A Survey of Twelve Factors Influencing Covering Power

Jeffrey Wagner

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A SURVEY OF TWELVE FACTORS INFLUENCING COVERING POWER

by

Jeffrey P. Wagner

1982

A thesis submitted in partial fulfillment of the requirements for the degree of Bachelor of Science in the School of Photographic Arts and Sciences in the College of Graphic Arts and Photography of the Rochester Institute of Technology

Signature of the Author............................ Jeffrey Wagner Photographic Science and Instrumentation

Certified by............................................ Robert B. Bayley Thesis Advisor

Accepted by.......................................... Ronald Francis Coordinator, Undergraduate Research
I, Jeffrey Wagner, prefer to be contacted each time a request for reproduction is made. I can be reached at the following address:
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ABSTRACT

A statistical screening design was used to test 12 emulsion and processing variables said to influence covering power in silver halide emulsions. Emulsion grain size was found to be the only statistically significant factor at an alpha risk of .10.
ACKNOWLEDGEMENTS

The author wishes to express sincere thanks to Mr. Robert Bayley who put much time and effort into the advisement of this thesis.

A great deal of appreciation goes to the E.I. DuPont Company of Rochester, NY, for providing the emulsions and analyzing portions of the data.

A thank you is also due the Central Intelligence Agency for funding the project.

Last but not least I would like to thank Richard Quattrini for writing a program which helped in analysis of the data.
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INTRODUCTION

Covering power of a silver particle suspension is defined as the ratio of optical density to weight of processed silver per unit area. Given normal processing conditions covering power of a fine grain film is about 120 and the covering power of an X-ray screen film, with shoulder exposure, is about 30.¹

There are several factors which affect the covering power of a given emulsion. One of the most important is silver halide grain size. It has been demonstrated that for chemically developed emulsions covering power increases as the size of the undeveloped grain is reduced². The same authors have found that this increase is limited. In subsequent research, Farnell and Solman³ have found that covering power is generally independent of mean grain area, for chemically developed silver. This is true when grain area is between .002 and .005 square micrometers and inversely proportional to its square root for larger sizes.

Processing conditions are also significant in determining the covering power of developed silver. The type of developer, chemical or physical, influences the density given by metallic silver. Common nonfilamentary silver particles, given by physical development, have less light stopping power than the tangled mass of filaments given by chemical development⁴, provided there are equal amounts of developed silver.
present. James and others\textsuperscript{3,5} have shown that covering power usually decreases with increased development time to a minimum at full chemical development. This is true due to the small size of the developed silver particles at short development times. Farnell\textsuperscript{3} states that covering power falls by a factor of two on passing from 10 percent to 100 percent reduction of the halide.

The temperature of the developer can also cause changes in the covering power of the developed silver. Nepela\textsuperscript{6} has shown that the change in covering power is a result of the temperature influence on the intermolecular hydrogen bonds of the gelatin. Napela experimented with temperatures in the 20-35°C range. There is sufficient energy at those temperatures to break hydrogen bonds in the gelatin matrix. Napela also found that covering power could be increased by employing a gelatin denaturing agent in the developer. This would work by chemically breaking down the gelatin matrix. Thus, covering power varies directly with the mechanical properties of the gelatin matrix which can be influenced by both temperature and chemical composition of the developer. With the matrix broken down, silver filaments are able to spread further out into the gelatin. This in turn will increase the covering power of the developed silver. There is additional experimentation showing that other characteristics of gelatin can effect covering power. Silver developed in the presence of gelatin is filamentous, and in its absence compact\textsuperscript{7,8}. As stated earlier, filamentous silver will produce higher covering powers than compact silver.

The hardness of the gelatin effects the covering power\textsuperscript{9,10}. The
softer the gelatin the more readily filamentous silver will extend beyond the silver halide crystal boundaries. Upon drying, a softer gelatin will tend not to compress the filamentary silver as much as will hard gelatin.

Exposure given to the emulsion has a direct effect on the covering power of the developed silver.\textsuperscript{11,12,13} With a low-intensity exposure the number rather than the size of silver particles increased with development time. At high-intensity exposures, many grains are reduced quickly, and the size rather than the number of silver particles increases with development time\textsuperscript{14}. Many large latent-image sites are formed with a high-intensity exposure. Because of this, developer induction decreases and developer solvent action increases\textsuperscript{15,16}. This increase in solvent action causes an increase in physical development which, as stated previously by Haist\textsuperscript{14}, will cause a decrease in covering power.

Lastly, the tone of the developed silver may result in apparent changes in covering power. When silver is not spectrally neutral covering power depends on the wavelength of the measuring light\textsuperscript{17}. Silver deposits are colored and scatter light differently, so the optical system and spectral composition of the light used in the densitometer must be specified.\textsuperscript{18}

There are compounds which, when added to an emulsion, can increase the covering power of developed silver. Work done in this area\textsuperscript{19,20,21,22,23,24,25} shows that many of the compounds are polysaccharides or polysaccharide derivatives. Polysaccharides are
carbohydrates made from chains of monosaccharides. Basic polysaccharide structures are cellulose or alpha-linked polysaccharides, and starches or beta-linked polysaccharides. The afore mentioned work has shown that when these compounds are added to an emulsion in amounts of 10-50%, by weight of the gelatin, covering power increases. The effect is greater in the 30-50% range. The literature\(^{19,20,21,22,23,24}\) also states that the compounds could be put in a separate layer, to be coated next to the silver halide layer, with the same results obtainable.

**OBJECTIVE**

In none of the earlier mentioned work were more than two factors studied at one time. The object of this experimentation was to find which of the twelve factors has the greatest effect on covering power. From this, the significance of changing any one factor to increase covering power may be determined. Earlier experimentation proposed to study the effects of a polysaccharide itself. This was abandoned in lieu of first determining what factors had an important effect on covering power.

**EXPERIMENTAL**

A two-level fractional factorial screening design was used to accomplish the objective. This design was chosen because a full factorial design with twelve factors would require far too many runs to be plausible. The Plackett-Burman\(^27\) was the specific design used. In twenty runs
the design yielded the required information. Each of the twenty runs had a different combination of the factors tested. Covering powers were calculated for each of the twenty runs. These covering power values were the response variables used in the algorithm that calculated the effect of each factor. The absolute value of the individual effects were used to numerically rank the factors. A significance cut-off value was calculated using the error terms and an alpha risk. The factors which were above this cut-off value were statistically significant, those below were not. Although they were ranked the factors below the cut-off were subject to experimental error. The noise may have caused a factor to be ranked incorrectly. For this reason the ranking of factors below the cut-off had to be scrutinized much more than those above. Only main effects are estimated, any interaction effects are confounded with the main effects. This minimizes the influence of interactions on the results. Testing twelve factors in twenty runs leaves seven degrees of freedom for error estimate.

The factors and the levels that were tested are listed in table 1 on the next page. Emulsions chosen for the experiment had to be different in grain size and similar in formulation. For these reasons the emulsion formulations used in PPHS-31227 (see appendix A table 1&2) were used in this experiment. These emulsions were quite different in grain size and equal in composition. Both emulsions were 97% bromide, 3% iodide, and had a pAg of 10.53. The large grain emulsion had a mean grain size of .0142 cubic micrometers and the fine grain emulsion had a mean grain size of .0002 cubic micrometers. The tremendous difference in grain size is more than large enough to meet the experimental design requirements, and
<table>
<thead>
<tr>
<th>Factor</th>
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<tr>
<td>gel-silver ratio</td>
<td>1.5:1</td>
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<td></td>
<td>1:1</td>
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<td>Covering power adjuvant (inulin)</td>
<td>35 parts per</td>
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<td>100 parts gel</td>
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<td></td>
<td>melt at 37°C</td>
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<td>hardening</td>
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</tr>
<tr>
<td>Grain size</td>
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<tr>
<td></td>
<td>.0002 μm³</td>
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<tr>
<td>Wash time</td>
<td>60min</td>
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<td>15min</td>
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results in a large sensitivity difference (see figures 1,2,3&4 in appendix A). The emulsions were prepared in the emulsion lab of the E.I. DuPont Co. of Rochester, NY. Procedures followed were the same used in PPHS-31227 with the exception that the final gelatin to silver ratio is 1:1 and that the make was 15 times larger. The 1:1 ratio was equal to many of those in the literature, it also lent itself very well to coating with the draw bar coater.

Developers had to differ in the amount of physical development, yet be as similar in composition as possible. D-76 and DK-50 are similar in amount of developing agents and significantly different in sulfite content (see appendix B table 1 for formulas). With 70g more sulfite in D-76 there would be more physical development in processed samples. This difference in development was sufficient to fulfill the design criteria.

Development times of 7 and 3 minutes were determined after experimentation with the experimental film samples and exposure ranges. With equal amounts of exposure on similar film samples, 3 minutes in DK-50 and 7 minutes in D-76 produced similar characteristic curves. Both developers were used undiluted at 20°C. The development temperatures were selected after consideration of Napela's work on developer temperatures and their effect on covering power.

The two levels of gelatin present were a 1:1 gel silver ratio and a 1.5:1 gel silver ratio. According to work done by Perry this difference is large enough to change the covering power.
Inulin was chosen as the polysaccharide adjuvant to increase the covering power (see appendix C for formula). It was chosen because of prior work done at DuPont by V.C. Chambers\textsuperscript{19}, and because it is commercially available. Chambers found that inulin had an optimum effect when the weight added was 35\% that of the binder present, so this amount was chosen as the high level. In the second case no inulin was added because the general effect of the additive was sought, rather than differences due to specific amounts of inulin.

Two emulsion hardness levels were used. The first was just hard enough to stand the $35^\circ$C development temperature, the other significantly harder with a melting point of about $80^\circ$C. Formaldehyde was the hardener used because it was easily available. The continued hardening with time did not pose a problem because of the small amounts used, and the length of time the samples were allowed to harden. The specific quantities of hardener added were found by varying amounts of 37\%, 3.7\%, and .37\% formaldehyde solutions added to the emulsion samples. The melting points were measured by immersing the dry coated sample in a beaker of deionized water and heating it until the emulsion started to melt off. The melting points were checked at several time intervals. Hardening was essentially completed in 24 hours. After 3 days, the melting points increased by only one degree in most cases, and not at all in others.

The difference in exposure intensity was accomplished by using $10^{-2}$ and $10^{-4}$ second exposure times on an EG\&G Mark IV sensitometer. The exposures produced intensities of 40,000 and 700,000
lux respectively. A 1.0 neutral density filter was used with the $10^{-2}$ second exposure to keep log exposures close to being equal.

The fixers used were F-5 and F-24 (see appendix B table 2 for formulas). F-5 was chosen because it is a hardening fixer and F-24 because it is non-hardening.

A 15 minute wash time was used because it is a standard wash time recommended for many films. A 60 minute wash time was considered long enough to demonstrate the effect of wash time on covering power.

Tests were performed to optimize coating parameters before running the experiment. All coatings were done on an acetate base using a Conselor® draw bar coater (see appendix D). Variables in the coating were air pressure setting on the coater, coating bar size, emulsion temperature, amount of and placement of emulsion sample. The coating bars are wrapped with wire, the coating weight may be varied by using bars with different wire diameters. After testing the #10, #20, and #30 bars, the #30 bar was chosen because it produced the most even coatings. With the #30 bar, the best coatings were produced with the pressure set at 50 p.s.i., coating and placement of sample in front of the bar in the down position. One ml of 7% saponin solution was added to each 40ml sample of emulsion to promote even coating.

Fog tests were performed on samples from each emulsion. A coated sample with no exposure was processed in total darkness. Samples were left 6 ft. away from a #1 wratten safelight for 15 minutes. These
samples were processed the same way. The safelight had no effect on the fine grain samples and raised the base+fog density by .03-.05 on the large grain samples. With these results in mind, it was decided that the handling and processing of samples could be done under a #1 wratten safelight.

All samples were tray processed with 10 seconds RIT standard tray rock agitation every minute. After development, samples were stopped for 30 seconds, fixed for 3 minutes, washed, photo-flowed\textsuperscript{R}, and dried.

Emulsion samples for the design were prepared in 20ml amounts. This was a convenient quantity and allowed 4 coatings. The samples which required inulin and or extra gelatin were heated to 50\textdegree C so the gelatin and inulin would melt. They were then cooled to 35\textdegree C and coated. The formaldehyde was added immediately before coating to minimize hardening before coating and evaporation. After coating the samples were permitted to dry for 24 hours before being exposed and processed.

The covering powers were calculated by dividing density by weight of silver per 100 cm\textsuperscript{2}. Silver weights were determined using X-ray spectroscopy, done at DuPont. Densities were read on a Cosar 50 transmission densitometer accurate to within \(\pm.05\) density units.

Samples ranged in density between .03 and 4.59 in the first run of the experiment. Corresponding covering powers ranged from 57 to 239. This did not include three samples because the densities were not sufficient to get silver weight readings. With such a wide range in
densities the quality of the results was questionable. A second run was done with an aim density of 1.0. When these samples were analyzed it was found that covering power was dependent on density. This may be partially explained by the extent of halide reduction. When a decreased exposure was given to the samples with the higher densities the amount and size of latent images was also decreased. As a result of this the amount of halide reduction during processing may have been reduced. With the smaller latent images there is also an increase in developer induction period. This will lead to increased amounts of chemical development. For these two reasons covering power will increase.

RESULTS AND DISCUSSION

The tested factors, in order of importance, are listed in table 2 on the following page. Factors above a particular alpha risk value were statistically significant, for that alpha, those below were not. Grain size was the factor with the largest effect on covering power. Covering powers of the two emulsions did not cross at any point. The highest covering power for the large grain emulsion was not as high as the lowest covering power for the fine grain emulsion. This demonstrates the dominance of grain size on covering power. Grain size had an effect of 112, it was the only factor to lie above the significance cut-off value of 36.53. See appendix E for data and calculations. Grain size, wash time, amount of physical development, exposure intensity, amount of gelatin, development time, and fixer all had a negative effect. This means that the low levels of those factors worked to increase covering power. The
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<tbody>
<tr>
<td>1.</td>
<td>Grain size</td>
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<tr>
<td>2.</td>
<td>Wash time</td>
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<tr>
<td>3.</td>
<td>Developer solvency</td>
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<td>4.</td>
<td>Exposure</td>
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<td>5.</td>
<td>Developer temp.</td>
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<tr>
<td>6.</td>
<td>Gel-silver ratio</td>
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<td>7.</td>
<td>Emulsion hardness</td>
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<td>8.</td>
<td>Development time</td>
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<td>9.</td>
<td>Coated thickness</td>
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<td>10.</td>
<td>Fixer</td>
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<tr>
<td>11.</td>
<td>Quantity of adjuvant</td>
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<tr>
<td>12.</td>
<td>Drying temp.</td>
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remaining factors showed a positive effect.

The factors are ranked in order of effectiveness relative to the other factors. It may also be looked at as a way of attributing covering power changes to the factors. For example, if you were to use a low solvent developer and a shorter development time in an effort to increase covering power, a larger percentage of the increase could be attributed to the developer change than the development time change.

According to the results wash time has the greatest effect on covering power, behind grain size. This does not make sense when possible reasons for this are considered. Wash time had a negative effect, which meant shorter wash times would increase the covering power. The changes in covering power may be explained by a decreased optical density due to the effect of swelling and subsequent drying of the film. With a longer wash time there is more swelling in the emulsion. The swelling may pull the silver filaments apart and upon drying the optical density might be decreased. The combination of decreased density and consistent weights of silver present would cause decreases in covering power. If this were the case then emulsion hardness and drying temperature would have as much, if not more, of an effect on the covering power.

Because the type and amount of silver present are so critical to covering power, exposure and developer effects should be important. With the exception of development time these fell in the top 5 factors. The effect of emulsion hardness did not outweigh the effect of development time. It is likely that if the amount of hardener was increased, it might
eventually surpass the developer effects in importance.

Before the results are extrapolated to practical use there are several considerations to be taken. First, the emulsions used were model emulsions. There are not any commercial emulsions comparable to those used in the experiment, and therefore the levels of the factors (i.e. amount of hardener) used might not represent practical amounts. Because there were two levels of each variable tested the effect sought may have been missed in selection of levels. On the other hand, the levels chosen may have been so different as to overpower the results of the experiment. The effect of grain size may not have been so far above the others if the differences in grain size had been smaller.

Noise and interactions may have amplified the effect of some of the factors. This might have contributed to the high ranking of wash time.

Work done by Farnell and Solman\textsuperscript{12} states that the developer and development time have a larger effect on covering power than do the physical properties of the emulsion. James and Fortmiller\textsuperscript{5} state that the developer and developer temperature have a large effect on covering power. They found that this could be limited by the extent of emulsion hardness. This is supported by Nepela's\textsuperscript{6} work investigating the same factors. Results of this paper support the finding that developer effects are very important in determining covering power. It also shows that emulsion hardness dictates the extent to which this is true. It is probable that the levels of hardener used for this experiment were too close to show the extent of emulsion hardness effects. It is also
possible that if the amount of hardener was increased the effect of drying temperature might also have been increased.

Future work is suggested that would study the factors independent of grain size. Using results from this paper the levels of several factors should be changed. It may be a good idea to decrease the number of factors and increase the number of levels. This way interaction effects may be examined and their effects compared with the main effects.

CONCLUSIONS

The final covering power is a result of all the factors tested and is subject to change when any one or combination of those factors is changed. None of the factors were consistently able to change the covering power without significantly changing a more important characteristic of the film. If covering power changes are desired the most effective way of accomplishing this is to change the grain size.

Grain size had the largest effect on covering power. In this experiment the difference in grain size was enough to overpower the experiment. Other factors which proved to have an important effect on covering power were developer, exposure, and developer temperature. Each of these factors is important in determining the amount of silver produced. These factors out-weigh the effects due to emulsion hardness, but this may change upon increasing the amount of hardener added to the emulsion.
The polysaccharide adjuvant was added in the hope that it would have a significant effect on the covering power. The significance of the adjuvant fell very near the bottom of the ranking and therefore does not have a great value as a covering power adjuvant.
REFERENCES

15. ibid.
Appendix A
### Table 1

**Negative or Single Jet Emulsion**

**Large Grain**

Solution A:
- 7% phthaloyl gelatin 4.00g
- KI solution (36.76g/l) 10.00ml
- KBr, 99.6% 12.40g
- Distilled water to make 200.00ml

Solution B:
- AgNO₃ 17.00g
- Distilled water to make 450.00ml

Solution C:
- Inert gelatin 24.00g *
- KBr solution, 5 x 10⁻¹⁰N 400.00ml
- NaOH solution, 0.100N 8.00ml

* Published amount, 6.4g was added in the experimental emulsion.

### Preparation of Emulsion

1. Add solution B to solution A. Precipitation time 20 min.
2. Add 5.0g KBr.
3. Rippen for 20 min.
4. Coagulate.
5. Wash.
Steps 1-5 done at 65°C.
6. Redisperse (add solution C).
7. Add .0255g sodium thiosulfate.
8. Digest 60 min.
9. Add .030g 4-hydroxy-6-methyl-1,3,3,a,7- tetrazaindine.
Steps 6-9 done at 55°C.
Positive or Double Jet Emulsion
Small Grain

Solution A: 7% phthaloyl gelatin 4.00g
KBr (99.6%) 0.204g
Distilled water to make 370.00ml

Solution B:
AgNO₃ 17.00g
Distilled water to make 200.00ml

Solution C:
KBr (99.6%) 12.01g
KI 0.41g
Distilled water to make 200.00ml

Solution D:
Inert gelatin 24.00g*
KBr solution (5 x 10⁻⁴N) 390.00ml
NaOH solution (0.009N) 10.00ml
Distilled water to make 500.00ml

*Published amount, 6.4g was added in the experimental emulsion.

Preparation of Emulsion

1. Add solutions B&C to A. Precipitation time 15 min.
2. Coagulate.
3. Wash.
Steps 1-3 done at 60°C.
4. Redisperse (add solution D).
5. Add .051g sodium thiosulfate.
6. Digest 60 min.
7. Add .030g 4-hydroxy-6-methyl-1,3,3,a,7- tetrazaindine.
Figure 1

VOLUME WEIGHTED DATA

V(16) = 0.0213 μm³  D(16) = 0.279 μm  Vg⁻ = 3.99  MVD = 0.464 μm
V(50) = 0.0850  D(50) = 0.436  Vg⁺ = 3.40  VCV = 37.3
V(84) = 0.247  D(84) = 0.627  Vg⁰ = 2.90  SEQ = 0

Large Grain
Grain Size Frequency Distribution.

NUMBER WEIGHTED DATA

V(16) = 0.0043 μm³  D(16) = 0.161 μm  Ng⁻ = 3.30  MND = 0.276 μm
V(50) = 0.0142  D(50) = 0.241  Ng⁰ = 3.55  NCV = 44.9
V(84) = 0.0546  D(84) = 0.377  Ng⁺ = 3.84  CTS = 17169
Figure 2

VOLUME WEIGHTED DATA

\[ V(16) = 0.0002 \text{ \( \mu \text{m} \)}^3 \quad D(16) = 0.0544 \text{ \( \mu \text{m} \)} \quad V_{\sigma g^-} = 1.39 \quad \text{MVD} = 0.674 \text{\( \mu \text{m} \)} \]
\[ V(50) = 0.0002 \quad D(50) = 0.0612 \quad V_{\sigma g^0} = 1.51 \quad \text{VCV} = 40.7 \]
\[ V(84) = 0.0004 \quad D(84) = 0.0718 \quad V_{\sigma g^+} = 1.63 \quad \text{SEQ} = 0 \]

Grain Size Frequency Distribution.

NUMBER WEIGHTED DATA

\[ V(16) = 0.0001 \text{ \( \mu \text{m} \)}^3 \quad D(16) = 0.0530 \text{ \( \mu \text{m} \)} \quad N_{\sigma g^-} = 1.34 \quad \text{MND} = 0.0604 \text{\( \mu \text{m} \)} \]
\[ V(50) = 0.0002 \quad D(50) = 0.0586 \quad N_{\sigma g^0} = 1.39 \quad \text{NCV} = 14.3 \]
\[ V(84) = 0.0003 \quad D(84) = 0.0658 \quad N_{\sigma g^+} = 1.44 \quad \text{CTS} = 4337 \]
Figure 4
Small Grain Emulsion
### Table 1

**KODAK Developer DK-50**
- KODAK ELON developing agent: 2.5 grams
- Sodium sulfite: 30.0 grams
- Hydroquinone: 2.5 grams
- Balanced alkali: 10.0 grams
- Potassium bromide: 0.5 grams
- Water to make: 1.0 liter

**KODAK Developer D-76**
- KODAK ELON developing agent: 2.0 grams
- Sodium sulfite: 100.0 grams
- Hydroquinone: 5.0 grams
- Borax: 2.0 grams
- Water to make: 1.0 liter

### Table 2

**KODAK Fixing Bath F-5**
- Sodium thiosulfate: 240.0 grams
- Sodium sulfite: 15.0 grams
- 28% acetic acid: 48.0 milliliters
- Boric acid: 7.5 grams
- Potassium alum: 15.0 grams
- Water to make: 1.0 liter

**KODAK Fixing Bath F-24**
- Sodium thiosulfate: 240.0 grams
- Sodium sulfite: 10.0 grams
- Sodium bisulfite: 25.0 grams
- Water to make: 1.0 liter
Appendix C
Figure 1
Inulin

\[ n = \text{approximately 35} \]

approximate molecular weight 5000
Appendix D
Figure 1

Conselor® coater
Figure 2
Appendix E
Table 1

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<th>SAMPLE #</th>
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<td>2.</td>
<td>108</td>
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<tr>
<td>3.</td>
<td>90</td>
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<td>4.</td>
<td>86</td>
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<td>5.</td>
<td>80</td>
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<td>6.</td>
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Figure 1

Two-way Plackett-Burman Design
Table 2

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<th>SUM—</th>
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<td>1943.94</td>
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<td>1316.88</td>
<td>1446.21</td>
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CALCULATION OF SIGNIFICANCE VALUE
Significance value=(SFE)x(alpha risk)

\[ SFE = \sqrt{\frac{1}{7}[(30.97)^2+(18.72)^2+(9.55)^2+(17.95)^2+(2.78)^2+(29.95)^2+(6.55)^2]} \]

SFE=19.33

Student t value for an alpha risk of .10=1.89

cut-off=(19.33)x(1.89)=36.53

If diff./10 is greater than the cut-off value, it is significant.
VITA

The author was born on November 27, 1960 in Buffalo New York. At the age of 10 he moved to Bethlehem Pennsylvania, which is his current permanent address. Upon graduation from high school in 1978 he enrolled in the Rochester Institute of Technology. While at RIT he studied Photographic Science and Instrumentation. He was also involved with the RIT Outing Club for four years. Two of the four years he was president of the club.