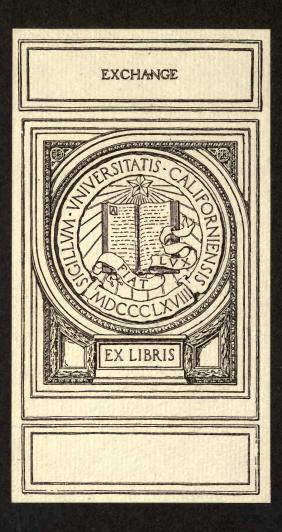
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HYPERSENSITIZING COMMERCIAL PANCHROMATIC PLATES

A DISSERTATION

SUBMITTED TO THE BOARD OF UNIVERSITY STUDIES OF THE JOHNS HOPKINS UNIVERSITY IN CONFORMITY WITH THE RE-QUIREMENTS FOR THE DEGREE OF DOCTOR OF PHILOSOPHY

SAMUEL M. BURKA

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HYPERSENSITIZING COMMERCIAL PANCHROMATIC PLATES.

BY

SAMUEL M. BURKA, Ph.D.

INTRODUCTION.

ORDINARY dry plates, which owe their sensitivity to the silver halides alone, are sensitive only to the violet and blue regions of the spectrum. If, however, a suitable dyestuff be added, the emulsion becomes sensitive to other regions, the particular region depending on the dye used. Thus erythrosin sensitizes to the green and greenish yellow, pinaverdol to the green and yellow, pinacyanol to the orange and red and dicyanin to the extreme red and infra-red.

The dyestuff is applied to the halides in one of two ways. In the commercial orthochromatic (sensitive to green and yellow) and panchromatic (sensitive to all colors) plates, the dyes are incorporated in the emulsion and the mixture flowed over the glass plate. The second method consists of bathing an ordinary bluesensitive plate in a dilute solution of the dye and allowing the plate to dry.¹

The bathed plates are in general faster than the commercial plates ² and, of course, have the advantage that the plates can be sensitized for any desired region. Certain dyes, moreover (dicyanin, for example), cannot be successfully incorporated in the emulsion. The bathed plates, however, if not used soon after bathing, or if kept at a high temperature, are, in the case of most dyes, more susceptible to chemical fog. Dicyanin and pinacyanol bathed plates when prepared so as to produce the greatest sensitivity are useless after three or four days.

It has long been known that, though at the expense of keeping qualities and freedom from chemical fog, the speed of bathed plates can be increased by the addition of ammonia to the dye-

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¹Eder, "Handbuch der Photographie," 1902, vol. iii, p. 169 *et. seq.* For the Bureau of Standards methods see Bulletin Bureau of Standards, 14, 371, 1917.

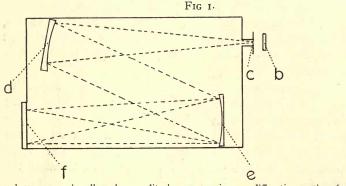
² Eder, "Handbuch der Photographie," 1902, vol. iii, p. 169. Reference to Schumann, Oct., 1885; Phot. Wochenbl., 1885, p. 395.

bath. Since this increase is quite marked in the case of some dyes, it was hoped that in this way commercial plates could have their speed increased, and, at the suggestion of Dr. P. W. Merrill, a study was made at the Bureau of Standards of the influence of ammonia on commercial orthochromatic and panchromatic plates.

SENSITOMETRY.

Three methods of sensitometry were used: first, the threecolor screen method; second, the spectrograph method; and third, the Hurter and Driffield method.

The first method, based on Abney's method,³ which, however,



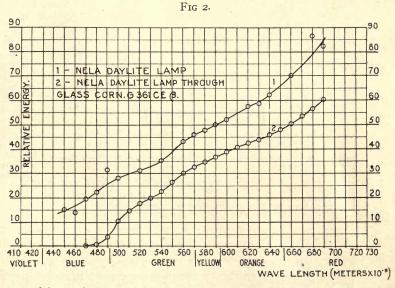
a, lamp source: b, yellow glass; c, slit; d, concave mirror; e, diffraction grating; f, plate to be tested.

was used only as a qualitative method at first, consists of exposing the plates to a constant light source (usually a nitrogen-filled tungsten lamp corrected by color screens so as to have an energy distribution similar to daylight) behind a so-called trichromatic sensitometer plate. This plate is a neutral screen having four strips of squares of increasing density, each square having twice the density of the preceding. One of these strips is left white, while on the others are placed a red, a green, and a blue filter intended to be of the same luminosity and mutually exclusive. The plates to be tested are thus exposed to varying intensities of white, red, green and blue light.

The second method avoids the difficulty of obtaining filters transmitting pure spectral colors, by exposing the plate in a spectrograph.

³ Phot. Jour., June, 1895; Eder, "Phot. Korr.," 1903, p. 426. Chapman Jones, Photo. Jour., 1901, 256.

The apparatus (Fig. 1) consists of a concave grating of 50 cm. radius with 20,000 lines per inch, mounted in parallel light with a 100-watt "daylight" tungsten lamp on 110-volt A. C. circuit as a source. For use with color sensitive plates a piece of yellow glass (7 mm. thick, Corning G 351 CE) barely transmitting the hydrogen blue line with wave-length 4861A was placed in front of the slit to cut out the second order blue. The region photographed was from about 4800A to the limit of sensitivity of the plate. A pair of cross-hairs just in front of the plate at

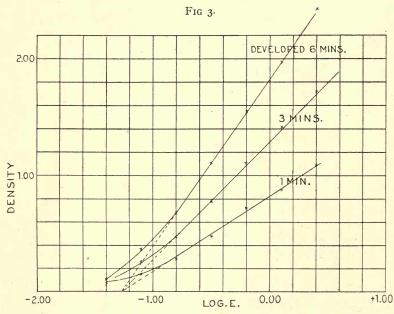


the position of the red line of hydrogen (wave-length 6563A) and of the blue line (4861A) served as reference marks. Fig. 2 gives the energy distribution of the lamp and screen combination. The energy was determined in the spectrophotometric laboratory, visually on a König-Martens Spectrophotometer, by a substitution method of comparison with a radiometrically calibrated Mazda lamp.

The photographic densities were measured on a Hartmann Microphotometer. This method gives immediately the sensitivity of the plate to all parts of the spectrum; but since, during the course of the work, minor changes (change of slit, resilvering of mirror, etc.) were made, the exposure times on different sets of plates were not always equivalent.

Furthermore, since the plates were of different kinds and showed markedly different rates of development, they were, in this part of the work, developed by tray until they showed the amount of fog allowable in plates to be used in general photography. The density of the silver deposit obtained depends on the time of development, so that in the curves obtained the shapes (maxima and minima) are the really important parts and the density is only approximately a measure of the speed.

The method generally adopted for the measurement of the



Ilford special rapid panchromatic (5630N). Untreated; no filter; speed, 4.45.

speed is that of Hurter and Driffield.⁴ Specimens of the plate to be tested are exposed for the same time, behind a sectored wheel, to a standard light source. The transmissions, T, of the images obtained are measured and their densities, defined as $-\log T$, are plotted as ordinates and the logarithm of the time of exposure, E, as abscissas.

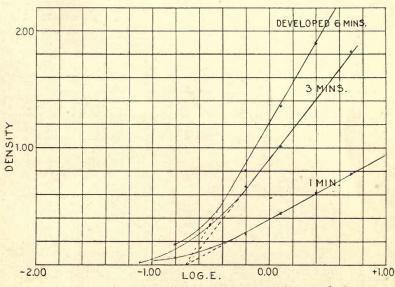
The curve so obtained is known as the characteristic curve of the plate. It consists of three parts: the first part is concave

⁴ Hurter and Driffield, Jour. Soc. Chem. Industry, May, 1890, p. 455.

HYPERSENSITIZING PANCHROMATIC PLATES.

upward, the second is a straight line and the third is concave downward. Prolong the straight line part of the curve until it intersects the axis of abscissas. The numerical value of the exposure at the point of intersection is known as the inertia, i, of the plate and the speed is defined as 1/i. Hurter and Driffield showed that this inertia of a plate is independent of the kind of developer used (except for strong pyro and for the presence of free bromide) and of the time of development (see Figs. 3 and 4). Free bromides, either in the film or in the developer, shift the point of

FIG 4.



Ilford special rapid panchromatic (5630N). Treated; no filter; speed, 18.2.

intersection of the straight line portions of the curves to a point below the axis. The plates for which Fig. 5 gives the characteristic curves contain free bromide in the emulsion.

The Hurter and Driffield speeds for several ammoniated and unammoniated plates were obtained, using an apparatus designed and built by Mr. R. Davis of the Bureau of Standards. The light source (a calibrated tungsten lamp) is corrected for daylight, and means are provided for accurately controlling the current through the lamp, the speed of the sectored disc and the total time of exposure.

PROCEDURE.

A series of Cramer Spectrum Process and Ilford Special Rapid Panchromatic plates were bathed for four mintes at 16° to 18° C. in water containing increasing amounts of ammonia, and were then exposed in the spectrograph. Both Metol-Hydroquinone and Pyro developers were used. It was soon found that the plates showed fog in development unless they were kept cool while drying and were dried rapidly. The plates showed a progressive increase in speed with increase in ammonia concentration

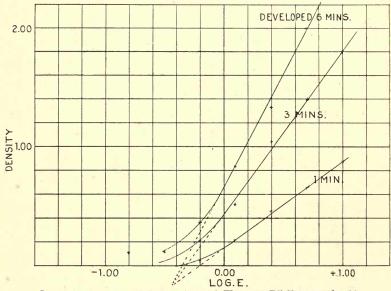


FIG 5.

Cramer spectrum process (1439). Treated; Wratten "F" filter; speed, 1.88.

until, with a bath containing 4 c.c. of ammonia water (containing 20 per cent. NH_3) in 100 c.c. distilled water, the plates began to fog so badly in development as to be useless.

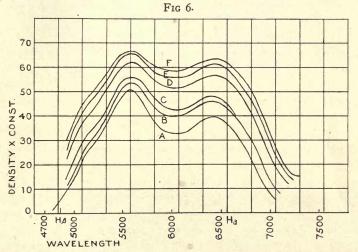
The ammonia was then added to water-alcohol mixtures of varying concentrations. The plates bathed in the alcoholic ammonia did not have their sensitivity increased as much as in the corresponding water baths, but were very much cleaner working. Seventy-five parts of water to twenty-five of ethyl-alcohol gave the best results, and this proportion was adhered to thereafter. Sometimes the plates showed a slight network of fog (mottling)

HYPERSENSITIZING PANCHROMATIC PLATES.

similar to the drying marks shown when plates are dipped (not soaked) in alcohol to hasten drying. Washing in 95 per cent. alcohol for 20 or 30 seconds after the ammonia bath prevented this, but this final wash is not usually necessary, especially if the plates are not forced in development.

Fig. 6 gives the spectral sensitivity curves of a series of Cramer Spectrum Process plates in baths containing $1/_{40}$, $1/_{10}$, $1/_{2}$, 1 and 3 c.c. of 20 per cent. NH₃ ammonia water to an alcohol-water mixture of 25 parts of alcohol and 75 parts of water.

Three to 31/2 c.c. of the strong ammonia to 75 c.c. of water and



Cramer spectrum process (1439). A, untreated; B, $^{1}/_{40}$ c.c. ammonia; C, $^{1}/_{10}$ c.c. ammonia D, $^{1}/_{2}$ c.c. ammonia; E, I c.c. ammonia; F, 3 c.c. ammonia; 75 c.c. water; 25 c.c. alcohol. Same exposure and development.

25 c.c. alcohol was adopted as the combination to be used, and except for a few trials on various plates, the plates were all treated with this same mixture.

The time of bathing, 2, 4, or 6 minutes, had no appreciable effect, provided that the film was bathed long enough to get thoroughly soaked. Four minutes was the time used for bathing in all subsequent experiments.

For a study of the keeping qualities of the plates, a number of Cramer Spectrum Process and Ilford Panchromatic plates were treated and samples of the treated plates exposed and developed after being stored for various lengths of time. The Cramer plates showed deterioration after a week but were still usable. The Ilford plates were useless after 3 or 4 days. The plates bathed without the use of alcohol deteriorated more rapidly.

The bath of 3 to $3\frac{1}{2}$ c.c. of ammonia water (20 per cent. NH₃), 25 c.c. of alcohol and 75 c.c. of water is recommended. Three and one-half c.c. of ammonia water to 100 c.c. of water is used where the maximum increase of sensitivity is desired, but the plates are much more susceptible to fog in development and must be used within a few hours of drying. Development should be carried out, using the Wratten Safelight No. 3 and over-development carefully guarded against.

ORDINARY PLATES.

As is well known, the speed of the silver halide emulsion can be increased by treatment before it is flowed on the plate. This treatment, known as "ripening," is usually either to keep the emulsion at a high temperature for some time or to add ammonia. This ripening by ammonia is effective even after the plates are ready for use.⁵

On ripened plates such as the Seed 30 and Central Special, and on the Seed 23, there is no appreciable increase in speed when they are bathed in ammonia. In some cases there is rather a slight decrease in speed. On one plate known to have been ripened by the ammonia process before coating (Central Dry Plate Co.) which, however, was not fresh, this increase in speed on subsequent bathing in ammonia was quite apparent.

Microscopic examination of the treated and untreated plates showed no difference in the size of the grain.

ORTHOCHROMATIC PLATES.

A number of plates sensitive to the green and yellow were tried. Most of them showed no appreciable change in sensitivity. Some had the sensitivity decreased, *e.g.*, Cramer Commercial Isonon. The sensitivity of the Seed Aero Ortho was increased slightly.

Fig. 7 is a print from treated and untreated Cramer Commercial Isonon negatives obtained through the tricolor sensitometer plate. Fig. 8 is a similar set on the Seed Aero Ortho. It will be noted that the change in sensitivity can be observed in all the strips.

⁵ Eder, "Handbuch," iii, p. 63.





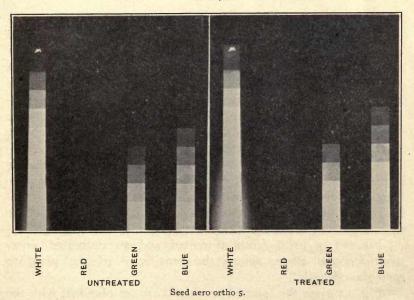


FIG 7.

The Cramer "Trichromatic" showed an increase in sensitivity comparable to that observed in the Spectrum Process.

Treatment of Eastman and of Ansco N. C. films, which are orthochromatic, showed no change larger than the differences attributable to experimental errors.

PANCHROMATIC PLATES.

Every panchromatic plate tried showed remarkable increase in speed. The very fast plates, several of which were developed only recently in response to the need for fast color sensitive plates for aerial photography, showed a somewhat smaller increase than the slower and process plates.

The plates examined were :

Cramer Spectrum Process; Ilford Panchromatic (Special Rapid); Five of the Eastman Special Experimental Panchromatic plates; Wratten and Wainwright Special Red Sensitive; Wratten and Wainwright R. F. C. Panchromatic; Wratten and Wainwright Panchromatic; Wratten M; Cramer Spectrum No. 11; Cramer G D I, G D II, G D III; Central Experimental Panchromatic.

Wherever possible several different emulsions were used.

Figs. 9, 10, and 11 are prints from pairs of Wratten and Wainwright Special Red Sensitive, one of the Eastman Experimental emulsion and Ilford Panchromatic plates. The three sets of prints were exposed and developed exactly alike on Artura Iris paper. Even though the paper cannot reproduce all the gradations of the original negative, they do give fairly well the relative speeds of these three brands of plates and show quite well the great increase in speed effected by the ammonia treatment.

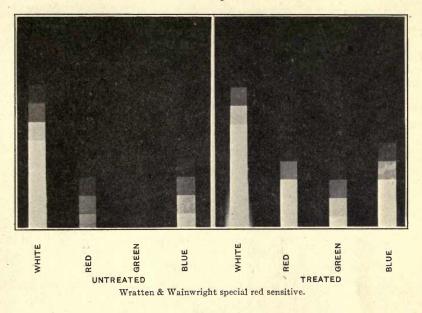
This sensitometer plate method gives the relative speeds of plates towards filters, but since the filters do not transmit pure spectral colors (the blue transmits a band in the red and the transmission of the three filters overlap) more can be learned of the color sensitivity from the spectrograph curves.

Fig. 12, a print from a treated and an untreated Spectrum Process Plate, shows the sensitivity of the plate to each wave-length.

Fig. 13 gives the spectral sensitivity curves obtained with the



Fig 9.





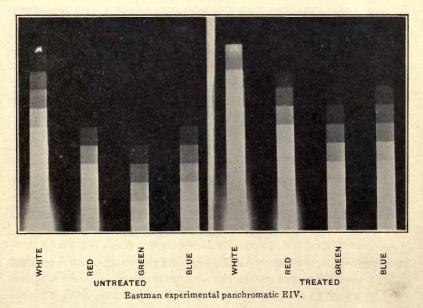
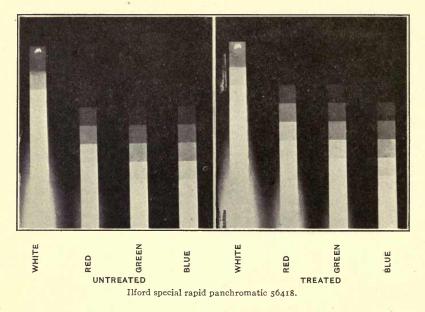
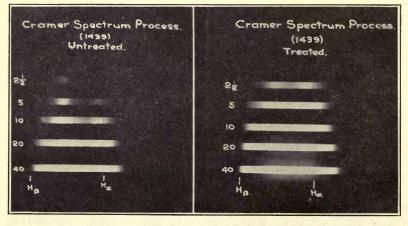


FIG 11.







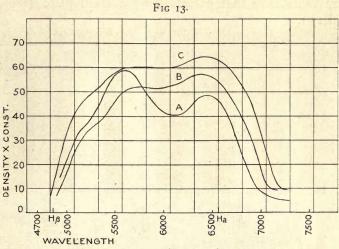
Cramer spectrum process (panchromatic).

Spectrum Process plate: 5- and 10-second exposures on the treated and 10 seconds' exposure on the untreated plate.

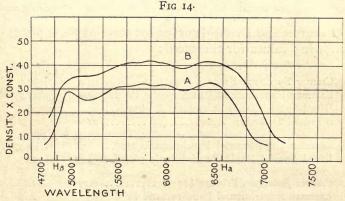
Fig. 14 gives the curves for one of the Eastman Kodak Com-

pany's special experimental Panchromatic Plates. Both curves are for 10-second exposures.

In Fig. 15, curve A is for the 10-second exposure on the un-



Cramer spectrum process (1439). A, untreated, 10-second exposure; B, treated, 5-second exposure; C, treated, 10-second exposure.

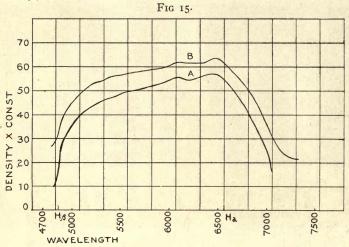


Eastman special experimental panchromatic (EIV 2165D). A. untreated, 10 seconds exposure; B, treated, 10 seconds exposure.

treated Ilford Panchromatic plate. Curve B is for the 5-second exposure on the treated plate.

One of the most striking changes produced by the animonia treatment was the action on old panchromatic plates. A Wratten and Wainwright plate marked "use before September 15, 1915," was exposed on October 21, 1918, in the spectrograph. As was

to be expected, the plate was badly fogged on the edges and the sensitivity of the emulsion was much below that of a fresh plate. Treatment with ammonia brought the sensitivity up to an even greater value than that possessed by a fresh plate (see Figs. 16 and 17).



Ilford special rapid panchromatic (56418). A, untreated, 10 seconds exposure; B, treated, 5 seconds exposure.

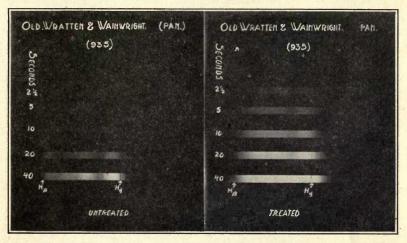
The Hurter and Driffield curves for some of these plates gave the following values for the speed:

υ	ntreated Whi	Treated te Light
Cramer Spectrum Process (1439)	10.2	25.1
Eastman Experimental Panchromatic IV d	12.6	17.7
Ilford Special Rapid Panchromatic 5630 N	4.45	18.2
Standard Orthonon	23	1. 1. 37
	Minus Blue Filter	
Eastman Experimental Panchromatic 2199	1.91	4.37
Ilford Special Rapid Panchromatic 56,416	4.37	13.5
	F Filter	
		1 11001
Cramer Spectrum Process (1439)		1.88

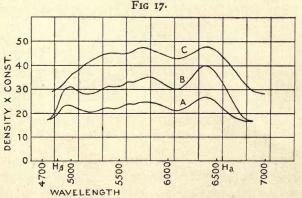
The figures give the absolute speeds of the plates to white light and through the Wratten Minus Blue and F Filters on the Hurter and Driffield scale. The value for the speed of the Standard Orthonon plate to white light obtained on the same apparatus is given for comparison. The Minus Blue, a deep yellow filter, cuts out all the blue and violet light, while the F, a deep red, transmits only the long red wave-lengths.

These figures show the remarkable increase in speed of the plates, particularly in the long wave-length region. Thus the Cramer Spectrum Process plates are increased in speed 150 per

FIG 16.



Wratten & Wainwright panchromatic. Expiration date, September 15, 1915. Used October 21, 1918.



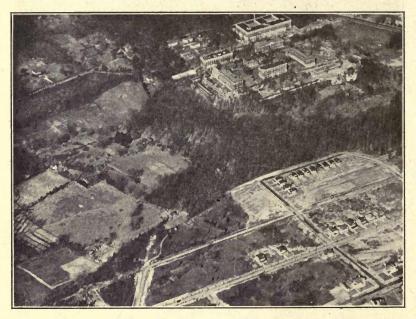
Wratten & Wainwright panchromatic (935). Expiration date, September, 1915. Used, October, 1918. A, untreated, 10 seconds exposure; B, untreated, 20 seconds exposure; C, treated. 10 seconds exposure.

cent. to white light and nearly 400 per cent. to the red. Through the Minus Blue filter one of the Ilford plates is increased 300 per cent.

These plates were used in experiments in airplane photog-

raphy, where for photographing through the haze a fast redsensitive plate is necessary. Fig. 18 is a set of prints from the Ilford plates. Prints a and b were printed for the same time from the untreated and treated plates. Print c is from the untreated negative timed to give the best print. The negatives were given the same exposure in the air and were exposed within a few seconds of each other through the Minus Blue filter.

FIG. 18a.



AMMONIATED PLATE. PRINTED 21 SECONDS. Hypersensitized Ilford plate. $1_{3\sigma}^1$ second exposure. Minus blue filter. Oblique from 4,000 feet.

The increased speed of the treated plate is apparent. It will be seen, also, that the treated plate is less contrasty and gives much better detail in the shadows.

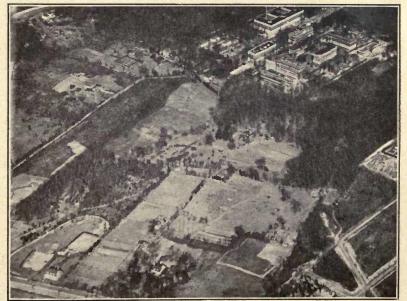
Fig. 19*a* is from a Seed 30 plate used with no filter at an altitude of 17,000 feet on a slightly hazy day. Fig. 19*b* shows the same scene taken on a treated Spectrum Process plate through the A filter. The pictures were taken simultaneously in a multiple lens camera. The increased haze penetration obtainable by the use of the red filter is clearly shown.

FIG. 18b.



UNTREATED PLATE. PRINTED 21 SECONDS. Ilford panchromatic plate. 130 second exposure. Minus blue filter. Oblique from 4,000 feet.





UNTREATED PLATE. PRINTED 8 SECONDS. Ilford panchromatic plate. 130 second exposure. Minus blue filter. Oblique from 4,000 feet.

ACTION ON MINIMA.

On every plate which had an irregular curve of spectral sensitivity, the minima were raised and in many cases smoothed out entirely. For example, note the minima at 6100A (Fig. 13), at 5200A and 5850A (Fig. 14), and at 5200A and 6100A (Fig. 17). This property of the ammonia treatment adds greatly to the value FIG. 10a.



SEED 30 PLATE, F/11. NO FILTER. 30° oblique from 17,000 feet. Hazy day. the second exposure. Taken simultaneously with 19b in a multiple lens camera.

of the plates in the photography of spectra, especially as the sensitivity is extended some 200 Angström units further into the red at the same time. The bad effect of a minimum is, however, smoothed out to some extent in ordinary photography through filters.

PLATES WITH KNOWN DYESTUFFS.

The difference in behavior between the ordinary and orthochromatic plates and the panchromatic plates shows clearly that . CARDER COTANIN . INAL STAR TAD

manual 100 moth and an 1 1 to prove be ready to t

the sensitivity increase dealt with here is not that observed by Eder, which is due to "ripening," but is an action associated with the dyestuff.

The effect of the ammonia is to increase the sensitivity of the emulsion to the incident light and not to increase the developability of a latent image already formed, since a plate treated with



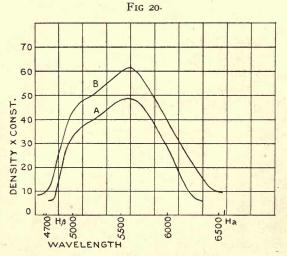
HYPERSENSITIZED CRAMER SPECTRUM PROCESS PLATE. F 4.5. WRATTEN A FILTER. 30° oblique from 17,000 feet. Hazy day. $\frac{1}{190}$ second exposure. Taken simultaneously with 19a in a multiple lens camera.

ammonia after exposure but before development showed no increase in sensitivity. That the action is not due to the alkalinity of the bath is shown by the fact that bathing in a solution of sodium hydroxide having the same concentration of OH ions had no effect on the speed of the plate.

Part of the increase in speed is, probably, due to the fact that some of the dyestuff in the gelatine film is washed out and

its screening effect diminished. The ammonia bath becomes colored with use and a color difference is observed between the untreated and treated plate before development.

The pinaverdol dyes sensitize for the green and yellow and an orthochromatic plate containing one of this series of dyes has the sensitivity increased by the ammonia treatment. Fig. 20 is a plate made up by the Cramer Dry Plate Co., using pinaverdol Pv. I., made by the Bureau of Chemistry, and marked by Cramer "similar to Trichromatic." The Trichromatic plate has the sensitivity curve of a pinaverdol dyed plate and Fig. 21 gives the curve before and after ammonia treatment.



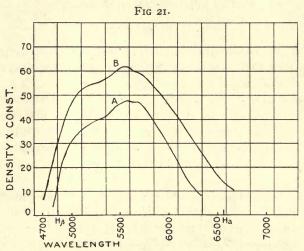
G D IV. PvI (similar to trichromatic); A, untreated, 10 seconds exposure; B, treated, 10 seconds exposure.

Mixtures of pinaverdol and pinacyanol are the dyes usually used in the preparation of panchromatic plates intended for spectrum photography. The mixture of these two dyes alone leaves a strong minimum at about 6100A. The ammonia treatment smooths out the minimum. In many plates intended for general photography other dyes are added to sensitize in this region (see Fig. 17).

Fig. 22 gives curves for a plate (similar to their Spectrum Plate) made up by the Cramer Co., using a mixture of the Bureau of Chemistry's pinacyanol, Pc. XII., and pinaverdol, Pv. I. It

will be noted that the addition of pinacyanol lowers the maximum due to the pinaverdol.

The dyes used in sensitizing are nearly all basic dyes, and it is not probable that the ammonia changes the dye itself. On the



Cramer trichromatic, No. 21. A, untreated, 10 seconds exposure; B, treated, 10 seconds exposure.

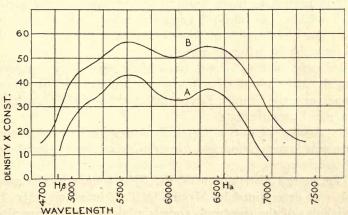


FIG 22.

G D III. PcXII and PvI; A, untreated, 10 seconds exposure; B, treated, 10 seconds exposure.

addition of ammonia to the dye solution before bathing, there is no color change. It is possible that the solvent action of the ammonia on the silver halide facilitates the reaction between the

dyestuff and the silver salt, in addition to its softening effect on the gelatin. This, however, does not account for the raising of the minima. It is possible that there is the formation of a $AgNH_3Cl + Dyestuff$ molecule with a photosensitiveness slightly different from that of the original molecule.

SUMMARY.

Since ammonia, when added to the dye bath in preparing bathed plates, increases the sensitizing action of the dye, its action on commercial plates was investigated.

In the course of the work three methods of sensitometry were used. Of these, one was used as a first qualitative test; the second, the spectrograph method, was used to study the effect of the ammonia on the sensitivity of the plate to each wave-length; the third, the Hurter and Driffield method, gives the absolute value of the speed of the plate.

It was found that by bathing commercial panchromatic plates in a solution of 25 c.c. ethyl-alcohol, 75 c.c. water, and 3 c.c. of strong ammonia water (20 per cent. NH_3) for four minutes at 18° C. and drying rapidly, the speed to white light is increased 100 per cent. in nearly all cases, and the sensitivity in the red extended one hundred or more Ångström units. The speed in the red is increased, in many cases, 400 per cent. If the plates be bathed without the alcohol (100 c.c. water, $3\frac{1}{2}$ c.c. ammonia water) the speed is still more increased, but the plates should be used immediately after drying.

Ordinary plates do not have their sensitivity appreciably changed. Most brands of orthochromatic plates are not improved, although one, the Cramer Trichromatic, showed the same increase as the panchromatic plate.

My thanks are due to Dr. J. S. Ames for his supervision of the present investigation and for valuable advice rendered during many conferences.

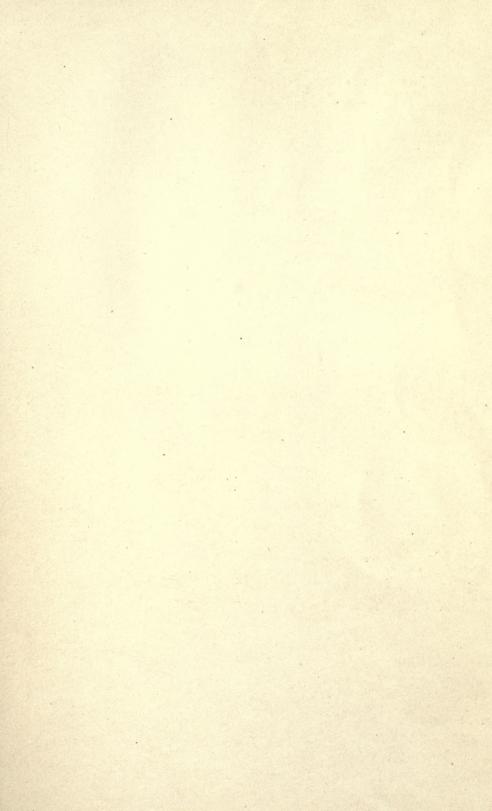
The energy distribution of the light source in the spectrograph was determined by Mr. E. P. T. Tyndall and Mr. H. J. McNicholas of the spectrophotometric laboratory. I am also indebted to Mr. R. Davis and Mr. F. M. Walters, Jr., through whose help the Hurter and Driffield curves were obtained, and to Dr. C. C. Kiess, under whose direction and coöperation most of the work was done.

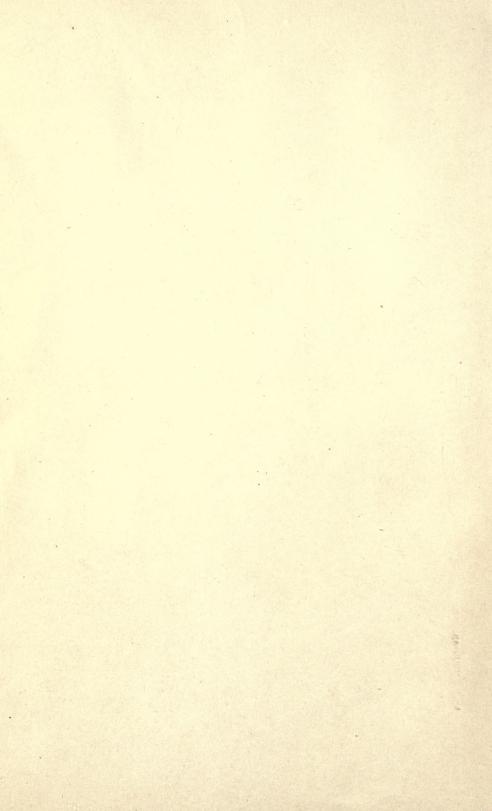
BIOGRAPHICAL SKETCH.

Samuel Moses Burka, son of Meyer and Ida (Zinberg) Burka, was born May 26, 1891, in Union Springs, Ala. He entered the undergraduate department of the Johns Hopkins University from the Baltimore City College in 1909. He received the baccalaureate degree in 1913 and entered the Chemistry Department of the graduate school, attending lectures by Professors Morse, Jones, Acree and Lovelace. In 1914 he entered the Physics Department, taking Physics, Physical Chemistry and Astronomy, attending lectures by Professors Ames, Wood and Pfund in Physics, Professor Anderson in Astronomy, and Professor Cohen in Mathematics. He received the degree of Master of Arts in Physics in 1916, the title of his essay being "Radioactive Atoms."

During the years 1913–1914 and 1914–1915 he held Hopkins Scholarships. In the summers of 1910, 1911 and 1912 he was assistant to Professor Gilpin in the Summer School. During the years 1911–1912 and 1912–1913 he was Student Assistant in Chemistry, and during 1915–1916 and 1916–1917 was Student Assistant in Physics and Lecture Assistant to Professor Ames. In the spring of 1917, under leave of absence from the University, he received an appointment as Laboratory Assistant in the Spectroscopy Section of the Bureau of Standards, and in February, 1918, was promoted to Assistant Physicist.



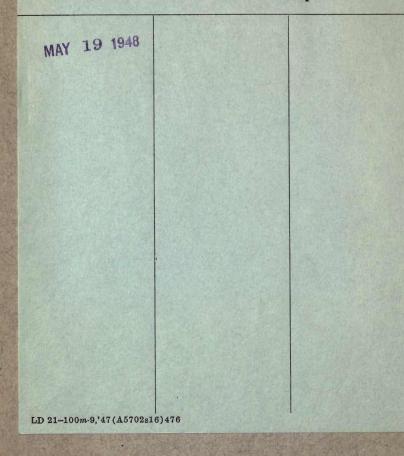






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