# **A Comparison of the Errors in the Determination of Ink Strength by Various Methods of Specimen Preparation, Measurement and Analysis**

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Abstract: NPIRl has introduced a new standard bleaching white in North America for use in evaluating the strength of an ink by creating a pastel tint by diluting an ink or flush with the bleach. While standardizing on the composition of the bleaching white may improve the reproducibility of a given methodology it does not address all of the remaining variables. The methods by which one prepares the specimens, measures the optical properties and analyzes the measurement data are equally critical. Since strength is an economically important property of an ink, a full understanding of the correspondence between these methods is imperative. This report documents some preliminary findings using statistical experimental design to assess and identify the critical sources of errors and differences in the various methods of strength determination.

Introduction

The use of the bleaching or tinting process for the determination of the color strength of inks and concentrated pigment dispersions, such as flushes and pigment bases, has been an industry standard for many years. In recent years, the National Printing Ink Research Institute, "NPIRI", has assembled a task force to write a recommended procedure for the evaluation of the color strength of flushed colors. In their paper (NPIRI, 1996) a procedure is recommended using a new standard bleaching white identified as NPIRI Universal Bleach 95, which was made available to the industry that year. Other authors have written on techniques for establishing and measuring color strength in both liquid and paste

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inks (Saylor,l990; O'Boyle, 1986; ASTM, 1997). In addition, a technique for assessing print color strength instead of using a bleach, was described by Braun ( 1989) in which a standard film thickness proof was made of an ink on a printability tester using a standard paper stock. The proof was then measured with a spectrophotometer and the color strength assessed using a special FAIF computer program. The ASTM (1994, 1997) has also published two standards bearing on color strength and tinting strength, Standard D 387 and Standard D 2066. In these methods, standard techniques are described for instrumental as well as visual methods of comparing the color strength of a sample versus a reference standard. It is our purpose in this paper to identify the sources of error that can arise in color strength determinations, particularly when instrumental techniques are used.

# Theory

Most of the determinations of colorant strength of printing inks and color concentrates have traditionally been done by diluting the concentrated ink with a standard white tinting base. The purpose of this was to view the small differences between batches and standards in a way that would make them easily visible. Typically, a visual assessment was made after diluting both the reference standard ink and the batch ink with the white and then drawing them down sideby-side in a thick film and noting whether they appear to be of equal color. If one were judged to be stronger, this bleach would be diluted with a small amount more white to bring it to an equal color. When both specimens appear equal, then the percentage of white added would be equal to the percentage difference in strength between the reference standard and sample.

In recent years, the proliferation of spectrophotometric instruments in the printing industry has resulted in the use of instrumental measurements of bleaches in an attempt to get quantitative strength differences directly, without having to add additional bleaching white to the strong sample, thus saving laboratory time. To accomplish this, the spectral reflectance factor measurements must be related to the absorption properties of the ink. In addition, there is also a desire on the part of quality control people to use a stored instrumental reference standard color measurement, rather than having to remake a bleach of the reference standard every time a sample of a new batch is bleached. This procedure could save nearly 50% of the laboratory effort in making color strength assessments. However, past attempts to do this have met with some experimental difficulties in repeatability of results and of reproducibility between communicating laboratories.

The most widely used model to relate reflectance to absorption is the Kubelk-Munk (1931) two-flux, single constant model. This model assumes that the absorbing material is dispersed in a perfectly diffusing medium. The mixtures of a small amount of colored ink into a large volume of white bleach matches this

assumption to a very close approximation as documented by Johnston-Feller and Bailie ( 1982). In the Kubelka-Munk model, the absorption of the ink relative to the scattering of the bleach is assumed to be proportional to the concentration of the ink, in much the same way that the absorption of an analytical solute dissolved in a solvent is assumed to be proportional to the concentration of the solute by Beer's Law. Thus, the relative strength of two mixtures can be determined by taking the ratio of the Kubelka-Munk values of each mixture. A percent strength is then obtained by scaling of the ratio so that equal concentrations yield a I 00% strength value. Equation 1 shows the relationship between reflectance and the ratio of absorption  $(K)$  and scattering  $(S)$ . Included in the equation are also terms to adjust the values for the first surface (specular) reflectance of the bleaching cell and for the light that does not escape from the cell but is reflected back into the cell by the surface due to total internal reflectance. These corrections were first suggested by Saunderson (1942).

$$
R_{Theoretical} = \frac{R_{Measured} - r_e}{(1 - r_e)(1 - r_i) + r_i(R_{Measured} - r_e)}
$$
(1)

$$
F(R) = \frac{K}{S} = \frac{\left(1 - R_{Theoretical}\right)^2}{2 \cdot R_{Theoretical}}
$$
 (2)

where  $r_e$  is the surface or specular reflectance outside of the ink film and  $r_i$  is the surface reflectance inside of the ink film.  $R_{Measured}$  is the reflectance factor measured on the spectrophotometer and R<sub>Theoretical</sub> is the reflectance inside of the ink film. K is the absorption due to the ink film and S is the scattering from the bleach.

Since the K to S ratio, often identified as  $F(R)$  in spectroscopy, is assumed to be linear with changes in concentration, the earliest method of colorant strength determination, pioneered by textile dyers, involved finding the wavelength at which the absorption (F function) was maximal and then taking the ratios of the F value of the test dye to the F value of the standard dye. This method worked well if the spectral signatures of the test and standard are identical. To compensate for small spectral variations, due to impurities in the commercial grade dyes, it has been suggested that one sum the F function over all measured wavelengths. In the regions where there is little or no absorption the F value adds little to the sum while in the region of maximum absorption, the F value contributes strongly to the sum. In the limit of spectral identity, this method should give identical results to the single wavelength method. In the case of small spectral differences this method will "smooth" out the differences using the weighted sum. Noting that the sum of the F function tended to emphasize all parts of the spectrum equally, it was found that colorants with multiple peaks, such as magentas or deep blues, were often producing strengths that did not agree well with visual evaluations. It was then suggested that the sum of the F function be weighted by the sum of standard observer functions. The sum of the standard observer functions produced a three peak weighting functions that was supposed to approximate the human visual response. This method has been termed the "Integral" method. Garland (1973) proposed that the "Integral" method should include the light source under which the visual judgements of strength were performed, since the amount of available light will further deemphasize certain regions of the visible spectrum. His method has come to be known as the "Integ" method. The final method, in use today and recommended in the NPIRI report and ASTM standards, involves treating the tristimulus values as filter functions, much like the red, green and blue response filters of a reflection densitometer, and converting the color response to an absorption value using the F function but substituting the X, Y or Z for the  $R_{\text{Measured}}$  value. In this case, it is usually recommended that X be used to evaluate cyan inks, Y be used for magenta ink and Z be used to evaluate yellow inks.

In the following sections, we will describe some of the parameters that need to be carefully controlled if this procedure is to be successfully followed. The results of a set of designed experiments and a statistical evaluation of them will be given.

### Experiment

For this experiment, a quantity of NPIRI 95 Universal White Bleach was obtained. The bleach was used with a set of modem heat-set, process inks for offset printing. A standard was formed by preparing a 50:1 mixture of bleach to ink as recommended in the NPIRI study (NPIRI 1996). Bleaches were made by taking  $10$  grams of white bleach and  $0.2$  grams of ink  $(50.1)$ . Five replicate bleaches were made using the cyan ink to evaluate the repeatability of the weighing and mixing process. The results of those replicate weighings are shown in Table 1. Differences in ink strength were synthesized by increasing or decreasing the amount of ink mixed with the 10 grams of white bleach. The actual concentrations in the series are: 0.1695, 0.1796, 0.1898, 0.2000, 0.2102,  $0.2204$ ,  $0.2307$  grams. This represents a concentration series of: 1.6667%, 1.7643%, 1.8626%, 1.9608%, 2.0587%, 2.1565%, 2.2550% respectively. If one then assumes that the ratio of the concentration represents the strengths, then the relative strengths will be: 85.00%, 89.98%, 94.99%, 100.00%, 104.99%, 109.98%, 115.00%. This forms a series of 15% weak and 15% strong bleaches.

All weighings were made on a precision analytical balance, with fully enclosed weighing pan. The balance displayed weights down to 0.1 milligram. All weighings were made to within 0.0004 grams of the required weights, both for the white bleach and the chromatic inks. This means that the potential errors,

due to weighing would be 0.004% for the bleach and between 0.17% and 0.23% for the inks. All of these errors are well below the threshold for visual detection and, based on Table 1, below the errors for specimen preparation and presentation to the instrument.

The weighings were made on small, tempered glass plates and transferred to a larger glass plate for mixing. The large quantity of white bleach was always weighed first then the balance was tared and the small amount of ink added to the bleach. This technique was used to avoid the possibility of leaving a film of ink on the weighing plate that cannot be mixed with the white bleach. As shown above, small errors in the weight of white bleach results in errors of no significance in the bleaching mixture. The weighed bleach and ink were then transferred to the large mixing plate, being careful not to disturb the pool of ink on top of the bleach. Mixing was carried out for two to three minutes by pulling the white bleach up and folding it over the pool of ink. This process was repeated until the mixture was a uniform color and no streaks of white or color could be seen in the mixture.

At that point, a one gram aliquot was removed from the mixture and placed in the center of a custom bleaching cell. The cell was prepared from a plate of clear acrylic plastic (90 mm wide by 140 mm long by 4 mm thick) onto which a thin plate (0.6 mm) of galvanized steel was taped from one end to form a hinge. In the center of the steel plate was a circular aperture, 40 mm in diameter. After the aliquot of ink was placed in the center of the aperture, a large microscope slide (Coming 2947, 75 mm x 50 mm x 1 mm) is placed over the aperture and pressed down to flatten the ink and uniformly fill the cell. The microscope slide was chosen to cover the cell rather than the more common microscope slide cover glass for two reasons. First, the slides chosen are wider (50 mm rather than the more common 30 mm ) and much thicker. A slide cover glass will often bend and become convex rather than flatten the ink completely. Such curvature can intrude into the measurement port of an instrument in such a way as to influence the spectral reflectance factor readings. Figure 1 shows the transmittance of the glass slides indicating closeness to spectral neutrality of the glass used in the slides. After positioning the slide onto the cell and forcing the ink down into the sample well the bleaching cell was carried to waiting spectrophotometer. The time from assembly of the cell to the time that the cell is mounted on instrument and is ready for measurements to begin can be no more than 10 or 12 seconds. Thus readings can begin as soon as 15 seconds after the glass cover makes contact with the bleach mixture. This is important, since the NPIRI report and ASTM standards recommend that readings be taken within 45 seconds of the time the cell was prepared. This recommendation is given, in spite of the table in the NPIRI report that showed no significant difference in measured strength for readings taken at 45 seconds and at 60 minutes after assembling the cell. All readings were taken on an Applied Color Systems (now Datacolor International) ChromaSensor CS-5 spectrocolorimeter.

The final design for this experiment was 3 colors (cyan, magenta, yellow) by 5 analysis methods (wavelength of maximum absorption, the sum of the K/S values, the "Integ" value, the "Integral" value and the tristimulus value) by 7 synthetic strength levels (85%, 90%, 95%, 100%, 105%, 110%, 115%). This represents a  $3 \times 5 \times 7$  fixed effect 3-way ANOVA experiment. In addition, each level was replicated three times for a total of 315 experiments. For the results given below, the values of  $r_a$  and  $r_i$  were set to 0.04 and 0.60 (4% and 60%) respectively.



Figure 1. The spectral transmittance of the Corning 2947 microscope slide glass used to cover the bleaching cell.

# Results

Table 1 shows the relative strength, in terms of the ratio of K/S at 630nm relative to the average, of making five replicate bleaches of the cyan ink at the nominal concentration of 0.2 grams of ink in 10 grams of bleach. Note that four of the five specimens were read within 30 seconds of sealing the cell while one was made within 60 seconds. That cell required more time to correctly position the microscope slide. As a result, its value was slightly further from the average than the other four specimens. Based on the NPIRI report, such a large difference should not have been observed in the strength of that bleach.



The results in Table 1 indicate that the weighing, mixing, filling and reading procedures are adequate. The maximum color difference is 0.3 CIELAB color difference unit, well below the visual threshold for a blue. If one drops the 60s reading from the set then the average decreases to 99.5 and the standard deviation to 0.57. The jump in reading for the 60s time lapse (nearly  $3\sigma$ ) was a cause for concern. Subsequent bleaches were then made and allowed to set on the instrument for a period of time while readings were taken without moving the cell. Results from one such test are shown in Table 2.





Figure 2 shows similar time dependent strength changes for three different colored inks. All strength estimates are based on using the tristimulus values appropriate for the color ink being tested. Strengths are relative to the frrst reading of the nominal 100% specimen. The results were very surprising. The data have been plotted on a semi-logarithmic basis showing relative strength versus the log of time and the linear regression fit is excellent.



Figure  $2.$  Time Series Plots of Relative Strength Changes for Readings Taken from the Time the Cells Were Assembled for the Three Process Color Heat-Set. Offset Inks. Color Strength is relative to the First Reading in Each Series.

Each color ink showed a similar time dependent behavior in the strength build, with moderate changes in the rate of change (slope). Figures  $3 - 5$  show the spectral reflectance factor curves for these bleachings indicating that the absorption region does indeed exhibit a variation in reflectance factor as a function of time. The apparent strength of the bleach does change as it is being measured on the spectrophotometer.

To further investigate this behavior, time series readings were taken on several different concentrations and on replicate weighings of the same concentration. Figures 6 and 7 contain the strength versus logarithmic time plots for several different absolute ink concentrations in the bleach (shown as relative strength values) all referenced to the same initial reading of the nominal 100% level. Note that the different concentrations form approximately parallel lines indicating that the initial readings and subsequent readings maintain the relative strength relationship to the nominal concentration and that the change in strength with time follows approximately the same time course for any given colorant. Different colorants have different slopes but different concentrations do not. It is also important to note that replicate weighings fall on or very nearly on top of the previous curves, again indicating that not only is the strength consistent from replicate to replicate but also the time dependence.

To test whether this behavior was due only to the type of ink that had been chosen for the experiment, a cyan UV-curing ink was bleached and the reflectance drift logged in the same way as had been done with the heat-set inks. The re-

suits were very similar. Figure 8 show the time dependent change in strength of the UV curing ink bleached in this similar manner. Also shown is a replicate run that came from a separate weighing performed on a different day by the same operator.



Figure 3. Spectral Reflectance Curves of a Series of Yellow Ink Bleaches Measured as a Function of the Time Elapsed from the Time the Cell Was Assembled.



Figure 4. Spectral Reflectance Curves of a Series of Magenta Ink Bleaches Measured as a Function of the Time Elapsed from the Time the Cell Was Assembled.



Figure 5. Spectral Reflectance Curves of a Series of Cyan Ink Bleaches Measured as a Function of the Time Elapsed from the Time the Cell Was Assembled.



Figure 6. Three different concentrations of magenta, heat-set ink in NPIRI 95 Bleach, measured for change in strength with time. The open square symbol shows a replicate weighing of the same 100% nominal concentration.



Figure 7. Two different concentrations of yellow, heat-set ink in NPIRI 95 Bleach, measured for change in strength with time. The cross symbol shows a replicate weighing of the same 100% nominal concentration.



Figure 8. A cyan UV-cured ink in a 50:1 mixture with NPIRI 95 bleach and tested for strength drift. The test was replicated on a different day with a similar cyan ink.

Based on the experience gained from the temporal dependence of the bleaches described above, the specimens that were used for the statistically designed experiment were prepared and measured in the first 30 seconds after assembly of the bleaching cell. The 3-way fixed effect ANOVA table is shown in Table 3.



Table 3. 3-Way Analysis of Variance for Fixed Effects of Color, Concentration and Method of Calculation. (1=COLOR, 2=CONC, 3=METHOD), \*

The analysis of variance shows that there is a significant effect due to the concentration, which is good – indicating that the  $15\%$  weak bleach measures significantly less than the average bleach which would be near 100%. There is also a significant difference between the colors. This implies that the errors associated with bleaching a cyan, a magenta and a yellow are not equal. This is also not unexpected. It is well known that dispersing the different pigments is more or less difficult.so it is reasonable to find that it is more or less difficult to keep the pigments dispersed. Surprisingly, there is not a significant effect due to changing the method of calculating the strength.

# Discussion

While the presence of flooding of colors in bleaches has been observed before, the linear dependence of the strength of an ink with the log of Time, as determined from a white bleach was totally unexpected. Neither ASTM nor the NPIRI report had reported such a fmding. What is also surprising is the lack of significance in the ANOVA results for the two different methods of strength computation. Both of these results need further experimental work.

The bleaching process uses a paste ink and mixes it with a white dispersion loaded with about 50% by weight of titanium dioxide pigment. Shear thinning temporarily lowers the viscosity of the bleach so that the ink can be evenly and uniformly dispersed into the large volume of bleach. The recovery of the original viscosity can be fairly rapid, as measured on a rheometer. However, it was observed that the bleach sample picked up after the mixing and placed into the cell would often flow easily off of the ink knife, while neither the bleach nor the ink, by themselves did so. The bleaching cell is normally placed on the instrument port with the cell in a vertical position. This could allow the bleached ink to sag within the cell, during the reflectance factor measurements. To evaluate this hypothesis, the CS-5 instrument was mounted to operate in the vertical mode, with the specimen port pointing downward. The cyan heat-set ink was

again bleached, placed in the cell and positioned under the instrument up against the port, with the cell in a horizontal position. Figure 9 shows the cvan UVcured ink with the cell in a vertical position, the cyan heat-set ink with the cell in a vertical position and the cvan heat-set ink with the cell in a horizontal position. There is no significant difference in the bleaches taken in the two orientations and both of the slopes are consistent with the bleached cyan UV-cured ink. The presence of the flooding effect, even when the cell is held horizontal and flat indicates that the pigment is migrating to the surface of the bleach at a similar rate.



Figure 9. Comparison of the temporal drift of a cyan heat-set ink bleach and a cvan UV-cured ink bleach. The heat-set ink bleach cell was presented to the instrument in a normal vertical position, as was the UV-cured ink. The heatset bleach was also presented in a horizontal position, where the bleach would not be able to flow within the cell.

Based on the temporal measurements it might reasonably be hypothesized that placing of the microscope slide over the bleach in the cell initiates the migration. To test this, a single bleaching was used to fill a second or third cell after remixing the bleach. The relative strength number shown in Table 4 would seem to indicate that the migration takes place during the mixing phase, continues after mixing is stopped, and is not reversed by re-mixing. This last speculation is in contrast to much of historical experience and needs to be studied further.

The one process that appears to be consistent is the linear behavior of the strength increase with the logarithm of the elapsed time. Since the process is logarithmic much of the drift occurs in the first few minutes. This drift with the logarithm of the time seemed familiar to the senior author of this paper. In a paper given at a TAGA meeting in 1987, Bassemir and Krishnan reported on the migration of a surfactant through a fountain solution, which was also related to the log of time. Since most pigments use surfactants as aids in dispersion and flushing, it is seems logical that the drift of the pigment may be related to the diffusion of the surfactant from the surface of the pigment and into the bleach. As the surfactant migrates away, the pigment flooding to the surface is facilitated. This migration could be dependent on the surfactant and any surface treatment present and the morphology of the pigment particles, thus developing a different slope for each color ink. Another possibility is that surface treatment with resins, which is a particularly common surface additive on yellow and magenta pigments, can affect migration rates due to the similarity of the resin treated to the polymers in ink vehicles. Confirmation of these hypotheses will require further studies that utilize pigments with various morphologies and surface treatments but different chemistries.



A method to minimize the effect of the temporal drift would be to place the cell on the instrument and collect reflectance factor readings each minute for the frrst three minutes. The time of collection should be logged with a stop watch or computer software, some color measurement packages will allow the instrument to be set up to collect timed readings. Readings could be taken more often if desired, say every 30 seconds, and the strength determined at each measurement point versus the stored standard. Then one could use a least squares routine to fit a linear function of  $log(t)$  versus strength and report the strength at the middle time point, for example at the two minute point. That predicted strength, can be given as a single value or as a statistical range with one, two or three sigma confidence levels on the interval. This would put relative strength determinations on a solid statistical basis. Further, if a custom bleach is developed which does not exhibit the same level of temporal drift as the NPIRI 95, the least squares estimate becomes the best unbiased estimator of the average strength.

The reason for lack of significance in the method of strength analysis is more difficult to quantify. 1f there were some, unidentified systematic variable, that ANOVA has missed, it will usually be detected in a Normal probability plot of the residuals. Figure 10 illustrates such a plot for the  $3 \times 5 \times 7$  3-way ANOVA.



Figure 10. Normal probability plot of the residuals after application of the ANOVA model. The straight line represents the standard Normal probability distribution. The more closely the residuals follow the line, the more likely they are to have come from a random Normal distribution.

The fit of the residuals to straight line predicted by the standard Normal distribution is quite good. Since this line represents the cumulative Normal probability, which assumes the full population extends to plus and minus infmity, while our experimental sample does not, the top and bottom ends of the residual will often show deviations from the line. On the other hand, if one determines the predicted strength from the known concentration by linear regression, the standard error of the predicted strength ranges from 1.7% for the cyan to 8.1% for the yellow with the error for the magenta falling in between at 3.5%. Only the cyan has a standard error in the range that is desired (less than 2%).

As the yellow had the highest standard error, it was hoped that further analysis of it would lead to a better understanding of the un-modeled random errors. In performing a thorough review of the measurement data, it was observed that the third replicate bleaching of the yellow ink did not agree with the two previous bleaches. The only identifiable difference in procedure between the second bleaching of the yellow and the third was that the mixing was carried out, not on the large glass plate, as previous, but on the smaller glass weighing plate. It was believed that this procedure generated less mixing energy and thus resulted in a poorer dispersion of the ink in the bleach. A fourth replicate bleaching was then performed on the yellow ink. This time the weight components were transferred to the large glass plate and the earlier mixing procedure performed as before. The results was a replicate bleaching that was in excellent agreement with the earlier bleach, as illustrated in Figure 7.

The results of the mixing test, now indicate that the source of the remaining random error is the mixing procedure. The procedure of mixing for 2 to 3 minutes, until visually uniform is shown to be adequate for the cyan ink, barely acceptable for the magenta ink and unacceptable for the yellow ink. Neither time nor visual appearance alone was enough to signify when an ink had been thoroughly mixed into the bleach. It remains a task for future work, to develop an operator independent, highly reproducible method for mixing an ink or flush into the standard bleach.

#### Conclusions

- 1. The most critical parameter in instrumental measurements of bleaches is the careful measurement of time elapsed after placing the cover slide on the bleach until the color measurement is made. This is essential due to the rapid orientation of colorant at the interface, an effect known as flooding.
- 2. It has been found that the flooding rate of various pigments is quite different even though diluted in the same standard bleaching white. The color strength has been found to be a linear function with the logarithm of time, and the slope of the resulting line is a measure of the rate of increase of color strength with time .. The ratio of the rates of flooding of heatset weboffset inks in NPIRI Standard 95 Bleach was found to be in the order of cyan=l, magenta=l.5, and yellow=2.5.
- 3. The measurement of bleaches at elapsed times of less than 1 minute is extremely difficult to repeat and should be avoided due to the rapid rate at which flooding occurs during the zero to one minute time period. Variations in timing of even 10 seconds during this period can result in significant errors in strength.
- 4. A suggested procedure for minimizing errors during these measurements, is to make at least three successive color measurements on each sample at known times of about one, two and three minutes after covering the bleach with a slide; and then plotting these three measurements vs log of time to predict the actual strength at exactly 2 minutes.
- 5. The preferred method of calculating strength differences is the use of the selected tristimulus value technique. It has been found that this gives predicted strengths close to the actual strength differences.
- 6. A statistical analysis of all of the data, taken at various strength levels indicate that the standard errors for the three process colors were l. 7% for cyan, 3.5% for magenta and 8.1% for yellow.
- 7. The use of the maximum wavelength of absorbance method for strength assessment gives very similar results, except in the case of the cyan ink where the strengths appear somewhat lower than the actual differences.
- 8. Newer spectrophotometers often have additional options for strength determination, which are shown to give varying results when compared to the actual color strength; and are not generally as useful as the two methods described above.

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