

THE SYNERGISTIC FASTNESS EFFECTS OF SOME
DISPERSE DYES ON NYLON 66

A THESIS

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Approved:

[Signature]
Date approved by Chairman:

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DEDICATION

To my wife, Barbara

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SUMMARY

A very limited amount of research has been performed on the fastness of dye mixtures relative to their components taken alone. Even less work has been published on the measurement of color changes due to hue and concentration changes by spectrophotometric and colorimetric techniques.

The purpose of this research was to determine if dyes in mixture were more fast or less fast to carbon arc light and nitrogen dioxide than they are when used alone.

Four common disperse dyes were used in this research on nylon 66 carpet fiber. Eleven single and multi-dye mixtures were examined at the following concentrations; 0.13 per cent, 0.67 per cent, and 2.0 per cent owf (on weight of fiber).

The results revealed that for lightfastness there does exist certain dyes which in combination with one another have greater lightfastness than either dye used alone. In no case was a mixture of dyes of less lightfastness than either component dye alone.

The results of the gasfastness research revealed an opposite trend in that in no case was a mixture of greater gasfastness than either dye alone and in some cases was less fast to nitrogen dioxide.

The color changes which took place during exposure to a carbon arc were primarily a combination of hue and concentration changes. However, on exposure to gas the color changes which resulted were due almost entirely to hue changes.

Such results are in agreement with the theory that molecular

masking may play a part when dye aggregates are exposed to light. In such a situation, the inner molecules of the aggregate are shielded from the light energy by the outer molecules and are not as subject to fading. When the same aggregate is exposed to a gas, the inner molecules are not afforded any protection since the gas may diffuse through the dye aggregate.

CHAPTER I

INTRODUCTION

Purpose

The purpose of this research was to determine if, in two and three dye combinations, four disperse dyes frequently used for dyeing nylon 66 carpet fiber would result in dyeings with increased or decreased lightfastness and gasfastness as compared to either dye used alone; to quantitatively present the degree of positive or negative synergism recorded; and to relate the synergism recorded to hue and/or concentration changes. Positive and negative synergism as referred to in this research may be defined as a respective increase or decrease in fastness due to the presence of other dyes in the fiber.

Statement of Problem

Fastness to light is one of the most important properties of a commercial dye. With each dye placed on the market, technical data are supplied describing the fastness properties of the dye. However, when in combination with other dyes, the rating for a specific dye alone may not apply. In combination with other dyes in a dye-fiber system, the lightfastness of the dye may be increased or decreased by interaction between the dyes or with other parts of the system. Many carpet manufacturers use dyes according to their fastness ratings, i.e., they specify a certain minimum fastness the dye must possess before it will be used. It is probable that if the fastness of a dye were reduced in

a mixture, customer complaints would result and if fastness were increased in a mixture, more economical or otherwise acceptable dyes could be used.

However, difficulties in the measurement of changes in the color, as estimated by changes in hue or concentration of the faded dyes have compounded the problems of fastness evaluation when no interaction of the dyes in the fiber is considered. Small changes in concentrations in certain dye mixtures may lead to seemingly large changes in hue. A good example of this is a mixture of blue and yellow to make a green. Assume the blue is of good fastness and loses an insignificant amount of its color on fading alone. Assume the yellow loses half of its tinctorial strength upon fading alone. If, in the mixture, the extent of fading of both dyes is the same as when separate, then the original green mixture will change from green to a more bluish hue. The total change in color of the mixture appears much greater than the change in either the two dyes considered separately because of the eye's greater sensitivity to hue than to concentration changes. The casual observer might conclude that the blue increased the rate of destruction of the yellow, and that the dyes fade more rapidly in mixture than alone when, in fact, neither of these conclusions is valid.

Review of the Literature

Work on the fastness of binary and trinary dye combinations has been hampered by the general lack of understanding of the exact nature of fading of single dyes. Though much has been written on the subject of lightfastness and gasfastness, only those publications that concern themselves with the variables encountered in this research will be noted.

Schaeffer (1) pointed out that illumination behind glass required one to deal with photo-oxidation. Illumination causes the moisture in a fiber to cleave into H atoms and OH radicals. The OH radicals then form H₂O₂ to form a system containing; fiber, dye, H⁺, H₂O₂, and air. To this is added whatever elements that exist around the system at the time of illumination.

Time is another factor which must be considered: that for illumination and that in which there is no illumination. For example, there is direct sunlight for only a few hours a day and then there are hours of darkness. Fading experiments must duplicate these conditions since some dyes are reduced on illumination and then reoxidized during periods of no illumination.

Basic Laws of Photochemistry

Photochemical reactions are governed by two fundamental laws: (1) the Grotthus-Draper Law which states that only radiations which are absorbed by the reactants of the system produce chemical change, and (2) the Stark-Einstein Law which states that each molecule taking part in a chemical reaction induced by light absorbs one quantum of radiation.

Once this energy has been absorbed, any one or all, of the following reactions may be possible:

- (a) The activated molecules break down into atoms and radicals which react with other molecules and/or radicals which may lead to chain reactions.
- (b) Two molecules put into an excited state by the absorption of energy may react with each other,
- (c) activated molecules may react with non-activated molecules, and

(d) The previous reaction may produce a third compound which may be able to enter into more reactions.

Lightfastness Variables

Other variables that must be included in any discussion of lightfastness and gasfastness include: (a) intensity and duration of the light source, (b) type of illumination, (c) temperature of the dyed specimen, (d) dye concentration, (e) moisture content of specimen, (f) properties of the substrate, (g) structure of the dye and substituent groups, and (h) the physical state of the dye.

Giles (2) noted that there was a difference between fading results obtained by artificial light and those produced by sunlight. Cooper and Hawkins (3) pointed out, however, that an artificial source of energy was most commonly used for lightfastness studies. This artificial source is the enclosed-flame carbon arc fade-ometer. Its output resembles sunlight to the extent that the proportion of radiant energy emitted between 3000 and 4300 A. (Angstroms) is almost the same as that of noon summer light. In other regions of the spectrum, the energy distributions are quite different.

The American Association of Textile Chemists and Colorists (AATCC) (4) has performed the most thorough study of the relation between light intensity and fading and concluded that there is no threshold light intensity below which fading does not occur and that the amount of dye faded is proportional to the illumination intensity.

Morton (5) found that increases in temperature increased the rate of fading of dyes. Giles, Baxter, and Rahman (6) in their studies of fastness of dyes on hydrophobic fibers pointed out that high heats of

illumination may initially breakdown dye aggregates causing an apparent increase in the color depth. This effect can mask fading in the early stages and is favored by dry fading conditions.

A similar situation exists for the relation between humidity and fading. McLaren (6) pointed out that fading is more severe in cellulose in overcast conditions than in sunshine, confirming the fact that high humidities lead to higher rates of fading. Eaton, Gordon, and Giles (8) found that fading is directly proportional to dye concentration and attributed this fact to aggregation of the dye within the fiber.

The nature of the substrate and its effect on dye fading was pointed out by Cummings, Giles, and McEarhan (9). They found that fading was caused by reduction on proteins and oxidation on non-proteins. Giles (10) stated that the lightfastness of hydrophobic fibers will increase as their standard regain increases.

Baxter, Giles, McKee, and Macauley (11) investigated the physical state of dyes in relation to their lightfastness and concluded that, in general, fading is reduced as dye aggregates increase in size and number.

Asquith and Peters (12) found a correlation between the fading of nitrodiphenylamine disperse dyes and their structure and ultraviolet absorption spectra. This relation was established on the basis of the contributions made to their structure by various charge-transfer forms. Fourness (13), 12 years earlier, found what appeared to be no definite relation between the chemical structure of disperse dyes and their fastness to light. He did indicate, however, that substituent groups, on anthraquinone disperse dyes, had effects caused both by their nature and position.

Gas Fading

Salvin, Paist, and Myles (14) have reviewed and published results of theoretical and practical studies of gasfading of dyes through 1951. Included are a number of significant statements. For example, the substrate largely determines the gas fading character of dyes. Dyes on nylon and viscose are faded to a lesser extent than the same dyes on acetate and polyester. The reason given was that nitrogen dioxide is highly soluble in the latter fibers and thus are free to diffuse through those fibers and attack the dye. It was found that in nylon, the gas is held by salt formation or nitrosation of the amide links.

Fourness (15) found that burnt gas fumes do not normally cause fading of anthraquinone disperse violets and blues on nylon, though the reason at the time was not understood.

Fastness of Dye Mixtures

Though there is considerable literature on dye mixtures in relation to dyeing behavior, few have mentioned their behavior in relation to lightfastness and gasfastness.

For example, Fourness (16) reports a positive synergistic effect in dyeing with two anthraquinone dyes on secondary acetate (the two dyes produced a darker shade in admixture than could be attained with either alone). However, he does not mention the effect of fading on the same dyes in mixture.

Cady (17) reported certain anomalies in light fastness tests in which certain yellow dyes were improved by mixing them with certain blue dyes. On the other extreme, he reported that some blues which were lightfast alone, degraded rapidly in combination with one another.

Morton (18) briefly refers to mixtures of two dyes (class not reported) and concludes that, in general, fastness for mixtures of dyes increase over prolonged periods of time.

Burgess (19) has published results of a most significant study on the fading of dye mixtures. He pointed out the difficulty of determining fading, due to simultaneous loss of depth and change in hue. The green shades produced by blues and yellows have provided the worst instance of this difficulty. As a general conclusion, however, green mixtures result in shades less fast than that of its constituent dyes. (The weight given to hue and depth changes was not reported.) He also reports that in binary systems, a dye's fastness is rarely worse than the least fast of the components, though it is commonly less fast than the mean of the components.

Evaluation of Lightfastness

Barker, Hirst, and Lambert (20) were the first to publish results showing the relation between exposure time and per cent loss of color. They expressed the results in the general form:

$$T = aL^n ,$$

where T = time of exposure,

L = per cent loss of dominant color,

a = constant characteristic of each dyestuff, and

n = constant of value about 1.9 or 2.0.

Mathematically, the calculated curves were not in good agreement with the initial portions of the experimental curves but fit reasonably

well over long periods of exposure.

Cunliffe and Lambert (21) refined the above equation to a relation between time of exposure and concentration to the general form:

$$F_t = F_{\infty}(1 - e^{-kt}) ,$$

where F_t = concentration at time t , and

k = constant which varies with the dye used.

This equation has the general form characteristic of photodegradation (22).

Formulae for Evaluation of Data. Judd and Wyzsceki (23) review the terms to be defined in the measurement of chromaticness. Based on MacAdams' original work, Chickering (24) has published the most recent and most optimized mathematical relationships for measuring color difference comparing lightness, hue, and saturation. (See Appendix.)

To evaluate the color difference of the carpet samples as compared with the unfaded state, use was made of the "MacAdam-Modified 1965 Frielle Color-Difference Formula" (25). In 1961, Frielle (26) introduced a color difference metric which was later optimized and modified by MacAdam. Chickering (27), in 1967, optimized the new Frielle-MacAdam formula to fit the well-known Nutting data. This most recent formula has been selected by the CIE Committee on Colorimetry to replace the earlier MacAdam PQS color-difference formulae (28). The Frielle-MacAdam-Chickering metric as modified by Chickering adjusted the relative weighting of chromaticity and lightness differences and adjusted the size of color difference units so as to be a function of lightness level. The

metric of these color differences is in MacAdam units such that a value of one, i.e., one PQS unit, is that difference in color that is just perceivable to the eye of the average observer and a value of two units represents twice this amount. (See Appendix for formulae and computer programs used to make calculations.)

To evaluate concentration changes, use was made of spectrophotometric color matching with the least squares technique (29). This method allows one to back-calculate to the concentrations of each dye and its fading postcursor needed to match the color of the mix. This value was then divided by the exhaustion (computed from solution measurements) of the single dyes used in the mix to obtain the amount of dye actually in a sample.

This procedure was accomplished by converting reflectances (R) to K/S where K is the absorption coefficient and S is the scattering coefficient, according to Kulbulka Munk's Law (30). Accordingly,

$$K/S = \frac{(1 - R)^2}{2R} .$$

R is the fractional reflectance at a given wavelength. The total K/S for a combination of n colorants ($i = 1, 2, 3, \dots, n$) used in a match is given by (31):

$$(K/S)_{\text{Match}} = \sum_{i=1}^n \frac{(K/S)_i^0}{c_i^0} c_i \quad (1)$$

where C_i = the concentrations of the colorant i ,
 $(K/S)_i^0$ = the K/S for dye number i alone at known concentration
 C_i after subtraction of the K/S of the substrate, and
 $(K/S)_{Match}$ = the K/S of the match after subtraction of the K/S of
the substrate.

See Appendix for long form of Equation (1). The above formula holds for the following assumptions:

- (a) that K/S values are directly proportional to colorant concentrations,
- (b) that each dye in a mix acts independently of the others,
- (c) that the substrate is responsible for the majority of the light scattering expressed in the scattering coefficient S , and
- (d) the sample is non-fluorescent.

To obtain a spectrophotometric match, the above equation (1) must be satisfied at all wavelengths, in this case, 16 wavelengths ($\lambda = 1, 2, 3, \dots, 16$) between and including 400μ and 700μ in intervals of 20μ . This is actually obtained by minimizing the sum of the squares of the difference between the computed match and the measured standard (corrected for substrate) as expressed in the following equation:

$$\Delta = \text{Min} \sum_{\lambda=1}^{16} \left[(K/S)_{\text{Standard}} - (K/S)_{\text{Match}} \right]^2 .$$

This minimization is obtained by setting partial derivatives of C_i equal to zero which gives a reduced form of simultaneous linear equations (See Appendix).

CHAPTER II

INSTRUMENTATION AND CHEMICALS

Dyeing Machine

Fiber specimens were dyed in an open-pot dyeing machine with a 10 pot (600 ml. per pot) capacity. The machine was built by the Edwin L. Wiegand Company of Pittsburgh, Pennsylvania. It was equipped with steam and electric heat. Temperature was controlled by a Chromalox thermostat between 50° and 250°F.

Beckman DB-G Grating Spectrophotometer

The Beckman DB-G works on the double beam principle and is equipped with a tungsten lamp for transmittance measurements in the visible region. It was manufactured by Beckman Instruments, Incorporated of Fullerton, California. One centimeter silica cells were used for all measurements. (See Appendix for calibration procedure.)

Beckman IR 10 Infrared Spectrophotometer

The Beckman IR 10 also built by Beckman Instruments, is a double beam grating instrument operating in the wavelength range of 250-4000 μ . (See Appendix for calibration procedure.)

Color-Eye

All reflectance measurements were taken from a large sphere Color-Eye, Signature Model, built by Instrument Development Laboratories. It is designed for the measurement of highly directionally-surfaced

materials with the sample and standard being diffusely illuminated within an 18-inch diameter integrating sphere and viewed 8° from normal. (See Appendix for calibration procedure.)

Atlas Fade-ometer

The Model 18F compact fade-ometer was built by the Atlas Electronic Devices Company of Chicago, Illinois. It is an enclosed carbon-arc machine equipped with humidity and temperature control devices. Black panel temperatures may be controlled between 130° and 190°F. The humidifier uses an atomizer unit to control relative humidity between 20 and 60 per cent. (See Appendix for calibration procedures.)

Despatch Oven

The Despatch Oven, style LTC2-12, was built by the Despatch Oven Company of Minneapolis, Minnesota. The oven is equipped with a thermostat to control temperature between 100° and 450°F. It is equipped with recirculating and exhaust fans which may be operated independently. This instrument was modified for this research as described in the Gas Fading procedures and as shown in the schematic diagram. (See Appendix.)

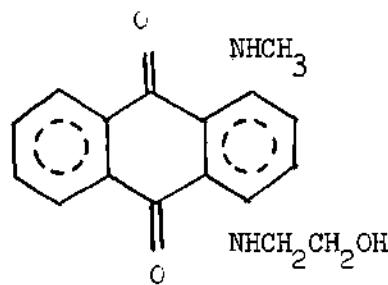
Nylon 66

Nylon 66 (see Appendix for IR characterization) was chosen for this research since it still represents the greatest percentage (by fiber type) of the carpets produced today. The particular fiber used was a nylon 66 type, 1300 denier/68 filaments multifilament, regular dye yarn. The yarn was tufted into Tapar (DuPont) polypropylene primary backing.

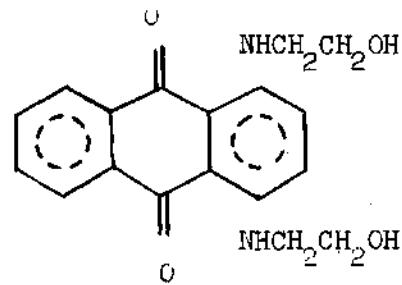
Disperse Dyes

Four disperse dyes were used in single, binary, and trinary combinations. The dyes used were: C.I. Disperse Blue 3 (I.C.I.), C.I. Disperse Blue 7 (DuPont), C.I. Disperse Yellow 3 (DuPont), and Latyl Cerise Y (DuPont).

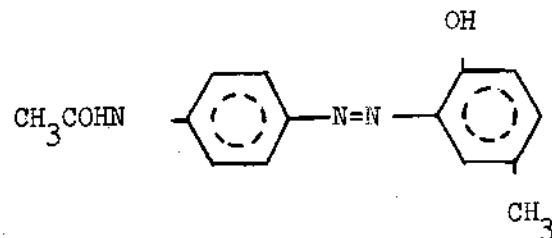
Table 1. Dye Structures



C.I. Disperse Blue 3



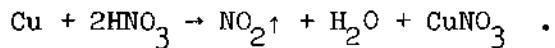
C.I. Disperse Blue 7



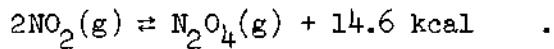
C.I. Disperse Yellow 3

Nitrogen Dioxide

The NO₂ used in this research was made by reaction of granular copper (Mallinckrodt) with nitric acid (Technical). The equation for the reaction is as follows:



The nitrogen in the gas formed, exists half as NO₂ and half as N₂O₄ in the following equilibrium:



at 60°C and 1 atm. pressure which was also the condition under which the gas fading experiments were carried out. Higher temperatures favor the formation of more NO₂. The NO₂-N₂O₄ mixture exists as a brown poisonous gas and is a strong oxidizing agent. The mixture dissolves in water to form HNO₃ and NO. Therefore, part of the nitric acid used up in the formation of the gas is regenerated. (See Appendix for calculation of equilibrium concentration for gas fading.)

CHAPTER III

PROCEDURES

Dyeing

Stock dye solutions were made to a strength of one gram per liter by dissolving 0.500 grams of dye in 10 ml. of 25 per cent acetone/75 per cent water. Once dissolved, additional acetone/water solution was added to bring the total volume to 500 milliliters.

Calibration curves were then prepared by recording the absorption spectra of each dye on the Beckman DB-G Grating Spectrophotometer at four different concentrations, i.e., 0.005 g/l., 0.01 g/l., 0.02 g/l., and 0.04 g/l. The absorbance at the highest peak of each curve was then plotted against concentration to establish a curve (see Appendix).

Ten dye baths were then prepared for an 0.13 per cent owf (on weight of fiber) using 6 ml. total dye solution for each bath. Dyeings consisted of 5-binary solutions and 2-trinary solutions. Table 2 contains the various mixtures used to dye samples. All mixtures contained equivalent amounts of each dye.

Carpet samples were cut in squares measuring approximately 3" x 3" and weighing exactly 3 grams each. This size and weight was found most convenient for color measurement on the Color-Eye and for dyeing in a 600 ml. container. The actual fiber weight disregarding the polypropylene backing was 77 per cent of the total weight of each sample. The backing absorbed a negligible amount of dye and could be disregarded in exhaustion calculations. Two only 3 gram carpet samples

Table 2. Dye Mixtures

-
1. Yellow 3 and Red 55
 2. Blue 7 and Red 55
 3. Blue 7 and Yellow 3
 4. Blue 3 and Red 55
 5. Blue 3 and Yellow 3
 6. Blue 7, Yellow 3, and Red 55
 7. Blue 3, Yellow 3, and Red 55
-

previously wetted out were dyed in each pot.

Temperature rise was controlled at 2°C per minute. Dyeing was carried out at 90°C for one hour. Samples were stirred repeatedly and kept submerged for the complete dye cycle. They were then drained and rinsed in warm water before being dried at 105°C. They were then allowed to condition to normal regain before light exposure in the fadeometer began.

The above sequence was repeated for 0.67 per cent and 2.0 per cent dyeings of mixtures. It was then repeated for the single dye mixtures at the three given concentrations.

Measurement of Dye Exhaustion. Dye exhaustion was measured on the single dyeings by measurement of the absorbance of each exhausted dyebath at the wavelength of peak absorbance. These wavelengths were 357 μ , 510 μ , 620 μ , and 670 μ which correspond to yellow 3, red 55,

blue 3, and blue 7, respectively. Dyebaths were refilled to their initial volumes to compensate for loss of water due to evaporation, before any of the exhausted bath was removed. The carpet samples were drained of excess liquor over the pot before rinsing and drying. Those exhausted solutions that registered absorption readings below 0.10 were concentrated by a factor of 4 or from 400 ml. to 100 ml. and remeasured.

The formulas used in the calculation of exhaustion were as follows:

$$C = KA$$

where C = the concentration in grams/liter,

K = slope of calibration on curve for each dye,

A = absorbance at peak wavelength, and

$$\%E = \frac{C_I - C_E}{C_I} \times 100 ,$$

where %E = per cent exhaustion,

C_I = initial concentration of dye bath in grams/liter, and

C_E = equilibrium concentration of dye bath in grams/liter.

Table 3. Exhaustion of Dyes Used Alone

Dye	%E @ 0.13% owf	%E @ 0.67% owf	%E @ 2.0% owf
Blue 3	83	85	68
Red 55	83	85	83
Yellow 3	83	85	83
Blue 7	87	82	78

Light Fading

The fade-ometer was calibrated according to the standard test method of the AATCC (33) (see Appendix). Each dyed carpet sample was trimmed to a width of 2.75 inches. Two samples were placed in a standard size sample holder with a face opening of 1.75 inches by 5 inches by 5 inches. The pile of the samples was opposite the face opening of the holder. The holders were hung in the fade-ometer with the back side of the specimen holder facing the globe in order to expose the complete width of the specimen. Rubber bands were utilized to support the samples in the specimen holder. The bands were replaced every eight hours to prevent their breakage due to loss of strength caused by the carbon-arc illumination.

Back panel temperature was controlled at $145^{\circ} \pm 5^{\circ}$ F. The relative humidity of the air inside the machine was controlled at 30 ± 5 per cent. Fading intervals were 0.5, 1, 2, 4, 8, 16, and 32 hours with at least an 8 hour dark period between each fade interval. The humidity of the

atmosphere around the samples during the dark periods was held at 30 per cent with the use of a desiccator charged with a 52 per cent solution of sulfuric acid.

An undyed carpet sample was exposed with the dyed samples. All samples were run in the fade-ometer at the same time, for identical periods, and subjected to the same conditions during dark periods.

Gas Fading

The carpet samples used in this experiment were exposed to nitrogen dioxide which was generated outside of the oven in a 1000 ml. flask stoppered to allow air to be pulled in through the top of the flask, over the reactants and into the oven with hoses through a side port in the oven.

To bring the concentration of the nitrogen dioxide inside the oven to equilibrium concentration, 0.77 grams of copper was added to 25 ml. of N/2 nitric acid in a 50 ml. Erlenmeyer flask and allowed to react inside the oven without circulation. Five minutes were allowed for the reaction after which the recirculating fan was turned on and allowed to run for five minutes. The 50 ml. flask was then removed via a small door with the recirculating fan off to prevent disturbing the established equilibrium.

The nitrogen dioxide generator was made by adding 80 ml. of nitric acid (technical) to 170 ml. of water. The intake hole of the flask stopper contained a 7 inch length of 1/4 inch glass tubing. A straight length of copper tubing of 1/8 inch outside diameter and 1/16 inch inside diameter was entered into the glass tubing of the intake

hole of the stopper and the end allowed to rest on the bottom of the flask. In this manner, as the copper reacted with the HNO_3 solution, the weight of the copper in the glass tubing formed a gravity feed so that a constant length of the copper was exposed to the acid at all times. The copper tubing was ground to point with the length of the slope of the point equal to the depth of the nitric acid solution in the flask. This allowed a constant weight of copper to react per unit time.

The carpet samples were entered in the oven at 140°F for 4 hours to allow them to reach equilibrium desorption without gasfading. The reflectance of the samples was then measured on the Color-Eye to obtain the reflectances of the unfaded samples. When not being measured or exposed, the samples were kept in a desiccator charged with a 61.0 per cent sulfuric acid solution to maintain the relative humidity at 15 per cent. The relative humidity inside the Despatch Oven at 140°F was observed to be 14-17 per cent.

After each fading interval the samples were kept in a desiccator for at least 8 hours before measurement.

Color Measurement

The Color-Eye was calibrated according to the manufacturer's specifications as described in the Appendix.

A glass plate coated with a layer of barium sulphate was first used as a standard and the reflectance at 16 wavelengths plus x, X, Y, and Z values of a standard vitrolite plate was measured. This standard reference material was then used as a reference and the carpet samples and standard fading strips were measured relative to it.

Reflectance measurements and tristimulus filter values were taken at the end of each fading interval and dark period and recorded to nearest hundredth of a unit. Correction from the vitrolite to a theoretically absolute white was made mathematically in both the color difference and least squares matching computer programs.

Measurement of Concentration Changes

Concentration changes were measured by fading constants calculated for the unfaded compound, with the smallest concentration value for the unfaded compound equal to zero and its largest value equal to one after normalization.

The sequence of operations performed by the fading rate program are as follows; all reflectance values are converted to K/S and corrected for vitrolite and substrate. The K/S values at each time are then plotted and transformed for a curve fit to

$$F_t = F_{\infty}(1 - e^{-kt}) .$$

An iterative technique was used to obtain F_{∞} , i.e., the program calls for the value of K/S at the longest fading time for its first estimate of the extrapolated value, and then uses this value to compute the next value until the last value selected is within 0.1 per cent of the previous value.

Once the K/S of the faded compound is computed at 16 wavelengths for each dye, these values then form the spectrum of the faded dye at F_{∞} . A matrix is then set up for a least squares color match, first with the single dyes and then with the dye mixtures. For the single

dyes the least squares match computes the match at each time interval using the dye alone and the faded compound at F_{∞} as the second dye. Mixtures of three dyes would actually involve 3 actual dyes, and 3 pseudo dyes in the form of a faded compound for each dye at infinity.

The program is also adjusted at this point to compensate for any zeros in the matrix by reducing the matrix one row and one column for each zero that occurs in the matrix. The NxN system of equations is then solved for the concentration of each component needed to match the recorded color at each time interval.

The answers obtained for the matches are then normalized such that the answers lie between and include zero and one. Concentration is then plotted against time and the resulting curves fitted to the general form

$$C_t = C_0 (1 - e^{-kt}) .$$

However, since $C_0 = 1$ by normalization, an iterative technique is not required. The curve is reduced to a linear function by taking the natural log of the concentration at each time t. This function is then fitted by least squares. The slopes of these final curves are then taken as a synergistic constant for the rate of fading for each dye alone and for each dye in a mixture.

CHAPTER IV

DISCUSSION OF RESULTS

PQS Color Difference

The results of the PQS color differences revealed both positive and negative synergistic effects though most mixtures changed to a difference within the range of the component dye changes.

Carbon Arc Exposure

Samples subjected to a carbon arc revealed conclusive evidence of the existence of positive synergism, i.e., some dye mixtures changed less than any of the component dyes of the mixture. (See Table 4 for comparison of PQS values after 32 hours.)

C.I. Disperse Blue 7, the least fast of the four dyes used, was not in any mixture that exhibited positive synergism. The single dyes may be listed in the following order of fastness:

Yellow 3 > Red 55 > Blue 3 > Blue 7 .

Gas Exposure

Samples subjected to gasfading were either within the range of the changes in the components or exhibited negative synergism, i.e., they changed more than either component alone. (See Table 5 for comparison of PQS values after 8 hours.)

Table 4. Comparison of Dye Mixtures and Components
as Measured by PQS Color Difference

Carbon Arc Exposure

Conc.	Mixture	Mixture Change	Component Dye Changes			
			B ₃	Y ₃	R ₅₅	B ₇
1	Y ₃ -R ₅₅	5.37		4.40	6.64	
2		1.50		1.50	3.22	
3		.28(+)		.93	2.63	
1	B ₇ -R ₅₅	8.15			6.64	10.89
2		7.94			3.22	7.97
3		3.79			2.63	5.18
1	B ₇ -Y ₃	6.95		4.40		10.89
2		4.19		1.50		7.97
3		3.30		.93		5.18
1	B ₃ -R ₅₅	5.11(+)		7.92		6.64
2		4.04		5.23		3.22
3		4.45		4.22		2.63
1	B ₃ -Y ₃	3.77(+)		7.92	4.40	
2		2.76		5.23	1.50	
3		1.76		4.22	.93	
1	B ₇ -Y ₃ -R ₅₅	6.34		4.40	6.64	10.89
2		3.64		1.50	3.22	7.97
3		1.72		.93	2.63	5.18
1	B ₃ -Y ₃ -R ₅₅	3.65(+)		7.92	4.40	6.64
2		2.33(+)		5.23	1.50	3.22
3		2.43(+)		4.22	.93	2.63

(+) = +Synergism, i.e., the mixture changes less than either component dye alone.

Table 5. Comparison of Dye Mixtures and Components
as Measured by PQS Color Difference

Gas Exposure

Conc.	Mixture	Mixture Change	Component Dye Changes			
			B ₃	Y ₃	R ₅₅	B ₇
1	YR	2.19		2.11	8.73	
2		3.08		.85	4.64	
3		2.11		.73	4.32	
1	B ₇ R	9.12			8.73	12.36
2		12.51(-)			4.64	11.12
3		7.21			4.32	7.50
1	B ₇ Y	16.09(-)		2.11		12.36
2		14.43(-)		.85		11.12
3		10.33(-)		.73		7.50
1	B ₃ R	20.20(-)	14.25		8.73	
2		10.56(-)	9.85		4.64	
3		10.21(-)	6.69		4.32	
1	B ₃ Y	12.02	14.25	2.11		
2		11.25(-)	9.85	.85		
3		8.01(-)	6.69	.73		
1	B ₇ YR	11.85		2.11	8.73	12.36
2		14.93(-)		.85	4.64	11.12
3		9.73(-)		.73	4.32	7.50
1	B ₃ YR	11.17	14.25	2.11	8.73	
2		10.60	9.85	.85	4.64	
3		10.19(-)	6.69	.73	4.32	

(-) = -Synergism, i.e., the mixture changes more than either component dye alone.

Concentration Changes

Concentration changes of dyes faded by light and as measured by synergistic fastness constants revealed that changes in color of mixtures are due both to hue and concentration changes.

Generally, dyes in mixtures faded at a higher rate than alone with C.I. Disperse Yellow 3 having the lowest average rate of fading and incidences of fading, and C.I. Disperse Red 55 having the highest average rate of fading and incidences of concentration change.

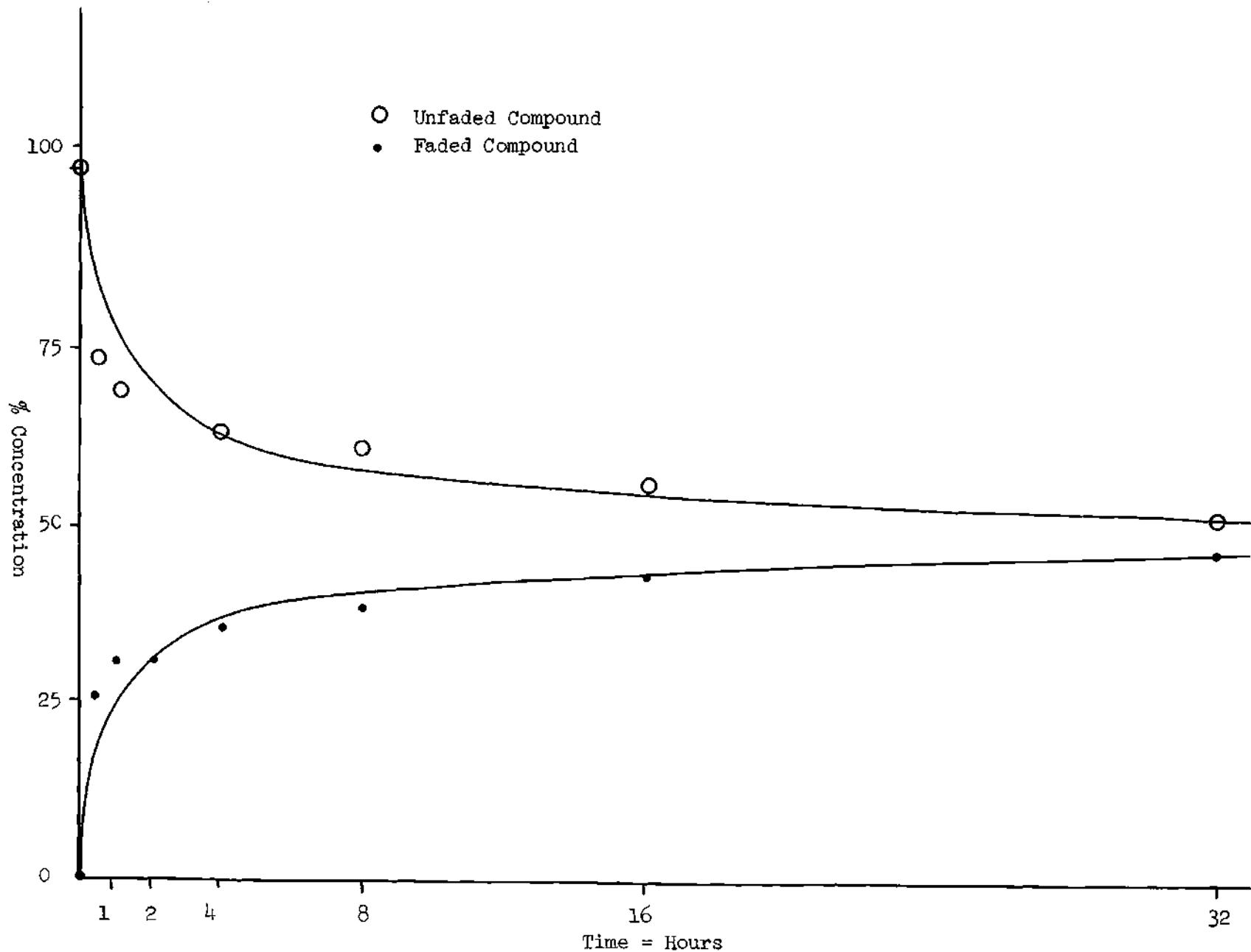
Dyes subjected to gasfading revealed almost no color changes due to changes in concentration. Those dyes which did have measurable changes had values so small as to be insignificant.

Table 6. Comparison of Dye Mixtures and Components
as Measured with Synergistic Constants

Carbon Arc Exposure

Mix	Concentration = .13%				Concentration = .67%				Concentration = 2.0%			
	B ₃	Y	R	B ₇	B ₃	Y	R	B ₇	B ₃	Y	R	B ₇
1				0				0				0
2				.45				0				0
3			.13				A					0
4	0						.57				0	
5	0	0	∞		.23	A	∞		0	∞	∞	
6	0	0	∞	∞		0	∞	∞	0	0	0	∞
7	0	0			.16	A			.21	0		
8	0		∞		.17		∞		A		∞	
9	0			∞		A		0	0		0	
10			.06	A			A	0		0	0	
11	0		∞			0	∞		0	∞		

- (A) indicates an anomalous value.
 (∞) indicates that sample changed to its final concentration within the first time period.
 (0) indicates no change in concentration took place.
 All other values are synergistic constants such that the higher the constant the higher the rate of fading.



CHAPTER V

CONCLUSIONS

This research establishes the existence of synergistic fastness effects, i.e., it is possible for dyes in a mixture to have greater lightfastness than the same dyes alone. However, when the same dyes are used under varying conditions, such as when exposed to gas fumes, the effect can be reversed such that the mixture is less fast than the single components.

This anomaly may be explained by considering the masking effect of dyes in mixture. It is possible that when various dye molecules aggregate; apart from this condition causing increased fastness, the molecules on the outer portion of the dye particle may absorb the light energy before it can reach the inner molecules.

Since nitrogen dioxide may diffuse through the fiber, the inner molecules of the aggregate cannot be protected from the effects of the gas and this could explain the more radical effect on the gas-faded samples.

Finally, dyes tend to fade more rapidly in a mixture when exposed to light though the combined hue and lightness change may not be significant. However, when exposed to gas fumes, dyes change color predominantly by a hue change mechanism rather than by one of concentration.

The numerous anomalous results shown as synergistic constants points out the difficulty of measuring such changes spectrophotometrically and colorimetrically. Such results arise from the researcher's

inability to handle certain characteristics revealed by the raw data such as a dye first increasing in concentration on initial exposure to a carbon arc and then decreasing in concentration over longer exposure periods.

CHAPTER VI

RECOMMENDATIONS

In further studies of this subject, dyes should be exposed over much longer periods of time to insure better consistency of results, to allow for more contradictory data, and to determine if the relationship between hue changes and concentration changes remain constant for prolonged periods. In addition, a broader spectrum of dyes should be investigated and measured at shorter intervals of exposure time to adequately define when changes begin to occur.

Research with dye solutions alone should be studied and compared with these results to verify the significance of the masking theory. The effects of different dyed substrates and their reaction to gas fading could reveal masking caused by the substrate if the gas could not diffuse through the fiber.

APPENDIX

Equipment Calibration Procedures

Beckman DB-G Grating Spectrophotometer. The Beckman spectrophotometer was calibrated according to the manufacturer's instructions (Beckman Instructions 566-F).

The reference standard consisted of a 25 per cent acetone/75 per cent water solution. An opaque block was placed in the sample beam and the meter needle adjusted to zero on the per cent transmittance scale. The slit program selector was set at one and the slit opening adjusted manually to 0.0133 inches. The opaque block was removed and replaced with a sample silica cell containing the above reference standard solution and the recorder set at 100 per cent T on the chart paper. The sample cell was then replaced with the exhausted dye solutions and the required charts recorded. Only the visible range between 400 and 700 μ was examined.

IR 10 Infrared Spectrophotometer. The nylon yarn used in this research was characterized by making a thin film of the fibers and measuring its infrared spectra. The per cent T scale was set at zero by blocking the sample beam with opaque cardboard and adjusting the pen to 0 per cent T. The machine was then set to scan the entire spectra (250-4000 μ) to insure that the 0 per cent T would hold. This was repeated with the sample beam unblocked and the pen set at 100 per cent T. A thin film of polystyrene was then placed in the sample beam and scanned to check the wavelengths of the chart paper against the known peak wavelengths of polystyrene. With the chart calibrated, the nylon film was then scanned.

Atlas Fade-ometer. The fade-ometer was calibrated according to the procedures outlined in the AATCC Manual with the use of light sensitive paper and standard faded strips all furnished by the Office of Standard Reference Materials, National Bureau of Standards, Washington, D.C. Light sensitive paper was exposed under test conditions for twenty hours. By visual examination it was determined that this amount of fading was approximately equivalent to 18 Standard Fading Hours (SFH). All fade hours given in the previous data are fade-ometer hours and may be converted to SFH by multiplying by .9. During experiments, new carbon electrodes (No. 20 Cored and No. 70 Solid) were installed after each 15 hours of operation. The globe was washed and re-replaced after the same time intervals.

Color-Eye. This instrument was supplied with two vitrolite standards marked "A" and "B" with "A" as the working standard. A barium sulphate standard was also supplied.

The machine was calibrated using the manufacturer's recommended procedures as given in Instruction Manual No. 5055. The lamp voltage specified for this particular machine was 8.3 and was set accordingly. The zero spread check was made with vitrolite standards in the ports, "A" in front and "B" in back. Reflectance readings were then taken for X, Y, and Z. The standards were then reversed and the previous values measured again. The spread was calculated and corrected accordingly and true zero set. High and low sensitivities were then checked and calibrated. Light Source C was used for all measurements.

Calculation of Equilibrium Gas Concentration

The first step in the calculation of the concentration of the

$\text{NO}_2 - \text{N}_2\text{O}_4$ mixture in the system was to measure the flow of air through the oven by measuring the volume being exhausted. This was done by a liquid displacement technique in which the air being exhausted from the oven was allowed to bubble into an inverted, water-filled 1000 ml. container and the time for displacement measured to the nearest second.

The next step was the calculation of the amount of gas formed by the reacting copper tubing with the acid solution for a given amount of time, weighing the tubing and taking the difference in weight as the amount reacted per unit time. Several checks were made to obtain an average.

The molar volume of the gas was then calculated at 1 atm. and 60°F using the ideal gas equation. By using the air flow in liters/minute measured earlier and simple proportional algebra it was possible to calculate the concentration of the gas in parts per million.

The equilibrium concentration was found to be approximately 400 parts per million.

Table 7. MacAdam-Modified 1965 Friele
Color-Difference Formula

The modified formula recommended for the calculation of color differences is:

$$(\Delta E)^2 = 0.0778(\Delta L/a)^2 + (\Delta C_{r-g}/a)^2 + (\Delta C_{y-b}/b)^2$$

where

$$a^2 = 17.3 \times 10^{-6} (P^2 + Q^2) / 1 - 2.73 P^2 Q^2 / (P^4 + Q^4)$$

$$b^2 = 3.098 \times 10^{-4} (S^2 + 0.2051 Y^2)$$

$$\Delta C_{r-g} = (Q\Delta P - P\Delta Q) / (P^2 + Q^2)^{\frac{1}{2}}$$

$$\Delta L = (P\Delta P + Q\Delta Q) / (P^2 + Q^2)^{\frac{1}{2}}$$

$$\Delta C_{y-b} = S\Delta L / (P^2 + Q^2)^{\frac{1}{2}} - \Delta S$$

$$\Delta P = P_{\text{Sample}} - P_{\text{Standard}}$$

$$\Delta Q = Q_{\text{Sample}} - Q_{\text{Standard}}$$

$$\Delta S = S_{\text{Sample}} - S_{\text{Standard}}$$

and, where the values P, Q, and S of the standard unfaded sample are given by:

$$P = 0.724X + 0.382Y + 0.098Z$$

$$Q = -0.480X + 1.370Y + 0.1276Z$$

$$S = 0.686Z$$

where X, Y, and Z are CIE tristimulus values.

Table 8. Expanded Formulation for Least Squares
Color Matching

$$(K/S)_{400} = \frac{c_1}{c_1^o} (K/S)_{1,400}^o + \dots + \frac{c_N}{c_N^o} (K/S)_{N,400}^o + (K/S)_{W,400}$$

$$(K/S)_{420} = \frac{c_1}{c_1^o} (K/S)_{1,420}^o + \dots + \frac{c_N}{c_N^o} (K/S)_{N,420}^o + (K/S)_{W,420}$$

*

*

*

$$(K/S)_{700} = \frac{c_1}{c_1^o} (K/S)_{1,700}^o + \dots + \frac{c_N}{c_N^o} (K/S)_{N,700}^o + (K/S)_{W,700}$$

This formulation may be simplified by letting

$$M = \begin{bmatrix} (K/S)_{400} \\ (K/S)_{420} \\ * \\ * \\ * \\ (K/S)_{700} \end{bmatrix} \quad P = \begin{bmatrix} (K/S)_{W,400} \\ (K/S)_{W,420} \\ * \\ * \\ * \\ (K/S)_{W,700} \end{bmatrix} \quad C = \begin{bmatrix} c_1/c_1^o \\ c_2/c_2^o \\ * \\ * \\ * \\ c_N/c_N^o \end{bmatrix}$$

(Continued)

Table 8. Expanded Formulation for Least Squares
Color Matching (Concluded)

and $A =$

$$\begin{bmatrix} (K/S)_{1,400}^o & (K/S)_{2,400}^o & \dots & (K/S)_{N,400}^o \\ (K/S)_{1,420}^o & (K/S)_{2,420}^o & \dots & (K/S)_{N,420}^o \\ * & * & * & * \\ (K/S)_{1,700}^o & (K/S)_{2,700}^o & \dots & (K/S)_{N,700}^o \end{bmatrix}$$

The above may be expressed in matrix notation as

$$M = AC + P$$

The least squares technique requires that the sum (Δ) of the squares of the residuals be a minimum. In matrix notation this is equivalent to

$$\Delta = \text{Min } ((M-P) - AC)^T ((M-P) - AC)$$

Taking the partial derivative yields

$$A'AC = A'(M-P)$$

the solution of which is

$$C = (A'A)^{-1}A'(M-P) .$$

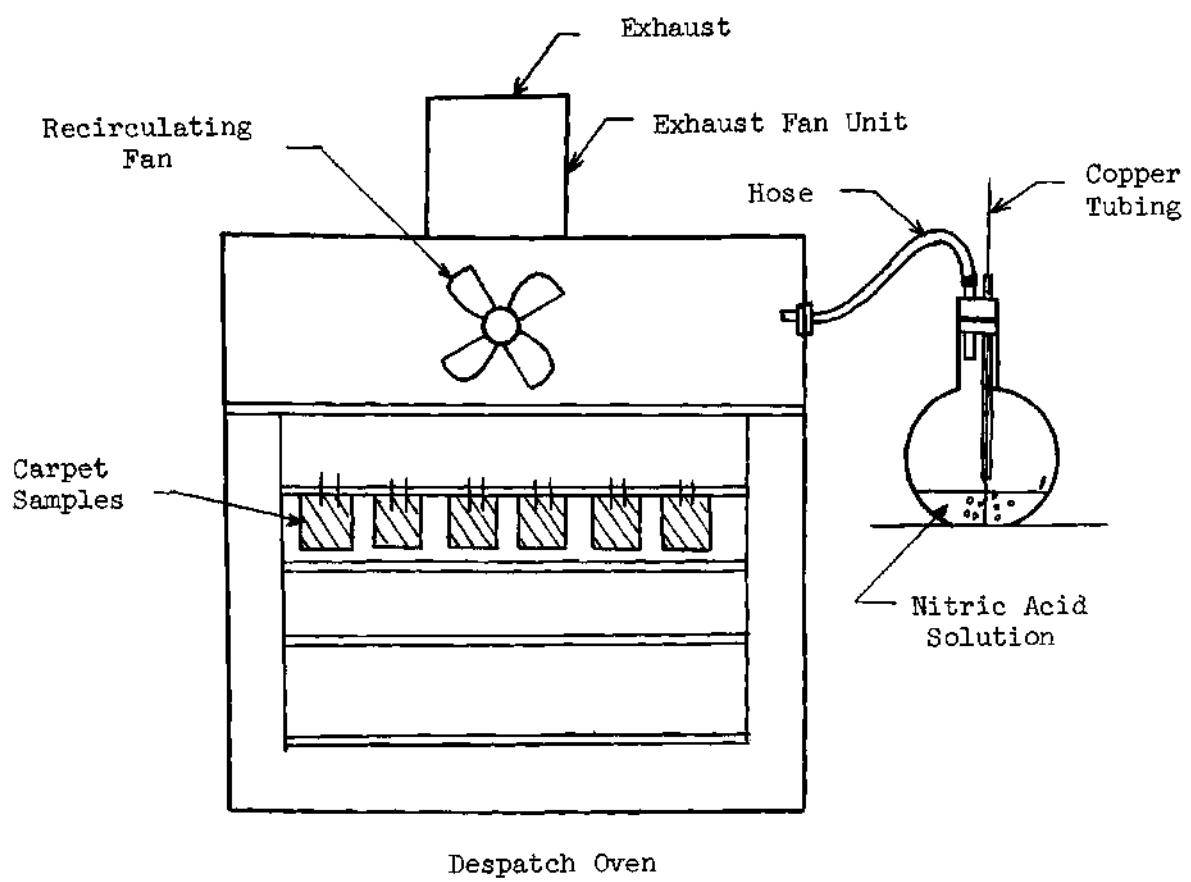


Figure 2. Schematic of Nitrogen Dioxide Generator on Despatch Oven.

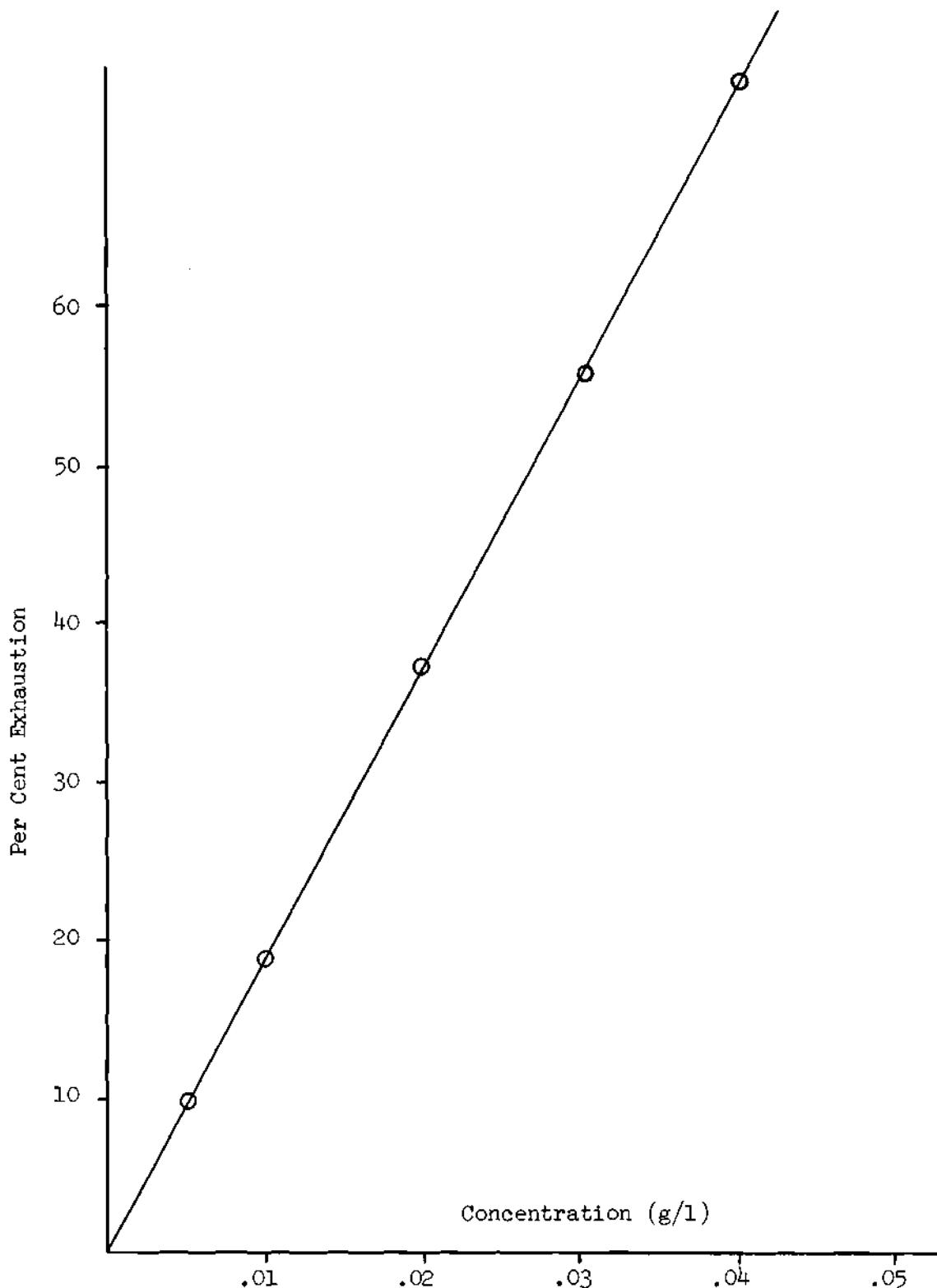


Figure 3. Calibration Curve for C.I. Disperse Blue 3.

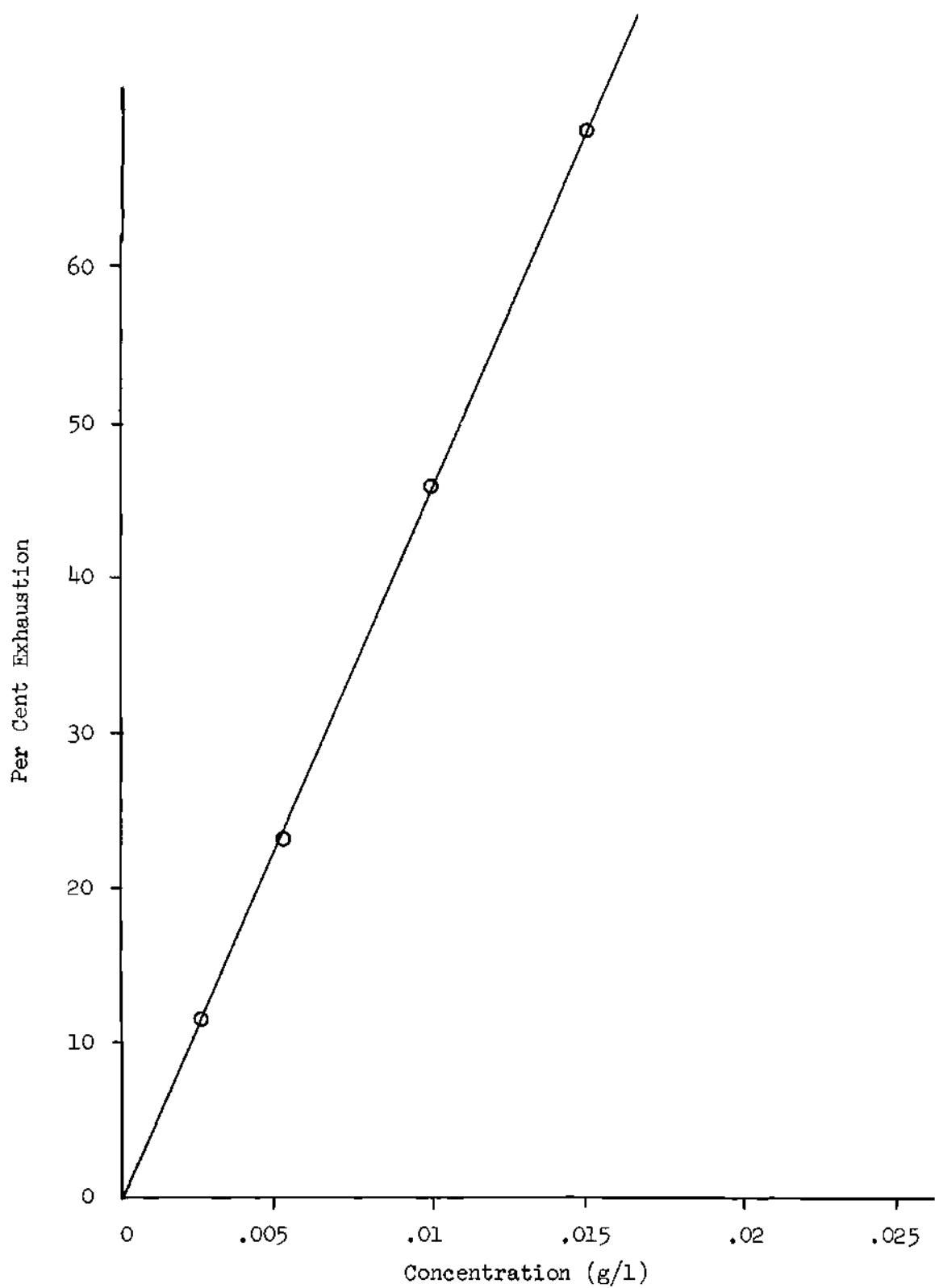


Figure 4. Calibration Curve for C.I. Disperse Yellow 3.

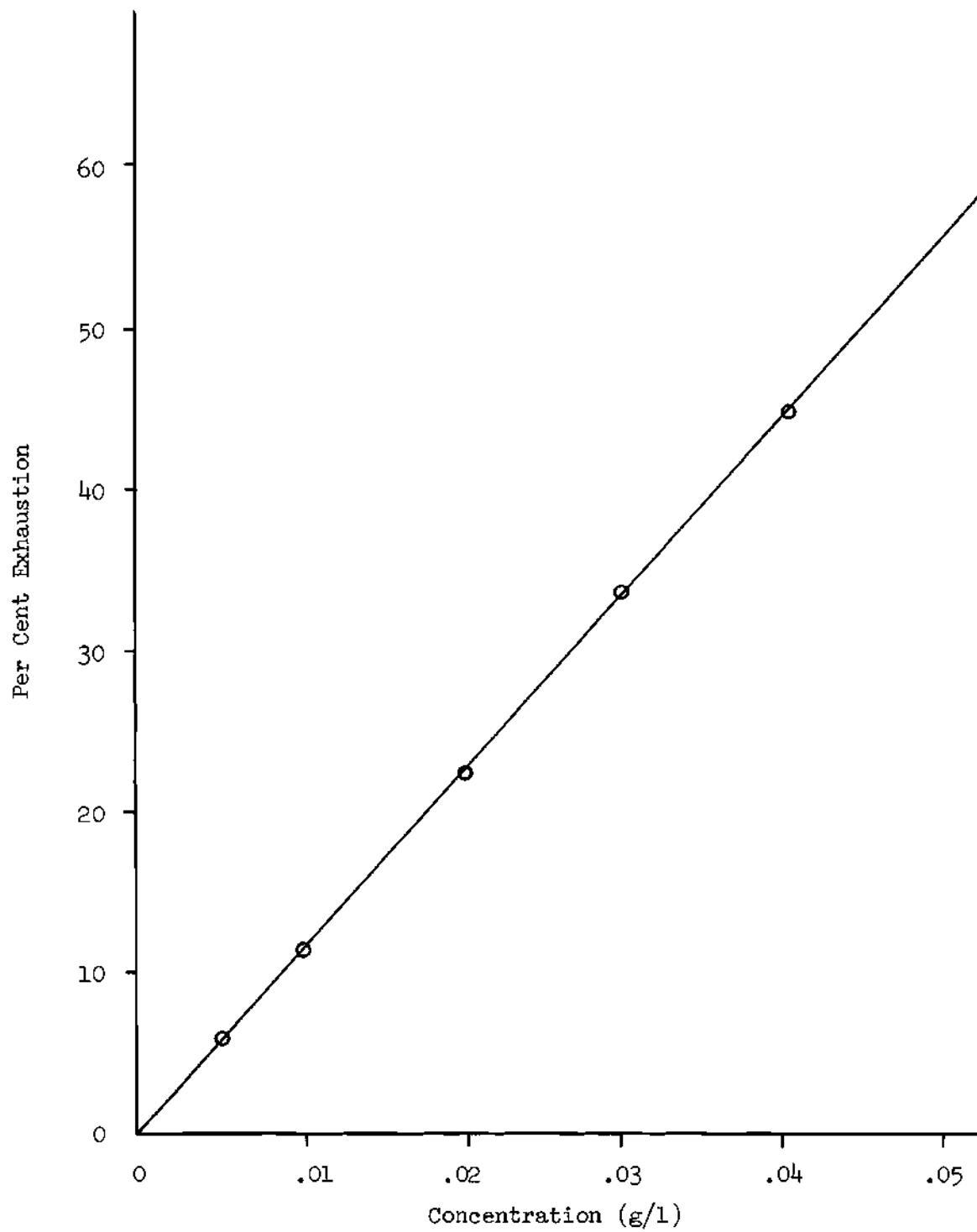


Figure 5. Calibration Curve for C.I. Disperse Red 55.

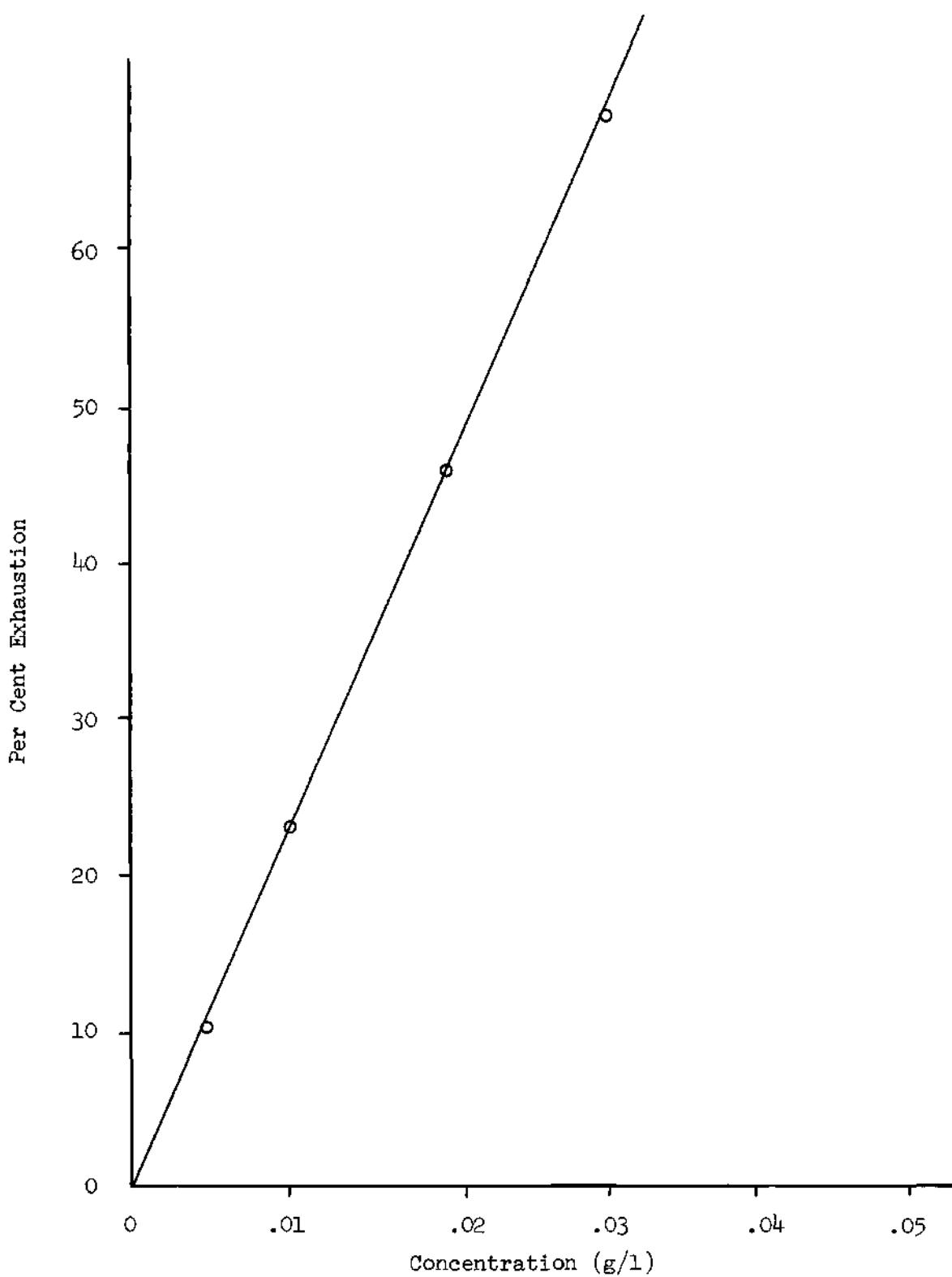


Figure 6. Calibration Curve for C.I. Disperse Blue 7.

Note: The following data are included in this thesis for the use of subsequent researchers for further consideration such as for statistical analysis or other such evaluation.

All columns are reflectances taken as a decimal rather than as a per cent (decimal is omitted to allow room for all data for a sample for one time period to fit on one data card) on the Color-Eye.

Each row represents one data card and the rows are in order as used behind the computer programs. However, the reader should be aware that minor changes would be required in the programs to switch from lightfastness data to gasfastness data.

Table 9. Dye Mixtures by Number

Mix Number (M)	Dyes in Mix (C.I. Disperse)
1	Yellow 3-Red 55
2	Blue 7-Red 55
3	Blue 7-Yellow 3
4	Blue 3-Red 55
5	Blue 3-Yellow 3
6	Blue 7-Yellow 3-Red 55
7	Blue 3-Yellow 3-Red 55
8	Blue 3
9	Yellow 3
10	Red 55
11	Blue 7

Table 10. Key to Symbols

For Lightfastness	For Gasfastness
$T_1 = 0$ Hours	$T_1 = 0$ Hours
$T_2 = .5$ Hours	$T_2 = 4$ Hours
$T_3 = 1$ Hour	$T_3 = 6$ Hours
$T_4 = 2$ Hours	$T_4 = 7$ Hours
$T_5 = 2$ Hours	
$T_6 = 8$ Hours	
$T_7 = 16$ Hours	
$T_8 = 32$ Hours	
	$C = \text{Concentration}$
	$C_1 = .13\% \text{ owf}$
	$C_2 = .67\% \text{ owf}$
	$C_3 = 2.0\% \text{ owf}$

Table 11. Computer Program for Calculation of PQS Color Differences

```

00101    1*      DIMENSION RV(4)*R(4)
00103    2*      IN=5
00104    3*      IO=6
00105    4*      333 READ(IN,100) IMC,ICC,ITC
00112    5*      WRITE(IO,103)
00114    6*      IF (IMC) ,3,2
00117    7*      2 READ(IN,104) (RV(IW),IW=1,4)
00125    8*      DO 5 IM=1,IMC
00130    9*      DO 5 IC=1,ICC
00133   10*      ITT=1
00134   11*      DO 5 IT=1,ITC
00137   12*      READ (IN,101) (R(IW),IW=1,4)
00145   13*      X=R(1)*RV(1)+R(2)*RV(2)
00146   14*      Y=R(3)*RV(3)
00147   15*      Z=R(4)*RV(4)
00150   16*      P=0.74*X+0.382*Y-0.098*Z
00151   17*      Q=-0.48*X+1.37*Y+0.1276*Z
00152   18*      S=0.685*Z
00153   19*      IF (ITT-1) 15,14,15
00156   20*      14 PS=P*P
00157   21*      QS=Q*Q
00160   22*      YS=Y*Y
00161   23*      XSTD=X
00162   24*      YSTD=Y
00163   25*      ZSTD=Z
00164   26*      PSQSS=PS+QS
00165   27*      PSQSR=SQRT(PSQSS)
00166   28*      YC=YS*Y
00167   29*      YT=YC*Y
00170   30*      A=SQRT((17.3E-6*(PSQSS))/((1.0+2.73*PS*QS)/(PS*PS+QS*QS)))
00171   31*      B=SQRT(3.09E-4*(S*S+0.2015*YS))
00172   32*      AK=.55669+.04943*Y-,82575E-3*YS+.79172E-5*YC-.30087E-7*YT
00173   33*      BK=.17546+.027556*Y-.57262E-3*YS+.6389E-5*YC-.26731E-7*YT
00174   34*      PSTD=P
00175   35*      QSTD=Q
00176   36*      SSTD=S
00177   37*      ITT=2
00200   38*      GO TO 5
00201   39*      15 DP=P-PSTD
00202   40*      DQ=Q-QSTD
00203   41*      DS=S-SSTD
00204   42*      DLS=(PSTD*DP+QSTD*DQ)/PSQSR
00205   43*      DL=BK*.279*DLS/A
00206   44*      CRGA=((QSTD*DP-PSTD*DQ)/PSQSR)/A
00207   45*      CYBA=((SSTD*DLS/PSQSR)-DS)/B
00210   46*      DC=(SQRT(CRGA*CRGA+CYBA*CYBA))*AK
00211   47*      DE=SQRT(DL*DL+DC*DC)
00212   48*      WRITE(IO,102) DL,DC,DE,IT,IC,IM
00222   49*      5 CONTINUE
00226   50*      GO TO 333
00227   51*      3 STOP
00230   52*      100 FORMAT(3I3)
00231   53*      101 FORMAT(64X,4F4.2)
00232   54*      102 FORMAT(3F6.2,3I5)
00233   55*      103 FORMAT(33H LIGHT CHROM TOTAL TIME CONC MIX5)
00234   56*      104 FORMAT(64X,2F4.4,F3.3,F5.4)
00235   57*      END

```

END OF COMPILEATION:

NO DIAGNOSTICS.

Table 12. Computer Program for Calculation of Concentration Changes

```

// JOB      0001
LOG DRIVE   CART SPEC    CART AVAIL  PHY DRIVE
 0000        0001          0001        0000
V2 M05      ACTUAL 16K  CONFIG 16K

PAGE      1
// JOB
LOG DRIVE   CART SPEC    CART AVAIL  PHY DRIVE
 0000        0001          0001        0000
V2 M05      ACTUAL 16K  CONFIG 16K
// XEQ ON
// DUP
*DELETE      CENCH
CART ID 0001    DB ADDR 5859    DB CNT  0094
// FOR
*LIST SOURCE PROGRAM
*ONE WORD INTEGERS
*I0CS(CARD,1403 PRINTER)
  DIMENSION RS(16),R(16,8,11),AZS(16,11,2),ND(3)
  DIMENSION A(7,8),AN(6),IZERT(6)
  DIMENSION ANS(3),ANN(6,8,11),RV(16)
101  FORMAT(16F4.4)
100  FORMAT(4I3)
103  FORMAT(2E15.4,3I3,F3.1)
105  FORMAT(F8.4,2I3)
  IN=2
  I0=5
2223 READ(IN,100) IMC,ICC,ITC
  IF (IMC) 6571,6570,6571
6570 STOP
6571 IC=ICC
  IC=ICC
  READ(IN,101) (RV(IW),IW=1,16)
  DO 5 IT=1,ITC
  READ(IN,101) (RS(IW),IW=1,16)
  DO 2 IW=1,16
  RS(IW)=RS(IW)/RV(IW)
  2 RS(IW)=((1.0-RS(IW))**2)/(2.0*RS(IW))
  DO 5 IM=1,IMC
  READ(IN,101) IR(IW,IT,IM),IW=1,16)
  DO 3 IW=1,16
  T=IR(IW,IT,IM)/RV(IW)
  3 RI(W,IT,IM)=(((1.0-T)**2)/(2.0*T))-RS(IW)
  5 CONTINUE
  READ(IN,100) IMMI,IMMX
  DO 8 IM=IMMI,IMMX
  DO 8 IW=1,16
  FI=R(IW,IT,IM)
  ICNT=0
  IF(R(IW,1,IM)-R(IW,IT,IM)) 9731,9731,22
9731 AZ=R(IW,1,IM)
  GO TO 21
22 CONTINUE
  ICNT=ICNT+1
  SY=0.0
  SX=0.0
  SXS=0.0
  SXY=0.0
  Q=0.0

```

```

DO 77 IT=1,ITC
TV=R(IW,IT,IM)-FI
IF (TV) 7,7,10
10 Y=FLN(TV)
X=2.0**FLOAT(IT-3)
IF (IT-1) 12,13,12
13 X=0.0
12 SY=SY+Y
SX=SX+X
SXS=SXS+(X*X)
SXY=SXY+(X*Y)
Q=Q+1.0
7 CONTINUE
77 CONTINUE
RVALA=SX*SX
RVALB=Q*SXS
RVALC=SX*SXY
RVALD=SY*SXS
RVALE=RVALB-RVALA
RVALF=RVALD-RVALC
RVAL=RVALF/RVALE
RVALG=EXPIRVAL)
RVALF=R(IW,1,IM)
AZ=RVALF-RVALG
IF(INCT-99) 99,21,21
99 CK=ABS((FI-AZ)/AZ)
IF (CK-0.001) 21,21,20
20 ICKV=0
DO 97 IT=1,ITC
TV=R(IW,IT,IM)-AZ
IF(TV) 98,98,97
98 ICKV=ICKV+1
97 CONTINUE
IF(ICKV-ITC+2) 93,93,92
92 AZ=(FI+AZ)/2.0
IF(AZ-FI-.00009) 21,21,20
93 CONTINUE
FI=AZ
GO TO 22
21 IF(AZ) 423,424,424
423 AZ=0.0
424 CONTINUE
AZS(IW,IM,1)=AZ
8 AZS(IW,IM,2)=R(IW,1,IM)
123 READ(IN,100) NDU,(ND(I)),[=1,3]
IF (NDU) 2221,2223,2221
2221 N=NDU*2
NP=N+1
NPER=N
DO 67 IT=1,ITC
ITAG=1
KKK=0
3176 DO 48 I=1,NDU
IMI=ND(I)
IIN=(I-1)
DO 48 L=1,2
II=I+L-1+IIN
A(II,NP)=0.0
DO 46 IW=1,16
46 A(II,NP)=R(IW,IT,IMI)*AZS(IW,IMI,L)+A(II,NP)
DO 48 J=1,NDU
IMJ=ND(J)
JIN=(J-1)
DO 48 K=1,2
JJ=J+K-1+JIN
A(II,JJ)=0.0
DO 48 IW=1,16
48 A(II,JJ)=AZS(IW,IMI,L)*AZS(IW,IMJ,K)+A(II,JJ)
IF(KKK) 2178,3177,2178
2178 DO 3271 K=1,KKK
II=IZERT(K)
DO 3271 KK=II,N
DO 3271 IJK=1,NP
NT=KK+L
3271 A(KK,IJK)=A(NT,IJK)
DO 3272 K=L,KKK
II=IZERT(K)
DO 3272 KK=II,N
DO 3272 IJK=1,N

```

```

NT=KK+1
3272 A(IJK,KK)=A(IJK,NT)
NPV=NP-KKK
DO 3273 IJK=1,N
3273 A(IJK,NPV)=A(IJK,NP)
N=N-KKK
NP=N+1
ITAG=0
3177 IF(N-1) 3118,777,3118
3118 LL=N-1
KK=1
II=1
GO TO 555
666 LL=2
KK=N
II=-1
M=N+1
555 L=LL
333 IF (L-KK+II) 111,999,111
111 JJ=L+II
K=KK
222 IF (K-L-II) 202,221,202
202 MM=K+II
RA=A(K,JJ)
S=A(MM,JJ)
DO 188 J=1,M
188 A(K,J)=A(K,J)/RA-A(MM,J)/S
A(K,JJ)=0.0
K=K+II
GO TO 222
221 L=L-II
GU TO 333
999 IF (KK-1) 777,666,777
777 IF(ITAG) 778,779,778
779 JSTEP=0
ISTEP=1
DO 785 K=1,N
JSTEP=JSTEP+1
IF(K-IZERT(ISTEP)) 780,8739,780
8739 IF(ISTEP-KKK) 7778,7778,780
7778 ISTEP=ISTEP+1
AN(JSTEP)=0.0
K=K-1
GO TO 785
780 AN(JSTEP)=A(JSTEP,NP)/A(JSTEP,JSTEP)
785 CONTINUE
IF(JSTEP-NPER) 1234,5678,1234
1234 AN(NPER)=0.0
5678 CONTINUE
KKK=0
DO 8372 K=1,NPER
IF (AN(K)) 8373,8373,8372
8373 KKK=KKK+1
IZERT(KKK)=K
8372 CONTINUE
IF(KKK) 6799,6799,8176
8176 DO 8793 K=1,NPER
IF (AN(K)) 8379,8793,8793
8379 NP=N+1
N=NPER
ITAG=1
GO TO 3176
8793 CONTINUE
6799 DO 7815 I=1,NDU
ANS(I)=0.0
KIN=I-1
DO 7816 J=1,2
K=I+J-1+KIN
7816 ANS(I)=AN(K)+ANS(I)
ANN(K,IT,IC)=AN(K)/ANS(I)
K=K-1
ANN(K,IT,IC)=AN(K)/ANS(I)
7815 K=K+1
GO TO 68
778 KKK=0
DO 69 I=1,NDU
ANS(I)=0.0
KIN=(I-1)
DO 821 J=1,2

```

```

      K=I+J-1+KIN
      AN(K)=A(K,NP)/A(K,K)
      IF (AN(K)) 3173,3173,821
3173 IF (AN(K)+.05) 3178,3178,3126
3126 AN(K)=0.0
      GO TO 821
3178 KKK=KKK+1
      IZERT(KKK)=K
821  ANS(I)=AN(K)+ANS(I)
      ANN(K,IT,IC)=AN(K)/ANS(I)
      K=K-1
      ANN(K,IT,IC)=AN(K)/ANS(I)
69   K=K+1
      DO 65 K=1,N
65   CONTINUE
      IF(KKK) 68,68,3176
68   ITAG=1
      N=NPER
      NP=N+1
67   CONTINUE
      DO 88 K=1,NDU
      I=K*2-1
      FI=1.0
      SY=0.0
      SX=0.0
      SXS=0.0
      SXY=0.0
      Q=30.0
      DO 711 IT=1,ITC
      WRITE(10,105) ANN(I,IT,IC),IT,IC
      TV=FI-ANN(I,IT,IC)
      IF (TV) 711,711,1011
1011 Y=FLN(TV)
      X=FLOAT(2***(IT-3))
      IF (IT-1) 1211,1311,1211
1311 X=0.0
      1211 SY=SY+Y
      SX=SX+X
      SXS=SXS+(X*X)
      SXY=SXY+(Y*Y)
      Q=Q+1.0
      711 CONTINUE
      AZ=((SY*SXS)-(SX*SXY))/((Q*SXS)-(SX*SX))
      AONE=((N*SXY)-(SX*SY))/((N*SXS)-(SX*SX))
      AZ=EXP(AZ)
      AONE=-AONE
      88 WRITE(10,103) AZ,AONE,ND(K),IC
      GO TO 123
      END

FEATURES SUPPORTED
  ONE WORD INTEGERS
  IOCS

CORE REQUIREMENTS FOR
  COMMON          0  VARIABLES    4876  PROGRAM    2210

END OF COMPILATION

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Table 13. PQS Color Differences for all Exposure Periods - Carbon Arc Exposure

LIGHT	CHROM	TOTAL	TIME	CONC	MIXS
-.04	.41	.41	2	1	1
.59	.39	.70	3	1	1
.80	1.15	1.40	4	1	1
1.36	.72	1.54	5	1	1
1.22	.83	1.48	6	1	1
.41	3.03	3.05	7	1	1
-.22	5.37	5.37	8	1	1
-.08	.06	.10	2	2	1
.23	.46	.52	3	2	1
.31	.12	.34	4	2	1
.55	.16	.58	5	2	1
.72	.28	.77	6	2	1
.25	.87	.90	7	2	1
.07	1.50	1.50	8	2	1
-.09	.11	.14	2	3	1
.06	.09	.11	3	3	1
.13	.42	.44	4	3	1
.17	.27	.32	5	3	1
.23	.39	.45	6	3	1
-.02	.63	.63	7	3	1
-.07	.27	.28	8	3	1
.22	.42	.48	2	1	2
.95	.66	1.16	3	1	2
.77	.56	.95	4	1	2
1.39	1.20	1.84	5	1	2
1.61	2.48	2.95	6	1	2
2.17	4.44	4.94	7	1	2
3.19	7.49	8.15	8	1	2
.05	.29	.30	2	2	2
.28	.83	.87	3	2	2
.37	.61	.71	4	2	2
.47	1.60	1.67	5	2	2
.57	1.89	1.97	6	2	2
.72	4.67	4.73	7	2	2
1.76	7.75	7.94	8	2	2
.31	1.10	1.14	2	3	2
.46	1.05	1.15	3	3	2
.37	.68	.78	4	3	2
.41	.63	.75	5	3	2
.29	.99	1.03	6	3	2
.36	1.82	1.85	7	3	2
.90	3.69	3.79	8	3	2
.41	.09	.42	2	1	3
.67	1.11	1.30	3	1	3
.85	.29	.90	4	1	3
1.02	1.10	1.50	5	1	3
1.19	2.35	2.64	6	1	3
.50	3.58	3.61	7	1	3
.96	6.88	6.95	8	1	3
-.06	.19	.20	2	2	3
.36	.55	.66	3	2	3
.26	.16	.30	4	2	3
.26	.80	.84	5	2	3
.17	1.45	1.46	6	2	3
-.25	2.37	2.39	7	2	3
-.37	4.18	4.19	8	2	3

-.20	.73	.76	2	3	3	3
.13	.03	.14	3	3	3	3
.00	1.03	1.03	4	3	3	3
.00	.67	.67	5	3	3	3
-.23	1.00	1.03	6	3	3	3
-.70	2.43	2.52	7	3	3	3
-.99	3.14	3.30	8	3	3	3
.22	.68	.71	2	1	4	4
.72	.41	.83	3	1	4	4
.52	1.27	1.38	4	1	4	4
1.18	.68	1.36	5	1	4	4
1.40	1.91	2.36	6	1	4	4
1.98	2.77	3.40	7	1	4	4
2.58	4.41	5.11	8	1	4	4
.29	.29	.41	2	2	4	4
.62	.29	.69	3	2	4	4
.72	1.09	1.31	4	2	4	4
1.09	.69	1.29	5	2	4	4
1.10	1.43	1.81	6	2	4	4
1.54	2.29	2.76	7	2	4	4
2.53	3.15	4.04	8	2	4	4
-.10	1.73	1.73	2	3	4	4
.29	1.58	1.60	3	3	4	4
.22	1.96	1.97	4	3	4	4
.40	2.80	2.82	5	3	4	4
.24	1.73	1.75	6	3	4	4
.38	3.46	3.48	7	3	4	4
.75	4.39	4.45	8	3	4	4
-.31	.53	.61	2	1	5	5
.36	.43	.56	3	1	5	5
.32	.60	.67	4	1	5	5
.45	.42	.62	5	1	5	5
1.02	.88	1.35	6	1	5	5
.47	1.97	2.02	7	1	5	5
1.23	3.56	3.77	8	1	5	5
-.38	.51	.64	2	2	5	5
.05	.61	.61	3	2	5	5
.15	.25	.29	4	2	5	5
.15	.84	.86	5	2	5	5
.25	1.26	1.29	6	2	5	5
-.01	2.11	2.11	7	2	5	5
.20	2.75	2.76	8	2	5	5
-.34	.13	.36	2	3	5	5
.07	.75	.75	3	3	5	5
.04	.61	.61	4	3	5	5
.15	.37	.39	5	3	5	5
-.01	.98	.98	6	3	5	5
-.19	1.06	1.07	7	3	5	5
-.15	1.75	1.76	8	3	5	5
-.14	.53	.55	2	1	6	6
.06	.90	.90	3	1	6	6
.44	.69	.82	4	1	6	6
.60	1.04	1.20	5	1	6	6
.61	1.71	1.82	6	1	6	6
.77	3.26	3.35	7	1	6	6
1.62	6.13	6.34	8	1	6	6
-.29	1.32	1.35	2	2	6	6

-.03	1.43	1.43	3	2	6
.05	.75	.75	4	2	6
.31	1.50	1.53	5	2	6
.26	1.56	1.58	6	2	6
.31	2.68	2.70	7	2	6
.63	3.59	3.64	8	2	6
.00	.28	.28	2	3	6
.15	.69	.71	3	3	6
.31	.99	1.03	4	3	6
.10	.43	.44	5	3	6
.20	.60	.63	6	3	6
.03	2.19	2.19	7	3	6
.23	1.70	1.72	8	3	6
-.44	.29	.52	2	1	7
.33	.15	.36	3	1	7
.42	.47	.63	4	1	7
.57	.18	.60	5	1	7
.95	.44	1.05	6	1	7
.51	.75	.91	7	1	7
1.64	3.26	3.65	8	1	7
-.27	.34	.43	2	2	7
.13	.40	.42	3	2	7
.25	.66	.71	4	2	7
.27	.21	.34	5	2	7
.38	.48	.61	6	2	7
.52	.37	.64	7	2	7
1.06	2.07	2.33	8	2	7
-.36	1.98	2.01	2	3	7
-.01	1.84	1.84	3	3	7
.05	2.55	2.55	4	3	7
-.22	1.68	1.69	5	3	7
-.11	2.28	2.28	6	3	7
-.06	1.92	1.92	7	3	7
.11	2.42	2.43	8	3	7
-.17	.87	.89	2	1	8
.33	.70	.77	3	1	8
.36	.91	.98	4	1	8
.47	1.31	1.39	5	1	8
.41	1.63	1.68	6	1	8
.61	4.20	4.25	7	1	8
1.71	7.73	7.92	8	1	8
.21	.44	.49	2	2	8
.49	.72	.87	3	2	8
.32	.85	.91	4	2	8
.43	1.41	1.48	5	2	8
.18	1.88	1.89	6	2	8
.25	3.63	3.63	7	2	8
.74	5.18	5.23	8	2	8
.10	.36	.37	2	3	8
.10	.80	.81	3	3	8
.17	.86	.88	4	3	8
-.02	1.59	1.59	5	3	8
-.25	1.97	1.99	6	3	8
-.27	3.56	3.57	7	3	8
-.04	4.22	4.22	8	3	8
-.36	.43	.57	2	1	9
.05	.49	.50	3	1	9

LIGHT	CHROM	TOTAL	TIME	CONC	MIX5
-.54	.35	.65	5	3	11
-1.01	1.02	1.44	6	3	11
-1.73	3.09	3.54	7	3	11
-2.13	4.72	5.18	8	3	11
64.09	.00	64.09	9	1	1
9.73	14.93	15.34	10	1	1
47.91	33.40	58.40	11	1	1
47.76	33.31	58.23	12	1	1
47.59	33.30	58.08	13	1	1
46.99	32.35	57.05	14	1	1
45.79	30.73	55.14	15	1	1
44.02	29.27	52.86	16	1	1
39.99	27.52	48.54	17	1	1
31.46	24.03	39.59	18	1	1
64.09	.00	64.09	19	1	1

Table 14. PQS Color Differences for all Exposure Periods - Gas Exposure

LIGHT	CHROM	TOTAL	TIME	CONC	MIXS
.45	1.23	1.30	2	1	1
.64	2.57	2.65	3	1	1
.99	1.95	2.19	4	1	1
.65	1.68	1.80	2	2	1
.99	2.55	2.73	3	2	1
1.26	2.81	3.08	4	2	1
.41	1.25	1.32	2	3	1
.66	1.62	1.75	3	3	1
.73	1.98	2.11	4	3	1
<u>-.61</u>	<u>5.97</u>	<u>6.00</u>	<u>2</u>	<u>1</u>	<u>2</u>
<u>-.10</u>	<u>7.21</u>	<u>7.21</u>	<u>3</u>	<u>1</u>	<u>2</u>
<u>-.41</u>	<u>9.11</u>	<u>9.12</u>	<u>4</u>	<u>1</u>	<u>2</u>
<u>-.15</u>	<u>14.05</u>	<u>14.05</u>	<u>2</u>	<u>2</u>	<u>2</u>
<u>.08</u>	<u>10.87</u>	<u>10.87</u>	<u>3</u>	<u>2</u>	<u>2</u>
<u>-.11</u>	<u>12.50</u>	<u>12.51</u>	<u>4</u>	<u>2</u>	<u>2</u>
<u>-.25</u>	<u>3.21</u>	<u>3.22</u>	<u>2</u>	<u>3</u>	<u>2</u>
<u>-.17</u>	<u>4.53</u>	<u>4.53</u>	<u>3</u>	<u>3</u>	<u>2</u>
<u>-.40</u>	<u>7.20</u>	<u>7.21</u>	<u>4</u>	<u>3</u>	<u>2</u>
<u>-1.57</u>	<u>9.56</u>	<u>9.69</u>	<u>2</u>	<u>1</u>	<u>3</u>
<u>-.1.50</u>	<u>13.19</u>	<u>13.28</u>	<u>3</u>	<u>1</u>	<u>3</u>
<u>-2.22</u>	<u>15.93</u>	<u>16.09</u>	<u>4</u>	<u>1</u>	<u>3</u>
<u>-2.37</u>	<u>8.65</u>	<u>8.97</u>	<u>2</u>	<u>2</u>	<u>3</u>
<u>-2.59</u>	<u>11.64</u>	<u>11.93</u>	<u>3</u>	<u>2</u>	<u>3</u>
<u>-3.20</u>	<u>14.07</u>	<u>14.43</u>	<u>4</u>	<u>2</u>	<u>3</u>
<u>-1.75</u>	<u>5.32</u>	<u>5.60</u>	<u>2</u>	<u>3</u>	<u>3</u>
<u>-2.48</u>	<u>8.29</u>	<u>8.65</u>	<u>3</u>	<u>3</u>	<u>3</u>
<u>-.3.10</u>	<u>9.65</u>	<u>10.33</u>	<u>4</u>	<u>3</u>	<u>3</u>
<u>2.77</u>	<u>9.33</u>	<u>9.73</u>	<u>2</u>	<u>1</u>	<u>4</u>
<u>4.76</u>	<u>15.03</u>	<u>15.77</u>	<u>3</u>	<u>1</u>	<u>4</u>
<u>5.73</u>	<u>19.37</u>	<u>20.20</u>	<u>4</u>	<u>1</u>	<u>4</u>
<u>1.15</u>	<u>5.69</u>	<u>6.00</u>	<u>2</u>	<u>2</u>	<u>4</u>
<u>1.91</u>	<u>9.11</u>	<u>9.30</u>	<u>3</u>	<u>2</u>	<u>4</u>

88.44918	07937	.21	4	2	4
.35	5.24	5.25	2	3	4
.72	9.37	9.40	3	3	4
.98	10.17	10.21	4	3	4
.58	6.85	6.88	2	1	5
1.44	9.92	10.03	3	1	5
.70	12.00	12.02	4	1	5
.17	5.32	5.32	2	2	5
.71	9.79	9.82	3	2	5
.24	11.25	11.25	4	2	5
-.16	4.06	4.07	2	3	5
-.07	6.72	6.72	3	3	5
-.38	8.00	8.01	4	3	5
.93	6.99	7.05	2	1	6
1.14	8.93	9.00	3	1	6
1.78	11.71	11.85	4	1	6
.51	8.09	8.11	2	2	6
1.05	11.29	11.34	3	2	6
1.37	14.87	14.93	4	2	6
.15	3.62	3.63	2	3	6
.10	7.49	7.50	3	3	6
-.24	9.70	9.70	4	3	6
1.80	5.51	5.79	2	1	7
2.91	8.86	9.32	3	1	7
3.30	10.67	11.17	4	1	7
1.47	5.05	5.26	2	2	7
2.22	8.23	8.53	3	2	7
2.58	10.28	10.60	4	2	7
.60	4.87	4.91	2	3	7
1.36	6.94	7.08	3	3	7
1.69	10.05	10.19	4	3	7
.28	7.09	7.10	2	1	8
.12	11.52	11.52	3	1	8
.31	14.25	14.25	4	1	8
-.52	5.45	5.47	2	2	8
-1.18	7.77	7.86	3	2	8
-1.05	9.80	9.85	4	2	8
-.70	4.02	4.08	2	3	8
-1.13	5.17	5.29	3	3	8
-1.14	6.59	6.69	4	3	8
.14	.20	.24	2	1	9
.26	.64	.88	3	1	9
.46	.57	.73	4	1	9
.62	1.07	1.24	2	2	9
.52	1.43	1.52	3	2	9
.80	.26	.85	4	2	9
.80	.76	1.10	2	3	9
.92	2.00	2.20	3	3	9
1.03	1.84	2.11	4	3	9
2.25	6.05	6.46	2	1	10
1.43	2.98	3.30	3	1	10
3.20	8.12	8.73	4	1	10
.67	3.00	3.07	2	2	10
.78	4.29	4.36	3	2	10
.92	4.55	4.64	4	2	10
.37	3.20	3.22	2	3	10
.58	4.12	4.16	3	3	10

.51	4.29	4.32	4	3	10
2.22	6.90	7.25	2	1	11
3.01	9.84	10.29	3	1	11
3.43	11.87	12.36	4	1	11
2.68	5.66	6.26	2	2	11
3.82	8.91	9.70	3	2	11
4.17	10.30	11.12	4	2	11
1.63	4.11	4.42	2	3	11
2.68	5.67	6.27	3	3	11
3.06	6.85	7.50	4	3	11
<hr/>					
LIGHT	CHROM	TOTAL	TIME	CONC	MIXS
.21	.83	.85	2	1	1
.30	1.65	1.68	3	1	1
.54	2.30	2.36	4	1	1
64.06	.00	64.06	5	1	1
45.47	47.97	66.10	6	1	1
41.78	13.27	43.84	7	1	1
45.48	31.42	55.28	8	1	1
45.82	37.14	58.99	9	1	1
64.06	.00	64.06	10	1	1

Table 15. Per Cent Reflectance of Carpet Samples for Carbon Arc Exposure

93289031905691049052896790358967895888938724878487288684867286389105896790119079
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Table 16. Per Cent Reflectance of Carpet Samples for Gas Exposure

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