}} = -\mathbf{a}\mathbf{y}^2 + \frac{3}{2}\,\overline{\mathbf{V}}_{\mathbf{x}} \quad (10-3)$$

In this equation, a is an arbitrary constant to be evaluated later, and  $3/2 \ \overline{V_x}$  is the maximum gas velocity between the plates expressed in terms of  $\overline{V_x}$ , the average velocity of the gas between the plates which can be determined from the total flow of gas and the distance between the plate and the development electrode. The constant a can be evaluated by considering that at y = Y/2,  $v_x = 0$ ,  $a = 6\overline{V_x}/Y^2$ . The expression becomes:

$$\mathbf{v}_{\mathbf{x}} = -\frac{6}{\mathbf{Y}^2} \,\overline{\mathbf{V}}_{\mathbf{x}} \mathbf{Y}^2 + \frac{3}{2} \,\overline{\mathbf{V}}_{\mathbf{x}} \, \cdot \tag{10-4}$$



FIGURE 10-32. DISTRIBUTION OF GAS VELOCITY BETWEEN ELECTROPHOTOGRAPHIC PLATE AND DEVELOPMENT ELECTRODE

In this equation,  $\underline{y}$  is actually a function of time,  $\underline{t}$ , and  $\underline{y} = V_{\underline{y}}t$ , since it is assumed that the particle under consideration is moving parallel to the  $\underline{y}$  axis with a uniform velocity,  $\underline{V}_{\underline{y}}$ . Using this relationship, Equation 10-4 becomes:

$$v_x = -\frac{6}{Y^2} \overline{V}_x V_y^2 t^2 + \frac{3}{2} \overline{V}_x \cdot$$
 (10-5)

Now, the actual position,  $\underline{x}$ , of the particle starting from the origin of the coordinate system is given by:



10 - 90

$$\mathbf{x} = \int \mathbf{V}_{\mathbf{x}} d\mathbf{t} = -\frac{6}{\mathbf{Y}^2} \overline{\mathbf{V}}_{\mathbf{x}} \mathbf{V}_{\mathbf{y}}^2 \int t^2 d\mathbf{t} + \frac{3}{2} \overline{\mathbf{V}}_{\mathbf{x}} \int d\mathbf{t} + C \quad , \qquad (10-6)$$

or,

$$\mathbf{x} = -\frac{2}{Y^2} \overline{V}_{\mathbf{x}} V_{\mathbf{y}}^2 t^3 + \frac{3}{2} \overline{V}_{\mathbf{x}} t + C \quad . \tag{10-7}$$

In this equation, <u>C</u> equals zero, because it was assumed that <u>x</u> is zero when <u>t</u> is zero. Again, expressing <u>x</u> in terms of <u>y</u> rather than in terms of <u>t</u>,

$$\mathbf{x} = -\frac{2}{\mathbf{Y}^2} \frac{\overline{\mathbf{V}}_{\mathbf{x}}}{\mathbf{V}_{\mathbf{y}}} \mathbf{Y}^3 + \frac{3}{2} \frac{\overline{\mathbf{V}}_{\mathbf{x}}}{\mathbf{V}_{\mathbf{y}}} \mathbf{Y} \cdot$$
(10-8)

This same equation will hold for negative values of  $\underline{y}$  since the whole system is symmetrical with respect to the midplane between the two plates.

To obtain numerical results from Equation 10-8, it is necessary to know values for Y, the space between the electrophotographic plate and the development electrode;  $\overline{V}_{x}$ , the mean velocity of gas flowing in the development regions; and  $\underline{V}_{y}$ , the velocity of the particle normal to the plate as produced by the electrical field resulting from the electrical charge on the electrophotographic image.

Although calculated in a subsequent section of this report, the following values of these constants will be used here to preserve continuity in the practical implications of the present calculations -- Vy = 58 centimeters per second,  $\overline{Vx} = 450$  centimeters per second, and  $\underline{Y} = 0.038$  centimeter. Using these constants, Equation 10-8 becomes:

$$x = -10730 / y^3 + 11.62 y$$
 (10-9)

Figure 10-33 shows a plot of the results of this equation for the case of powder flowing between an electrophotographic plate and a development electrode. According to these assumptions, powder moves as far as three millimeters past the edge of an electrically charged area of the plate. However, it must be remembered that this represents only a transient condition, and that, as soon as powder builds up on the plate, the electrical field between the plate and the development electrode is reduced until it approaches zero for complete development. In this case, the flow of powder is seriously altered. Again, powder will not continue to move toward the plate after the powder passes beyond the boundary of an electrically charged area.

BATTELLE MEMORIAL INSTITUTE



RESTRICTED

÷

t.

۱

Ĵ

10-92

This calculation represents the first attempt to study the dynamics of powder-cloud development. It is heped that further calculations and experimental verifications will give greater understanding of the process and lead to practical improvements.

Electrical Charge on Particles. From past measurement, of powder particles, it is reasonable to assume a particle diameter of two microns and a specific electrical charge of 40 microcoulombs per gram of developer powder. To find the weight of a single particle of powder, it is necessary to know the density of the material. This was determined as approximately 1.45 by finding that particles float in a liquid having a density of 1.50 but sink in a liquid having a density of 1.40. Using the figure of 1.45, the mass of a two-micron particle was calculated to be 6.1 x  $10^{-12}$  gram, or there are  $1.64 \times 10^{11}$  particles per gram of powder. From this number, it can be calculated that there is an electrical charge of 24.4 x  $10^{-17}$ coulomb per particle. Arbitrarily, it was assumed that the particles to be studied would have three times the amount of this charge, this factor being used to allow for particles having zero or reverse-polarity electrical charges. With this factor, the charge on the particle is 2.16 x  $10^{-6}$ electrostatic unit.

Using a potential crop of 100 volts across the space of 0.038 centimeter between the electrophotographic plate and the development electrode gives a potential gradient which would produce 8.75 dynes force on a unit charge in the field. This, combined with the figure for the charge on the particle of powder, gives a force of 1.89 x  $10^{-5}$  dyne on the average particle.

A brief calculation of  $\exists f \in \zeta$  savitational zonce on this particle showed it to be negligible as compare on with the electrical force.

To calculate the velocity of motion in the development zone, a modification of Stekes' law was used in the form

$$\mathbf{F} = \frac{3\eta\mu \vee \mathbf{D}}{\mathbf{K}_{\mathbf{m}}} , \qquad (10-10)$$

where  $\underline{F}$  is the drag on the particle in dynes,  $\underline{\mu}$  is the viscosity of the fluid,  $\underline{v}$  is the velocity of the particle,  $\underline{D}$  is the diameter of the particle, and  $\underline{Km}$ is a correction factor whose value is 1.08 for the size of particle involved here.

Solving Equation 10-10 for the velocity of the particle using the above values, gives 58 centimeters per second for the velocity of the particle moving toward the plate in the development region. <sup>5</sup>It is interesting to note that, at this velocity, only about 0.7 millisecond will be required for the

BATTELLE MEMORIAL INSTITUTE

10-93

particle to travel the whole distance between the development electrode and the plate. Even so, calculations reported above indicate that the particle would have moved about three millimeters along the plate during its travel through the development zone.

Velocity of the Gas in the Development Zone. From previous data on the amount of gas flowing from the development zone, it can be calculated that the average velocity of the gas in this region is about 450 centimeters per second.

#### Prevention of Unwanted Deposition of Powder

<u>Constant-Velocity Transition Zone</u>. In the high-speed development unit, transition between the turbulent flow in the capillary tube and the laminar flow in the development region is carried out in a simple, relatively effective device. However, this device does not distribute the powder cloud uniformly and it collects a large quantity of powder. It had been proposed that this accumulation of powder might be prevented if the transition from turbulent to laminar flow could be carried out in two separate steps.

The first step would be a constant-velocity transition from turbulent to laminar flow, accomplished by using a fishtail-shaped transition section of constant cross-section area to pass from the circular tube to a flat, rectangular tube. No powder should deposit in this region because of the high velocity of the air flow. The second step would be a transition from the high-velocity laminar flow to the low velocity of gas useful in developing electrophotographs. No powder should deposit in this region because of the laminar flow.

This type of transition zone has been tested and found only partly satisfactory. The device constructed to test this type of transition was formed by clamping together two plates of cold-rolled steel 3/6 inch thick, about 6 inches wide by 15 inches long. Shims of the proper thickness and shape were placed between the edges of the two plates to form the desired fishtail-shaped ducts. The plates were clamped together with 1/4-inch screws spaced one inch apart to insure the plates being well seated on the shims. The surfaces of the plates were smoothed with emery cloth and polishing paper to remove any rough spots or burrs.

Figure 10-34 is a sketch showing the internal dimensions and shape of the fishtail duct. The design of the duct is somewhat arbitrary. Except for the important considerations that, in the first part of the duct, the cross-sectional area must be kept roughly constant and that, throughout the device, the direction and velocity of the flow must not change rapidly, the design was governed largely by duct thicknesses that could be produced

HATTELLE MEMORIAL INSTITUTE

:

5

į

<u>с.</u>с

c.



**RESTRICTED** 

TE

0

A- 1303

1

#### 10-95

by a slight deformation of rigid steel plates. The duct was intended to operate using a powder cloud of charcoal in air flowing at free-air-flow rates of less than six liters per minute. Approximate values of the Reynolds number are noted in Figure 10-34 for several points along the duct. These values are for a free-air-flow rate of about five liters per minute.

Testing the device consisted of passing a powder cloud through it for a minute or longer and then dismantling the device for inspection. In every trial, inspection revealed some accumulation of powder in the duct. Some powder deposited rather uniformly in the cross-hatched region shown in Figure 10-34. This deposition suggests that the diffuser portion of the duct expanded too rapidly to preserve laminar flow. Presumably, this difficulty might be overcome by lengthening the duct. A more troublesome deposition of powder occurred in the high-velocity, laminar-flow portion of the duct. There, for unknown reasons, islands of powder formed slowly, eventually bridging the narrow gap between the plates and causing pronounced streaking. However, no simple change in the design will eliminate this difficulty. For this reason, and since modifications of the device to eliminate deposition in the diffuser section would lead to a rather cumbersome device requiring high air pressures, no further work on this device is warranted at this time.

()

1

<u>Porous-Plate Development Electrode.</u> In the high-speed development unit, the most objectionable deposits of unwanted powder occur where the powder emerges from the capillary tubing and impinges on the plate and on the development electrode. This deposition possibly could be prevented by blowing air through the back of a porous metal electrode into the development chamber.

To try this, a portion of the development electrode was replaced by a sheet of porous stainless steel(1) 0.032 inch thick. During the development process, air was blown through this porous plate into the development chamber and a jet of powder from the capillary tube was arranged to impinge directly onto this porous plate. The amount of powder deposited on the porous plate was reduced substantially when air was passing through the plate. However, some powder did deposit on the plate. Microscopic examination of the porous steel plate revealed that the pores occupied only about one-fifth of the total area of the plate surface and that the pores were as much as 0.005 inch apart. A material having a greater porosity undoubtedly would give better results, but such a material has not been obtained. Two other manufacturers were contacted, but their products had still lower porosities.

If powder were fed into the development chamber parallel to the development electrode rather than directly against it, the present porous material might give satisfactory results. This approach has not been investigated.

(1) Manufactured by the Miero Metallic Corporation, 30 Sea Cliff Avenue, Glen Cove, New York,

BATTELLE MEMORIAL INSTITUTE

#### 10-75

tubes, the higher pressures gave inconveniently high flow rates, and because some of the lower pressures did not produce sufficient electrical charge on the powder cloud to be measured accurately. Between three and eight readings were made for each set of conditions.

Figure 10-26 is a plot of most of the data obtained in this series of experiments. In this plot, the specific electrical charge of  $t^{h}$  powder, that is, the amount of electrical charge per unit weight of powder in the cloud, is plotted against the Reynolds number for the flow through the different tubes.

The most obvious information obtainable from Figure 10-20 is that the data are not consistent. The variations in the electrical charge are undesirable in themselves, and they may obscure trends that otherwise might have been uncovered in this work.

The electrical charge produced on the powder cloud did not depend on the length of the tube within the limits of this test. The electrical charge obtained with the tube 1.45 inches long, for a given diameter and a given rate of flow, was about the same as the electrical charge obtained from the tube 7.85 inches long for the same rate of flow. This was true for tubes of all four diameters tested.

For a constant Reynolds number for the flow through the tubes, tubes having smaller diameters produced more electrical charge on the particles of powder than did the large tubes. For example, the amount of electrical charge produced for the same Reynolds number with flow through the tube 0.010 inch in diameter was roughly twice that produced by flow through the tube 0.020 inch in diameter. The mean velocity of the flow through the smaller tube was twice that through the larger tube. This observation suggests that the electrical charging may be chiefly a function of the force with which a particle strikes the wall of the tube, which, for highly turbulent flow with a Reynolds number much greater than 2200, would be proportional to the mean velocity of flow through the tube.

The results of these experiments suggested several modifications to the high-speed development technique. One such modification, shortening the capillary tube used in the development unit, has been tried. The capillary tube having a diameter of 0.023 inch, which was usually six to eight inches long, was shortened to 1.6 inches. The pressure of the Freon gas was then reduced until the flow rate through the shorter tube was about the same as with the longer tube. In all other respects, the development technique was left unchanged.

Figure 10-27 is a reproduction of an electrophotograph produced using the capillary tube only 1.6 inches long. The original of this picture

MEMORIAL

BATTELLE

INSTITUTE

AND THE PARTY OF A SAME AND A SAME

10--74



ĔŚŦŔĨĊŤĔ

R

A-1304

TUTE

#### 10-75 and 10-76

Ċ T 2 ah . 🖬 Í

98796

FIGURE 10-27. ELECTROPHOTOGRAPH DEVELOPED WITH POWDER-CLOUD UNIT USING CAPILLARY TUBE 1.6 INCHES LONG



BATTELLE MEMORIAL INSTITUTE

10-77

and all of the electrophotographs produced using the short capillary tube are apparently similar in quality to those obtained with the longer tube of the same internal diameter. As described later in this report, images of very high quality have been prepared using capillary tubes as short as onesixteenth inch. The need is evident for more fundamental information on the mechanism of charging particles of powder electrically while they are suspended in a gas.

#### Study of Characteristics of Development Process

<u>The Formation of Agglomerates in Aerosols</u>. Deposition of large particles of powder on the plate during powder-cloud development constitutes one of the difficulties yet to be overcome in electrophotography. These large particles, appearing on the final prints, may be large chunks of charcoal that existed in the original powder, or they may be agglomerates that either were not broken up in the capillary tubes, or that formed between emission of the cloud from the capillary tubes and deposition of the powder on the electrophotographic plate. The probability that agglomeration may be important led to the investigation of this general problem in handling aerosols. This study is still in progress and conclusive results cannot be given.

In an aerosol, the particles of material are in perpetual "Brownian" motion due to continual molecular bombardment. Whether the aerosol is at rest or moving, the Brownian motion provides ample opportunity for collisions between particles. In the study of aerosols, it has been generally assumed that all or most of such collisions result in a combination of the two particles to form an agglomerate. This almost universal coalescing of particles on contact may be due to electrical forces, Van der Waal forces, or mechanical adhesion.

The density of the powder cloud, the size of the particles of material, and the time interval between the forming and using of the cloud, are only a few of the factors known to determine the degree of agglomeration in an aerosol. A number of other factors are of importance.

When any two particles collide and remain in contact, a primary agglomerate is formed. This new particle increases the heterogeneity of the aerosol, and, since the heavier particle responds less readily to Brownian forces than does a single particle, there will exist a difference of velocity between the lighter and the heavier particles. In this fashion, additional particles strike the primary agglomerate and adhere to it. If the time interval is long enough and the electrical forces negligible, the larger particles may settle out, leaving a less densely populated volume in which new collisions can occur.

BATTELLE MEMORIAL INSTITUTE

#### 10-78

In 1917, M. Von Smoluckowski(10-1) made a theoretical study of the process of agglomeration in aerosols. Assuming a homogeneous distribution of uncharged particles and using only the diffusion caused by Brownian movements, he arrived at an equation for the rate at which agglomeration would reduce the number of particles originally present in the suspension. If short periods of time are considered, then this equation, with minor changes, can be used to determine the rate of formation of primary twoparticle agglomerates in the aerosol. In this form, the equation becomes:

$$\frac{\mathrm{dn}}{\mathrm{dt}} = \frac{2\mathrm{R'In_{C}}^{2}}{3\mathrm{N}_{i}} \qquad (10-1)$$

where <u>R</u> is the universal gas constant, <u>n</u> is the number of agglomerates, <u>T</u> is the absolute temperature (1, 2) the or much moder of particles per cubic centimeter, <u>N</u> is Avagadra 5 another, and <u>r</u> is the viscosity of the gas.

This equation gives the minimum rate at which agglomerates would form in an aerosol. It is interesting to note that the rate is independent of the size of the particles but is proportional to the square of the number of particles. The rate is proportional to the first power of the absolute temperature, which makes it relatively independent of normal temperature changes for usual ambient conditions.

If approximate values are substituted in Equation 10-1, an estimate can be made of a lower limit of agglomeration. Using 8.31 ergs per degree centigrade per gram molecular weight for R, an absolute temperature of 300 K, 186 x 10<sup>-6</sup> poise for the viscosity of the gas,  $\mu$ , and 6.023 x 10<sup>23</sup> for the number of molecules per gram molecular weight, Equation 10-1 becomes:

 $\frac{dn}{dt} = 1.49 n_0^2 \times 10^{-10} . \qquad (10-2)$ 

A value for no can be arrived at by logical assumptions concerning the powder used. The particles were assumed to be 2.5 microns in diameter, and the density of the particles was estimated at 1.45 by noting that the particles float in a liquid having a density of 1.50, but sink in one having a density of 1.40. Using these data, and the observed fact that in the cloth-belt powder-cloud generator, about 0.2 milligram of powder is blown into every 60 cubic centimeters of gas, there are about 2.8 x  $10^5$ particles per cubic centimeter of aerosol. Using this figure in Equation 10-2 gives a value of only 12 agglomerates formed per second for each

 (10-1). Von Smoluckowski, M., Versuch einer mathematischen Theorie der Koagulationkinetik Kolloider Lösungen: S. physik, Chem, Vol 92, 1917, pp 129-168.

BATTELLE MEMORIAL INSTITUTE

10-79

cubic centimeter of aerosol. This is negligibly small, which means only that there is a possibility that agglomeration is not a serious problem in the practical case; it certainly does not prove the absence of agglomeration from causes not accounted for in this calculation. For example, this consideration takes no account of the electrical charges known to be present on particles in powder-cloud development.

The attempted calculation of agglomeration in powder clouds was considerably more difficult than anticipated originally. Important factors such as turbulence in the cloud and the electrical charge on particles could not be taken into account. A number of tests were outlined to determine how the density of the powder cloud affects the amount of agglomeration. Images were developed with the high-speed, continuous-tone powder-cloud development device and examined under a microscope. A question arose immediately as to whether the large areas of powder observed were large single particles or agglomerates of smaller ones. To settle this question, the size of the original powder particles was determined by making a series of electron photomicrographs of particles of charcoal which had been ball milled for 48 hours.

Figure 10-28 is a typical example of one of these photomicrographs. The particles seem to be of all sizes, ranging from barely perceptible ones even at this magnification of 10,820X, to particles so large that a single one fills the entire field. A particle count and a particle-size determination were made on ten different fields containing a total of 993 countable particles. An analysis of this count showed that over 90 per cent of the powder particles had diameters of less than one micron. This indicated that most of the powder comes from the ball mill as small discrete particles, but it did not rule out the possibility of an occasional large particle. However, not enough large particles were observed to account for the many large particles seen on developed electrophotographic plates.

Following ball milling, the powder was put into a powder-cloud generator which metered and deposited it on a moving cloth belt. To determine if agglomeration is caused by the powder-cloud generator itself, photomicrographs were made of the output from the generator.

Figure 10-29 is a photomicrograph of the powder as it comes from the original powder-cloud generator. Agglomerates as large as 20 to 30 microns appear in this picture, showing that some agglomeration occurs in generating the powder cloud. These agglomerates are approximately the size of some of the "pepper" observed on developed electrophotographs.

Another experiment raised doubts as to whether the original powdercloud generator is the main source of agglomerates. In this experiment, the output of the powder-cloud generator was passed through a settling

BATTELLE MEMORIAL INSTITUTE

10-80

ç

chamber and then to the device used to coat the cloth belts for the developing apparatus. Photomicrographs were then made of the particles not removed by the settling chamber.

Figure 10-30 shows the strangement used to separate the large and small particles. Applying Stoken' for a could falling in a viscous medium, it was calculated that a volume of 12 hores of air per minute passing through this particular chamber, carrying powdered charcoal having a density of approximately 1.45 would cause settling of particles larger than five microns in diameter. Particles smaller than this would pass through the chamber to the device for impregnating the developer belt.

Figure 10-31 is a photonic regraph of the powder as it came from the settling drum. Comparing the figure with Figure 10-23, it is obvious that most of the larger particles had been removed from the cloud of passing through the settling chamber.

Particle counts and particle-size determinations on the samples used for Figures 10-29 and 10-31 revealed that 10 per cent of the powder particles entering the settling chamber had diameters of five microns or larger, while only 0.7 per cent of the particles leaving the chamber had a diameter larger than five microns.

Using the device just described, two belts were impregnated with charcoal powder, one with powder directly from the powder-cloud gene itor, the other with powder from the settling drum. Distinct differences were observed between the images developed with these two different belts.

Pictures developed with unseparated powder appeared "normal" as expected. Pictures developed with the smaller particles showed excessive contrast and some halo around dark objects. Both of these characteristics have been associated with low electrical charges on the powder particles. A possible explanation for this low charge is given later.

More important to the study at hand was the observation that the number of large agglomerates in the pictures developed with the fine particles was about the same as in pictures developed with the powder containing many larger particles. It was concluded that agglomeration of powder occurs somewhere in the development apparatus or as a result of the powder being deposited on the cloth belt. Further study must be made to locate the exact source of agglomerates. But it has now been established that the large particles appearing on the print are agglomerates, and that they are formed either during the coating of the belts or later in the development procedure.

0

5

; ;

#### 10-81 and 10-82

3



10820X 1.0µ

E-3208-A 95439

> . ได้มีสมับสีมายากการเป็นสาวารเรียงการเกิดเสียงไปประกัดเหลือการเกิดการเป็นการเป็นสาวารเกิดเรื่องเป็นสีมีสี

FIGURE 10-28. ELECTRON PHOTOMICROGRAPH OF CHARCOAL BALL MILLED FOR 48 HOURS



#### 10-83 and 10-84

. . ., .



450X

. . . 2

1

4

10.0μ

95440

FIGURE 10-29. PHOTOMICROGRAPH OF POWDER PARTICLES PRODUCED BY POWDER-CLOUD GENERATOR



# **`**Ŕ**Ĕ**ŚŤŔÌĊ**ŤĔ**Ď

FIGURE 10-30. POWDER-SETTLING DEVICE USED TO REMOVE LARGE POWDER PARTICLES FROM OUTPUT OF POWDER-CLOUD GENERATOR

A-3101

be block of the second of radius better in the second second second second second second second second second s



۰,

10-85

10 - 86

An explanation of the suple of 1s onlier charges on the finer particles may be found in a recent article by W. B. Kunkel(10-2). According to Kunkel, small particles are cushiored by the molecules of air and may not strike the capillary charging follow in the same moment or as frequently as larger particles, and, hence, should a quire a lower electrical charg In general, the charge on a possible construction rease almost as the "square of the diameter of the quire of the same almost as the

The Movement of Powder Particles in the Development Region. In powder-cloud development, a powder image is produced on the electrophotographic plate because electrically charged particles are drawn out of the powder cloud by the electrical field created between the plate and the development electrode. If the cloud were stationary during development of an image, the motion of powder particles would be simple, and probably would not be a factor in determining the quality of the images produced. However, since the cloud must move during development, the particles must describe curved trajectories in moving to the plate, and the final distribution of powder on the image must be the result of a complicated dynamic situation.

In performing the work reported in this section, it was assumed that an understanding of the behavior of in hvideal particles of powder in the development zone would provide a better understanding of the whole process of powder-cloud development, and, hence, would lead to practical improvements in powder-cloud development. Representing an initial attack on the problem of the dynamics of powder-cloud development, the following serves to open the field for future investigation, or addition to giving several interesting and valuable results.

In a first approach to the poblem t was assumed that: (1) the powder cloud moves in simple laminar flow between the plate and the development electrode, (2) the particles of powder are uniform in size and electrical charge, (3) the motion of the particles does not disturb the movement of gas, and (4) the movements of the particles at right angles to the flow of gas are completely independent of their movement with the laminar flow of gas. More-sophisticated ass in ptions may be possible later.

Figure 10-32 shows the region between the electrophotographic plate and the development electrode with the parabolic distribution of gas velocity characteristic of laminar flow. The origin of the coordinate system is placed midway between the plates for convenience. For this situation, where  $v_x$  is the velocity parallel to the flow of the gas between the plates,

(10-2). Kunkel, W. B., The Static Electrification of Dust Particles on Dispersion Into a Cloud: J. Applied Phys., Vol 21, August, 1950, pp 820-832.

BATTELLE MEMORIAL INSTITUTE

10-87 and 10-88



450X

1

95441

FIGURE 10-31. PHOTOMICROGRAPH OF POWDER PARTICLES LEAVING CYLINDRICAL SETTLING DRUM



#### 10-89

 $\underline{\mathbf{y}}$  is the distance from the center of the plates and at right angles to the plates,

$$\mathbf{v}_{\mathbf{x}} = -\mathbf{a}\mathbf{y}^2 + \frac{3}{2}\,\overline{\mathbf{V}}_{\mathbf{x}} \quad (10-3)$$

In this equation, a is an arbitrary constant to be evaluated later, and  $3/2 \ \overline{V_x}$  is the maximum gas velocity between the plates expressed in terms of  $\overline{V_x}$ , the average velocity of the gas between the plates which can be determined from the total flow of gas and the distance between the plate and the development electrode. The constant a can be evaluated by considering that at y = Y/2,  $v_x = 0$ ,  $a = 6\overline{V_x}/Y^2$ . The expression becomes:

$$\mathbf{v}_{\mathbf{x}} = -\frac{6}{\mathbf{Y}^2} \,\overline{\mathbf{V}}_{\mathbf{x}} \mathbf{Y}^2 + \frac{3}{2} \,\overline{\mathbf{V}}_{\mathbf{x}} \,\cdot \tag{10-4}$$



FIGURE 10-32. DISTRIBUTION OF GAS VELOCITY BETWEEN ELECTROPHOTOGRAPHIC PLATE AND DEVELOPMENT ELECTRODE A-2259

In this equation, y is actually a function of time, t, and  $y = V_y t$ , since it is assumed that the particle under consideration is moving parallel to the y axis with a uniform velocity,  $V_y$ . Using this relationship, Equation 10-4 becomes:

$$v_x = -\frac{6}{Y^2} \,\overline{V}_x \, V_y^2 \, t^2 + \frac{3}{2} \,\overline{V}_x \, .$$
 (10-5)

Now, the actual position,  $\underline{x}$ , of the particle starting from the origin of the coordinate system is given by:



10 - 90

$$\mathbf{x} = \int \mathbf{V}_{\mathbf{x}} dt = -\frac{6}{\mathbf{Y}^2} \overline{\mathbf{V}}_{\mathbf{x}} \, \mathbf{V}_{\mathbf{y}}^2 \int t^2 dt + \frac{3}{2} \, \overline{\mathbf{V}}_{\mathbf{x}} \int dt + C \quad , \qquad (10-6)$$

or,

$$\mathbf{x} = -\frac{2}{Y^2} \overline{V}_{\mathbf{x}} V_{\mathbf{y}}^2 t^3 + \frac{3}{2} \overline{V}_{\mathbf{x}} t + C \quad . \tag{10-7}$$

In this equation, <u>C</u> equals zero, because it was assumed that <u>x</u> is zero when <u>t</u> is zero. Again, expressing <u>x</u> in terms of <u>y</u> rather than in terms of <u>t</u>,

$$x = -\frac{2}{Y^2} \frac{\overline{\nabla}_x}{\nabla_y} Y^3 + \frac{3}{2} \frac{\overline{\nabla}_x}{\nabla_y} Y \cdot$$
(10-8)

This same equation will hold for negative values of y since the whole system is symmetrical with respect to the midplane between the two plates.

To obtain numerical results from Equation 10-8, it is necessary to know values for Y, the space between the electrophotographic plate and the development electrode;  $\overline{V}_{x_2}$ , the mean velocity of gas flowing in the development regions; and  $\underline{V}_{Y}$ , the velocity of the particle normal to the plate as produced by the electrical field resulting from the electrical charge on the electrophotographic image.

Although calculated in a subsequent section of this report, the following values of these constants will be used here to preserve continuity in the practical implications of the present calculations -- Vy = 58 centimeters per second,  $\overline{Vx} = 450$  centimeters per second, and  $\underline{Y} = 0.038$  centimeter. Using these constants, Equation 10-8 becomes:

$$x = -10730 / y^3 + 11.62 y$$
 (10-9)

Figure 10-33 shows a plot of the results of this equation for the case of powder flowing between an electrophotographic plate and a development electrode. According to these assumptions, powder moves as far as three millimeters past the edge of an electrically charged area of the plate. However, it must be remembered that this represents only a transient condition, and that, as soon as powder builds up on the plate, the electrical field between the plate and the development electrode is reduced until it approaches zero for complete development. In this case, the flow of powder is seriously altered. Again, powder will not continue to move toward the plate after the powder passes beyond the boundary of an electrically charged area.

BATTELLE MEMORIAL INSTITUTE



ţ

3
11-41

This calculation represent the first attempt to study the dynamics of powder-cloud development. It is heped that further calculations and experimental verifications will give greater understanding of the process and lead to practical improvements.

Electrical Charge on Particles. From past measurements of powder particles, it is reasonable to assume a particle diameter of two microns and a specific electrical charge of 40 microcoulombs per gram of developer powder. To find the weight of a single particle of powder, it is necessary to know the density of the material. This was determined as approximately 1.45 by finding that particles float in a liquid having a density of 1.50 but sink in a liquid having a density of 1.40. Using the figure of 1.45, the mass of a two-micron particle was calculated to be 6.1 x  $10^{-12}$  gram, or there are  $1.64 \times 10^{11}$  particles per gram of powder. From this number, it can be calculated that there is an electrical charge of 24.4 x  $10^{-17}$ coulomb per particle. Arbitrarily, it was assumed that the particles to be studied would have three times the amount of this charge, this factor being used to allow for particles having zero or reverse-polarity electrical charges. With this factor, the charge on the particle is 2.16 x  $10^{-6}$ electrostatic unit.

}

Using a potential orcp of 100 volts across the space of 0.038 centimeter between the electrophotographic plate and the development electrode gives a potential gradient which would produce 8.75 dynes force on a unit charge in the field. This, combined with the figure for the charge on the particle of powder, gives a force of 1.89 x  $10^{-5}$  dyne on the average particle.

A brief calculation  $f \to c \in g$  so taxonal torde on this particle showed it to be negligible in comparison with the electrical force.

To calculate the velocity of motion in the development zone, a modification of Stekes' law was used in the form

$$\mathbf{F} = \frac{3\pi\mu \vee \mathbf{D}}{\mathbf{K}_{\mathbf{m}}}, \qquad (10-10)$$

where <u>F</u> is the drag on the particle in dynes,  $\underline{\mu}$  is the viscosity of the fluid, <u>v</u> is the velocity of the particle, <u>D</u> is the diameter of the particle, and <u>Km</u> is a correction factor whose value is 1.08 for the size of particle involved here.

Solving Equation 10-10 for the velocity of the particle using the above values, gives 58 centimeters per second for the velocity of the particle moving toward the plate in the development region. It is interesting to note that, at this velocity, only about 0.7 millisecond will be required for the

BATTELLE MEMORIAL INSTITUTE

1 () - 43

particle to travel the whole distance between the development electrode and the plate. Even so, calculations reported above indicate that the particle would have moved about three millimeters along the plate during its travel through the development zone.

Velocity of the Gas in the Development Zone. From previous data on the amount of gas flowing from the development zone, it can be calculated that the average velocity of the gas in this region is about 450 centimeters per second.

#### Prevention of Unwanted Deposition of Powder

1

<u>Constant-Velocity Transition Zone</u>. In the high-speed development unit, transition between the turbulent flow in the capillary tube and the laminar flow in the development region is carried out in a simple, relatively effective device. However, this device does not distribute the powder cloud uniformly and it collects a large quantity of powder. It had been proposed that this accumulation of powder might be prevented if the transition from turbulent to laminar flow could be carried out in two separate steps.

The first step would be a constant-velocity transition from turbulent to laminar flow, accomplished by using a fishtail-shaped transition section of constant cross-section area to pass from the circular tube to a flat, rectangular tube. No powder should deposit in this region because of the high velocity of the air flow. The second step would be a transition from the high-velocity laminar flow to the low velocity of gas useful in developing electrophotographs. No powder should deposit in this region because of the laminar flow.

This type of transition zone has been tested and found only partly satisfactory. The device constructed to test this type of transition was formed by clamping together two plates of cold-rolled steel 3/6 inch thick, about 6 inches wide by 15 inches long. Shims of the proper thickness and shape were placed between the edges of the two plates to form the desired fishtail-shaped ducts. The plates were clamped together with 1/4-inch screws spaced one inch apart to insure the plates being well seated on the shims. The surfaces of the plates were smoothed with emery cloth and polishing paper to remove any rough spots or burrs.

Figure 10-34 is a sketch showing the internal dimensions and shape of the fishtail duct. The design of the duct is somewhat arbitrary. Except for the important considerations that, in the first part of the duct, the cross-sectional area must be kept roughly constant and that, throughout the device, the direction and velocity of the flow must not change rapidly, the design was governed largely by duct thicknesses that could be produced

HATTELLE MEMORIAL INSTITUTE



· .

1

ŕ

FIGURE 10-34. INTERNAL CONFIGURATION OF FISHTAIL DUCT

A- 1303

١

Û

#### 10-95

by a slight deformation of rigid steel plates. The duct was intended to operate using a powder cloud of charcoal in air flowing at free-air-flow rates of less than six liters per minute. Approximate values of the Reynolds number are noted in Figure 10-34 for several points along the duct. These values are for a free-air-flow rate of about five liters per minute.

Testing the device consisted of passing a powder cloud through it for a minute or longer and then dismantling the device for inspection. In every trial, inspection revealed some accumulation of powder in the duct. Some powder deposited rather uniformly in the cross-hatched region shown in Figure 10-34. This deposition suggests that the diffuser portion of the duct expanded too rapidly to preserve laminar flow. Presumably, this difficulty might be overcome by lengthening the duct. A more troublesome deposition of powder occurred in the high-velocity, laminar-flow portion of the duct. There, for unknown reasons, islands of powder formed slowly, eventually bridging the narrow gap between the plates and causing pronounced streaking. However, no simple change in the design will eliminate this difficulty. For this reason, and since modifications of the device to eliminate deposition in the diffuser section would lead to a rather cumbersome device requiring high air pressures, no further work on this device is warranted at this time.

<u>Porous-Plate Development Electrode</u>. In the high-speed development unit, the most objectionable deposits of unwanted powder occur where the powder emerges from the capillary tubing and impinges on the plate and on the development electrode. This deposition possibly could be prevented by blowing air through the back of a porous metal electrode into the development chamber.

To try this, a portion of the development electrode was replaced by a sheet of porous stainless steel(1) 0.032 inch thick. During the development process, air was blown through this porous plate into the development chamber and a jet of powder from the capillary tube was arranged to impinge directly onto this porous plate. The amount of pcwder deposited on the porous plate was reduced substantially when air was passing through the plate. However, some powder did deposit on the plate. Microscopic examination of the porous steel plate revealed that the pores occupied only about one-fifth of the total area of the plate surface and that the pores were as much as 0.005 inch apart. A material having a greater porosity undoubtedly would give better results, but such a material has not been obtained. Two other manufacturers were contacted, but their products had still lower porosities.

If powder were fed into the development chamber parallel to the development electrode rather than directly against it, the present porous material might give satisfactory results. This approach has not been investigated.

(1) Manufactured by the Miero Metallie Corporation, 30 Sea Cliff Avenue, Glen Cove, New York.

BATTELLE MEMORIAL INSTITUTE

10-96

<u>Heated Development Electrode.</u> Because of known effects of thermal gradients on the motions of powder particles, attempts were made to reduce the amount of powder deposited on the development electrode by creating a thermal gradient between it and the electrophotographic plate during the development procedure. Three temperatures were used: the electrode was heated to 130 F, held at 80 F, and cooled to 40 F. In all cases, the electrophotographic plate was at 80 F. On observing the amounts of powder deposited on the electrode in the three cases, no differences attributable to the thermal gradients could be observed. It is assumed that other, stronger forces, resulting f, om the high velocity of the air and the electrical field, masked any effect of the thermal gradient.

#### Device for Impregnating Cloth Belt With Powder

A cloth belt impregnated with powdered charcoal(1) is used with the powder-cloud generator of the high-speed development unit. The cloth belt originally used in this generator consisted of a strip of cotton flannel 72 inches long and about 1-1/4 inches wide, into which powdered charcoal had been brushed by hand. This technique for impregnating the cloth belts produced belts which were entirely satisfactory, but it has a number of limitations. The amount of powder forced into the cloth depends on the person preparing the belt, on the way he handles the brush, and on the vigor with which he brushes.

To eliminate these variables, an automatic machine was built for impregnating cloth belts with powder. It consists of an arrangement for blowing the powder cloud produced by a disc-type powder-cloud generator through the cloth belt at a low enough velocity that most of the powder particles deposit in the cloth.

Figure 10-35 is a photograph of the belt-impregnating device. The mechanism has a constant-speed capstan for driving the cloth belt; two spool holders, one driven through a slipping clutch, the other arranged to provide tension for the belt by means of a friction brake; and a powdercloud-supply tube ending at a rectangular orifice in the smooth, cylindrical plate over which the belt is pulled. The impregnating device is used with the cloth-disc-type powder-cloud generator<sup>(2)</sup>. The amount of powder forced into the cloth can be varied by changing the speed of rotation of the disc in the powder-cloud generator or the rate at which the cloth belt moves. At present, belts are fed twice through the device. The device works well, and eliminates the human element in preparing powder-impregnated belts.

(1) Final Progress Report, December 31, 1951, pages 1-92 and 1-93.

(2) Final kepolt, December 31, 1951, pages 1-86 and 1-87.

BATTELLE MEMORIAL INSTITUTE

#### 10-97



ŗ

FIGURE 10-35. DEVICE FOR IMPREGNATING CLOTH BELTS WITH POWDER

BATTELLE MEMORIAL INSTITUTE

#### 10--98

#### New Type of Powder-Gloud Generator

A potentially simple method of producing powder cloud involves spray vaporization of a dispersion of charcoal powder in liquid Freen 12. This method was un sable in the specific form tested, although it might be satisfactory under other conditions.

The method was tested by constructing a small Freen bomb from a six-inch length of wo-inch-diameter pipe, to the base of which was fixed an outlet tube and control valve leading to an orifice 0.004 inch in diameter, or to an 8.0-inch length of 0.025-indu-diameter capillary tubing. The bomb was filled with liquid Freen 12, to which had been added one per cent by weight of pulverized charcoal. This concentration is approximately equal to the concentration of the powed c clouds used in developing good pictures.

In the first trials of the bornb-type generator, the valve was opened so that the liquid dependent under full bomb pressure was supplied to the orifice and the tube. The conducted consisted of two components, a well-dispersed cloud of charcoal particles, and a spray of droplets of the mixture of liquid Freon and charcoal. If the cutput of the generator was directed at a white card one foot away from the orifice, the card was enveloped in a cloud of fine particles, but it would also be well spattered with spots formed by the comparatively live droplets of liquid Freon.

Freon droplets are present in the stream because the enthalpy of liquid Freon is considerably less than its heat of vaporization. Consequently the stream of Freon leaving the or fice will vaporize explosively at first, breaking the stream into droplets this explosive vaporization stops as soon as the liquid is cooled to its boiling point. In the case of Freon 12, this temperature will be reached when less than one-third of the liquid in the stream has vaporized. After that, the comparatively slow process of evaporation from the surface of the droplets waponized the Freon, a process which would not tend to brain a roplets. Two things might be done to overcome this difficult  $\mathbb{A}^{(1)}$  the Freon-charcoal dispersion might be heated enough to vaporize all the Freon, which would require raising the temperature of the mixture to about 100 C and using pressures of several hundred pounds per square inch, or (2) the mixture might be heated after spraying. In the latter case, it might not be possible to transfer the required amount of heat during the relatively short time available.

One attempt to heat the Freon as it was vaporizing consisted of releasing the liquid dispersion through a capillary tube instead of from an orifice. Here it was hoped that the dispersion could acquire heat from the capillary tube as the dispersion moved along the tube under gradually

MEMORIAL

RESTRICTED

INSTITUTE

RATTELLE

#### 10-99

decreasing predsures. Here again, a large proportion of the spray consisted of liquid droplets, and the number of droplets was not appreciably decreased by leating the capillary tube electrically until it was hot to the touch.

Other a trangements provided spraying the mixture into a small chamber to which was attached a short capillary tube. However, with this arrangement, he system soon clogged, apparently because of the powder remaining when the spray droplets evaporated on the walls of the chamber.

A promising modification of this method involves substituting liquid carbon dioxide for Freon 12. The enthalpy of liquid carbon dioxide should be great erough that the liquid would vaporize before its temperature drops near its triple use -57 C. Such an arrangement would require a thickwetted be sumilar to the cylinders used for carbon dioxide fire extinguishers. Such an arrangement has not been tested.

#### Micromanifold Development Device

BATTELLE

It earlier studies(1) of the harging of powder clouds flowing through capillary tubes, the charging of the powder cloud was found independent of the length of the tubes, for constant flow rates. Also, the quantity of charge generated on the particles of the cloud was roughly proportional to the average velocity of flow, at least when the flow was turbulent. These considerations suggested that powder particles might be charged using a row of short capillary ducted small diameter instead of the long capillary tube used earlies. Such a "micromanifold" of capillary ducts might have the fractical advantages of increasing the uniformity of development across the width of the plate, and of making it possible to design development devices for processing much wider plates than the four-inch span developable with the single capillary tube. Also, such a micromanifold might reduce the clogging problem, or at least restrict unwanted deposition of powder to areas which are easy to clean. The micromanifold developed at Battelle appears to possess these advantages.

The internal diameter of the capillary ducts in such a micromanifold was originally conceived as being perhaps 0.002 inch to 0.003 inch. However, because it was comparatively easy to fashion the ducts by casting metal around wire of 0.0035-inch diameter and then withdrawing the wire, the device was designed around this size.

(1) Seventh Quarterly Progress Report to The Haloid Company on "Continuous Tone Electrostatic Electrophotography", March 31, 1952.

MEMORIAL

RESTRICTED

INSTITUTE

#### 10-100

To insure turbulent flow through the small tubes, a Reynolds number of 8000 was selected for the flow. The flow rate necessary to produce such a Reynolds number was calculated from the conventional equation

$$N_{Re} = \frac{\rho V D}{\mu} , \qquad (10-11)$$

where N<sub>Re</sub> is Reynolds number,  $\underline{\rho}$  is the density of the fluid (in g/cm<sup>3</sup>, for instance), V is the average velocity of the flow (in cm/sec), D is the duct diameter (in cm), and  $\underline{\mu}$  is the absolute viscosity (in poises or in dynesec/cm<sup>2</sup>).

This equation has been converted into a specific form for convenience in making calculations by substituting for the average velocity the value indicated by the equation relating velocity and flow rate in a circular crosssection tube giving:

$$V = \frac{4F}{\pi D^2}$$
, (10-12)

where  $\underline{F}$  is the flow rate (cm<sup>3</sup>/sec). For this equation, the variables of flow rate and tube diameter were converted to units used in the laboratory measurements, and values were substituted for the density and viscosity of Freon 12 at 27 C and atmospheric pressure. These were 4.92 x 10<sup>-3</sup> g/cm<sup>3</sup> for the density and 1.25 x 10<sup>-4</sup> poise for viscosity. The resulting equation is:

$$N_{Re} = 330 \frac{f_{F12}}{d}$$
, (10-13)

where  $\underline{f_{F12}}$  is the flow rate in liters/min of Freon 12, and <u>d</u> is the internal diameter of the duct in inches.

Another equation used frequently and derived in the same manner relates Reynolds number to the flow rate of Freon 12 between parallel plates:

$$N_{Re} = 520 \frac{f_{F12}}{w}$$
, (10-14)

where  $f_{F12}$  is the flow rate in liters/min of Freon 12, and <u>w</u> is the width in inches of the flow stream -- not the interplate distance.

Similar formulas for dry air at 27 C and atmospheric pressure are:

$$N_{Re} = 55.0 \frac{f_a}{d}$$
, (10-15)

and

 $^{\circ}$ 

ţ

$$N_{Re} = 86.0 \frac{t_a}{w}$$
, (10-16)

BATTELLE MEMORIAL INSTITUTE

#### 10-101

where fa is the flow rate in liters/min of dry air.

)

Using Equation 10-13 and values for tube diameter and Reynolds number of 0.0035 inch and 8000, respectively, the flow rate required was found to be:

$$f_{F12} = \frac{N_{Re}d}{329}$$
, or (10-17)

$$f_{F12} = \frac{8000 \times 0.0035}{329} = 0.085 1/min$$
 (10-18)

It follows directly from the above calculations that, in order to supply the 12 liters of gas per minute used in the single-tube development unit, there would be required 12/0.085 or 142 ducts across the four-inch width of the development region, or about 35 ducts per inch.

The maximum length of the ducts to pass 0.085 liter of gas per minute, without requiring pressures greater than 60 psi as available from the cylinder of Freon, may be calculated by relating pressure difference to flow rate, length of tube, and tube diameter. In the case of turbulent flow through relatively smooth tubes, an empirical relation is:

$$p = k \frac{lf^2}{d^5}$$
, (10-19)

where <u>p</u> is the pressure difference, <u>l</u> is the length of the tube, <u>f</u> is the flow rate through the tube, and <u>k</u> is a coefficient depending on friction. Assuming the cast ducts have about the same smoothness as the drawn hypodermic-needle tubing, data from the pressure drop through hypodermicneedle tubing can be used to determine a usable length for the cast ducts. The following values, measured using hypodermic-needle tubing, were substituted in Equation 10-19: f = 2.3 liters per minute, d = 0.023 inch, l = 4.5 inches, and p = 34.4 psi. This gives a value of  $9.3 \times 10^{-9}$  for <u>k</u>. Now, solving for <u>l</u>, and using 60 psi as a practical maximum for the pressure and 0.0035 inch as the inside diameter of the duct, we get:

$$1 = \frac{pd^5}{9.3 \times 10^{-9} f^2}, \qquad (10-20)$$

$$1 = \frac{(60) (0.0035)^5}{(9.3 \times 10^{-9}) (0.085)^2} , \qquad (10-21)$$

$$1 = 0.47$$
 inch . (10-22)

This is a pratical maximum for the length of the duct. The pressure required is proportional to the length of the tube, so that a tube only one-third

BATTELLE MEMORIAL INSTITUTE

10-102

as long, about 0.16 inch long, would require only 20 psi to produce a flow rate of 0.085 liter per minute of Freon 12.

A trial micromanifold was made to specifications differing slightly from those just given. This manifold was one inch long and contained 30 ducts, each 0.0035 inch in diameter spaced 0.031 inch apart. The micromanifold width and therefore the duct length, was 0.1 inch. Designed to be one of the three walls between the plate and development electrode bounding the development region, the micromanifold was made 0.030 inch thick, the spacing between the plate and electrode.

The micromanifold was made by first forming a grid with 32 wires to the inch. Then, two strips of brass, each one inch long, 0.1 inch wide, and 0.01 inch thick, were pressed together with the grid between, using a force of about 20,000 pounds. The brass strips then were soldered together and the wires pulled out.

ş

ļ

٩

Figure 10-36 is a sketch of the arrangement used to test the micromanifold. This arrangement was not designed as a practical development device, but merely to test whether or not the micromanifold would charge and deagglomerate the powder cloud. The powder cloud is conveyed from the cloth belt to the micromanifold through a comparatively large conduit which, because of the low-velocity flow, would collect much powder.

Figure 10-37 is a reproduction of a picture produced with this test arrangement in a development time of ten seconds using Freon 12 at 50 psi with powdered charcoal. The quality of the picture is comparable with the best quality produced with the single-tube developing unit. The original had the brown tone associated with the fine-grain pictures produced with well-electrified charcoal.

An interesting indication of the effectiveness of the micromanifold was given in one test in which the plate was accidently sealed improperly to the micromanifold. In this case, the powder cloud leaked into the development region without passing through the capillary ducts and developed a picture with the black color, the halo, and background deposits characteristic of pictures made with poorly charged and agglomerated powder.

i

Powder still deposited at the outlets of the ducts. In fact, there appears to be about the same quantity as obtained with the single-tube unit. However, the powder is deposited where it can be cleaned from the electrode when the plate is changed.

Deposition of powder near the inlet side of the ducts is caused by the low-velocity turbulent flow in that region. In a practical development unit, this would be overcome by making the conduit to the capillary ducts a

BATTELLE MEMORIAL INSTITUTE



О.



FIGURE 10-36. ARRANGEMENT USED TO TEST MICROMANIFOLD DEVICE

1

**RESTRICTED** 

and a section of

10-104

high-velocity region, or by generating the cloud at the inlets of the capillary ducts. However, because of the smallness of the ducts as compared with the rectangular slot used in the present cloth-belt-type powder-cloud generator, there was some question as to whether a flow of gas into a hole 0.0035 inch in diameter would remove much powder from a belt. In order to resolve this question, the existing powder-cloud generator was modified to provide a take-off device having a tube of 0.0035-inch internal diameter touching the cloth belt. The tube was about 0.3 inch long, and, after various pressures had been applied to the generator, it was found that 40 psi was needed to produce a flow rate of 0.085 liter per minute. This checks well with the calculated value of 38 psi, using the value of k determined for a hypodermic needle. The concentration of the powder in the cloud, 0.15 per cent to 0.30 per cent by weight, was somewhat lower than the concentration obtained with larger tubes--from 0.4 per cent to 1.0 per cent by weight. However, in the arrangement used to measure the amount of powder produced by the modified generator, a substantial fraction of the powder was deposited on the walls of the short tube leading to the collecting device, so that the numbers obtained are not valid. Such a generator should produce sufficiently concentrated powder clouds for rapid development of continuous-tone electrophotographs.

#### Practical Multitube Development Device

15

Use of a single capillary tube in the high-speed development device is limited to developing a plate not more than four inches wide. During 1952, some effort was applied to designing a unit that would be applicable to developing larger plates.

Previous work had shown that, when the powder cloud entered the development chamber at more than one point, streaks occurred on the image wherever the adjacent streams of powder impinged on one another. However, using the micromanifold arrangement described above, streaking appeared near the outlets of the tubes but the streams of powder quickly merged to give a uniform flow of powder cloud through the development region.

To collect additional data on the manner in which the streams of powder merge, a device was built in which several tubes were fed from the powder-cloud generator and the effective spacing of the tubes at the development chamber could be varied from 0.5 inch to 4.0 inches in increments of 0.5 inch. The values chosen for tube diameter, tube length, and maximum number of tubes were the results of computations similar to those described in the preceding section of this report. It was assumed that the total flow through the development region should be 0.5 liter per second, that the velocity of the cloud through any tube should be approximately 1000 feet

BATTELLE MEMORIAL INSTITUTE

10-105 and 10-106



0 0

92700

FIGURE 10-37. ELECTROPHOTOGRAPH DEVELOPED WITH MICRO-MANIFOLD TEST ARRANGEMENT



BATTELLE MEMORIAL INSTITUTE

10-107

per second, and that air would be used at 40 psi. With this information, families of curves were plotted giving the number of tubes which could be used for different diameters and lengths of tubes. The Reynolds number in the tube was calculated for each case. The values assumed above were obtained from previous conditions which gave good electrophotographs.

Figure 10-38 shows the results of these computations for 6 to 250 tubes across the four-inch width of the development zone. Several factors influenced the final choice of eight tubes, the principal factor being the length of tube required to reach from the powder-cloud generator to the sides of the development electrode. Since the length required is approximately three inches, no more than nine tubes could be used. The availability of tobing with an internal diameter of 0.020 inch suggested the use of seven tubes. As a compromise, eight tubes were chosen and the length of each tube increased to 3.25 inches. This gives a Reynolds number in the tubes of approximately 12,000.

Figure 10-39 is a sketch of the eight-tube powder-cloud development device. The tubes lead directly from the powder-cloud generator to the development area and enter it at an angle of 10 degrees to the electrode rather than the 90 degrees used in the one-tube devices. Although not obvious on the drawing, all capillary tubes have the same length.

With the eight parallel capillary tubes, 0.5 inch apart, entering the development chamber, the powder cloud in the chamber appears to become uniform about seven-eighths of an inch beyond the ends of the tubes. After testing this arrangement, the spacing between the tubes was increased by 0.5-inch steps until they were 2.0 inches apart. At this spacing, the powder cloud did not become uniform until it had travelled 1.75 inches from the ends of the tubes. Under each condition, images of good quality were obtained after the streams had merged.

Figure 10-40 is a photographic copy of an electrophotograph developed using the unit shown in Figure 10-39. For this image, air was used at a pressure of 100 psi and the cloud of powder flowed from the bottom to the top of the image. <u>Image development was completed in 0.8 second</u>. The short development time resulted from modification in the powder-cloud generator discussed later in this report.

This multiple-tube development device should, by simple extension, develop areas of any reasonable width.

## RESTRICTED

BATTELLE

MEMORIAL INSTITUTE



FIGURE 10-38. RELATIONSHIP BETWEEN TUBE LENGTH, INSIDE DIAMETER. AND REYNOLDS NUMBER FOR DIFFERENT NUMBERS OF CAPILLARY TUBES TO GIVE SAME AIR-FLOW CONDITIONS

**RESTRICTED** 

A-4344



ELECTROPHOTOGRAPHIC POWDER-CLOUD DEVELOPMENT FIGURE 10-39. MULTITUBE DEVICE

A-4345

њ. њ. "

RESTRICTED

ľ

And and a state of the state of

#### 10-110

### Effect of Powder-Cloud Density of Rate of Image Development

One of the major objectives of this phase of the project was to reduce the time required to process electrophotographs. Through various modifications made earlier in the development process, development time had been reduced to approximately 10 to 15 seconds for complete development of a fairly dark subject. However, it was desirable to reduce this time still further, perhaps to less than one second.

One of the factors influencing the time required to develop an image is the density of the powder cloud in the development chamber. This, in turn, depends on the amount of powder released per unit time and on the volume of gas used to release this mass of powder.

<u>Concentration of Powder on Cloth Belt in Generator</u>. To determine how development time is affected by powder-cloud density, a number of experiments were performed in which the cloth belt for the powder-cloud generator was impregnated with various amounts of powder to vary the density of the powder cloud.

The first test was made using the belt-impregnating device described in Figure 10-35. The cloth belt was passed through this coating apparatus once and then put into the powder-cloud generator. A known volume of Freon 12 at 40 psi was forced through the belt, carrying the powder through the capillary tube either to a collector where the mass and specific charge of the powder were determined, or to the development area where development time was measured to obtain a standard image. The technique used to measure the mass and electrical charge of the powder, and the standard image used to measure development time have been described in an earlier report<sup>(1)</sup>.

Three more tests were made by passing the belt through the coating apparatus two, three, and four times before it was used in the development apparatus. In a fifth test, the coater was modified slightly and the belt was passed through once. In a sixth test, the speed of the belt through the modified coater was decreased to allow more powder to be deposited per unit area of belt.

Table 10-4 gives results of these tests. Increasing the powder loaded into the belt increases, somewhat, the density of the powder cloud in the development chamber. This is accompanied by a corresponding change in the time required for development. Also, the amount of electrical charge on the particles of the powder cloud depends on the concentration of the cloud.

(1) Final Progress Report to The Haloid Company on "Continuous Tone Electrostatic Electrophotography", December 31, 1951, pp 1-115 through 1-118.

BATTELLE MEMORIAL INSTITUTE

HATTELLE MEMORIAL INSTITUTE

#### FIGURE 10-40. PHOTOGRAPH OF ELECTROPHOTOGRAPH PRODUCED USING THE EIGHT-TUBE POWDER-CLOUD DEVELOPMENT DEVICE

98733



ţ

10-111 and 10-112

 $\odot$ 

i

٤

			10-	113		
for complete image devel- opment, seconds(1)	17.5	12.5	17.5	15.0	12.5	7.5
<pre>&gt;pecific charge on particles, microcoulombs per gram(1)</pre>	70. 5	76.0	66. 0	75. 0	94. 0	90°0
Powder-cloud density, grams per liter(1)	23.5	32. 5	21.5	28. 0	20.0	47.0
Method of impregnating cloth belt	l pass, l orifice, <b>nor</b> mal speed	<b>2 passes</b> , l orifice <b>nor</b> mal sp <b>ee</b> d	<b>3 pa</b> sses, l orific <b>e</b> , <b>nor</b> mal speed	<b>4 pa</b> sses, l orifice, <b>nor</b> mal speed	<b>l pa</b> ss, 3 orifices, <b>nor</b> mal speed	l pass, 3 orifices, one-sixth normal speed
Test	-	2	ß	4	ŝ	Q

(1) Each value is the average of from three to six determinations.

#### 10-114

0

1

While some decrease in development time follows increased loading of the cloth belt with powder, this method does not appear promising as a way of reducing development time significantly.

Rate of Travel o loth Belt Through Generator. Another way of increasing the density of the cloud produced by the powder-cloud generator is to increase the speed of the powder-impregnated belt past the exit orifice. In the generator used in the past for developing pictures in 10 to 15 seconds, the belt runs through the generator at a rate of about 0.2 inch per second. This speed was increased 15 times, to 3.0 inches per second. As the rate increased, the density of the cloud became greater and the time required for development of an image decreased until, at a speed of 3.0 inches per second, only one second was required to give complete development. However, with decreased development time, image quality decreased slightly. As a result, later work was directed toward improving the quality of the images produced when development was completed in one second.

It was the use of this powder-cloud generator at a belt speed of 3.0 inches per second that made it possible to obtain complete image development in less than one second when using the eight-tube device described earlier in this report.

Effect of Air Added to Powder Cloud After Generation. In all recent work on powder-cloud development using the single capillary needle to convey the cloud from the generator to the development region, the powder passes through an air-aspirator device described and pictured in Figure 1-42 of the Final Report to The Haloid Company on "Continuous-Tone Electrostatic Electrophotography, December 31, 1951. The function of this device was never fully understood, but it does improve the quality of the images produced. Because this device has an effect on the density of the powder cloud in the development region, experiments were conducted to investigate its function more fully, and the design of the aspirator was modified to give positive control of the amount of air added.

Figure 10-41 is a sketch of the modified device with the action of the mixing chamber changed from aspiration to positive addition of air. In this way, a known flow rate of air could be maintained and larger volumes of air could be added.

Using this device with the side air-inlet tube closed so that no air was added to the powder cloud after it was generated, a number of images were made with the belt traveling through the generator at the rate of 3.0 inches per second. The development time was one second, and the Freon 12 was supplied to the generator at pressures ranging from 40 psi to 67 psi. At all pressures of Freon 12 used, the images obtained were below average

BATTELLE MEMORIAL INSTITUTE

10-115



### FIGURE 10-41. MODIFIED POWDER-CLOUD MIXING CHAMBER USED ON HIGH-SPEED DEVELOPMENT UNIT A-4346

in quality, with those produced at the lowest pressures being the poorest. The images contained large particles of powder, had a coarse grain, and showed a considerable amount of "halo".

The next experiments were performed with the pressure of the Freon held constant at 60 psi and with air added to the mixing chamber through the side tube at rates ranging from 10 liters to 60 liters per minute. The development time was again held at one second. As the amount of air added was increased, the quality of the images increased markedly; when air was added at the rate of 60 liters per minute, the quality of the images produced was excellent.

Additional images were produced using a similar arrangement with the exception that the Freon to the generator was maintained at 40 psi; and, instead of supplying air to the mixing chamber, Freon 12 was added through the side tube at rates again ranging from 10 liters to 60 liters per minute. All of the images produced were poor in quality, although some improvement was evident when the flow rate of the added Freon was 60 liters per minute.

BATTELLE MEMORIAL INSTITUTE

#### 10-116

These images were characterized by a coarse grain, "halo", dark overall tone, and excess powder deposited in highlight areas.

To continue this series of experiments, air was substituted for the Freon in the powder-cloud generator. With the side tube to the mixing chamber closed, the pressure of the air to the generator was varied from 40 psi to 100 psi. The images obtained were poor in quality, similar to those obtained when Freon was used in the generator and the side inlet tube was closed. Somewhat improved images were obtained when the air was at 100 psi, a pressure greater than that available easily with Freon, and with the side inlet tube closed.

The next and final experiments in this group were performed using air in the generator at 40 psi and adding air to the mixing chamber at rates of from 10 liters to 60 liters per minute. The quality of the images improved with increased volumes of added air until, when air was added at the rate of 60 liters per minute, images showed very fine grain and a quality considerably above average. These images, developed in one second, were of a quality at least as good as those produced with the original high-speed development unit, in which the development times were 15 to 20 seconds.

From these experiments, it was concluded that other factors being constant, air supplied through the powder-cloud generator or added in the mixing chamber gave better quality images than did Freon 12. In addition, the more air added through the side inlet tube to the mixing chamber, the better the quality of the images obtained. The reason for these results are not well established, but some possible explanations have been suggested.

With all other conditions constant, because of the different values of the viscosity and density of air as compared with Freon 12, the Reynolds number for Freon 12 flowing through a passage will be approximately six times greater than that for air flowing through the same channel. This increased Reynolds number, which implies a greater tendency toward turbulent flow, may be the reason for denser pictures and the powder being deposited in highlight areas when Freon was the only gas entering the development region. There is a remote possibility that Freon may have a different effect on the electrical charge of the powder particles than does air.

The improved image quality noted with higher volumes of gas passing through the development chamber may be a result of increased charging of the powder particles because of their higher velocities, either through the powder-cloud generator or in the mixing chamber. However, a more likely explanation is that the increased volume of gas associated with the powder may simply dilute the cloud to such an extent that the amount of agglomeration occurring in the cloud is decreased and a finer grained image is produced.

BATTELLE MEMORIAL INSTITUTE

#### 10-117

The greater flow rates may cause a finer dispersion of powder to be created before it enters the development chamber. Further work is required to determine the validity of these arguments.

Effect of Removing Capillary Tube From Development Unit. Because of the pronounced effect of the rate of gas flow on the quality of the images obtained when developing images in one second, the function and importance of the capillary tubing leading from the powder-cloud generator to the mixing tube was questioned. This capillary tube, 0.023-inch inside diameter, was removed and a tube, 0.25-inch inside diameter, put in its place. However, the air still had to pass through an opening 0.046 inch in diameter but only 0.063 inch long. Using air, the flow rate of gas through the generator was adjusted to the same value used when the capillary tubing was in place. Air was then added to the powder cloud through the side inlet tube at rates up to 60 liters per minute. Using this arrangement, the quality of the images improved with increasing flow rates until, when air was added at the rate of 60 liters per minute, images of superior quality again were obtained in a development time of one second. In addition to having very fine grain, barely visible under twenty-power magnification, these images had a black tone rather than the sepia tone normally associated with electrophotographs developed with powdered charcoal.

It is now evident that high-quality images can be developed in one second or less by several different modifications of the powder-cloud technique. No attempt has been made to decrease the development time still further by running the cloth belt through the generator even faster, but there is no evidence that any limit has been reached. However, there is little practical advantage, at present, in reducing the development time much more because of the longer periods required for some of the other steps of the processing cycle.

#### Resolution of the Electrophotographic Process

BATTELLE

0

Measurements on developed images show that the process can now resolve at least 56 lines per millimeter on the plate, and at least 40 lines per millimeter on the final print. This resolution is better than any obtained previously. The resolving power may now be limited by the quality of the lens used in the laboratory, a 12-inch f/4.5 Radar Anastigmat, by vibration in the building, or by other uncontrolled conditions.

MEMORIAL

RESTRICTED

INSTITUTE

11

#### 10-118

#### Electrophotographic-Transfer Materials

During the period covered by this report, a search was made for a material other than damp dye-transfer paper or relatively short-lived pressure-sensitive tapes for use as final print material. Dampened dyetransfer paper makes good images, but the fact that it must be dampened before use and dried afterwards is a major drawback in a field application. For this reason, other types of materials have been sought which might give suitable transfer of electrophotographic images.

#### Waxed Paper

An attempt was made to transfer an electrophotographic image to a sample of waxed paper produced by the Munising Company of Chicago, Illinois, and provided by Mr. S. Levinos of the Signal Corps. The image transferred to this waxed paper was of approximately the same poor quality as that obtained when transferring to dry bond paper. This waxed paper is considered unacceptable as an image support.

#### Pressure-Sensitive Adhesive-Coated Paper

A fairly comprehensive search was made in previous work to find a commercial pressure-sensitive tape which would be satisfactory as an electrophotographic-transfer medium. As described in the Final Progress Report dated December 31, 1951, conventional commercial tapes have quite the opposite characteristics of those desired for electrophotographic transfer mediums. For example, a transfer tape should have little tack, while most commercial tapes are formulated to have maximum adhesive strength and tack. The adhesive should have a particularly smooth and unmarked surface, while in most commercial tapes the condition of the surface is either of little concern or purposely textured to impart special properties to the tape.

However, a new adhesive-transfer material was obtained which has many of the characteristics needed for making high-quality transfers of continuous-tone electrophotographic images. This new material is not an adhesive tape in the usual sense. Rather, it is a commercial paper coated with a very thin layer of a material that imparts a slight tack to its surface. Several modifications of this material have been especially prepared by the Champion Paper and Fibre Company of Hamilton, Ohio. All samples were based on types of a high-gloss, cast-coated paper known as "Kromekote". These papers have a particularly smooth surface which makes them well suited for a backing sheet of an electrophotographic transfer medium. After

BATTELLE MEMORIAL INSTITUTE

#### 10-119

coating these sheets with various formulations of ädhesives, their tack is so low that they show little tendency to stick to other materials and, hence, can be handled without interleaves. In spite of this low tack, these coated papers transfer a large fraction of the powder image from the electrophotographic plate. However, somewhat greater pressure is required to transfer powder to these materials than to dampened dry-transfer paper.

During this work, several variables were investigated. The weight and type of the raw paper stock, the type of adhesive used, the percentage of adhesive in the coating material, the thickness of the coating of adhesive **applied**, and the amount of stabilizer added to the adhesive have all been considered.

No specific information concerning a given sample has been supplied by the manufacturer, but the results of the tests on each of the coded papers have been reported to him. The manufacturer, in turn, has made what his technical people believed to be appropriate modifications to improve the characteristics of the paper. As a result of this work, definite improvements have been made both in the ability of the paper to provide a transfer image of good fidelity, and also in the stability of the adhesive coatings used.

As mentioned previously, the pressure required in making a transfer with these new materials is somewhat greater than that required when using dampened dye-transfer paper. To determine the pressure needed to produce a satisfactory transfer of the powder image from an electrophotographic plate to these different coated papers, a transfer device was constructed which could apply known and adjustable forces to a transfer roll.

Figure 10-42 is a sketch of this pressure-transfer device. By changing the amount and position of the weights on the lever arm, a wide range of forces is available. The rollers used have a center of steel and are covered with a firm rubber blanket. This rubber, when on the steel center, has a hardness of 87 as measured on a rubber hardness meter designated as the Rex Model A, manufactured by the Naugatuck Chemical Division of the United States Rubber Company(1). The force on the top roller can be adjusted from 0 to 1,000 pounds. A force of from 300 pounds to 800 pounds on the roller gave an acceptable transfer on most of the samples tried. A more compact unit is under construction which will use springs rather than a lever arm to obtain the force necessary to give a good transfer.

Figure 10-43 presents a comparison of the quality of images transferred to dampened dye-transfer paper and to a specially coated "Kromekote"

MEMORIAL

RESTRICTED

INSTITUTE

BATTELLE

1

 $\sim$ 

<sup>(1) 150</sup> Elm Street, Naugatuck, Connecticut.



RES

1

¥ 1 T

RI

A-4347 FIGURE 10-42. DEVICE FOR MEASURE FORCE REQUIRED TO TRANSFER POWDER IMAGES TO ADHESIVE - COATED PAPER

CTED

1

10-121 and 10-122



IMAGES TO DYE-TRANSFER PAPER AND TO A COATED KROMEKOTE **WHESIVE TRANSFERS OF ELECTROPHOTOGRAPHIC** COMPARATA PAPER FIGURE 10-43.

I N ÷., T 1.1.0.1.1

H A T

τF 1

R

LE

12

MEMORIAL

ESTRICTE

2.55

ì

#### 10-123

paper(1). The transfer to the "Kromekote" paper was made on the device described above using a force of 400 pounds on the top roller. The Manufacturer states that the white dust spots on the paper would be eliminated if production methods were used to prepare such a paper.

A larger quantity of this paper will be obtained and further tests will be made under various conditions of temperature and humidity. At present, it appears that this material will be suited for use in a field camera; it gives good images, requires no moistening or drying, and is reported to be no more expensive than dye-transfer paper.

#### Fixing of Transferred Images

No special attention has been given during the year to the problem of fixing an electrophotographic image after it has been transferred. For many purposes, the unfixed print may be satisfactory, as the image is relatively well attached to the transfer paper and even dark portions of the image will not shake off nor smudge easily. Fixing can be done easily, however, by spraying the print with a thin lacquer or other plastic. In the laboratory, Krylon in a Freon-pressurized dispenser has given good results.

#### CONCLUSIONS

The following general conclusions can be drawn from the past year's work on this project:

1. The addition of tellurium to the selenium on an electrophotographic place can increase the over-all photographic speed by a factor of 17, and can extend the spectral response to cover all of the visible spectrum and some of the near infrared.

2. Plates made with a layer of selenium-tellurium mixture over a thicker layer of selenium not only exhibit the same photographic speed and as much panchromaticity as one-layer selenium-tellurium plates, but they can be reproduced more consistently.

3. Concentrations of tellurium in the top layer of a two-layer plate may be at least 20 per cent to obtain the desired panchromatic response.

BATTELLE MEMORIAL INSTITUTE

<sup>(1)</sup> Code 2F as described in letter dated November 5, 1952, from Dr. W. P. Taylor, Champion Paper and and Fibre Company to W. E. Bixby, Battelle Memorial Institute.

#### 10-124

4. Of the factors believed to affect the electrical characteristics of two-layer plates, concentration of the tellurium has been found to be very important, rate of deposition of the film much less important, and temperature of the backing plate of comparatively little importance in the region between 60 C and 80 C. Other conditions have not been studied completely enough to warrant positive conclusions.

5. The electrical characteristics of both single-layer and two-layer selenium-tellurium plates depend on sensitizing conditions. Some plates show a marked improvement in quality when charged with corona currents higher than usual and for times longer than usual.

6. Microscopic examination of selenium films is a practical tool for determining the extent of crystallization in the films. Crystallinity appears necessary to produce red sensitivity in plates containing selenium alone, but it is not necessary in obtaining panchromaticity in seleniumtellurium plates.

7. Suitable apparatus has been constructed to process seleniumtellurium plates, even though they may have rapid dark-decay rates. Processing of plates from sensitization through development can be completed now in less than three seconds.

8. Selenium-tellurium plates appear to have inherent properties that will give images of a quality equal to that obtained on selenium plates. In addition, these plates have speeds approximating an ASA rating of 20 to 30, daylight.

9. A practical plate-sensitizing unit was built which does not require a bulky power supply for its operation. It employs a radioactive material and miniature batteries, and can sensitize a plate in 15 seconds. This completely eliminates the need for a bulky power supply in a portable camera,

10. Satisfactory electrophotographic images can be developed now in less than one second.

11. Plates considerably wider than the standard four-inch plate used previously can be developed now using an extension of an image-developing unit devised this year.

12. An adhesive-coated paper has been found which appears to have the characteristics necessary for a suitable medium to which electrophotographic powder images can be transferred. It requires no moistening or interleaving, and is relatively inexpensive.

BATTELLE MEMORIAL INSTITUTE

#### 10-125

#### RECOMMENDATIONS

As a result of improvements made during 1952, a number of recommendations can be made concerning possible use of these improvements at present and as to the course future research should follow.

#### Plates

In the work of the past year, it has been shown that two-layer selenium-tellurium plates having about ten times the photographic speed of selenium plates, and having a spectral response extending into the red, can be produced about as consistently as selenium plates. Also, seleniumtellurium plates can be made with 15 to 20 times the photographic speed of selenium plates, with a maximum sensitivity in the green, and with high sensitivity in the red. This is not necessarily the best that can be achieved, but it is felt that it represents fulfillment of at least one phase of the program. Selenium-tellurium plates can produce picture quality comparable to that obtainable with selenium plates, but selenium-tellurium plates are more difficult to use because their electrical characteristics are markedly affected by exposure to light, by repeated charging, and possibly by other actions.

In general, two-layer selenium-tellurium plates have the characteristics needed to make electrophotography practical for military purposes. It is true that additional research is needed on such plates, but such research should be directed largely to learning why such plates vary in properties even though made under apparently identical conditions. If reproducible plates can be produced routinely, with the spectral response and photographic speed of the best plates made during 1952, the plate problem can be considered solved while research is being devoted to other phases of electrophotography.

#### Sensitization

Additional work will be required to determine the best techniques and conditions for using radioactive-sensitizing units. Problems involving rates of charging under various spacing and voltage-gradient conditions have been considered in only a preliminary way, and no attempts have been made to determine the effect on charging rate of changes in the housing surrounding the source and the plate.

RESTRICTED

MEMORIAL INSTITUTE

BATTELLE

10-126

More information is needed on the effect of continued exposure of plates to a radioactive source, and how this effect may be related to the intensity of the radiation. The possibility of contamination from the radioactive material should be considered under various conditions that might be encountered in the field.

#### Development

To decrease the time required to develop a continuous-tone electrophotograph, it is recommended that the driving mechanisms on existing belt-type powder-cloud generators be modified to turn the capstan within the generator at approximately two revolutions per second. This will provide a cloud of powder capable of developing a four-inch by five-inch electrophotographic plate in one second. To maintain a high quality image, it is recommended also that the system of transporting the cloud of powder from the generator to the development zone be modified in one of two ways. If air is added as described in this report, an image of high quality can be obtained. Increasing the number of capillary tubes transporting the powder to the development region also will improve the quality of the image.

ş

ł

Because practical developments have outstripped the understanding of the processes involved, it is recommended that further work be done to determine some of the basic characteristics of the powder cloud. The mechanism by which the cloud is charged electrically is more uncertain now than at the beginning of the project. The effect of density of the powder cloud on the rate of agglomeration becomes increasingly important as denser clouds are used. A better understanding of the way in which mixtures of gases and solids flow in small channels would make the design of future units simpler, and might lead to the development of a device well suited for use on a continuous basis. The entire subject of powder-cloud generation needs more consideration. While the present belt-type works well, it is not simple to build or service, and is not applicable to continuous operation.

The fast dark-decay rates of selenium-tellurium plates require some type of device to carry the plate quickly through the various processing steps required when making an electrophotograph. Further work is needed to refine the unit which has been built, or to design an entirely new unit which might be more useful than the present one. Eventually, a unit suitable for use in the field should be designed and constructed.

It is recommended that materials other than powdered charcoal be investigated for use as developer materials. Although charcoal gives images

BATTELLE MEMORIAL INSTIT**UTE** 

RESTRICTED

۴.

#### 10-127

of good quality, it has a sepia tone rather than a true black, it cannot be fixed easily using heat or solvents, and it may not transfer as readily from the plate as might some other materials.

#### Sensitometry

Work should be continued on studying all the variables affecting the quality of the finished electrophotographic print. Such things as the thickness of the photoconductive layer, initial potential of the plate, the residual potential of the plate, and the specific charge on the developer powder, are all known to influence the quality of the image produced. With selenium-tellurium plates, proper settings of these variables becomes even more critical, and it is recommended that they be investigated in detail.

#### Transfer

Further tests should be made with the type of adhesive-coated papers which have shown promise recently. Aging tests, transfer quality at various temperatures and humidities, and modifications of the base stock bearing the adhesive are all recommended. In addition, modifications of the adhesive itself might prove valuable.

#### **IDENTIFICATION OF TECHNICIANS**

The following is a list of personnel contributing to the work presented in this report, including a brief statement of the background of each.

Andrus, P. G.; Principal Physicist. Mr. Andrus has had five years' research experience in electrophotography and the graphic arts.

Bixby, W. E.; Principal Physicist. Mr. Bixby has had six years' research experience in electrophotography, including work with special applications of the process.

Cherry, L. V.; Principal Chemist. Mr. Cherry has had four years' research experience in physical chemistry and one year's experience in graphic arts research.

RESTRICTED

BATTELLE MEMORIAL

#### 10-128

(2x)

Cleland, B. B.; Physicist. Miss Cleland has had two and one-half years' experience in metallurigcal research and nine months' experience in electrophotography.

Goldis, B. A.; Principal Electrical Engineer. Mr. Goldis has had one year's experience in electronic engineering and three months' experience in electrophotography.

Kriss, R. S.; Technician. Mr. Kriss has had four years' training in machine shop and drafting and four months' experience in electrophotography.

Matthews, E. P.; Principal Physicist. Mr. Matthews has had five years' experience teaching physics and mathematics, and two and one-half years' experience in electrophotography and graphic arts.

Medley, H. C.; Principal Mechanical Engineer. Mr. Medley has had two years' experience in mechanical engineering and two months' experience in electrophotography.

Paris, B.; Chemist. Mr. Paris has had research experience in organic chemistry and electronic engineering, and one and one-half years<sup>†</sup> experience in electrophotography.

Quinty, Gladys, H.; Principal Chemist. Dr. Quinty has had three years' experience in academic research on the chemistry of thorium and the rare earths, and five months' experience in electrophotography.

Reid, W. T.; Supervisor. Mr. Reid has been engaged in research for 23 years, 12 years of which have been in a supervisory capacity.

Ricker, E. C.; Technician. Mr. Ricker has had five years' research experience in electrophotography, and special training in silver halide photography.

Schaffert, R. M.; Research Consultant. Dr. Schaffert has had 22 years' research experience in the physical sciences and the graphic arts and has been actively engaged in Battelle's research on electrophotography since its inception in 1944.

Stockdale, J. L.; Chemist. Mr. Stockdale has had four years' experience in electrophotography.

Trask, R. K.; Principal Physicist. Mr. Trask has had two years' experience teaching college physics and one year's research on the

BATTELLE ME'MORIAL INSTITUTÉ

#### 10-129 and 10-130

metallurgy of binary alloys. <sup>(b</sup>He has had one and one-half year's experience in electrophotography.

Ullrich, O. A.; Principal Physicist. Mr. Ullrich has been engaged in research on electrophotography for four years, and has had seven years' experience in the fields of optics and high vacuum.

Walkup, L. E.; Assistant Supervisor. Mr. Walkup has had six years' experience in electrophotography and 18 years' experience in engineering research.

Wilgus, H. M.; Technician. Mr. Wilgus has had six years' experience in engineering research and one and one-half years' experience in electrophotography.

\* \* \* \*

Data for this report are recorded in the following Laboratory Record Books:

5934, pages 68 through 90 5966, pages 17 through 29 6089, pages 29 through 100 6091, pages 43 through 99 6099, pages 66 through 100 6264, pages 54 through 83 6393, pages 3 through 5 6400, pages 21 through 22 6909, pages 1 through 99 6916, pages 1 through 100 7319, pages 1 through 33 7382, pages 1 through 33 7398, pages 1 through 6 7493, pages 1 through 7 7611, pages 1 through 6

ĥ.

WEB:PGA:OAU:LEW:RMS:WTR/PL:et January 19, 1953

BATTELLE



MEMORIAL

INSTITUTE

. . .

### APPENDIX A

. 4

О

 $\widehat{}$ 

.

.

BATTELLE MEMORIAL INSTITUTE

RESTRICTED

÷.,
#### 10-A1

#### APPENDIX A

#### MINUTES OF SPONSOR MEETING

#### October 16, 1952

Subject: "Continuous-Tone Electrostatic Electrophotography" Contract DA-36-039-SC-123

Persons Present: Steve Levinos, U. S. Army Signal Corps James M. Chapman, Wright Air Development Center John H. Dessauer, The Haloid Company Harold E. Clark, The Haloid Company William T. Reid, Battelle Lewis E. Walkup, Battelle
R. M. Schaffert, Battelle
Paul G. Andrus, Battelle
William E. Bixby, Battelle
Edwin E. Graves, Battelle

This meeting was held on October 16, 1952, to discuss the present status of the work on this project, and to plan work to be done between now and November 20, 1952, the tentative date of the next meeting. This meeting, too, will be held at Battelle.

The statements set forth in these minutes shall not be considered as evidence of inventorship nor as constituting recognition of the novelty or originality of any of the ideas or suggestions proposed by any of the participants.

#### **Plates**

Paul Andrus reported on research done on plates during the past four weeks. This work can be divided into three parts:

- 1. Rechecking sensitivity and spectral response of selenium-tellurium plates made about one year ago.
- Preparing single-layer, 93 per cent selenium 7 per cent tellurium, plates by evaporating seleniumtellurium mixture from a single source.

BATTELLE MEMORIAL INSTITUTE

- 10-A2
- Preparing two-layer plates in which the top layer, consisting of 93 per cent selenium and 7 per cent tellurium, is approximately ten per cent of the thickness of the lower layer of selenium.

#### Remeasurement of Selenium-Tellurium Plates Made in 1951

BATTELLE

ţ

<u>Plates Containing Less Than Five Per Cent Tellurium</u>. Andrus explained that when we started investigating the effects of small additions of tellurium to selenium in electrophotographic plates a year ago, nearly all plates showed such rapid dark decay that it was impractical to determine their spectral response. With plates containing less than five per cent tellurium, dark-decay rates were from 10 seconds to 42 seconds when the plates were charged to initial potentials of 200 to 300 volts.

Recent chec's show that the dark-decay rate of each of these plates now is much lower. These plates at present require an average of about 300 seconds to decay from 200 to 100 volts in darkness when charged to an initial positive potential of 400 to 500 volts under the newer charging conditions involving higher corona currents. While a lower dark-decay rate was obtained when charging the plates under conditions differing from those used a year ago, the change in dark-decay rate is considered primarily the result of changes in the selenium-tellurium mixture itself. This has been confirmed by charging a few plates under the old charging conditions where lower currents flow from the corona wires. In answer to a question from Clark, Andrus reported that initial potentials are measured approximately three seconds after charging the plate.

Reid suggested measuring the current flowing from the backing plate to ground rather than measuring the current flowing to the corona wires.

Clark questioned the effect on dark-decay rate of charging the plates to higher potentials. While admitting that charging plates to higher **potentials** does decrease dark decay, Andrus believes that the differences noted in retested plates are too great to be explained by this effect.

Andrus explained further that the top layer of these particular seleniumtellurium plates may be rich in tellurium, because these plates were made by evaporating a physical mixture of powdered selenium and tellurium.

MEMORIAL

RESTRICTED

INSTITUTE

#### 10-A3

Plates Containing About Five Per Cent Tellurium. Plates were prepared by evaporating a premelted mixture of 95 per cent selenium and 5 per cent tellurium. When first made, these plates exhibited dark-decay rates ranging from 4 seconds to 60 seconds when the plates were charged to initial positive potentials of about 200 volts under the older charging conditions. Dark-decay rates for these plates are now one-fourth to one-fifth those measured a year ago. For example, the dark decays of typical plates have changed during the year from 4 seconds to 22 seconds, from 40 seconds to 100 seconds, and from 25 seconds to 300 seconds. Andrus reported that one plate of this group is now more nearly panchromatic than any other plate tested to date.

Harold Clark questioned the number of times these plates had been charged, pointing out that repeated charging might affect the characteristics of the plate. Andrus replied that these plates had been charged a relatively small number of times, and that there was no evidence that recharging could explain the observed results.

Reid raised a question of what might be expected in the way of sensitivity and spectral response if plates containing 95 per cent selenium and 5 per cent tellurium were made today under the same conditions used for preparing plates a year ago. Andrus replied that we cannot reproduce plates well enough to permit such a comparison.

Reid suggested that it might be well to study the effect of aging by storing a series of selenium-tellurium plates under known conditions. No decision was made to do this, 'ut the value of such testing was recognized.

Selenium Plates Containing More Than Seven Per Cent Tellurium. Plates containing more than seven per cent tellurium were made by evaporating a premelted selenium-tellurium mixture from a single source. When measured a year ago, dark-decay rates for these plates varied from 10 seconds to 40 seconds, some plates showing slight sensitivity to red light. Other plates in this group had too rapid dark decay to permit measuring spectral response. When remeasured recently, the dark-decay rate for these plates was about 50 seconds, one of the group being the most sensitive plate tested to date.

Andrus pointed out that, one year ago, residual potential on many of these plates was high enough to interfere with measuring their sensitivity. Residual potentials are now lower and do not interfere. Clark said that the lower residual potentials might be the result, at least in part, of charging the plates to higher initial potentials. Andrus feels that the lower residual potential is due primarily to a change in the structure of the seleniumtellurium layer.

MEMORIAL

RESTRICTED

BATTELLE

INSTITUTE

#### 10-A4

Dessauer asked the numerical value of the minimum practical darkdecay rate. Andrus stated that a dark-decay rate of 20 seconds is practical now, and, with techniques and equipment permitting more rapid development, a still faster dark-decay rate will be practical.

Andrus pointed out that the best plates do not show much, if any, residual potential. Reid stated that the important question is "How does aging affect the plates, and can we preage plates and obtain the same results?" Schaffert stated that aging would be expected to cause denucleation in the selenium-tellurium layer.

#### Single-Layer Plates Made During Past Month Containing 93 Per Cent Selenium and 7 Per Cent Tellurium

At the beginning of the present period, a water-cooled platen was used in making selenium-tellurium plates, and both the postevaporative heat treatment and the rate of deposition of the selenium-tellurium mixture were being varied. The premelted materials were evaporated from a single source. Most plates made under these conditions exhibited residual potentials of 400 volts to 600 volts. Such residuals made the plates appear insensitive to light under ordinary testing conditions. However, when charged to 1000 volts, these plates were sensitive to light.

Attempts were made to produce four-inch by five-inch seleniumtellurium plates, for use in making actual pictures. These plates were heat treated for 10 to 20 minutes at 80 C after evaporation of the seleniumtellurium mixture. Again, the plates exhibited residual potentials of about 400 to 600 volts. These plates will be rechecked periodically to see if residual potentials change as the plates age.

With some of these plates, negative sensitization gave a much lower residual potential than if the plates were charged positively. Dark-decay rate was about the same regardless of the polarity to which the plates were charged.

#### Two-Layer Plates Made During Past Month Using 93 Per Cent Selenium and 7 Per Cent Tellurium

According to Andrus, multilayer plates offer promise, particularly in giving lower residual potential and more nearly panchromatic response. Several two-layered 50-micron plates were made during the past month, the bottom layer of 45 microns being selenium, and the top 5-micron layer consisting of 93 per cent selenium and 7 per cent tellurium. The selenium

BATTELLE MEMORIAL INSTITUTE

÷)

1

1

#### 10-A5

layer was deposited in ten minutes, and the thinner selenium-tellurium layer was deposited over a period of ten minutes, both with the plate at a temperature of 80 C. Plates were heat treated for about two minutes at 80 C and then cooled, not quenched. These plates had low residual potential and spectral-sensitivity cutoff at about 575 millimicrons, as compared with a cutoff of about 550 millimicrons for single-layer selenium plates.

Schaffert stated that he felt the information indicated that the top layer of selenium-tellurium is not thick enough, and that better results might be obtained by making a three-layer plate consisting of a layer of seleniumtellurium, a layer of selenium, and then a thin, top layer of a seleniumtellurium.

Andrus pointed out that on two-layer plates made this month, onehalf of the plate was covered with grease before depositing the selenium and selenium-tellurium, that the photoconductive layer could be removed for measuring its optical absorption. Chapman questioned the effect of such a layer of grease on the properties of the resulting plate. Andrus reported that dark-decay rates are approximately half as fast on the layers deposited on grease as for the layers deposited directly on clean brass. There was very little, if any, difference between the spectral sensitivity of layers deposited on grease and on brass.

Dessauer suggested that selenium-tellurium layers be deposited directly on glass to permit measuring the optical properties of the films. Walkup pointed out the difficulties in doing this.

Dessauer suggested that perhaps higher tellurium contents would be of value. Andrus said that, in earlier work on single-layer selenium plates, extremely rapid dark-decay rates were obtained with higher percentages of tellurium. Nevertheless, Andrus suggested that this might not be true for the two-layer plates, and that we will make other plates having a top layer containing as much as 15 per cent tellurium.

Clark suggested, and it was agreed, that the absorption of red light by the selenium-tellurium layer was of major importance. Clark then suggested that the top layer of selenium-tellurium should contain the same total quantity of tellurium as is present in a single-layer selenium-tellurium plate exhibiting good sensitivity and panchromatic response. He pointed out, if it is necessary to use very thick top layers of selenium-tellurium to get proper absorption of red light, that plates probably will have high dark-decay rates. Andrus pointed out that the addition of tellurium also makes plates more sensitive to blue light. He feels that red sensitivity in selenium plates is caused by absorption at the interface, between the selenium and the brass, of light which has passed through the selenium layer. Selenium-tellurium layers may absorb red light nonphotoelectrically so that it does not reach the selenium-brass interface.

BATTELLE MEMORIAL INSTITUTE

#### 10-A6

In concluding this phase, Dessauer suggested that we make an effort to find out specifically what is done in making selenium rectifiers as a possible guide to changing the sensitivity of selenium plates.

#### Sensitization

Bixby explained that work on sensitization is being directed toward reducing the time required to sensitize plates with a radioactive source. A weak alpha-ray source now being used requires about two minutes to charge a four-inch by five-inch plate. A one-millicurie radium-D source would cost about \$100. A ten-millicurie polonium source has been obtained at a cost of \$50. This source having a half-life of 149 days, should make possible the sensitization of a four-inch by five-inch plate in approximately five seconds.

#### Camera Exposure on Selenium-Tellurium Plates

Using a plate containing 93 per cent selenium and 7 per cent tellurium, actual photographs were made at a photographic speed of about ASA 30. However, the dark-decay rate of this plate was so high that it was necessary to expose and develop the plate within a few seconds after sensitization.

To provide such rapid processing, Bixby sketched a device which is being built and will be attached to the camera and in which exposing and developing can take place very rapidly. Levinos approved of this. Bixby pointed out that, as part of the improved sensitivity of selenium-tellurium plates may be due to the use of higher charging currents in corona charging, and because the alpha-ray source provides only low current, seleniumtellurium plates having high dark-decay rates may not give good results. Clark emphasized strongly that it might be well to incorporate a high-speed corona-charging unit in the camera, and that a determination should be made of the effect on dark-decay rates of charging with different corona currents and with the radioactive source. Bixby pointed out that the beneficial effects of charging at higher corona currents may last long enough to allow subsequent chargings to be done with lower currents. It was agreed that the camera attachment, in due time, should be provided with means for charging the plates either with a high-speed corona unit or with the radioactive source.

BATTELLE MEMORIAL INSTITUTE

i

#### 10-A7

#### Development

Bixby reported on studies of the effect of powder-cloud density on the speed of developing images with a high-speed development unit. By running the belt 15 times faster than has been used for 10-second to 15second development, an image was developed in one second. Speed of development thus appears approximately proportional to the density of the powder cloud. Images made in one second were somewhat inferior in quality, there being some halo and some streaking of the image. Producing a more dense cloud appears to decrease the electrical charge on the powder particles; therefore attempts were made to increase the charge on the particles.

Experiments were run to determine the maximum rate of flow of powder cloud between the exposed electrophotographic plate and the development electrode. As yet, this maximum has not been reached or determined, although clouds have been blown across the plate at high velocities. A number of interesting observations have been made as by= products of this work.

In the present high-speed developing device, Freon-12 gas is used to blow the powder from the cloth belt and through the capillary tube that supposedly breaks up agglomerates of powder and gives the particles an electrical charge. On leaving the capillary tube, the cloud flows through an aspirator where it draws room air into the stream. It was found that closing off this secondary air decreases picture quality, as does the introduction of Freon 12 at this point instead of air. Adding additional air at this point by using compressed air improved the quality of pictures developed in one second to equal the quality of pictures developed in 15 seconds.

;

0

Walkup suggested replacing Freon 12 in the generator by air; this has not been, but will be, tried.

Bixby stated that powder particles may be charged electrically after the powder leaves the capillary tube.

After considerable discussion of the entire subject of powder-cloud development with the capillary-tube device, everyone agreed that it is particularly important at this time to study the aerodynamics of the system. Clark pointed out, for example, that the amount of air introduced as a secondary gas may affect recombination (agglomeration) of powder particles in the clouds, and that such recombination may reduce the electrical charge on some of the powder particles after they leave the capillary tube.

BATTELLE MEMORIAL INSTITUTE

4

The second secon

#### 10-A8

Bixby stated that a combination photographic subject is being made up for use in the study of development. Reid suggested that a composite, still-life subject, on which lighting could be varied as desired, might be more suitable for these studies. Levinos backed this suggestion.

Levinos reported that a diffusion screen over the camera lens improves the quality of portraits made by electrophotography. This will be tried at Battelle.

#### Transfer

Bixby explained that 20 more samples of adhesive-coated papers have been received from the Champion Paper and Fibre Company and tested for use in adhesive transfer. Most of these papers appear to have greater tack than those received in the past. Champion is now checking the stability of the papers, and feels that this property can be controlled satisfactorily. Champion has stated that these papers should not cost more than dye-transfer papers.

Levinos suggested that instability of the white base paper, if serious, might be overcome by using a strong ultraviolet absorber in the transparent protective coating used in making the sandwich.

Chapman reported on a new adhesive material available for splicing magnetic tapes. This adhesive apparently is nonslipping and noncreeping. It might work satisfactorily as a transfer adhesive.

Bixby stated that, in addition to evaluating the results obtained by transfers for the new papers received recently from Champion, tests will be made to determine how much pressure is required for good transfer.

#### Future Work

It was agreed that research for the coming five weeks should continue along the same lines as in the recent past. No changes were suggested.

MEMORIAL

RESTRICTED

INSTITUTE

BATTELLE

T

1 n n

•

#### APPENDIX B

 $\odot$ 

#### 10-BI

#### APPENDIX B

#### MINUTES OF SPONSOR MEETING

November 14, 1952

5 ....

85

Subject: "Continuous-Tone Electrostatic Electrophotography" Contract DA-36-039-SC-123

Persons Present: Allen Rahm, U. S. Army Signal Corps James M. Chapman, Wright Air Development Center Harold E. Clark, The Haloid Company William T. Reid, Battelle
R. M. Schaffert, Battelle
William E. Bixby, Battelle
Paul G. Andrus, Battelle
Eugene C. Ricker, Battelle
Edwin E. Graves, Battelle

This meeting was held on November 14, 1952, to discuss the present status of the work on this project, and to plan work to be done between now and December 17, 1952, the tentative date of the next meeting, also to be held at Battelle.

The statements set forth in these minutes shall not be considered as evidence of inventorship nor as constituting recognition of the novelty or originality of any of the ideas or suggestions proposed by any of the participants.

#### Sensitization

Bixby described the new radioactive polonium source obtained from Canadian Radium and Uranium Corporation. This source consists of a rectangle of nickel-plated metal covered with four-inch by five-inch area of polonium-210, gold plated, and covered with a plastic coating. While we requested 0.0005 inch of polonium, which would provide 10 millicuries, the source actually provides 20 millicuries.

The source was checked to assure that it did not contaminate the selenium, as did an earlier polonium source. In this connection, Bixby read a report by United States Radium Company on what might have occurred

BATTELLE MEMORIAL INSTITUTE

#### 10-B2

when the original source contaminated the selenium. A check of the new source revealed no contamination of the selenium, the container, or the camera back in which it was used. The charging current with this source is 0.41 microampere with a potential difference of 200 volts and the source one centimeter from a bare metal plate. At potentials higher than 200 volts, only slightly more current flows. Following are the currents obtained with various plate-to-source spacings and voltages:

	Potential	Charging
Spacing,	difference,	current,
centimeters	volts	mic roamperes
2.0	350	0, 58
2.5	500	1.0
4.0	1200	1.1
6.7	2250	1.2

These data suggest that the gold and plastic coverings retard the alpha particles so that they do not act over their natural range of 3.6 cm. For this case, a reasonable space is about 2.5 cm and a reasonable voltage about 500.

With this radioactive source in the special camera back designed for testing plates with high dark decay, with the selenium plate spaced 0.75 inch from the source, and with a potential difference of 600 volts, the potential on the plate at different times and the charging current were as follows:

Charging time, seconds	Potential on plate, volts	Charging current, microamperes
0	0	0.9
5	175	0.8
10	300	0.54
15	400	0.48
30	400	0.42

Bixby stated that some of this current was not effective in charging the plate, being dissipated elsewhere in the system.

Allen Rahm asked if there was any correlation between the data for current flowing to a bare metal plate and times required for charging a selenium plate. Bixby replied that such correlation had not been determined and that these data were taken only for the actual charging of a selenium plate.

Bixby then reported that there was no practical difference between the light-decay and the dark-decay rates for selenium plates charged by

BATTELLE MEMORIAL INSTITUTE

i

ł

ſ

#### 10-B3

corona and by alpha particles. Similarly, there was little difference between light-decay and dark-decay rates for two-layer selenium-tellurium plates charged by corona and by alpha particles. These two-layer plates comprised a 25-micron layer of selenium covered with 25 microns of a mixture of 93 per cent selenium and 7 per cent tellurium. With a spacing of 0.75 inch between the plate and the source, and with alpha charging for 30 seconds at 300 volts, the plate accepted a potential of only 60 volts and lost po' ntial rapidly in darkness. At the same spacing and charging time, but with a potential difference of 600 volts, the plate accepted a potential of 250 volts and dark decay was much slower. It was concluded that higher initial potentials, as with corona charging, give slower dark decays. Also, Bixby reported that dark decays for two-layer sellurium plates were about the same for both corona and alpha charging, if the plates were charged to the same initial potential.

Clark suggested that the same measurements be made on a plate which has been difficult to charge in the past, to determine whether there is any effective difference in charging such plates with alpha particles and with rapid corona charging. Bixby replied that we have some data indicating that alpha charging of a plate "medium hard to charge" is equally as effective as charging the plate by the rapid corona method.

Bixby stated that more data are necessary to determine why some plates show much more rapid dark decay when charged to a low initial potential, either by corona or by alpha particles.

In the discussion which followed, Rahm indicated that more data are needed on sensitization. Reid raised the question again as to the adequacy of expressing dark decays as halftimes. Schaffert replied that he believes that a few more data will enable him to define some basic constants for expressing both light-decay and dark-decay rates.

Rahm expressed the opinion that charging data are needed for a large group of plates, representative of the best plates spectrally, photographically, and electrically. He did not feel that the present test results would be adequate to show the direct effects of charging conditions on light-decay and dark-decay rates. Bixby replied that this work was aimed specifically at obtaining practical results as soon as possible and not at getting basic data. Andrus pointed out that we are just now in a position to produce plates having similar spectral, photographic, and electrical characteristics. Therefore, it is now possible to explore more fully the reproducibility of charging with alpha particles and to determine optimum charging conditions and voltages required to minimize dark decay.

RESTRICTED

INSTITUTE

BATTELLE MEMORIAL

10-B4

In answer to a question as to whether tests have been made on charging single-layer and two-layer selenium-tellurium plates to negative polarities, Andrus replied that such tests had been made only on one-layer plates. When charged negatively, one-layer plates are sensitive to light, but generally accept lower initial potentials and have much lower residuals than when charged positively. Detailed data on these effects were given in the minutes of the meeting held on October 16.

 $\langle \rangle$ 

In concluding his report on sensitization, Bixby stated that for use in the camera, a two-minute charging time was selected for the polonium source, to assure completely uniform charging of the plate.

At this point, Rahm asked if the special camera back made to permit photographic tests with plates having a rapid dark decay is to be delivered to the Signal Corps at the end of the year. In reply, it was pointed out that this camera was intended for experimental work only, and that probably it would be modified many times before reaching its final form. Rahm reminded us that an ion-charging unit is to be furnished to the Signal Corps, as well as two plate holders modified for use with both the charging unit and the developing unit. It was agreed that this would be kept in mind. Chapman stated that the Air Force would like the same type of equipment, including a powder-cloud generator.

ţ

Chapman brought up the matter of recycling plates without fatigue by using regeneration procedures. Clark pointed out the undesirability of using polonium in place of electrical charging because of differences in plate performance found with the two different sensitizing methods. It was agreed, in general, that the flexibility of corona charging makes it preferable for charging of plates in the laboratory, whereas polonium is better for field use, such as for military purposes. It was agreed that work in the laboratory should be continued with both types of charging, the immediate objective being to obtain some correlation between the two.

#### Development

Bixby reviewed the design of the capillary-tube developing unit and the general method of producing the powder cloud and for aspirating air into the development chamber. He reported that cutting off the supply of air, so that only Freon was present, resulted in marked deterioration of picture quality.

MEMORIAL

RESTRICTED

INSTITUTE

4

BATTELLE

#### 10-85

Increasing the speed of the belt 15 times gave poor picture quality, but development was obtained in about one second. Increasing the flow of air to 60 liters per minute resulted in pictures with as good quality as the best continuous-tone photographs made to date.

To determine the maximum flow rate in the development region, Freon was added in the aspirating section instead of air. Poor quality pictures resulted, possibly indicative of less charging of the powder cloud. The addition of secondary air in the aspirator when air was used to produce the primary powder cloud gave slightly better results than the addition of air to Freon. In this case, the total rate of flow of air was 60 liters per minute, the development time was one second, and the coated belt moved four inches per second.

Reid suggested that helium be tried in both generator and aspirator.

Ricker showed a series of pictures made with the development unit in which the capillary tube was removed, leaving a 1/16-inch length of 0.046-inch ID tube in the line. The pictures were of very good quality, again perhaps as good as with the usual single capillary tube. Of more importance, these pictures were black, instead of sepia. Reid mentioned that as far as deagglomeration of the powder particles is concerned, flowing the cloud through an orifice might be as effective as passing it through a capillary tube.

Plans have been made to determine the effect of cloud density, and to determine whether or not the short 0.046-inch-diameter tube has any real function in charging the powder cloud. In this connection, Rahm and Chapman suggested that a live subject be used where possible, rather than copying an existing print.

Bixby reported tests made with a multiplicity of capillary tubes to make possible the use of wider plates. Using eight capillary tubes uniformly spaced over a width of four inches, somewhat grainier images were obtained than when using a single capillary tube.

#### Plates

#### **One-**Layer Plates

BATTELLE

!

ţ

Andrus reviewed the type of plates being made experimentally. Onelayer plates composed of 93 per cent selenium and seven per cent tellurium, prepared at a platen temperature of 70 C, gave lower residual potentials of about 50 volts, as compared with residual potentials of about 600 volts for the same type of plate made at a platen temperature of 80 C. Plates

RESTRICTED

MEMORIAL INSTITUTE

#### 10-B6

made at the lower temperature were less panchromatic than those prepared at 80 C. The maximum wavelength at which any response is obtained for a selenium plate on a brass backing is about 550 microns, that for a singlelayer selenium-tellurium plate made at 70 C is from 600 to 650 microns, and that for a single-layer sellurium plate made at 80 C is about 750 microns.

#### Two-Layer Plates

Two-layer plates were prepared consisting of a bottom layer of selenium comprising 90 per cent of the total thickness of the plate, and an upper layer consisting of a selenium-tellurium mixture in which the tellurium concentration before coating was varied from 7 to 20 per cent. These plates were 5 microns thick. Increasing the tellurium content in the top layer extended the sensitivity toward the red end of the spectrum. Absolute sensitivity also appears to increase, with increasing tellurium content. However, dark-decay rates also increase, seven per cent tellurium giving a halftime in darkness of 600 seconds, and 17 per cent tellurium giving a halftime of approximately 10 seconds.

Decreasing the thickness of the top layer of selenium-tellurium from four microns to about one micron improved the dark-decay rate, but gave slightly poorer spectral response. In this case, the top layer contained seven per cent tellurium.

Rahm suggested that these two-layer plates be tested by sensitizing them to negative polarities, as residual potentials have not been a major problem with plates charged negatively.

Andrus stated that the plate containing 20 per cent tellurium in the top layer appeared to be about 15 times more sensitive than selenium plates. Also, varying the deposition time from 1-1/2 minutes to 70 minutes for a top layer containing 10 per cent tellurium showed no definite effect on sensitivity. The conclusion was that the rate of deposition of the top layer is not critical.

Andrus then reported that the large vacuum system had been changed to produce a photoconductive layer with an area of four inches by five inches on a five-inch by seven-inch blank of brass. In this case, the top layer consisted of a mixture of 80 per cent selenium and 20 per cent tellurium, and was two per cent of the total thickness of the plate. Four of six plates of this type made were consistently good, ranking about halfway between standard selenium plates and the best single-layer selenium-tellurium plate produced in the laboratory. These plates are about eight times as fast photographically as selenium plates, but they deteriorated when exposed to light.

MEMORIAL

RESTRICTED

BATTELLE

INSTITUTE

74 . ....

2

10-B7 and 10-B8

#### Adhesive Transfer

Bixby reported the evaluation of more adhesive-coated papers supplied by the Champion Paper and Fibre Company. The manufacturer has reported that there appears to be a correlation between the adhesive materials he uses and the results obtained at Battelle. Tests are now being repeated with a few of the better materials. Plans are being made to determine the forces required to produce satisfactory transfers with these materials.

# RESTRICTED

MEMORIAL

ELLE

PFENDIX C

÷ij

#### MINUTES OF SPONSOR MEETING

#### December 17, 1952

Subject: "Continuous-Tone Electrostatic Electrophotography" Contract DA-36-039-SC-123

Persons Present: Steven Levinos, U. S. Army Signal Corps James M. Chapman, Wright Air Development Center Lt. James Mozer, Wright Air Development Center

R. E. Hayford, The Haloid Company

H. E. Crumrine, The Haloid Company

- W. T. Reid, Battelle
- L. E. Walkup, Battelle
- P. G. Andrus, Battelle
- W. E. Bixby, Battelle
- H. E. Medley, Battelle
- E. C. Ricker, Battelle
- B. A. Goldis, Battelle
- R. K. Trask, Battelle

This meeting was held on December 17, 1952, to discuss the present status of work or this project, and to plan work to be done between now and January 29, 1953, the tentative date of the next meeting, also to be held at Battelle.

The statements set forth in these minutes shall not be considered as evidence of inventorship nor as constituting recognition of the novelty or originality of any of the ideas or suggestions proposed by any of the participants.

#### Radioactive Contamination of Selenium Plate

Levinos opened the meeting by asking about the explanation given by the U. S. Radium Corporation concerning the radioactive contamination which occurred when a very strong source of polonium-210 was left near a selenium-coated electrophotographic plate. Their explanation was that selenium vapor combined with the silver-coated base plate on which the polonium was deposited and formed silver selenide. This material then separated from the base plate, and in doing so released small amounts of

BATTELLE MEMORIAL INSTITUTE

#### 10-C2

polonium from the source which were carried by air currents to the electrophotographic plate and the walls of the container. Levinos stated that he thought this analysis was incorrect. He suggested that the reaction was one of decomposition of water vapor caused by the intense ionization, with the resulting free hydrogen combining with the polonium to form polonium hydride, a volatile compound which could then diffuse throughout the chamber. He suggested that we forward this explanation to U. S. Radium.

#### Two-Layer Plates

#### **Review of Past Work**

Andrus reviewed the work done prior to the last meeting on the preparation of two-layer selenium-tellurium plates. These plates were coated with 45 microns of selenium as the bottom layer, over which was deposited five microns of a mixture of selenium and tellurium as the top layer. The amount of tellurium in the mixture was varied from 7 per cent to 20 per cent. The sensitivity of these plates as a function of wavelength of radiation increased as the percentage of tellurium in the top layer was increased, up to the maximum concentration, or 20 per cent, of tellurium used. The area under the sensitivity curve for the plate containing 20 per cent of tellurium in the top layer was 17 times the area under the sensitivity curve for a selenium plate. This concept of the area under the sensitivity curve being an indication of the sensitivity of the plate is the same as has been used previously.

#### Effect of Tellurium Concentration on Spectral Response

To determine the change in spectral response of the plates as the percentage of tellurium was varied, a new measure of panchromaticity was applied to the data on these plates. This definition states: "The effective panchromatic cutoff point is at a wavelength where the sensitivity of the plate has decreased to one-fifth the maximum value it has at any point in the spectrum". After some discussion, it was agreed that, although arbitrary, this definition does have definite value and can be used to advantage in describing the spectral response of electrophotographic plates.

Using this definition, the effective cutoff wavelengths were plotted against the percentage of tellurium in the top layer of the various plates of this series. It was found that these cutoff points lie on a straight line, with the effective cutoff point moving to longer and longer wavelengths as the percentage of tellurium increases. For a selenium plate, this cutoff point is 505 millimicrons. For the plate containing 20 per cent tellurium in the top layer, the effective cutoff point is at 735 millimicrons, well beyond the limit of human visibility.

BATTELLE MEMORIAL INSTITUTE

#### 10-C3

#### Effect of Thickness of Top Layer on Spectral Response

Andrus reported on a series of plates made with the top layer of the plate representing only two to four per cent of the total thickness of the coating. The bottom layer again was of selenium, and all of these plates were made with a top layer containing 20 per cent of tellurium. The effective spectral cutoff point was consistently between 650 and 700 millimicrons. The sensitivity, of these plates, defined as the reciprocal of the time in seconds required for the plate to decay from 200 to 100 volts when exposed to light having an intensity of 0.030 watt per sq cm at 400 millimicrons, varied more widely, from 0.6 to 1.0. Dark-decay rates varied considerably, the time required for the potential to decay from 200 to 100 volts for different plates being from 15 seconds to 1000 seconds. Most of these plates, however, had dark-decay times between 100 seconds and 300 seconds, even though the top layer contained 20 per cent tellurium. The possibility of tolerating even faster dark decay suggests that higher concentrations of tellurium might prove advantageous.

#### Effect of Deposition Rate and Backing-Plate Temperature

Another series of plates was made to determine the effect and the importance of the rate at which the coating material is evaporated and of the temperature of the backing plate during deposition. Plates were made at backing-plate temperatures of 60 C, 70 C, and 80 C, the top layer being deposited at a rate of one-sixth, one-twentieth, and one-sixtieth gram of selenium-tellurium leaving the crucible each minute; three, ten, and thirty minutes, respectively, were required to powder the top layer. In each case, the bottom layer of selenium was deposited in 10 minutes, but at the temperature corresponding to that used in depositing the top layer. The top layer contained 20 per cent of tellurium and was two to three microns thick; the total thickness of the coating was approximately 60 microns. Three plates were produced at each of the nine possible combinations of variables, giving a total of 27 plates in this series. These plates were made in random order, to decrease the effect that progressive changes in equipment, room atmosphere, or other uncontrolled variables might have.

The maximum value of the sensitivity of these plates, as defined by the conditions described previously, varied from 0.88 to 1.2. This is considered to be a small variation, as it is less than observed among plates made under conditions which are assumed to be identical. It was concluded, therefore, that there is little or no significant effect on the sensitivity of plates due to variation in the rate of deposition or the temperature of the backing plate, within the fairly wide range of each variable investigated.

MEMORIAL

RESTRICTED

INSTITUTE

BATTELLE

#### 10-C4

The effective spectral sensitivity cutoff, defined again as that wavelength at which the sensitivity falls to one-fifth of its maximum value, showed some correlation with the temperature of the backing plate and the rate of deposition. This cutoff value was extended approximately 20 millimic rons toward the longer wavelengths when either the deposition time was increased from three minutes to 10 minutes, or when the temperature increased from 60 C to 70 C. When these conditions were used simultaneously, the cutoff was extended some 40 millimicrons toward longer wavelengths when compared with plates made at 60 C and a deposition time of three minutes. For plates made with the longest deposition time, 30 minutes, and the highest temperature of the backing plate, 80 C, the results were intermediate to those just described. A statistical analysis of these data revealed them to have a significance level of approximately 10 per cent, or, in other words, there is a 90 per cent probability that the trends noted are true effects of the variables studied. One other measurement made on these plates, but for which no trends could be established,  $\mathcal{O}$ was that of dark-decay rates. The halftimes of dark decays varied from 8 seconds to 600 seconds.

#### Effect of Other Variables

The next series of experiments on this problem is designed to investigate the importance of one other variable, the pressure, within the vacuum chamber during deposition. Also to be studied further is the effect of the thickness of the top layer applied to the selenium base layer, and of the concentration of tellurium in the top layer.

#### Measurement of Charge Applied to Plate

Andrus reported that a device has been added to one electrometer to permit measuring the amount of charge flowing to the plate during sensitization. This is done by placing a known capacity in series with the plate during sensitization and reading the potential which this condenser acquires. The electrophotographic plate is masked so that a known area is sensitized. Knowing this area, the capacity of the electrophotographic plate, and the voltage to which the plate is charged, the amount of charge remaining on the plate can be computed.

With a selenium plate, approximately 90 per cent of the charge which flowed to the plate was retained on the plate. Approximately 0.5 microcoulomb is required to charge an area of ten square centimeters of a 50micron-thick selenium plate to 500 volts. This method of measuring the charge on a plate should aid in improving the reproducibility of sensitization of plates being tested on the vibrating-probe electrometer.

MEMORIAL

RESTRICTED

BATTELLE

INSTITUTE

#### 10-C5

#### Availability of Selenium

Andrus indicated that some work will be done to find other sources of selenium, because the ARQ brand will apparently be discontinued and because of the ver-all shortage of all types of selenium. Hayford reported that Haloid 1. not nearly so worried about the selenium supply now as they were a month ago, and that they do not presently consider it a critical problem.

#### Adhesive Transfer

Bixby reported that the work on image transfer has continued on the testing of specially coated Kromekote paper. As shown at the previous meeting with the Sponsor, good transfer can be produced with these special papers. However, the force required to produce a satisfactory transfer was not known.

To determine the force required to transfer powder images, a transfer device was constructed in which the forces can be measured. Medley described this device, which consists of two 15/16-inch steel rollers covered with sleeves of rubber 5/64 inch thick and having a Rex durometer hardness of 80, measured with the sleeves on the rollers. The top roller is attached to a lever arm so that forces up to 1000 pounds can be applied to the roller.

Using this device, it was found that several of the papers produced good transfers when a force of only 300 pounds was applied through the transfer rollers. Samples of transferred prints were exhibited, and it was agreed that the picture quality was generally as good as could be obtained with dye-transfer paper. Bixby said that it is now planned to obtain a larger quantity of the better material, perhaps a roll of several hundred feet of paper, and asked what the most desirable weight and whiteness of the paper would be. Levinos suggested a weight and whiteness comparable to that of single-weight photographic paper.

#### Development

In a brief review of previous work, Bixby noted that Freon 12 and air produced distinctly different qualities of images when used as the principal gas in the powder-cloud development apparatus, air yielding the better images. To determine whether the difference in image quality might be due to the difference in the density of viscosity of the two gases, a third

BATTELLE MEMORIAL INSTITUTE

#### 10-C6

gas, helium, was used. The tests are still in progress, but the initial results indicate that helium also is somewhat inferior to air in producing high-quality images. Levinos suggested that the different temperatures obtained as the different gases expand might be a factor to consider, and asked if it would be useful to measure the temperature of the gases being emitted from the capillary tube. Reid suggested that the temperature of the gas could probably be calculated more accurately than it could be measured.

Pictures made using air, Freon 12, and mixtures of Freon and air, were shown by Ricker. He also showed pictures made using a multipletube development unit. This type of unit produces much more uniform development than the single-tube unit. Therefore, a multiple-tube unit is being installed in the rapid-processing camera used with selenium-tellurium plates, where nonuniformity of development is particularly noticeable.

1

# Sensitization

Bixby reported that prior to the last meeting polonium-ion sources had been used to sensitize plates, and that good images had been produced. Since then, experiments have been conducted to determine more specifically how plates can be charged using radioactive sources. The experiments consisted of measuring accurately the ion current flowing from the polonium to a metal plate, varying the distance between them and the voltage gradient. In the experimental arrangement used, the current measured was that flowing to a circular plate surrounded by a guard ring held at the same potential as the circular plate.

Trask presented graphs of current as a function of applied field strength for various spacings of the bare metal plate and the source. These curves showed that the saturation current increased as the spacing was increased to about 1.25 inches. The maximum current obtained between the polonium and the 30 square centimeter circular plate was about 0.20 microampere. Trask explained how these curves were than integrated numerically by an approximation method. These integrations showed how plate potential varied with time for different plate-to-source spacings and field strengths. These calculated curves were similar to the results obtained experimentally in spot checks made to determine the validity of these calculations. For long charging times, the calculated values were somewhat higher than the experimental results.

This work indicates that a 50-micron selenium plate can be charged to 200 volts in 11 seconds using a potential of 300 volts between the present polonium source and the plate when the separation is one inch. More rapid charging could be obtained using higher potentials, but with some sacrifice

#### BATTELLE MEMORIAL INSTITUTE

10-C7 and 10-C8

in control. Walkup pointed out that a cyclic charging procedure involving an initial high-voltage charging period and then a low-voltage period might shorten the charging time without sacrifice of control. Levinos suggested that such a charging procedure be tested.

Hayford asked whether further work had been done on comparing the effects of corona and radioactive sensitization on plate characteristics. Bixby reported that none had been done during the past month.

MEMORIAL

RESTRICTED

FILE

WEB:PGA:OAU:LEW:RMS:WTR/PE:ets January 19, 1953

p