APPROVED: Raymond T. Galvin
(Chairman)

P. Rajeswaran.
(Member)

Walter G. Cleveland
(Member)

ABSTRACT

AN EXPERIMENTAL INVESTIGATION OF INFRARED LUMINESCENCE IN BALLPEN AND LIQUID INKS

by William H. Storer

This study, of interest to questioned document examiners, discusses a non-destructive photographic technique which permits the detection of infrared luminescemce in ballpen and liquid inks. It is a technique that can be of assistance in the differentiation of writing inks, and in the restoration of obliterated or erased writings.

The phenomenon of infrared luminescence is that fluorescence which occurs in the near infrared spectral region. It is most easily detected by infrared photography or with an electronic viewing device such as the electronic image converter used in a military "sniperscope."

The equipment used in the study is as follows:

a) Light source: A 35mm 500 watt slide projector. While infrared luminescence can be induced by an ultraviolet excitation source, it is most efficiently induced by a high energy incandescent visible light source. Photofloods and similar incandescent bulbs can be used. However, the slide projector permitted better manipulation of the light source, and it generated less heat.

b) Film: Kodak 35mm High Speed Infrared. This film is approximately eight times faster than standard infrared film; it also exhibits a sensitivity that extends beyond that of standard infrared film. The film was developed for ten minutes at 68 degrees F. in Kodak D-76.

c) Camera: A 35mm Exakta with an f/1.9 lens.

d) Filters: A 10% aqueous solution of copper sulfate in a flat sided one pint wine bottle having a light path of 35mm was placed in front of the slide projector lens. The copper sulfate filter served to absorb the infrared that was generated by the slide projector bulb.

   A Wratten 87C infrared transmitting filter was used over the camera lens. Its function was to absorb any visible light that was reflected from the ink samples and from the various papers upon which the ink lines had been drawn.

   The use of the copper sulfate filter and the 87C filter ensured that only infrared luminescence and not reflected infrared or visible light was recorded on the film.

   A Kodak +2 close-up attachment was used on the camera lens in order to provide an image that was larger than that provided by the standard camera lens.

The technique provides a superior sensitivity to weak infrared luminescence in comparison to the results achieved with the electronic image converter, or with standard speed infrared film. In addition, previously reported film exposure times of from 15 to 30 minutes can by this technique be reduced to approximately 15 to 30 seconds. The use of the Kodak High Speed Infrared film in a 35mm camera at a lens opening of f/1.9 permitted the reduced exposure time.

One of the salient features of the technique is its capability of supplementing or even surpassing the results provided with ultraviolet examinations or with standard infrared photography. The study specifically demonstrated (1) that all inks do not exhibit infrared luminescence, (2) that inks of colors other than blue may exhibit infrared luminescence, (3) that inks which exhibit similar visual colors may exhibit distinctly different infrared luminescent properties, (4) that the color of the paper upon which an ink writing appears can significantly influence the infrared luminescent properties of the ink, (5) that the ultraviolet characteristics of a white paper stock apparently do not affect the infrared luminescent properties of an ink, (6) that caution is required when evaluating the infrared luminescent properties of a liquid ink, because a liquid ink from an unshaken bottle can exhibit inconsistent luminescent characteristics.

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By

William H. Storer

A THESIS

Submitted to
Michigan State University
in partial fulfillment of the requirements
for the degree of

MASTER OF SCIENCE

School of Police Administration and Public Safety

1964

ACKNOWLEDGMENTS

The author wishes to express his appreciation for the constructive criticism and invaluable guidance provided by Mr. George G. Swett in bringing the experimental phase of this study to completion.

The author wishes to acknowledge also the guidance and criticism provided by Mr. P. Rajeswaran and Mr. Ralph F. Turner both of whom contributed to the drafting of this study.

The study was conducted in the Laboratory Division of the St. Louis Metropolitan Police Department, Lieutenant Dell R. Watts, Laboratory Commander.

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CHAPTER I

THE PROBLEM AND DEFINITIONS OF TERMS USED

Several research workers in the field of questioned document examination have called attention recently to a relatively unexplored investigative technique. It is one whereby the phenomenon of infrared luminescence may be detected or shown to be absent in inks and other writing materials. The potential value of this technique as a means of detecting alterations and additions, as a means of restoring obliterations, or as a means of demonstrating differences between writing materials has been discussed in the literature (1,2,4,5,6,7,14). However, no formal detailed reports have as yet been presented.

I. THE PROBLEM

Statement of the problem. It was the purpose of this study (1) to present a simple technique which would produce effective infrared luminescence detection; (2) to determine the conditions and circumstances under which effective detection may be expected; (3) to determine the practical limits of the technique.

Importance of the study. The examination of questioned documents is usually limited to non-destructive techniques. Regardless of its form, the value of a questioned record or document may and usually does transcend its temporary role as evidence when it is under suspicion.

The evidential value of a bullet, a blood stain, a hair, or a paint fragment is created with the inception of its related offense. Their values normally cease when the judicial processes have been fulfilled. Not so with the questioned document; particularly a questioned document which eventually discloses its genuineness. The destruction, obliteration, or alteration of a document during the examination may be an act exceedingly more offensive than the suspected act under immediate consideration. Accordingly, chemical and similar destructive test methods are rarely permissible. The competent examiner of questioned documents strives to establish facts by thorough examinations. However, thoroughness does not preclude his responsibility for maintaining the evidential integrity of the document in question.

The elimination of chemical and other altering or destructive test measures from the category of investigative techniques intensifies the importance of interrelated optical and spectrum analyses--or more simply, the importance of the visual examination. Accordingly, the examiner finds himself concerned with the investigative powers of radiant energy in the form of light. He constantly seeks non-destructive techniques which will supplement or improve the current non-destructive methods, several of which are: high and low power magnification which has the capability of revealing information not discernible to the unaided eye, and which often indicates the results that may be obtained from subsequent methods of examination (8); oblique and transmitted illumination which have the capability of disclosing indented writings, erased areas, and watermarks (9); filter photography which has the capability of restoring obliterated writings and over-lapping multi-colored writings (10); masking techniques which have the capability of separating overlapping multi-colored writings or stamping inks (6,11); infrared photography which has the capability of restoring obliterated, charred, erased, or otherwise invisible writing (12); fluorescence induced by ultraviolet illumination which has the capability of detecting and deciphering chemical alterations or eradications, of differentiating certain writing materials through observable differences in color of fluorescence, and the detection of invisible writings by the photography of ultraviolet images (13); selective wavelength examinations through dichroic filters (14); the use of electronic image converter tubes as substitutes for straight infrared photography (15); and the adaptation of automatic recording microdensitometers in the recognition and evaluation of specular and physical characteristics of writing materials (16).

There are, unfortunately, instances when the investigative capabilities of the current methods fail to reveal any latent significant information that a questioned document may bear. Accordingly, there exists the necessity for the development of additional non-destructive investigative techniques. The phenomenon of infrared luminescence (that fluorescence which occurs in the near infrared region of the spectrum) provides an additional non-destructive method of examination. In the present study a technique was devised and employed to photographically detect and record the infrared luminescent capabilities of writing inks.

Hypothesis. The detection of infrared luminescence in writing materials offers the examiner of questioned documents a technique that can supplement or extend his current investigative potential.

II. DEFINITIONS OF TERMS USED

Luminescence. The general term "luminescence" includes the emission of light that results from all processes except incandescence. It is a phenomenon of radiation which occurs during and/or after which a material capable of luminescence has been exposed to some form of external photon radiation. Since the generic term (Latin, Lumen, "light" + -escence) denotes a process of generating radiation during and after excitation, luminescence includes fluorescence and phosphorescence. Fluorescence describes the emission of light at the same time as excitation; phosphorescence describes the emission of light that follows excitation.[1] According to Leverenz, luminescence should be limited to the emission of visible radiation (1.7 e.v. to 3.1 e.v. photons); but this traditional limitation is generally ignored because the human eye is but one of many photosensitive devices now being used to detect luminescence.[2] e.g., image tubes and photographic emulsions.[3]

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[1] Humboldt W. Leverenz, An Introduction to Luminescence of Solids (New York: John Wiley & Sons, 1950), pp. vii-viii.

[2] Ibid., p. 136.

[3] Ibid., p. 460-464.

Luminescence, as characterized by Leverenz, is the emission of radiation that is in excess of the thermal radiation produced by heat in a given material. Thermal radiation is radiation that is emitted after absorbed energy is converted into low-quantum-energy heat that diffuses through the material. Luminescence radiation is radiation that is emitted after an appreciable part of the absorbed energy is temporarily localized as relatively high-quantum-energy excitation of atoms or small groups of atoms.[4]

Luminescence results when an excited atom or group of atoms is raised to a higher energy level and then returns to a state of lower energy.[5] The optical energy difference between the two levels is emitted in the form of photons. In the case of optical excitation, the emitted radiation almost always has a lower frequency than the exciting radiation. Further, it is essential for the exciting radiation to be absorbed if it is to be re-emitted as some form of luminescence. According to the law of conservation of energy, the emitted light must have the

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[4] Leverenz, op. cit., pp. 1-2.

[5] Jack De Ment, Fluorescent Chemicals, (Brooklyn: Chemical Publishing Co., 1942), pp. 4-5; Humboldt W. Leverenz, An Introduction to Luminescence (New York:John Wiley & Sons, 1950), Chaps. I and V: Peter Pringsheim, Luminescence of Liquids and Solids (New York: Interscience Publishers, 1943), pp. 8-19.

same energy as or smaller than the incident radiation, i.e., it must have a longer wavelength.[6]

Familiar examples of luminescence which is visible to the unaided eye are the narrow spectral lines and bands of radiation emitted by (1) electronically excited gases, such as in lightning and neon lamps, (2) certain oxidizable organic matter in liquids exposed to air, for example in glowworms and fireflies, and (3) coatings of tiny phosphor cyrstals excited by invisible alpha particles, electrons, and ultraviolent, as in luminescent watch dials, television picture tubes and "fluorescent" lamps. In all these cases of luminescence, the temperature of the luminescing material is best maintained near or below room temperature. Also, the quality and quantity of luminescence radiation are strongly dependent on the nature of the emitting material; the quality and quantity of thermal radiation depend chiefly on the temperature rather than on the nature of the emitting solid material.[7]

Infrared luminescence. A phenomenon similar to visible fluorescence under ultraviolet light except that the emission occurs in the infrared region and hence is invisible to the human eye. Chapter II discusses two

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[6] De Ment, *op. cit.*, p. 1.

[7] Leverenz, *op. cit.*, p. 2.

relevent methods of detecting infrared luminescence. While infrared luminescence can be induced by violet and ultraviolet excitation, Barnes (4) and Godown (1) found that it is most efficiently excited by visible light from high-energy incandescent lamps.

According to Leverenz this luminescence phenomenon, which has as its excitation source <u>low-energy photons</u> (visible light or UV), would be a luminescence designated as <u>photoluminescence</u>.[8]

<u>Infrared spectrum</u>. That region of the spectrum that extends out indefinitely from the red end of the visible spectrum. As the wavelengths increase, the radiation emerges into heat waves and finally into radio waves. For the purpose of this report, the infrared spectrum will encompass that region from approximately 7000 A to 9000 A. This infrared region is that to which the infrared film used in this study was sensitive. Maximum sensitivity of the film was within the range 7700 A to 8400 A.[9]

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[8] Leverenz, <u>op</u>. <u>cit</u>., Table 10, p. 148.

[9] "Cine-Kodak and Kodak High Speed Infrared Film," Kodak Pamphlet No. M-9, Rochester: Eastman Kodak Company.

CHAPTER II

METHODS OF DETECTING INFRARED LUMINESCENCE

Infrared luminescence can be detected most conveniently by either of two methods. One method utilizes an infrared image converter tube of the type present in the military sniperscope. The second method utilizes any suitable camera loaded with infrared film. (Figure I).

I. INFRARED IMAGE CONVERTER TUBE

This device has an objective lens that focuses the primary image on a cathode which is so sensitized that it will give off electrons when illuminated by ultraviolet, visible, or infrared radiation. A typical cathode for this purpose is a silver-cesium oxide-cesium film. When an image is projected on the cathode, electrons will be emitted from its surface in proportion to the illumination at any point. The emitted electrons form an electronic image which is a reproduction of the light image. The electronic image is focused on a zinc silicate phosphor screen after the image has been accelerated to a high velocity by a series of high-voltage electric fields. The electrons (Electronic image) falling on the screen excite it to fluorescence, and a visible green image is seen on the screen. The image is viewed through an eyepiece. If a permanent record is desired, the image can be photographed with any green sensitive film.[1,2,3] Figure 2 illustrates the infrared image converter tube.

Since the military device is designed to focus on objects at a distance greater than four feet, it is necessary that a close-up attachment be fitted over the objective lens. A close-up attachment of approximately three diopters will permit a viewing distance of about nine inches. Supplementary lenses of other than three diopters may be used if different viewing distances are required.

The use of an image converter tube device for the detection of infrared luminescence requires a totally darkened room and a properly filtered light source to ensure that no stray light will fall on the object and interfere with the luminescence phenomenon. A source of high incandescent energy is used to illuminate the object and to excite the infrared luminescence which the object may be capable of producing. However, all incandescent lamps emit more energy in the infrared than in the visible

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[1] Walter Clark, Photography by Infrared (New York: John Wiley & Sons, 1946), pp. 123-125.

[2] David Barnes, "Infrared Luminescence of Minerals," Geological Survey Bulletin 1052-C (Washington: Government Printing Office, 1958

[3] Leverenz, op. cit., p. 460.

range of the spectrum. If infrared radiation is permitted to strike the object under observation, some of the infrared radiation will be reflected and will be observed on the screen. Such reflected radiation can either mask completely or at least weaken the sought for infrared luminescence. In addition, reflected infrared may be erronously identified as infrared luminescence. It is therefore necessary that the infrared radiation which is emitted by the light source be eliminated. Effective filtering may be accomplished by placing a solution of copper sulfate of suitable concentration in front of the incandescent light source. Proper precautions must be instituted to ensure that no stray unfiltered light strikes the object. If the copper sulfate solution is effective in filtering infrared from the light source, then only visible light will strike the object and will accordingly excite any latent infrared luminescence.

The infrared image converter is sensitive to visible light as well as to infrared. Therefore, any object which reflects visible light can be observed on the screen. If we desire to observe only infrared luminescence, the reflected visible light must be filtered before it strikes the image tube. Effective filtering is accomplished by attaching an appropriate infrared transmitting filter over the objective lens of the image converter tube.

II. PHOTOGRAPHIC METHOD

The photographic method is essentially the same as the image converter tube method. The major difference is that a camera loaded with infrared film is substituted for the image converter tube. The filtering procedure is identical to the already described. That is, the incandescent source is filtered through a copper sulfate solution, and the reflected visible light from the object is filtered by placing a suitable infrared transmitting filter over the camera lens. The photography is performed in a totally darkened room except, of course, for the filtered light source. (See Fig. 3).

One factor which must be considered with infrared photography is that of focusing compensation. Infrared rays, because of their longer wavelength, do not focus in the same plane as visible rays. It is usually necessary to make an increase in the lens-to-film distance in order to correct for the focusing difference between infrared and visible rays.[4] For best definition, infrared

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[4] Walter Clark, <u>Photography by Infrared</u> (New York: John Wiley & Sons, 1946), pp. 14-18.

photographs should be exposed with the smallest lens opening that conditions will permit. This is particularly important since adjusting the lens-to-film distance corrects only for longitudinal color aberration; ordinary photographic lenses are not corrected in other aberrations for the infrared part of the spectrum.

III. ADVANTAGES AND DISADVANTAGES OF THE TWO METHODS

The image converter tube method and the photographic method each offer distinct advantages and disadvantages. The prime merit of the image converter is its capability of rendering rapid infrared luminescence detection. The image converter tube and its necessary accessories require little more bench space than a standard laboratory microscope. It can thus always remain set up for immediate use; an examination with the image converter could probably be completed in the time it would take just to prepare a camera in the photographic method.

The undesirable features of the image converter method are more numerous. The physical hazards of working with an electrical device which generates high voltage is a most important consideration. Since most of the available image converters are secured through the surplus market, the purchaser must thoroughly check the entire unit to ensure that the wiring and other electrical connections are securely insulated and safe. At the time of this report surplus image converters are not readily available on the market. Those image converters which are available will often be of poor quality or in poor mechanical condition; the purchaser of a surplus "sniperscope" or "snooperscope" assumes the risk of an early mechanical breakdown. To add to the difficulties, replacement parts are practically non-existant.

The operator of an image converter will usually find that the sensitivity of the device to infrared luminescence is less than that of infrared film. The sensitivity of the operator's eyes are also a factor that can contribute to the problem. It is certainly conceivable that faint luminescence may go undetected when both of these sensitivity factors come into play. The resolution of the image converter is also less than that of the camera lens-infrared film combination. Since the phosphor screen of the image converter was not designed to produce needle-sharp images, the operator can expect to experience difficulty in deciphering restorations.

The photographic method offers few, if any, real disadvantages. Its capabilities of providing superior sensitivity and resolution outweigh its equipment requirements and lengthier manipulations.

Because of the anticipated advantages of superior sensitivity and resolution, the photographic method was used in the preparation of this study. An image converter was used to compare its own sensitivity with that of the photographic experimental method.

IV. INFRARED-SENSITIVE FILMS

Kodak High Speed Infrared Film (HIR402) was used in the preparation of this study. Since Kodak infrared materials are ordinarily used in the United States, this discussion will be limited to Kodak products.

Most photographic films, whether they are orthochromatic or panchromatic, are not sensitive to infrared. Only when the emulsion is treated with special dyes can infrared sensitivity be attained.[5] While some of these dyes have been known for a long time, it was not until 1931 that dyes were discovered which made infrared photography as simple as photography with normal materials.[6]

In 1925, when H. T. Clarke at the Kodak Research Laboratories was preparing the dye Kryptocyanine, it was noted by him that a second dye was formed during the reaction. This second dye was found to sensitize film from 6,500 A to 9,000 A; it was termed Neocyanine. The maximum sensitivity of Kryptocyanine extended only to 8,000 A.

[5,6] "Infrared and Ultraviolet Photography, " Kodak

By treating the Neocyanine sensitized plates with ammonia, it was possible to photograph the spectrum to beyond 10,000 A. In 1930 using Neocyanine plates, Babcock, of Mount Wilson Observatory, photographed the solar spectrum as far as 11,634 A, the farthest limit that had ever been photographed. Babcock also made the first photograph in total darkness using heat rays, the plate being sensitized with Neocyanine.[7]

During the years 1931-1935 a new group of sensitizers were developed. This group, called the tricarbocyanines, made it possible to photograph the spectrum to beyond 11,000 A. Meggers and Kiess, at the Bureau of Standards in Washington, succeeded in photographing hundreds of new spectral lines out to about 12,000 A.[8]

Since 1934-1935, development of the tetra- and pentacarbocyanines have permitted the sensitizing of photographic plates that will record infrared out to about 13,600 A.[9]

Kodak infrared materials such as Kodak Infrared Sheet, Kodak Infrared 35mm IR135, Kodak High Speed 35mm IR402, Cine-Kodak High Speed, Kodak Infrared

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[7] Clark, op. cit., pp. 77-90.

[8, 9] Clark, loc. cit.

Sensitive Plates are sensitive not only in the extreme red and infrared regions (6,300 A to 9,000 A) but are also sensitive in the blue region (3,500 A to 5,300 A). These ranges are approximate.[10]

With infrared sensitive materials it is necessary to use over the lens a deep orange or red filter to absorb blue and sometimes visible red light if the photograph is to be made only by infrared radiation. Several of these filters are discussed in Fig. 4.

"Photographs can be made with infrared films without a filter, but the rendering will be similar to that of a blue-sensitive film. The quality will be less satisfactory than that produced by either orthochromatic or panchromatic film. Reds, greens, and yellows will be reproduced darker than normal; blues, lighter."[11] The latitude of infrared materials is less than that of most panchromatic films. Therefore, correct exposure is more critical.[12]

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[10] "Infrared and Ultraviolet Photography," op. cit., p. 5.

[11] Ibid., p. 4.

[12] Ibid., p. 4.

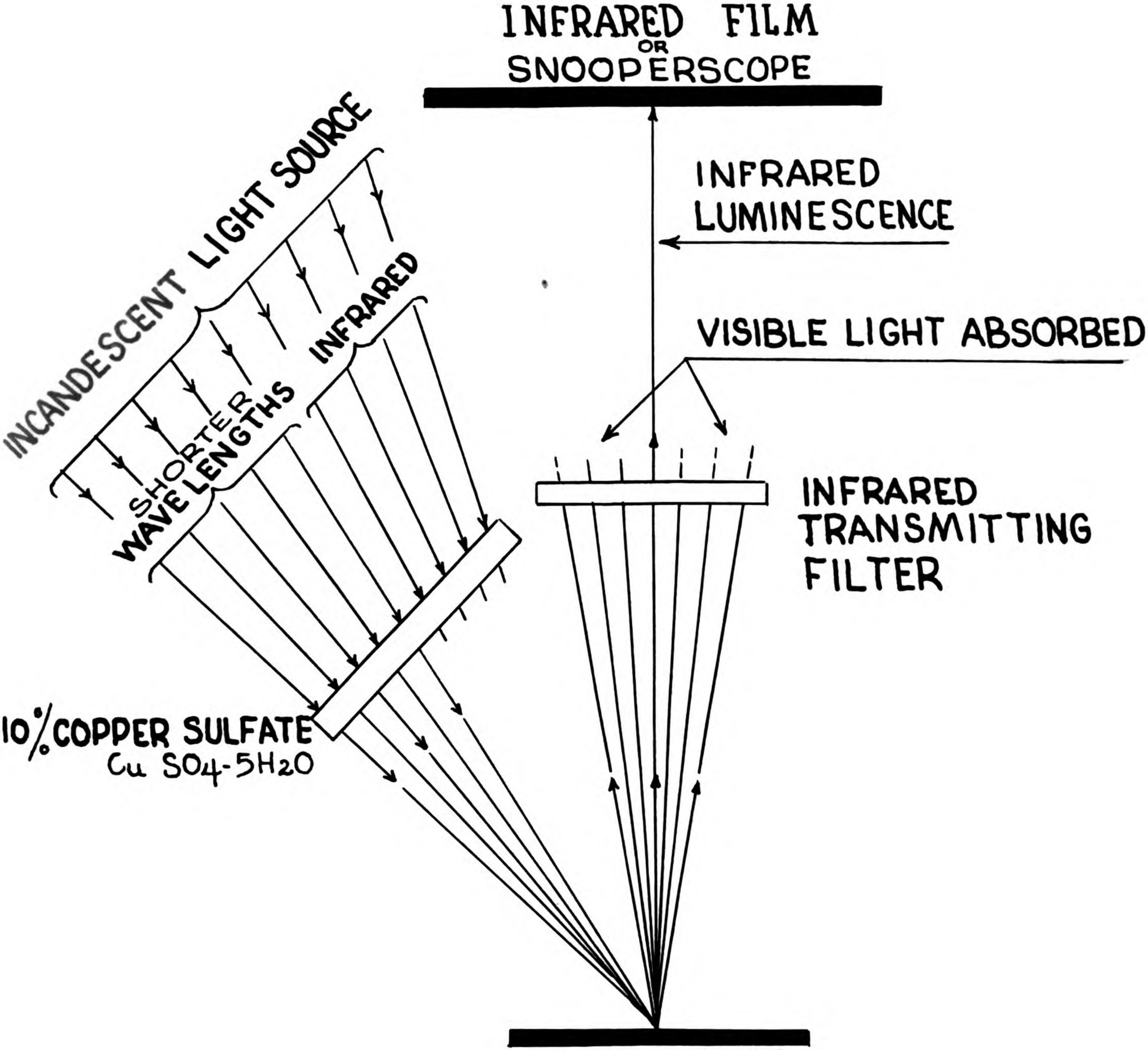

FIGURE 1
A SCHEMATIC OF THE EXCITATION AND DETECTION
OF INFRARED LUMINESCENCE

(Similar to Wilson, (5)

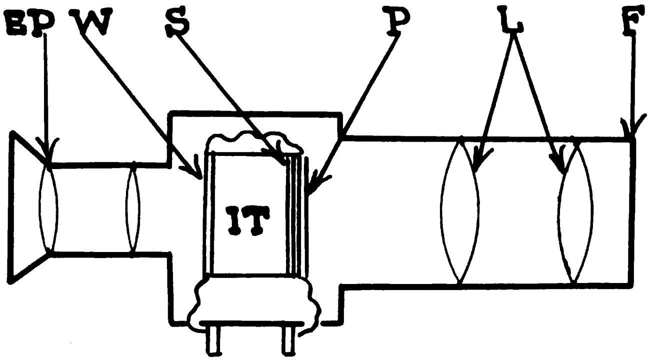

FIGURE 2
SCHEMATIC OF ELECTRONIC IMAGE CONVERTER[13]

Infrared radiation passes through an infrared transmitting filter (F). The radiation is focused by the optical lenses (L) upon the photocathode (P) of the image tube (IT). The electrons released by the photocathode (P) fall upon a fluorescent screen (W) covered with a phosphor of a composition similar to that of cathode-ray tubes. Electrons activate the phosphor (S) to a greenish Fluorescence and the electron image formed on the screen (S), is observed through a magnifying eyepiece (EP).

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[13] Zaboj V. Harvalik, "An Electronic Image Converter and Its Use in Chromatography," Anal. Chem., 22:1149, September, 1950.

CHAPTER III

REVIEW OF THE LITERATURE

The literature abounds with information and techniques which deal with the infrared spectrum and with standard techniques of infrared photography. The infrared spectrum and its photography are, of course, related to the present study. The literature review that follows will deal primarily with works which discuss infrared luminescence and its detection.

There have been included several summaries of papers which discuss the electronic image converter. These papers do not discuss the use of the image converter for the detection of infrared luminescence. However, these references have been included as information sources for those interested in the construction and in the criminalistics applications of the electronic image converter.

I. LITERATURE ON DETECTION OF INFRARED LUMINESCENCE

Godown was one of the first to report the potential value of detecting infrared luminescence in writing materials.[1] His preliminary experiments dealt with the electronic image converter (military surplus sniperscope) and with infrared photography. He achieved satisfactory results with a 5 percent aqueous copper sulfate solution in plastic cells to filter the light source, and Wratten 87, 87C, 88A, and 89B filters on the camera lens. Results were unsuccessful when a solution of nickel sulfate was used at the light source. No exposure or film type data was presented. Godown suggested various areas in which experimental endeavors might be directed. These first efforts by Godown were directly responsible for the inception of continued research in the "questioned document" area by this writer and others.

Somerford described the early work performed by Godown and credits Godown with making the first photograph showing infrared luminescence of writing

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[1] Linton Godown, "Infrared Luminescence," unpublished paper read at the American Academy of Forensic Sciences, Chicago, February, 1960.

materials.[2] Somerford also discussed John Gosling's (Kodak Research Laboratory) unpublished research which yielded satisfactory results with a 5 or 10 percent copper sulfate solution in a one centimeter glass or lucite chamber at the light source, and Wratten 87, 87C, 88A, and 98B filters on the camera lens. Gosling used Kodak Infrared Sheet Film, Kodak Infrared Aerographic Film, Kodak High Speed Infrared Film, and Kodak Photographic Plates. Gosling suggested a 500 watt projector lamp as the light source and an exposure approximately twelve times that required for a normal reflected infrared photograph. Somerford also reported that unpublished research by David Crown (San Francisco Postal Laboratory) "revealed that of 148 ballpen inks examined 66 exhibited luminescence. The latter group generally contained dyestuffs consisting of methyl violet and victoria blue. However, when these two dyes were blended with phthalocyanine dye, luminescence was not achieved. It follows that success in the application of IR-luminescence is dependent upon the presence of certain ink dye components and the absence of induline, and phthalocyanine blue dyes." Somerford included in his report several schematic drawings by Simeon Wilson

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[2] Albert W. Somerford, "Technique of Infrared Luminescence," <u>Identification News</u>, pp. 4-6, 10, July, 1961.

(Chicago Postal Laboratory) which depict the excitation and recording techniques of infrared luminescence.

Gosling presented a technique whereby various combinations of light filters, radiation wavelengths, and photographic films permit the selective discernment of overlapping multi-colored stampings on bank checks which are otherwise unreadable.[3] Gosling mentions that the use of infrared luminescence is one technique which may assist in the differentiation and separation of the unreadable stampings (imprints). For the photography of infrared luminescence he used an 8x10 view camera at 1:1 magnification, an 8-inch f/7.7 Kodak Ektar lens covered with a Wratten 87 filter, and two 500 watt projector lamps covered with glass cells (2 cm. thick) containing 10 percent copper sulfate in water placed at a distance of 24 inches and at an angle of 45 degrees from the copy board. Exposures were made on Kodak Infrared Aerographic Film for two minutes at f/7.7. Negatives were developed for five minutes in Kodak D-19 at 68 degrees F.

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[3] J. W. Gosling, "Photographic Separation of Colored Imprints by Masking Techniques," Identification News, 12:4-10, May, 1962.

Stoll discussed six components and characteristics of papers that provide means of paper identification: fibers, sizing chemicals, pigment fillers and coatings, physical characteristics, watermarks, and reflection properties.[4] In his discussion of reflection properties, Stoll mentioned that comparing the infrared luminescence of paper samples by photography can reveal differences. A photograph is presented which shows the infrared luminescence comparison of sixteen papers.

Barnes, in studying nearly 200,000 mineral specimens at the U.S. National Museum, found that 1,500 specimens would exhibit infrared luminescence.[5] He used a 500 watt photospot as an excitation source, and a surplus military sniperscope as a detecting device. A 5 percent aqueous solution of copper sulfate in a plastic chamber was used to filter the light source; four filters of varying infrared transmission were used in front of the image converter lens. The apparatus was supplemented with an ultraviolet light source and a weak infrared light source.

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[4] Robert P. Stoll, "Analysis and Identification of Paper," Identification News, 13:4-10, 12, September, 1963.

[5] David Barnes, "Infrared Luminescence of Minerals," Geological Survey Bulletin 1052-C (Washington: Government Printing Office, 1958.

The source of weak infrared radiation (25 watt bulb) was covered by an infrared transmitting filter. It was used primarily to distinguish infrared luminescence from reflected light that was leaked by the filters on the sniperscope.

Harvalik used an image converter tube to detect infrared luminescence, infrared reflection, and ultraviolet reflection on chromatographic columns.[6] Detection of infrared luminescence was achieved by a mercury arc source that was filtered by a combination of a Corning 3389 and a 4407 filter; a 5860 Corning filter was used when a higher excitation source was required. A Corning 2540 was used to cover the converter tube. Reflected infrared was observed by placing a Corning 2540 filter in front of the light source. Reflected ultraviolet was detected by using a mercury arc as a light source in connection with a Corning 5860 ultraviolet filter; a Corning 5860 filter was also attached to the converter tube in order to prevent any visible ultraviolet fluorescence from being observed. Harvalik also presented schematic diagrams of the converter tube and its power source.

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[6] Zaboj V. Harvalik, "An Electronic Image Converter and Its Use in Chromatography, "Anal. Chem., 22:1149-1151 (1950).

Zyuskin discussed the photography of ultraviolet fluorescence and infrared luminescence.[7] In his discussion of infrared luminescence he suggested that effective differentiation of some inks was possible by varying the copper sulfate concentration (9 to 120 grams/liter) in combination with radiation sources of various wavelengths and camera filters of varying wavelength characteristics. By use of the various filter and source combinations he found that it was possible to decipher and make readable overlapping and multi-colored ink stampings. The technique of Zyuskin is similar in concept to that set forth by Gosling's masking techniques. Much of the technical clarity has been lost in the translation from the original Russian; the Russian filter designations could not be related to American filters.

Hoover and MacDonell discussed glass filters that they found to be useful in infrared luminescence detection.[8]

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[7] N.M. Zyuskin, "The Luminescence Photography by Ultraviolet and Infrared Radiation," Translated from: Zhurnal Nauchoi i Prikladnoi Fotografii i Kinematografii 5 (4), 274-279, 1960. Through courtesy of Eastman Kodak, Rochester, New York; Translated by Carlo A. Bauman.

[8] H.L. Hoover and H.L. MacDonell, "Infrared Luminescence Using Glass Filters," Journal of Forensic Sciences, 9:98-99, January, 1964.

They described: filters that can be used in place of the liquid copper sulfate filter; sharp cut-off filters that are available for differentiating luminescence bands of intermingled substances; filters that are useful with long wavelength sensitive infrared films; and the use of photoflash and electronic flash as radiation sources. Wavelength vs. %Transmission curves for infrared films and filters are also presented.

II. LITERATURE ON ELECTRONIC IMAGE CONVERTERS

Kuhn in 1954 described his adaptation of a British type CV147 image tube for use in the infrared examination of questioned documents (as a substitute for standard infrared photography).[9] Two schematic diagrams, a parts list for construction of the image tube housing, and a parts list for the construction of a power supply are provided. Kuhn suggested that other applications of the image converter tube include its use in analytic chemistry, chromatography, the examination of inks, paper, hairs, fibers, and other objects by a comparison of their properties of reflection, absorption, and fluorescence in infrared and ultraviolet light as seen through the image tube.

Kuhn in 1959 discussed the modification and adaptation of a World War II army surplus sniperscope, RCA type 1P25 for use as a substitute for standard infrared photography in the examination of questioned documents.[10] A schematic show the electrical circuit of the sniperscope power supply. Kuhn compares the British CV147 tube with the RCA 1P25.

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[9] Richard Kuhn, "Infrared Examinations with an Electronic Image Converter," The Journal of Criminology, Criminal Law and Police Science, 45:486-489, No. 4, 1954.

[10] Richard Kuhn, "Recent Developments in the Use of Infrared Image Converters," Journal of Forensic Sciences, 4:11-17, No. 1, January, 1959.

Lechat discusses an apparatus called the "Hainaut H.S.L." which permits the examination of objects by direct and reflected light; it permits photography of normal size and moderate magnification as well as microphotographs.[11] The examinations can be made under infrared, ultraviolet, white, and sodium light. The British image converter tube type CV148 is used in the construction of the apparatus. Photographs, electrical circuits, and parts lists are included.

Edlin discussed his adaptation of a standard laboratory microscope and an electronic image converter for the direct infrared viewing of specimens.[12] Schematics and photographs illustrate the source of infrared radiation, the lens system, infrared filters, the image converter cell, and the high voltage power supply unit.

Harrison (9), O'Hara and Osterburg (18), and Kirk (17) mention in their texts that the electronic image converter is useful for infrared viewing of objects.

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[11] Rene Lechat, "Infrared Photoscopy," Inter. Crim. Police Review, 69:170-179, June-July, 1953.

[12] C.H. Edlin, "Infrared Microscopy Using Electronic Image Conversion," Inter. Crim. Police Review, 96:83-88, March, 1956.

CHAPTER IV

EQUIPMENT AND MATERIALS USED IN THE STUDY

I. PHOTOGRAPHIC

Camera. A 35mm Exakta camera equipped with a 50mm f/1.9 Xenon lens was used for all of the photography. A Kodak +2 close-up attachment was fitted over the camera lens. This attachment permitted the camera to be placed approximately thirteen inches from the object mounting board. At this distance the image size of the ink lines on the film was adequate for detecting the presence or the absence of infrared luminescence. The +2 attachment, however, will not provide a sufficiently large film image for most document problems. Attachments ranging from +6 to +10 would be more suitable for actual questioned document problems. These attachments provide a considerably larger film image and will permit the making of better quality photographic enlargements.

The initial focusing was performed by removing the camera back, placing a piece of onion skin paper over the image opening at the film plane, and then moving the camera to and from the object board until the ink lines were in sharp focus on the onion skin paper. The lens was set at f/1.9. No filter was on the lens during the focusing. The position of the camera was marked on the table so that the camera could be removed when necessary and returned to its correct position without re-focusing.

For the actual photography a Wratten 87C filter was fitted over the close-up lens on the camera. The 87C was selected because of its ability to adequately absorb any visible light which was reflected by the paper stock or ink lines. The 87C provides a sharp transmission cut-off at just below 8000 A. Tests with the 87, 88A, and 89B filters disclosed that they did permit some passage of visible light. (See Figure 4 for % Transmission vs. Wavelength curves). In instances of weak luminescence, the photographing of visible light could definitely mask the luminescence.

All of the photography was performed in a dark room in order to eliminate all light other than that being filtered by the copper sulfate solution. The camera lens was set at f/1.9. No infrared focusing compensation was employed. The negative images, even at this maximum lens aperture, were distinct and sharp without focusing compensation. Tests with smaller lens openings revealed that the slight increase in sharpness, if any, was not significant enough to warrant the necessity of an increased exposure time. Increased exposure time also increases the possibility of camera movement resulting from environmental vibrations; any camera vibration on a 35mm format can nullify the increased sharpness provided by smaller lens apertures. A simple hand magnifying glass was used to view the negatives.

Film. Kodak High Speed Infrared 35mm HIR 402 film was used for all of the photography. It was chosen (1) because of its faster film speed (8X that of regular infrared film), and (2) because of its extended sensitivity into the infrared range. Regular infrared film cuts off at approximately 8720 A; HIR 402 extends to approximately 9000 A.[1]

Exposure Time. Twenty seconds exposure at f/1.9 was used for all of the photography. This exposure combination provided negatives of optimum density.

The following procedure was used in order to avoid possible camera movement caused by the opening and closing of the shutter: A piece of black cardboard was held in front of, but not touching, the camera lens; the shutter was then opened and the hand removed from the shutter release; the black cardboard was removed from in front of the lens and twenty seconds were counted off; at the end of twenty seconds the black cardboard was again placed in front of the lens; the shutter was then closed.

Film Developer and Development. Kodak D-76 developer was used. It is the developer that is recommended by Kodak for use with HIR 402 film. It provided excellent tonal gradations; accordingly it was capable of revealing the presence of extremely faint infrared luminescence. The use of a contrast developer such as

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[1] "Cine-Kodak and Kodak High Speed Infrared Film," Kodak Pamphlet No. M-9. Rochester: Eastman Kodak Company.

Kodak D-19 is not recommended.

The film was developed in a daylight developing tank for ten minutes with 10 seconds agitation at one minute intervals.

Stop Bath and Fix. A standard acetic acid stop bath and Kodak Rapid Fixer were used. Films were washed for twenty minutes, dipped in a wetting agent bath, and hung to dry without being wiped.

II. LIGHT SOURCE AND $CuSO_4$ FILTER

Light Source. A 35mm Kodak slide projector Mod. 500 equipped with a 500 watt bulb was used. The projector lens was retained because it provided a more concentrated beam. A slide projector proved to be the most convenient and efficient excitation source. The built-in fan helped to eliminate the heat problem which is an inconvenient characteristic of photoflood bulbs. In addition, the projector housing eliminated the extraneous light that is difficult to eliminate when using photofloods. Microscope and similar illuminators were found to be inefficient excitation sources because of their low wattage.

The various papers which bore the ink lines were taped one at a time on a vertical backboard. The slide projector was placed at a 45 degree angle to the backboard; the distance from the projector lens to the backboard was 9 1/2 inches. Periodic exposure meter readings were made to ensure that the illumination remained constant throughout the study. The room was totally dark except for the illumination from the slide projector.

Copper Sulfate Filter. The filter consisted of a 10% aqueous solution of copper sulfate in a clear, semi-flat sided one pint wine bottle. The liquid light path was 35mm. The filter was placed in front of and in contact with the projector lens. The bottle provided complete coverage of the lens and the light leaks from the lens housing.

This filter provided efficient absorption of the infrared that emanated from the projector lens and lens housing. However, it was necessary to eliminate the light leaks from the lamp housing vents. The leaks were eliminated by covering the vents with a loose fitting box. The box was removed immediately after each exposure was made. This prevented the projector from overheating. The copper sulfate filter did not overheat at any time.

Figure 3 illustrates the photographic set-up. Figure 5 illustrates the absorption curve for 10% aqueous copper sulfate in a one centimeter layer.

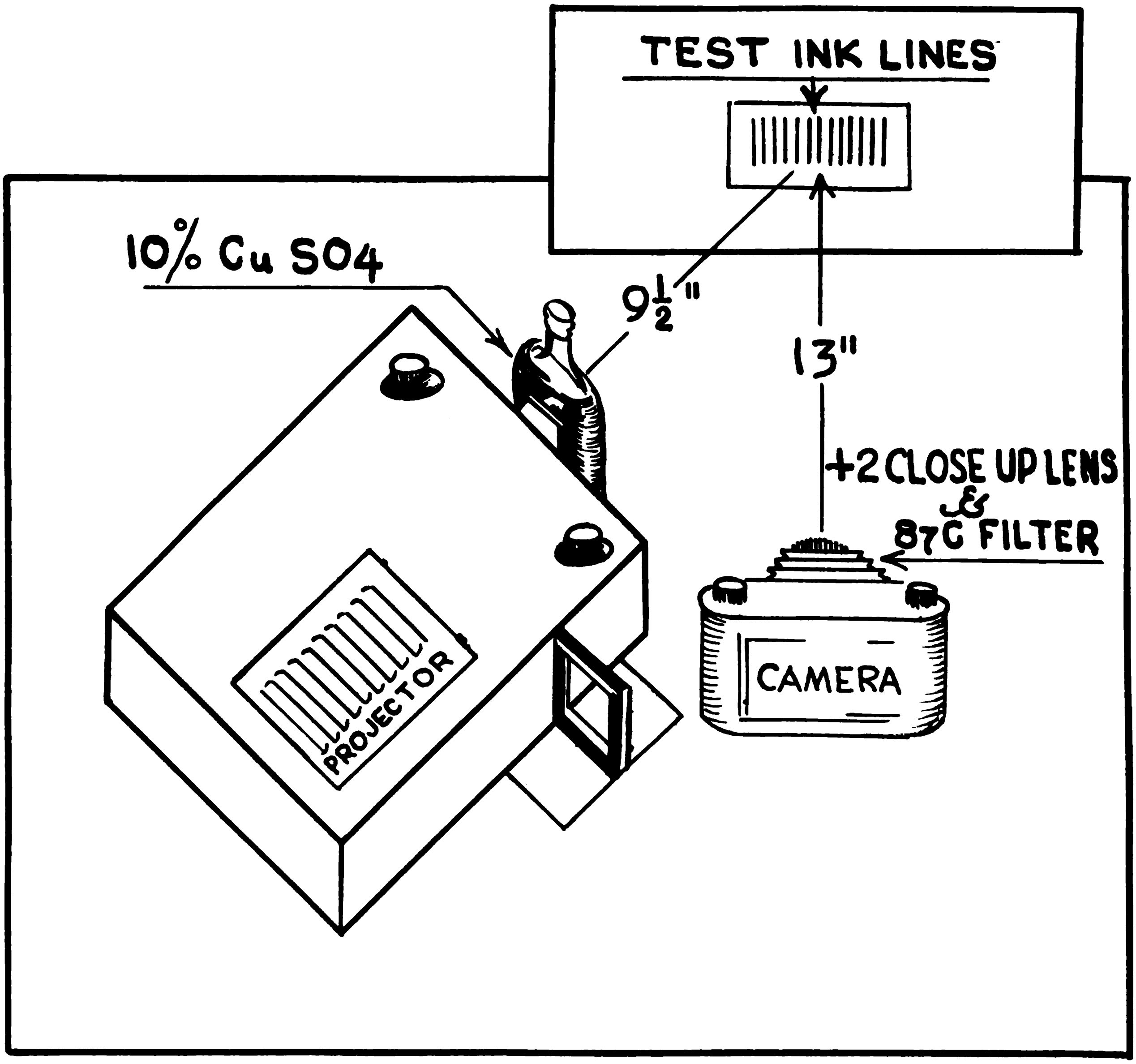
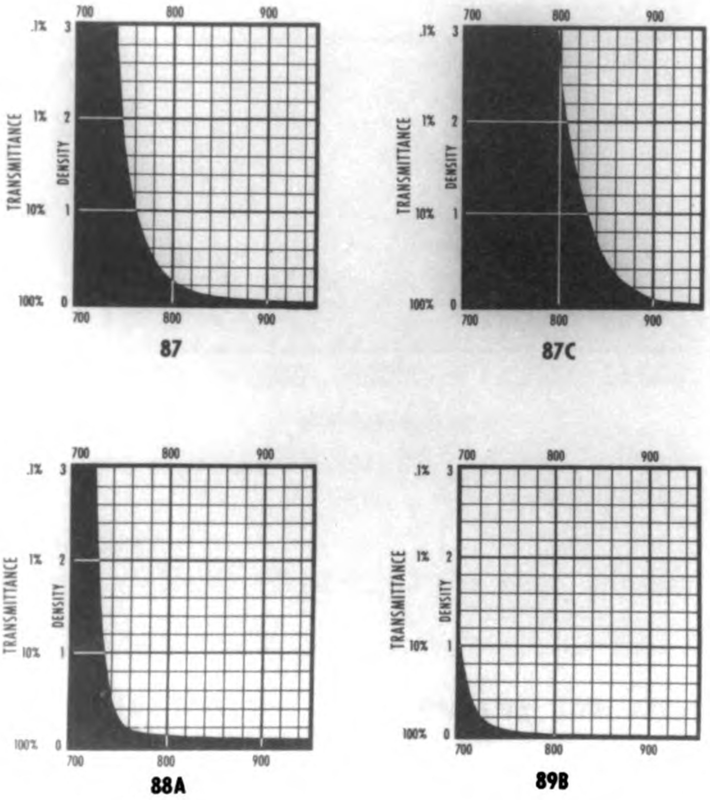


FIGURE 3
DIAGRAM OF PHOTOGRAPHIC SET-UP



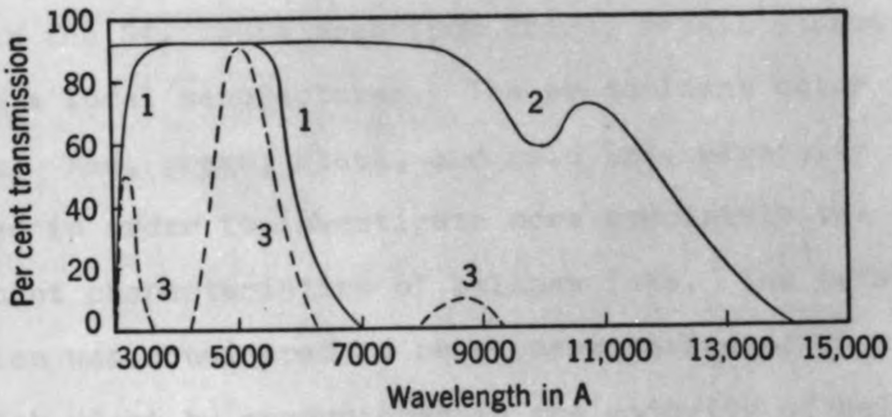
87

87C

88A

89B

FIG. 4 %TRANSMISSION VS. WAVELENGTH;
87,87C,88A AND 89B KODAK WRATTEN FILTERS.
(FROM "KODAK WRATTEN FILTERS FOR SCIENTIFIC
AND TECHNICAL USE", P. 77)



A. 1. Copper sulphate ($CuSO_4 \cdot 5H_2O$), 10 per cent solution in water, 1-cm layer.
2. Water, 2-cm layer.
3. Nickel sulphate ($NiSO_4 \cdot 7H_2O$), 50 per cent solution in water, 1-cm layer.

FIG. 5 % TRANSMISSION VS. WAVELENGTH; 10% AQUEOUS COPPER SULFATE, WATER, 50% NICKEL SULFATE.
(FROM WALTER CLARK, PHOTOGRAPHY BY INFRARED, P. 423)

III. WRITING MATERIALS

Ballpen inks. A total of 92 ballpens or ballpen refills were used. Each was assigned a number. They are listed in Appendix A. The pens and refills were collected in the St. Louis area from users, retail stores, and from a local manufacturer. The predominant color was blue. Red, green, black, and gold inks were also collected in order to investigate more completely the luminescent characteristics of ballpen inks. The total collection was considered to be representative of those inks which might be encountered in the majority of ballpen ink problems.

Liquid inks. A total of 20 bottles of blue and colored inks were collected. Each was assigned a number. They are listed in Appendix B. All of the inks were purchased from local retail stores.

Paper stock. A spiral shaped line of every ink was placed on each of the following 17 paper samples. The paper samples were cut to approximately 3 x 4 inches, and could accomodate ten ink lines.

1. White file cards (3 x 5 inches).
2. One brand of safety paper comprised of the colors yellow, green, and blue.
3. One brand of safety paper comprised of the colors yellow, green, blue, pink,and gray.

4. One brand of white bond comprised of eight different types.

   Each of the eight types exhibited a different ultraviolet fluorescence. They were specifically chosen because of this characteristic difference. The purpose was to determine whether or not differences in ultraviolet fluorescence would affect an ink's infrared luminescence.

The paper samples are listed in Appendix C.

CHAPTER V

EXPERIMENTAL PROCEDURE

The study was organized into six phases. They were (1) the experimental controls, (2) the photography of unaltered ink lines on the seventeen paper stocks, (3) the photography of eradicated ink lines on the seventeen paper stocks, (4) the photography of erased ink lines on the seventeen paper stocks, (5) the photography of obliterated ink lines on the seventeen paper stocks, and (6) the chemical testing of the blue colored ballpen inks for their presumptive constituents.

I. EXPERIMENTAL CONTROLS

The following control procedures were instituted in order to confirm that it was infrared luminescence and not reflected visible or reflected infrared that was being recorded on the film. (It should be recalled that the image of an ink line which exhibits infrared luminescence will be recorded as black or as a shade of gray on the film. Ink lines that are capable of reflecting infrared or visible will also record as black or as a shade of gray on the film).

A representative sampling of those ink lines which exhibited infrared luminescence was selected for rephotographing by the standard infrared photography technique.

(The film characteristic of the apparent luminescent lines was a dense black, or a shade of gray that was definitely darker than that of the paper stock). The standard infrared photographic technique consisted of using an 87C filter on the camera lens, and the slide projector light source without the copper sulfate filter in front of it. The results of this control photography disclosed that those ink lines which had exhibited apparent infrared luminescence were now recorded as either clear lines on the film, lines that exhibited less density than the background paper stock, or the lines were rendered invisible. These are the characteristics to be expected when little or no infrared or visible light is reflected from an object being photographed.

II. UNALTERED INK LINES

A spiral shaped line of every ink was placed on each of the 17 paper stocks. The identifying number that had been assigned to each ink was written at the head of each spiral. The lines were drawn in this irregular shape in order to evaluate the significance of an abrupt change in directional movement. The lines were then photographed by the experimental technique, and the presence or absence of infrared luminescence was determined by a visual reading of the photographic negatives.

III. ERADICATED INK LINES

A spiral shaped line of each ink which had exhibited luminescence was placed on each of the 17 paper stocks. The ball pen ink lines were then eradicated with a saturated solution of calcium hypochlorite. Light rubbing with a cotton tipped stick was necessary to complete the eradication.

The lines which were drawn with the liquid inks were eradicated with Carter's Rytoff eradicator solution. Except for one or two of the non-blue inks, no rubbing was required.

All of the eradicated ink lines were dried at room temperature, and were then photographed by the experimental technique.

IV. ERASED INK LINES

A spiral shaped line of each ink which had exhibited luminescence was placed on each of the 17 paper stocks. Some of the lines were erased with a standard ink eraser; others were erased with an electric eraser. Minimal destruction of the paper stock was attempted.

V. OBLITERATED INK LINES

A spiral shaped line of various inks was placed on the 17 paper stocks. Luminescent and non-luminescent inks were used in various combinations. e.g., non-luminescent inks obliterated with luminescent inks, luminescent inks obliterated with non-luminescent inks, inks that luminesced strongly obliterated with inks that luminesced weakly, inks that luminesced weakly obliterated with inks that luminesced strongly, and inks obliterated with an ink of a different color.

VI. CHEMICAL TESTS FOR CONSTITUENTS

A color spot chemical test procedure devised by Crown was used to classify each of the blue ballpen inks.[1] According to Crown, the procedure permits the presumptive detection of various ballpen constituents. After chemical testing, the inks were classified into specific groups.

Accordingly, ink lines from each of the sixty-nine blue ballpens or refills were drawn on filter paper. Using Crown's procedure, a drop of concentrated hydrochloric acid was added to each line. The resulting color and bleed (if any) was observed and noted. The spot was permitted to dry at room temperature. A drop of saturated

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[1] David Crown, et al., "Differentiation of Blue Ballpoint Pen Inks," The Journal of Criminal Law, Criminology and Police Science, 52:338-343, September-October, 1961.

aqueous sodium bicarbonate was added to the test area. The resulting color or colors were observed and noted. The colors were compared with the color reactions as set forth in Crown's scheme; The ink was then classified into its presumptive constituent group.

Crown's scheme is reproduced as part of Table III. The results of the chemical tests in the present study are set forth in Table III.

Crown's original paper provides a comprehensive description and interpretation of the color reactions.[2]

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[2] Crown, <u>loc</u>. <u>cit</u>.

CHAPTER VI

FINDINGS AND CONCLUSIONS

I. UNALTERED INK LINES

General. Fifty-three of the ninety-two ballpen inks exhibited some degree of infrared luminescence.

Nineteen of the twenty liquid inks exhibited some degree of infrared luminescence.

In addition to the blue inks, some of the reds, greens, blacks, and both golds luminesced. Therefore, the photographic detection technique is applicable to ballpens and liquid inks, and to the variety of colors which comprise them.

The intensity of luminescence was arbitrarily categorized into three degrees of film density: strong, medium, or weak. Luminescence was considered strong if it recorded as black on the film; medium if it recorded as a shade of gray; weak if it was barely perceptible.

Experimental photographic technique vs. converter tube technique. The photographic technique was found to be significantly more sensitive than the image converter tube (sniperscope). Those inks which exhibited a strong photographic luminescence were generally easily detected with the converter tube. However, those which exhibited a medium photographic luminescence were difficult or impossible to detect with the converter tube; inks which were barely perceptible on the film could not be detected with the converter tube.

White paper stocks. The eight white bond papers and the white file card revealed no significant differences in their effect on luminescence. Any ink that luminesced exhibited a consistant degree of luminescence on all nine of the white paper stocks. As previously stated, each of the nine white paper stocks fluoresced differently under ultraviolet light. Apparently there is no correlation between a paper's ultraviolet fluorescence and an ink's capability to exhibit infrared luminescence. At least no relationship was observed in this study.

Colored paper stocks. The study disclosed that a colored paper stock can significantly influence the luminescent capabilities of an ink. The colored safety papers used in this study produced some rather surprising results. For instance, ink #6 exhibited medium luminescence on the white stocks, and weak luminescence on Graham blue and Bergstrom yellow; it did not luminesce on any of the other colored safety papers. Inks #32 and #38 luminesced weakly on the white stocks, weakly on Graham blue, and medium on Bergstrom yellow; these two inks did not luminesce on the other colored safety papers. Another interesting variation was exhibited by ink #67. It exhibited weak luminescence on the white paper stocks, was rendered invisible on Graham blue and Bergstrom yellow, but recorded as a clear line on the remaining colored safety papers. These examples and many others to be found in Table I clearly demonstrate that inks comparisons which involve colored papers should be conducted on the same brand and color of paper. See also photographs in Appendix D.

Visual ink colors. The visual color of an ink provides no indication of the ink's capability to exhibit infrared luminescence. Inks which exhibited similar visual colors or hues were found to exhibit distinctly different luminescent properties. For instance, inks #11 and #12 appeared to be identical in color when viewed under a microscope. However, ink #11 luminesced strongly on all seventeen papers; ink #12 recorded as a clear line on all of the papers.

Directional changes in pen movement. Abrupt directional changes did not appear to alter a ballpen's luminescent capability. Luminescent ballpen lines exhibited no break in luminescence as a result of directional change. Pen pressure did cause a slight variation in intensity. The influence of pen pressure was noticable with those inks which exhibited weak luminescence. These changes in luminescence intensity could usually be accounted for by visually observing a change in the color intensity of the original ink line.

Old or poorly mixed liquid inks. A most significant change in luminescent properties occurred with liquid ink #18 (Waterman's Permanent Black). This bottle was purchased from a local stationary store which was disposing of old inks. The bottle had never been opened. As was the case with all of the liquid inks, the #18 bottle was not shaken prior to dipping the pen. An examination of the #18 negatives (white paper series) disclosed that #18 luminesced strongly, weakly, intermittently, and not at all. No other inks, ballpen or liquid, had exhibited these extreme differences on white paper stock. A visual examination of the original #18 ink lines disclosed that they were of equal color and density on the papers. Accordingly, the #18 bottle was shaken and lines were re-drawn on those papers upon which no luminescence had been detected. An examination of the new negatives disclosed that #18 now luminesced with no breaks in the line. However, there still occurred distinct changes in luminescence intensity of lines on the same paper. These changes occurred when the pen had not been re-dipped frequently. As before, the visual color intensity of the ink lines was similar on all of the papers.

These findings make it definitely conceivable that the liquid ink writing on a document can exhibit extreme variations in luminescence and yet be the same ink. Of course these variations are not likely to occur with fresh ink in which the constituents are homogeneous. The document examiner who is confronted with a liquid ink problem should exercise considerable discretion and caution when evaluation his luminescence observations.

II. ERADICATED INK LINES

The attempted restoration of ballpen inks by infrared luminescence was generally unsuccessful. Several attempts at restoration by standard infrared photography were likewise unsuccessful. In almost every instance the ballpen ink lines required rubbing in order to provide a complete eradication. It was found that even the most gentle rubbing with the hypochlorite solution caused excessive removal of the luminescent constituents.

Luminescence was detected in instances where visual fragments of the ink lines were permitted to remain. However, the experimental procedure was no more effective than the visual examination under a microscope.

No rubbing for eradication was required with the liquid inks. Accordingly, all of those inks which luminesced in the unaltered state could be restored after liquid eradication. Those Skrip inks which contained the fluorescing ingredient RC,35 were of course restorable under ultraviolet illumination also.

Successful restoration of eradicated inks ultimately depends upon (1) the capability of an ink to luminesce and (2) the degree of physical removal of the luminescent constituents.

III. ERASED INK LINES

Generally, the restoration of the ballpen and liquid inks was unsuccessful. As with the eradicated ink lines, success was dependent upon (1) the capability of an ink to luminesce and (2) the degree of physical removal of the luminescent constituents.

IV. OBLITERATED INK LINES

The most successful restorations occurred when both the obliterated and obliterating inks luminesced to different degrees. Strong over weak inks and weak over strong combinations permitted satisfactory restorations. Failure usually occurred when a non-luminescent ink covered a luminescent ink.

No definite rules can be established because of the many interrelated factors which govern the luminescence of the inks involved. The color of the paper stock and the luminescent characteristics of the overlaying ink are probably the most important governing factors. Obliterated ink problems, as a general rule, should be more easily resolved than the problems of erasure and eradication.

Several of the successful restorations are listed here.

#49 over #13 on Bergstrom yellow safety
#45 over #38 on Bergstrom yellow safety
#20 over #57 on Graham green safety
#38 over #53 on Graham green safety
#57 over #20 on Bergstrom blue safety

V. CHEMICAL TESTS FOR CONSTITUENTS

Following the scheme devised by Crown, the ballpen inks used in this study were found to belong to one of the following seven groups:

I. Methyl Violet - Victoria Blue
II. Phthalocyanine - Methyl Violet - Victoria Blue
III. Phthalocyanine - Victoria Blue - Rhodamine B
IV. Victoria Blue
V. Victoria Blue - Indulin
VI. Phthalocyanine - Methyl Violet
VII. Phthalocyanine - Rhodamine B

An evaluation of the chemical test findings disclosed that the presence of phthalocyanine apparently contributes to the non-luminescence capability of a ballpen ink. The presumptive presence of phthalocyanine was indicated in twenty-eight of the ballpen inks. Only four of the phthalocyanine containing inks exhibited luminescence: #32, #38, #67, #85. Inks #32, #38, and #67 exhibited very weak luminescence; #85 exhibited a slightly stronger luminescence. If phthalocyanine does inhibit luminescence, perhaps its quantitative presence in these four inks was so minimal that the luminescence was not entirely quenched.

Thirty-six of the ballpen inks contained the combination methyl violet and Victoria blue. All of these inks exhibited strong luminescence on white paper stocks. If the sampling in the study provided a representative collection, it would appear that the methyl violet - Victoria blue combination is the most prevalent in current use.

Those inks which were classified under Groups IV, VI, and VII did not exhibit luminescence.

The results of the chemical tests are outlined in Table III.

CHAPTER VIII

SUMMARY

A non-destructive photographic technique has been presented which permits the detection of infrared luminescence in ballpen and liquid writing inks. The method, which utilizes Kodak High Speed Infrared film, has demonstrated a superior sensitivity to weak luminescence when compared with results achieved with the electronic image converter or with standard speed infrared film. Previously reported film exposure times of from fifteen to thirty minutes may be reduced to fifteen to thirty seconds.

The study has demonstrated (1) that all colors of inks may exhibit infrared luminescence, (2) that colored papers can significantly influence the degree of luminescence, (3) that an ink will not luminesce on a colored paper if it will not luminesce on white paper, (4) that the UV fluorescent characteristics of a white paper do not affect luminescence, (5) that under suitable conditions successful restorations of erasures, eradications, and obliterations may be achieved, (6) that the accurate luminescence comparison of two inks must be made on the same paper stock, (7) that extreme caution is required when evaluating the luminescence of liquid inks in comparison problems,(8) that a comparison of luminescence between two or more ballpen inks may effectively differentiate them, (9) that inks which exhibit identical visual colors may exhibit distinctly different luminescent qualities, (10) that the detection of the presence or absence of infrared luminescence can serve as a valuable adjunct to the established non-destructive techniques.

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TABLE I

LUMINESCENCE CHARACTERISTICS OF BALLPEN INKS *

INK	WHITE STOCK	GRAHAM SAFETY					BERGSTROM SAFETY		
		YELLOW	GREEN	GRAY	PINK	BLUE	YELLOW	GREEN	BLUE
1	L	L	L	L	L	L	L	L	L
2	L	L	L	L	L	L	L	L	L
3	L	L	L	L	L	L	L	Lw	L
4	L	L	L	L	L	L	L	L	L
5									
6	Lm					Lw	Lw		
7									
8									
9									
10									
11	L	L	L	L	L	L	L	L	L
12									
13									
14									
15									
16									
17	L	L	Lm	L	Lw	L	L	Lm	L
18	L	L	L	L	L	L	L	L	L
19									
20	L	L	L	L	Lw	L	L	L	L
21									
22	L	L		L		L	L		Lw
23	L	L	L	L	L	L	L	L	L

*L denotes strong luminescence
Lm " medium "
Lw " weak "
Empty box denotes no luminescence detected

TABLE I (Continued)

LUMINESCENCE CHARACTERISTICS OF BALLPEN INKS

	WHITE	GRAHAM SAFETY					BERGSTROM SAFETY		
INK	STOCK	YELLOW	GREEN	GRAY	PINK	BLUE	YELLOW	GREEN	BLUE
24									
25									
26									
27	L	L	L	L	L	L	L	L	L
28									
29	L	L	L	L	Lw	L	L	Lw	L
30	L	L	L	L	L	L	L	L	L
31									
32	Lw					Lw	Lm		
33									
34									
35									
36									
37	L	Lm	Lw	Lm	Lw	Lm	L	Lm	Lm
38	Lw					Lw	Lm		
40									
41	L	L	L	L	L	L	L	L	L
42	L	L	L	L	L	L	L	L	L
43									
44	L	L	L	L	Lw	L	L	L	L
45									
46	Lw						Lw		
47	L	L	L	L	L	L	L	L	L

TABLE I (Continued)

LUMINESCENCE CHARACTERISTICS OF BALLPEN INKS

	WHITE	GRAHAM SAFETY					BERGSTROM SAFETY		
	STOCK	YELLOW	GREEN	GRAY	PINK	BLUE	YELLOW	GREEN	BLUE
48	Lw					Lw	Lw	Lw	Lw
49	L	L	L	L	L	L	L	L	L
50	L	L	L	L	L	L	L	L	L
51	Lm	Lw	L	L		L	L	Lw	Lw
52									
53	L	L	L	L	L	L	L	L	L
54	L	L	L	L	L	L	L	L	L
55	L	L	L	L	L	L	L	L	L
56									
57	Lw					Lw	Lw	Lw	Lw
58	Lw			Lw		L	L	L	L
59	L	L	L	L	L	L	L	L	L
60	Lw						Lw		
61									
62	L	L	L	L	L	L	L	L	L
63	Lw					Lw	L		
64	Lm	L	Lw	L	Lw	L	L	Lw	Lw
65	L	L	L	L	L	L	L	L	L
66	Lm	L	Lw	L	Lw	L	L	Lw	L
67	Lw								
68	L	L	L	L	L	L	L	L	L
69									
70									

TABLE I (Continued)

LUMINESCENCE CHARACTERISTICS OF BALLPEN INKS

	WHITE	GRAHAM SAFETY					BERGSTROM SAFETY		
INK	STOCK	YELLOW	GREEN	GRAY	PINK	BLUE	YELLOW	GREEN	BLUE
71									
72									
73									
74									
75	L	L	L	L	L	L	L	L	L
76									
77	L	L	L	L	L	L	L	L	L
78	L	L	L	L	L	L	L	L	L
79	L	L	L	L	L	L	L	L	L
80									
81	L	L	L	L	L	L	L	L	L
82	L	L	L	L	L	L	L	L	L
83									
84									
85	Lm	Lw		Lw		Lm	Lm		
86	L	L	L	L	L	L	L	L	L
87	L	L	L	L	L	L	L	L	L
88	L	L	L	L	L	L	L	L	L
89	L	L	L	L	L	L	L	L	L
90	L	L	L	L	L	L	L	L	L
91	L	L	L	L	L	L	L	L	L
92									

TABLE II

LUMINESCENCE CHARACTERISTICS OF LIQUID INKS

	WHITE	GRAHAM SAFETY					BERGSTROM SAFETY		
INK	STOCK	YELLOW	GREEN	GRAY	PINK	BLUE	YELLOW	GREEN	BLUE
1	L	Lw		Lm		Lm	L		Lw
2									
3	L	L	Lm	L	Lm	L	L	L	Lm
4	L					Lm	Lm		
5	L	L	L	L	L	L	L	L	L
6	L	L	Lw	L	Lw	L	L	L	Lm
7	L	L	Lw	L	Lw	L	L	L	Lm
8	L	L	Lw	L	Lw	L	L	L	Lm
9	L	L	Lm	L	L	L	L	L	Lm
10	Lm					Lw	Lm		Lw
11	L	L	Lw	L	Lw	L	L		
12	L					Lm	Lw		
13	L	L	Lm	L	Lm	L	L	Lm	Lm
14	L	Lm		L		L	L		Lw
15	L	Lm		L		L	Lm		
16	L	L	Lw	L	Lw	L	L	Lw	Lw
17	Lm	Lm	Lw	Lm	Lw	Lm	L	Lm	Lm
18	Lm					Lw			
19	L	L	Lm	L	L	L	L	L	L
20	L	L	L	L	L	L	L	L	L

TABLE III

"SCHEME FOR THE PRESUMPTIVE IDENTIFICATION OF BALLPEN INK FORMULATIONS" (after Crown)

"The ink line is spotted with concentrated hydrochloric acid, using a micropipette. The color of the ink line itself and the color of the "bleed", if any, should be observed. When necessary, the hydrochloric acid spot should be absorbed on filter paper and neutralized with saturated sodium bicarbonate solution, and the resultant colors observed. A second spot should be made on the ink line with N,N-dimethylformamide (or other solvents which release the red dye) and then observed under ultraviolet light, either short or long wavelength. All hydrochloric acid spots on the document itself must be neutralized afterwards, to prevent damage to the document. Should aberrant reactions be encountered, conventional chromatography, followed by spot testing, should be employed.

1. Non-phthalocyanine Inks--absence of greenish or olive color on ink line; no fluorescence.
   a. Indulin and Victoria blue--purple ink line.
   b. Iron blue and methyl violet--positive iron test with KCNS.
   c. Victoria blue--absence of violet coloration after neutralization.
   d. Victoria blue and methyl violet--vivid violet coloration after neutralization.

TABLE III (Continued)

1. Phthalocyanine Inks--greenish or olive coloration of ink line.
   2. Presence of rhodamine B--bright pink fluorescence.
      e. Phthalocyanine and rhodamine B--no blue or purple colors after neutralization.
      f. Phthalocyanine, Victoria blue and rhodamine B--purple, blue, and red colors after neutralization.
   2. Absence of rhodamine B--no pink fluorescence.
      3. No bleed in HCl.
         g. Phthalocyanine and alkali blue.
      3. Bleed in HCl.
         h. Phthalocyanine, Victoria blue and alkali blue--no violet coloration after neutralization.
         i. Phthalocyanine and methyl violet--pale green ink line, pale yellow bleed.
         j. Phthalocyanine, methly violet and Victoria blue--brownish bleed, violet color, after neutralization.

TABLE III (Continued)

CLASSIFICATION OF BALLPEN INKS INTO DYE CONSTITUENT GROUPS

The chemical tests indicated that each of the sixty-nine blue ballpen inks could be classified into one of seven dye constituent groups. The specimen number of each ink is listed under its respective group category. Inks which exhibited luminescence are parenthesized.

I. METHYL VIOLET - VICTORIA BLUE

(1)
(2)
(3)
(4)
(11)
(17)
(18)
(22)
(23)
(27)
(29)
(30)
(37)
(41)
(42)
(50)
(51)
(53)
(54)
(55)
(62)
(64)
(65)
(66)
(75)
(77)
(78)
(79)
(81)
(82)
(86)
(87)
(88)
(89)
(90)
(91)

Total 36

II. PHTHALOCYANINE - METHYL VIOLET - VICTORIA BLUE

10
12
14
15
19
25
28
(32)
33
(38)
39
40
52
61
69
70
71
72
80
(85)
92

Total 21

III. PHTHALOCYANINE - VICTORIA BLUE - RHODAMINE B

16
36
(67)
74
83

Total 5

TABLE III (Continued

IV. *VICTORIA BLUE*	
21	
31	
35	
73	Total 4
V. *VICTORIA BLUE - INDULIN*	
(49)	Total 1
VI. *PHTHALOCYANINE - METHYL VIOLET*	
9	Total 1
VII. *PHTHALOCYANINE - RHODAMINE B*	
26	Total 1

APPENDICES

APPENDIX A

LIST OF BALLPENS EXAMINED

Trade names have been listed only in those instances (1) when the refill bore identifying markings, (2) when a pen was known to contain its original refill, (3) when a pen was a non-refill type. All other pens or refills are listed as "No name."

Pens or refills other than blue are so designated.

Specimen

1. Rocket
2. Wonder-Rite
3. No name
4. Herald Square
5. Herald Square (black)
6. Herald Square (red)
7. Herald Square (green)
8. Wonder-Rite (green)
9. Wonder-Rite (black)
10. No name
11. No name
12. No name
13. Ready Riter (black)
14. No name
15. Sheaffer's refill
16. No name
17. No name
18. No name
19. Scripto T-010
20. Utility Pen (red)
21. Paper Mate C2 refill
22. Dixon Non-Skid Ball 1RB
23. No name
24. Carter's Laundry (black)
25. Scripto 0517AJ refill
26. No name
27. No name
28. Ritepoint refill

Specimen

29. Ritepoint refill
30. Fine Riter 21 refill
31. Paper Mate 19 refill
32. Sheaffer's 303 refill
33. Scripto refill
34. No name
35. Paper Mate refill
36. No name
37. No name
38. Sheaffer's 303 refill
39. Ritepoint 201 refill
40. Ritepoint 353 refill
41. Ritepoint 904 refill
42. Ritepoint 800 refill
43. Ritepoint 120 refill(black)
44. Ritepoint 324 refill(red)
45. Ritepoint 285 refill(green)
46. Ritepoint refill(gold)
47. Topriter refill(green)
48. Topriter refill(red)
49. Topriter refill(black)
50. Topriter refill(blue)
51. Wearever refill
52. Arnold PR92M refill
53. Alco refill

APPENDIX A (Continued)

Specimen

54. Alco refill
55. Alco refill
56. Alco refill (black)
57. Alco refill (red)
58. Ready Riter 440 (red)
59. No name (green)
60. Ready Riter 540 (gold)
61. Ready Riter 140 (blue)
62. No name
63. No name (red)
64. No name
65. No name
66. No name
67. No name
68. Eversharp KEC54 refill (black)
69. Eversharp KEC54 refill (blue)
70. Steno-Pen 467F
71. A.T. Cross X-Med.4 refill
72. A.T. Cross R-Med.8 refill
73. Paper Mate E1 refill
74. No name
75. No name
76. Utility 400M (green)
77. No name
78. No name
79. No name
80. Scripto Tele-Gauge refill
81. No name
82. No name
83. No name
84. Ready Riter 332 (green)
85. Gold Bond Kushion Karbide refill
86. No name
87. No name
88. No name
89. No name
90. No name
91. No name
92. No name

APPENDIX B

LIST OF LIQUID INKS EXAMINED

Specimen

1. Skrip Permanent Blue-Black #22
2. Skrip Permanent Jet Black #32
3. Skrip Washable Blue #42
4. Skrip Permanent Royal Blue #52
5. Skrip Washable Black #62
6. Skrip Washable Purple #82
7. Skrip Persian Rose #104
8. Sanford Penit - Violet
9. Sanford Penit - Green
10. Sanford Penit - Cardinal Red
11. Sanford Penit - Washable Royal Blue
12. Sanford Penit - Blue Black
13. Sanford Penit - Dubonnet
14. Sanford Penit - Brown
15. Carter's Permanent American Blue
16. Carter's Violet
17. Carter's Brown
18. Waterman's Permanent Black
19. Parker Super Quink - Permanent Blue
20. Parker Super Quink - Permanent Blue Black

APPENDIX C

LIST OF PAPER STOCKS

I. SAFETY PAPERS

Bergstrom Private Design Safety Paper.

Yellow
Green
Blue

Graham Exchange Safety Paper.

Yellow (Primrose)
Green
Blue
Pink (Rose)
Gray

II. WHITE BOND PAPERS

Graham #20 Bonds.

Paper 1. Old Deerfield
Paper 2. Ezerase
Paper 3. Miller Falls Opaque Parchment
Paper 4. Oriole Linen
Paper 5. Crane's Crest
Paper 6. Monetary
Paper 7. World
Paper 8. Requisition

APPENDIX D

PHOTOGRAPHS

Unless otherwise specified, the photographs that follow were exposed on Kodak High Speed Infrared Film for 15 seconds at f/1.9; development time was 10 minutes in Kodak D76 at 68 degrees F. All of the enlargements were made on the same contrast grade of paper.

Four blue ballpen inks were selected to demonstrate strong luminescence, medium luminescence, weak luminescence, and non-luminescence. Their identify is as follows:

1. John Williams -- strong luminescence
   Specimen #1, "Rocket Pen"
2. Ruth Roberts -- medium luminescence
   Specimen #51, "Wearever"
   refill
3. Dell Andrews -- weak luminescence
   Specimen #32, "Sheaffer's
   Dokumental 303 Skrip"
   refill
4. Donald Collins - non-luminescence
   Specimen #61, "ReadyRiter
   104 Pen"

The ink specimens were used to write the four names on a white file card and on each of the eight colored safety papers. The paper stock can be identified by the typewritten name and color which appears in each photograph.

APPENDIX D (Continued)

In comparing the photographs that appear in Appendix, the reader's attention is directed to:

a) Figures 7 and 8. Note that "Dell Andrews" exhibits luminescence in Fig. 7; it does not exhibit luminescence in Fig. 8 which was made with an 89B filter. This quenching of luminescence might be attributed to the reflected visible light that the 89B filter transmits. (see Fig. 4 for the transmission curve of the 89B filter).

b) Figures 9 through 13 which comprise the Graham Safety paper series. Note that "John Williams" and "Ruth Roberts" exhibit luminescence regardless of the color of the paper stock; the luminescence does vary in intensity; however, "Dell Andrews" exhibits luminescence only in Fig. 9 which is the blue paper stock.

c) Figures 13 and 14. Note that these two safety papers of similar color (yellow) but of different manufacturer produce a divergent luminescence effect in the "Dell Andrews" signature. "Dell Andrews" does not luminesce in Fig. 13 (Graham Safety); "Dell Andrews" does luminesce in Fig. 14 (Bergstrom Safety). The luminescence characteristics of the four inks on the Bergstrom yellow safety paper are similar to their luminescence characteristics on white paper stock (Fig. 7).

APPENDIX D (Continued)

d) Figures 15 and 16. Note that "Dell Andrews" in Fig. 15 has been rendered almost invisible. In Fig. 16 the "Dell Andrews" signature is distinctly readable.

e) Figure 17. Note that the absence of the copper sulfate filter at the light source results in the elimination of luminescence. Figure 17 illustrates the results of the standard infrared photographic technique.

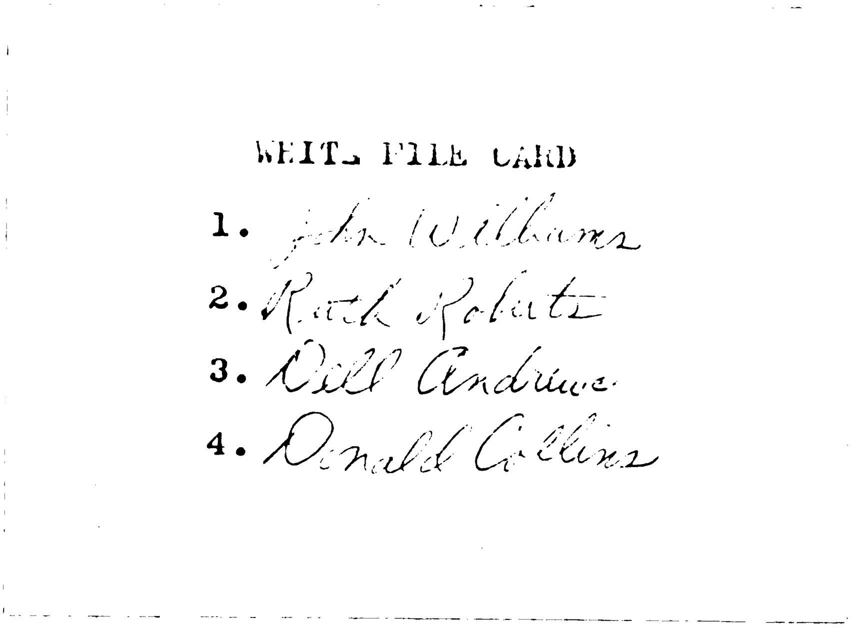
f) Figures 18 and 14. Here again is illustrated the elimination of luminescence in an ink when the 89B filter is used. Note that in Fig. 14, which was photographed with an 87C filter, the "Dell Andrews" signature exhibits luminescence. In fig. 18, which was photographed with an 89B filter, the "Dell Andrews" signature does not luminesce.

The series of photographs illustrates:

a) That colored paper stocks can affect the luminescent capability of an ink;

b) That the luminescent capability of an ink can be controled by the type of infrared transmitting filter used for the photography;

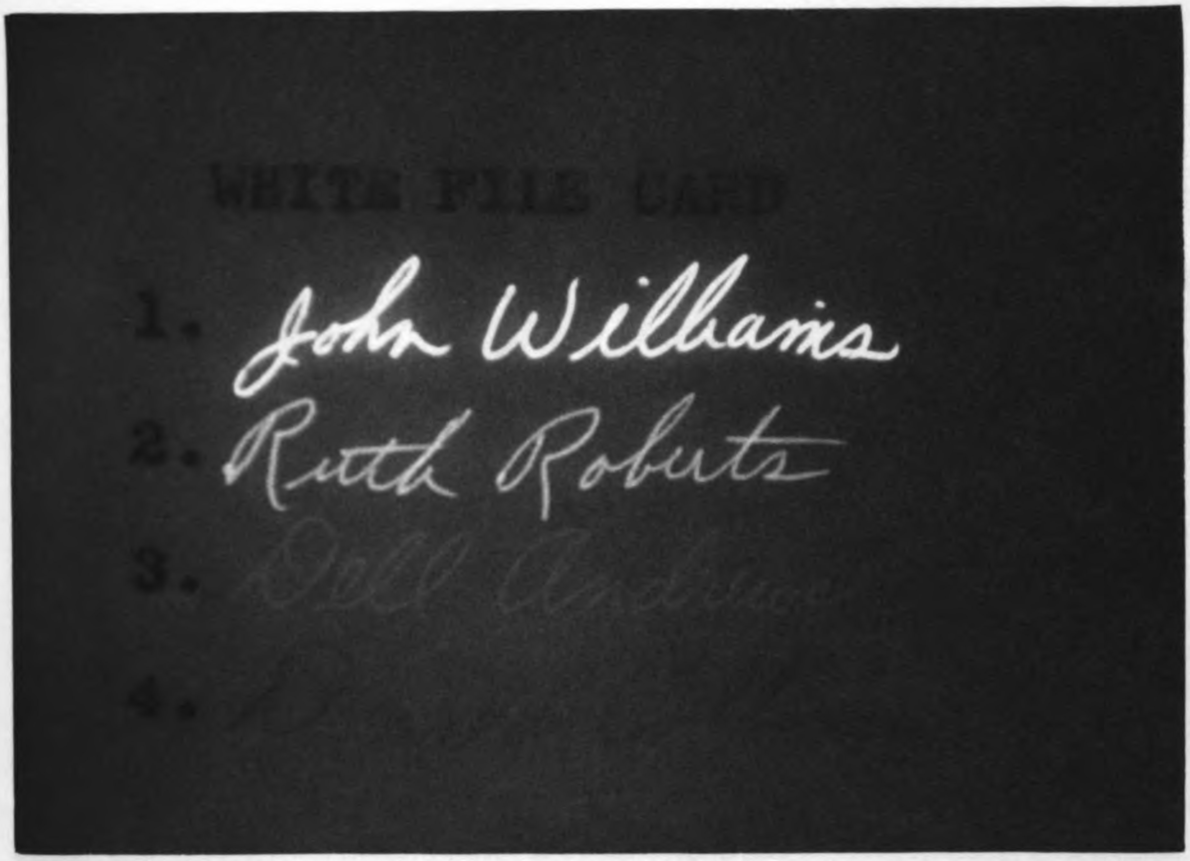
c) That blue ballpen inks may be differentiated by means of photographing their infrared luminescence characteristics.



WHITE FILE CARD

1. John Williams
2. Ruth Roberts
3. Bill Andrews
4. Donald Collins

FIG. 6 THE FOUR INKS AS PHOTOGRAPHED WITHOUT FILTERS ON A PANCHROMATIC, TYPE B SENSITIVITY FILM (POLARIOD 4X5 TYPE 55 P/N, ASA 50)



WHITE FILE CARD

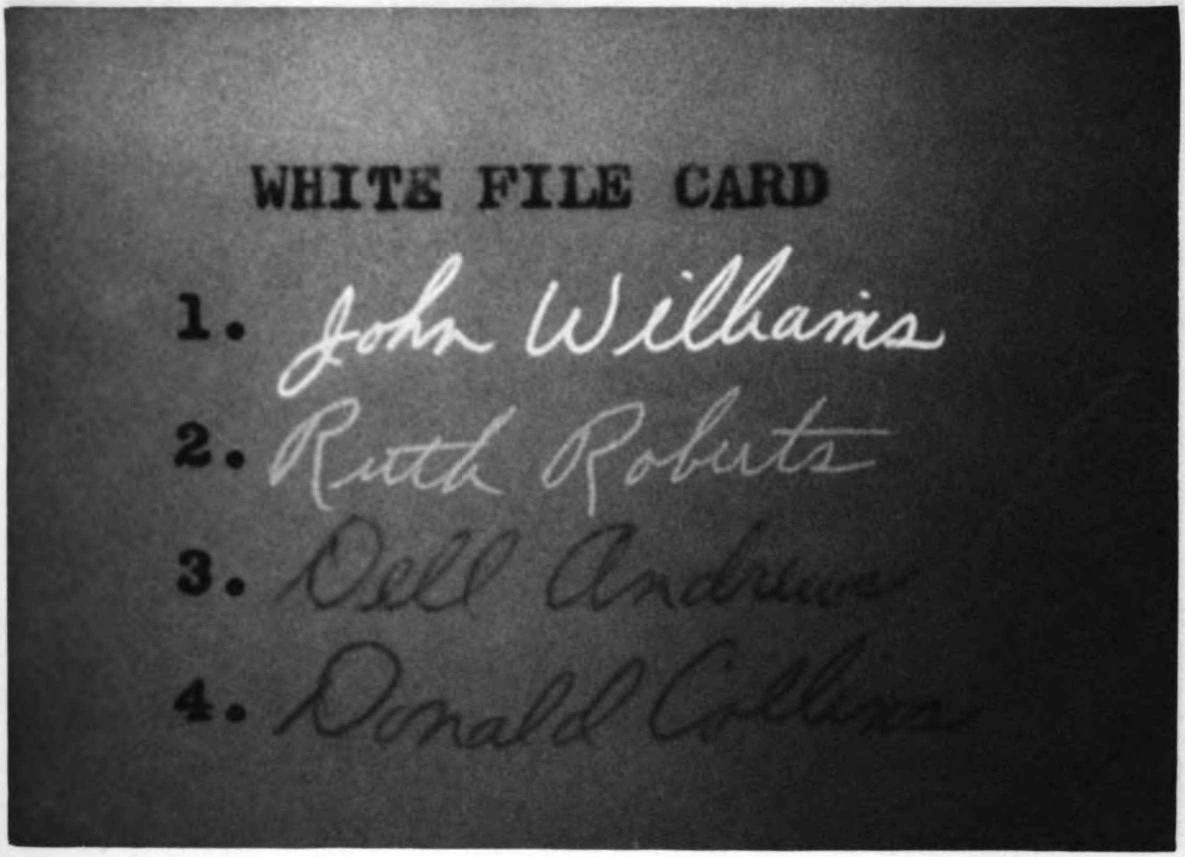
1. John Williams

2. Ruth Roberts

3. Dell Andrews

4.

FIG. 7 87C FILTER
COPPER SULFATE FILTER



WHITE FILE CARD

1. John Williams
2. Ruth Roberts
3. Dell Andrews
4. Donald Collins

FIG. 8 89B FILTER
COPPER SULFATE FILTER
2 SECONDS @ F/1.9

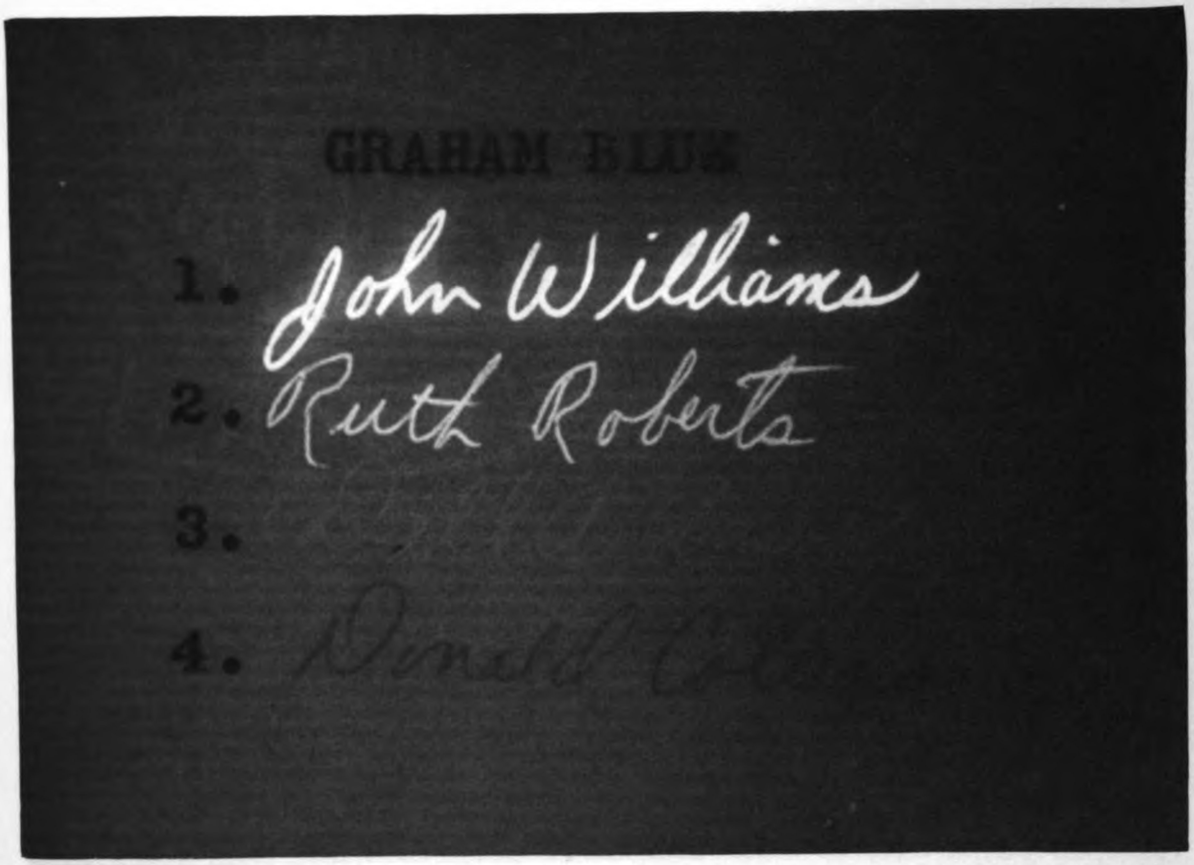


FIG 9 87C FILTER
COPPER SULFATE FILTER

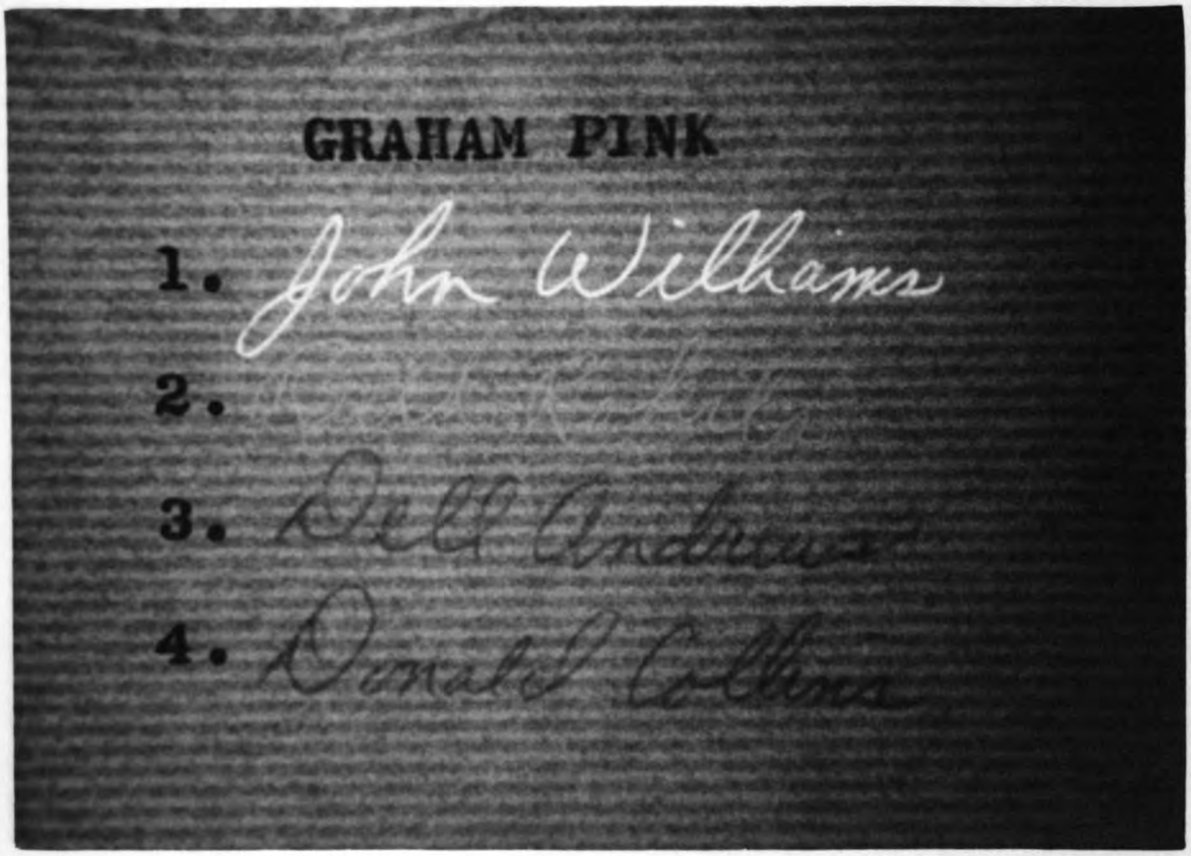
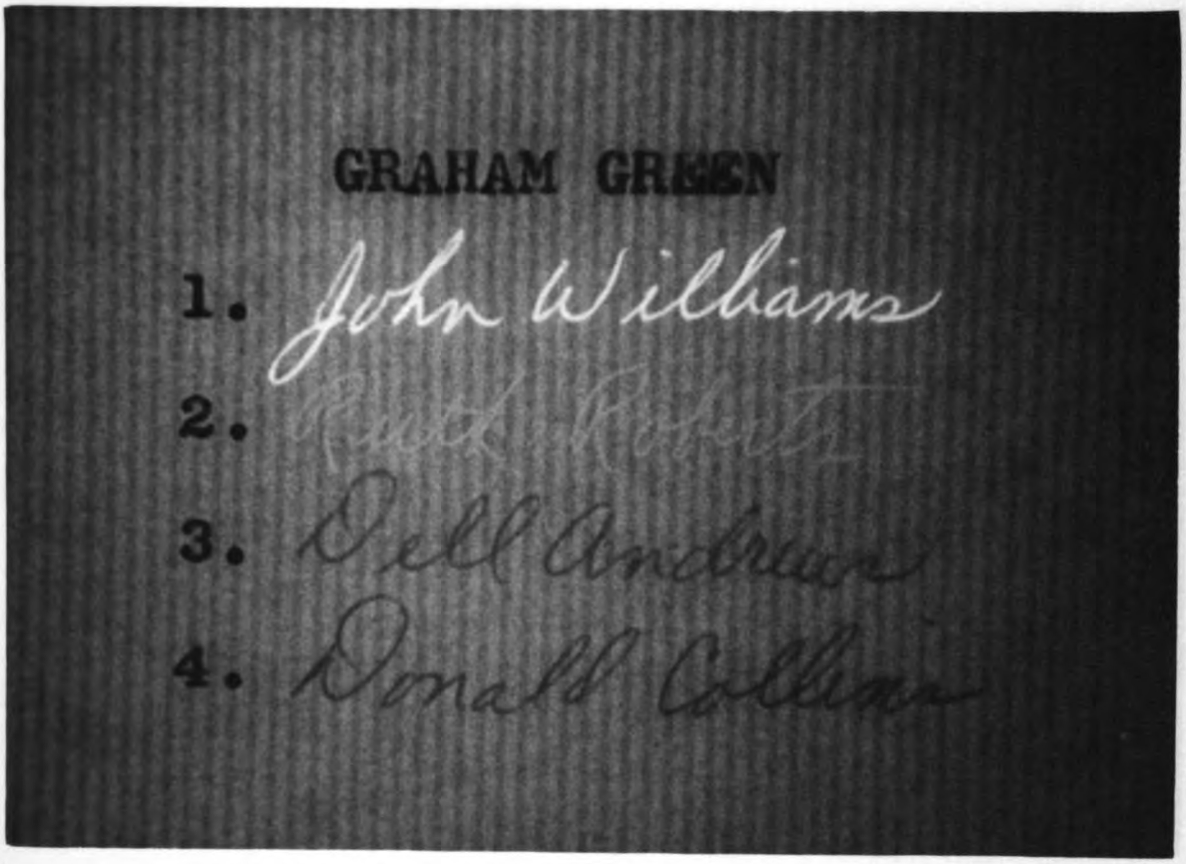


FIG. 10 87C FILTER
COPPER SULFATE FILTER



GRAHAM GREEN

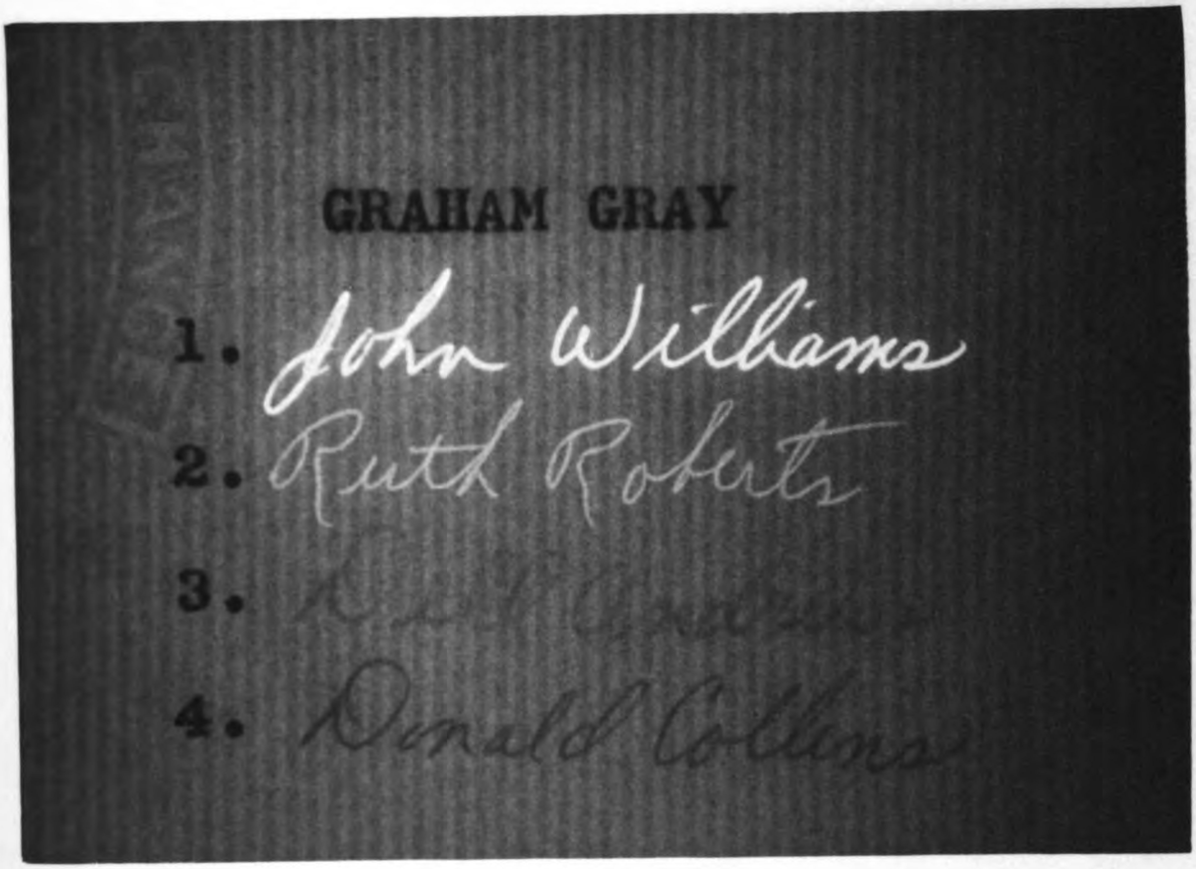
1. John Williams

2. Ruth Roberts

3. Dell Andrews

4. Donald Collins

FIG. 11 87C FILTER
COPPER SULFATE FILTER



GRAHAM GRAY

1. John Williams
2. Ruth Roberts
3. [illegible]
4. Donald Collins

FIG. 12 87C FILTER
COPPER SULFATE FILTER

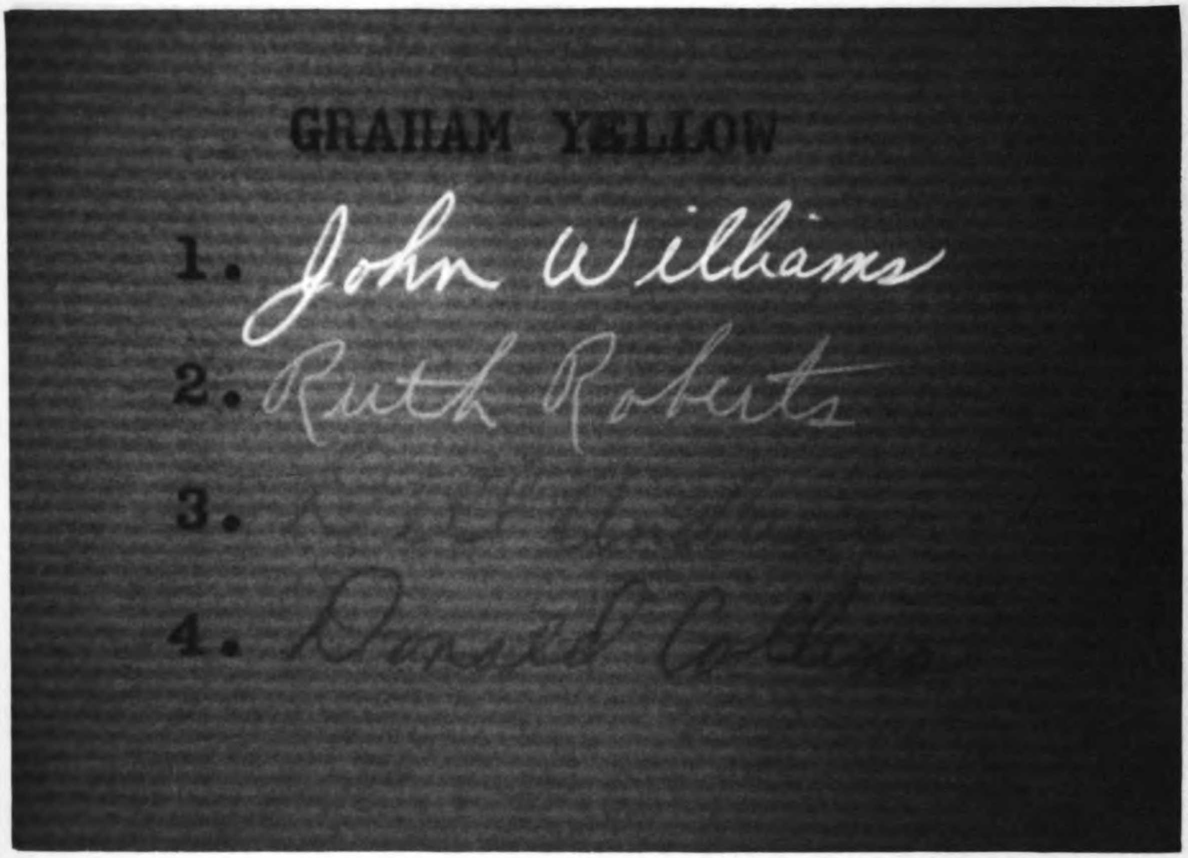
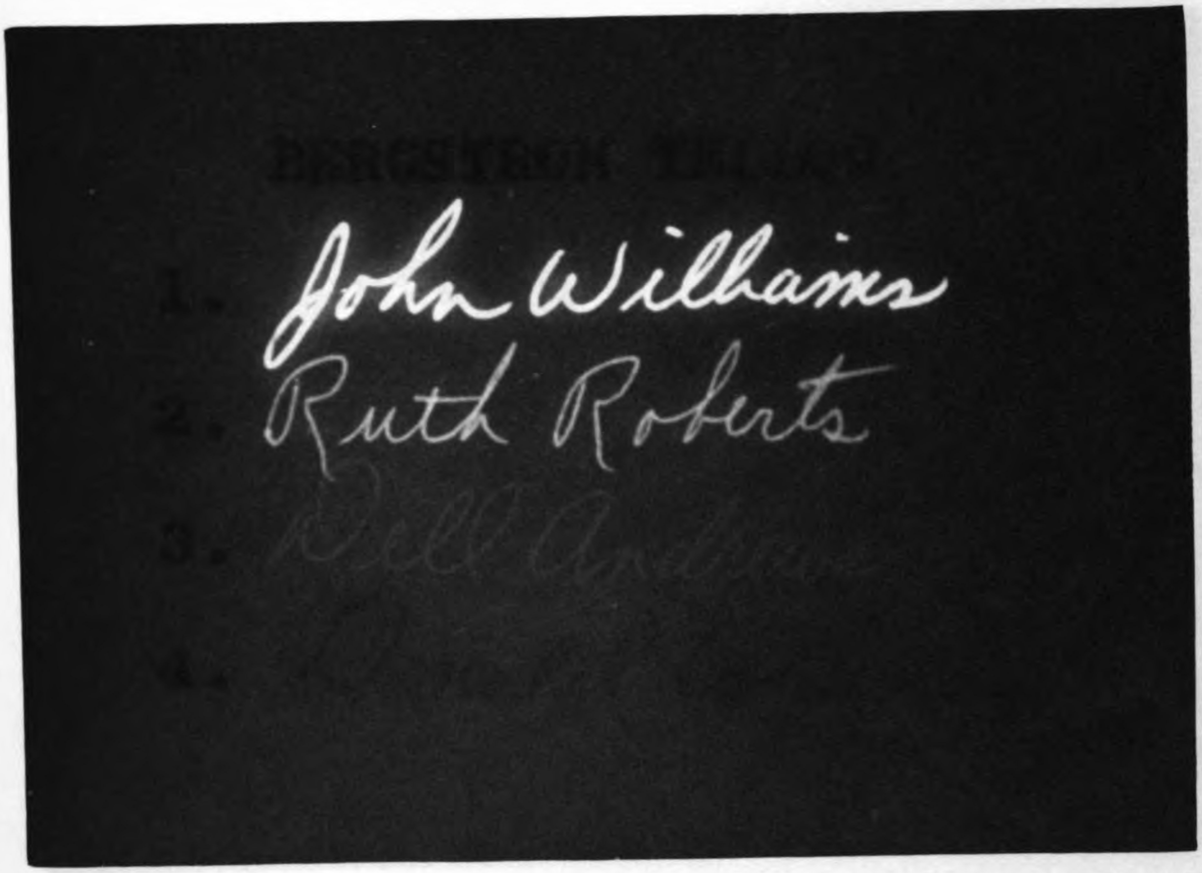


FIG. 13 87C FILTER
COPPER SULFATE FILTER

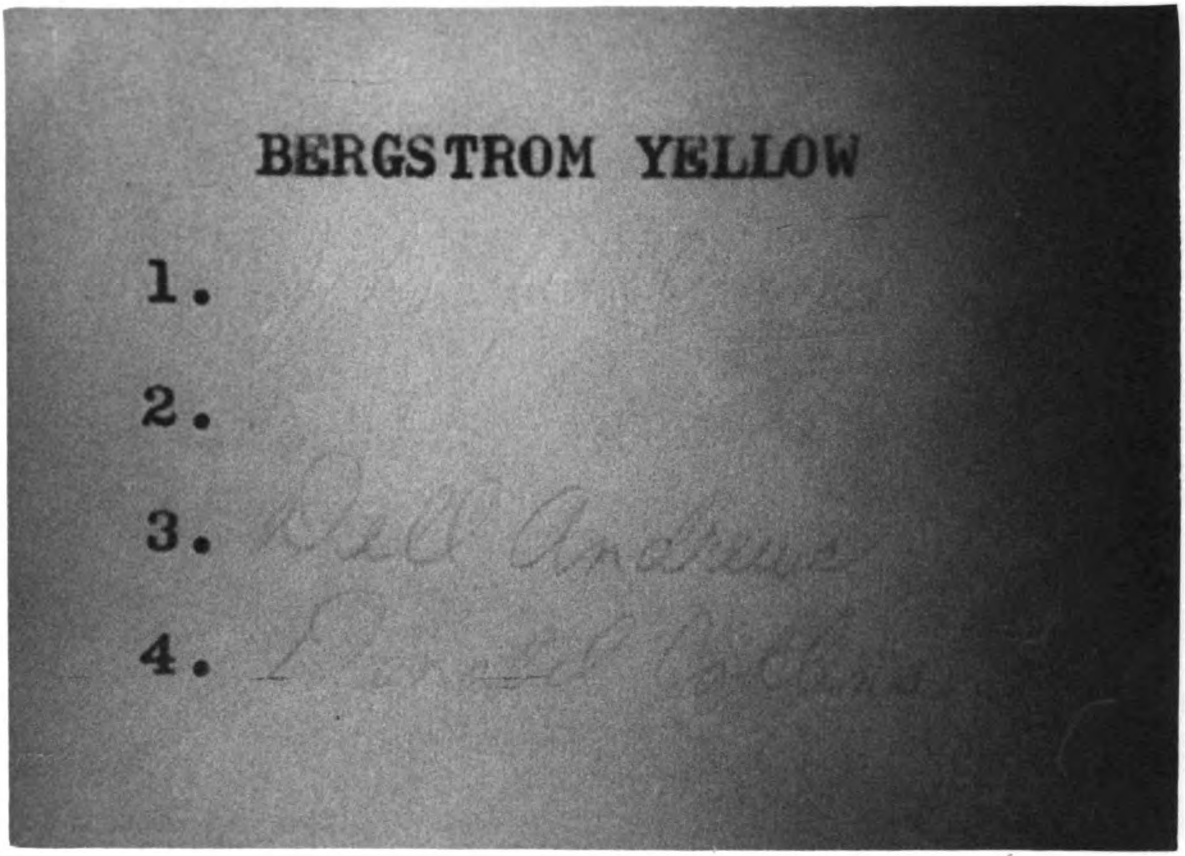


1. John Williams
2. Ruth Roberts
3. [illegible]
4.

FIG. 14 87C FILTER
COPPER SULFATE FILTER

FIG. 15   87C FILTER
          COPPER SULFATE FILTER

FIG. 16 87C FILTER
COPPER SULFATE FILTER



BERGSTROM YELLOW

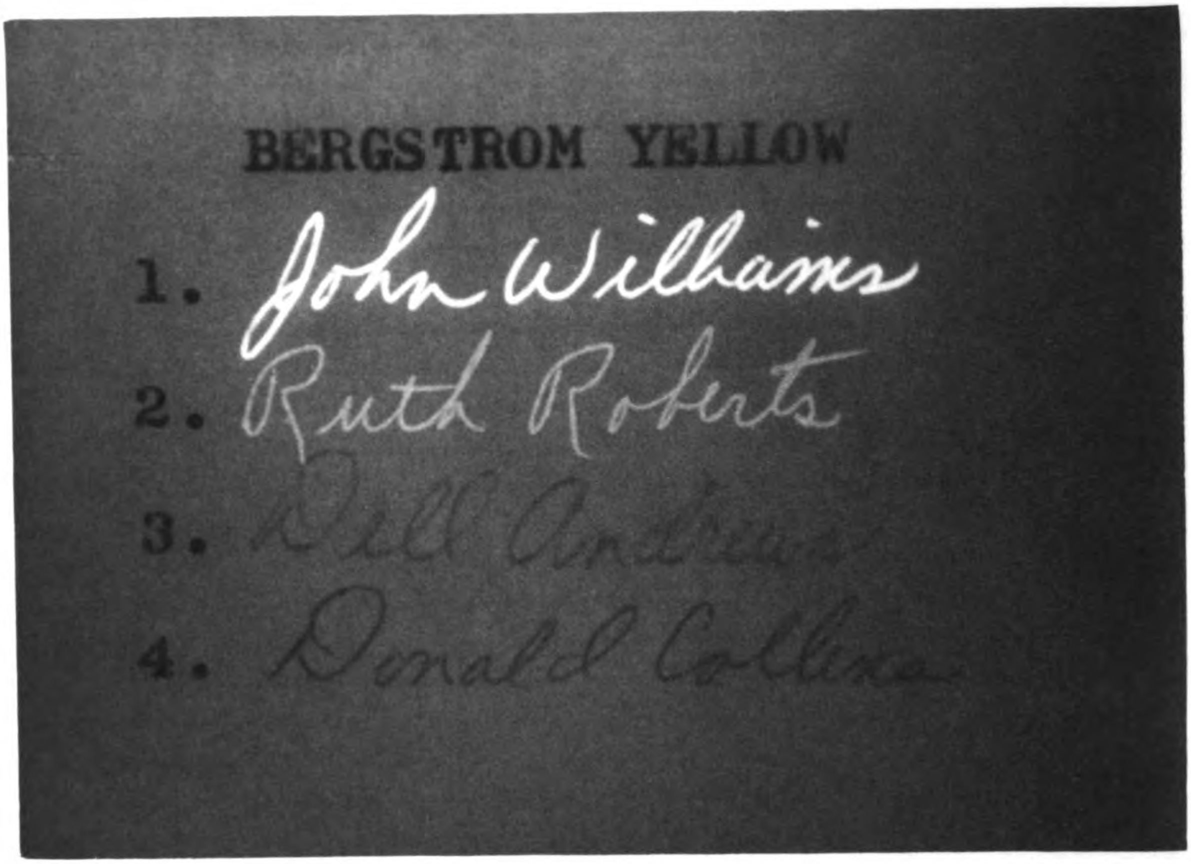
1.

2.

3. Dell Andrews

4. [illegible]

FIG. 17 87C FILTER
NO COPPER SULFATE FILTER
1/50 SECOND @ F/1.9



BERGSTROM YELLOW

1. John Williams
2. Ruth Roberts
3. Dell Andrews
4. Donald Collins

FIG. 18 89B FILTER
COPPER SULFATE FILTER
2 SECONDS @ F/1.9

WHITE FILE CARD

1. John Williams
2. Ruth Roberts
3. Dell Andrews
4. Donald Collins

BERGSTROM BLUE

1. John Williams
2. Ruth Roberts
3. Dell Andrews
4. Donald Collins

BERGSTROM YELLOW

1. John Williams
2. Ruth Roberts
3. Dell Andrews
4. Donald Collins

BERGSTROM GREEN

1. John Williams
2. Ruth Roberts
3. Dell Andrews
4. Donald Collins

GRAHAM PINK

1. John Williams
2. Ruth Roberts
3. Dell Andrews
4. Donald Collins

GRAHAM GRAY

1. John Williams
2. Ruth Roberts
3. Dell Andrews
4. Donald Collins

GRAHAM YELLOW

1. John Williams
2. Ruth Roberts
3. Dell Andrews
4. Donald Collins

GRAHAM GREEN

1. John Williams
2. Ruth Roberts
3. Dell Andrews
4. Donald Collins

GRAHAM BLUE

1. John Williams
2. Ruth Roberts
3. Dell Andrews
4. Donald Collins

ORIOLE LINEN BOND

CRANES CREST

MILLER FALLS OPAQUE PARCHMENT

WORLD BOND

EZERASE BOND

REQUISITION BOND

MONETARY BOND

OLD DEERFIELD BOND