

A STUDY OF THE EFFECT OF SURFACTANTS ON THE  
WASHFASTNESS OF DISPERSE-DYED FABRIC

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Approved

Chairman

Date approved by Chairman 7/26/71

DEDICATION

I dedicate this thesis to the memory of my father,

John J. Loti

## ACKNOWLEDGMENTS

I wish to particularly thank my advisor, Mr. Willard Ferguson, for his encouragement to pursue this thesis topic and for his help with the instrumentation used in this research.

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## SUMMARY

The purpose of this research was to determine the effects of various surfactants and washing conditions on the washfastness of disperse dyes applied to nylon 66. Fastness determinations were made using spectrophotometric techniques and the Friele-MacAdam-Chickering color difference formula.

Three disperse dyes were individually applied to Helanca nylon double-knit fabric. Factors under control during the wash tests were surfactant choice and concentration, time of wash cycle, and temperature. Significant differences in the washfastness qualities of individual dyes were found to vary according to surfactant choice. The standard for many years in washfastness testing, neutral soap, and a synthetic nonionic surfactant chosen for this work, Triton X-100, caused the most color change of the five surfactants tested. The addition of electrolytes as builders for the surfactant solutions caused additional color changes. The influence of surfactants and electrolytes appeared to be similar to that of additives in the dyeing process which increase the solubility of the disperse dye in the aqueous phase relative to the solubility of the dye in the fiber. The general controls over washfastness appear to be the same as in dyeing, washfastness being controlled by the partition between the fiber and aqueous phases and the diffusion of the dye through the fiber. The overall washfastness of the dyes improved with an increase in the molecular complexity of the dye and decreased with increasing wash bath temperature.

## CHAPTER I

### INTRODUCTION

#### Purpose

This research was conducted to evaluate the effects of washing conditions on the fastness properties of disperse-dyed nylon fabric.

#### History of the Problem

Determining the minimum fastness characteristics that will be acceptable to the textile consumer and the proper wet-treatment during production is a subject of concern for the manufacturer. Confusion exists in both the areas of washing conditions and evaluation of results for washfastness testing.

Lenz (1) suggests that the addition of perborate to a washfastness test alters the test results significantly with certain sensitive dyes. In research involving acid dyes on wool, Hine (2) expresses his concern over the differences of fastness and staining results between samples washed with soap and with synthetic detergents. In sharp rebuttal Skelly (3) defended the International Standards Organization (ISO) washfastness tests.

The Washing Fastness Subcommittee considers that these tests, augmented where necessary by the tests for washing in the presence of sodium hypochlorite and for sensitivity to peroxy compounds, provide adequate information on washing fastness over a very wide range of washing conditions.

Several years ago P. J. Fynn (4) defended the American Association of Textile Chemists and Colorists' position of using neutral soap rather

than synthetic detergents in washfastness testing. This decision was based on the results of testing 200 specimens for washfastness. About 10 per cent were judged as having ratings as great as one-half gray scale unit difference between the standard soap formula and five synthetic variations. A recent vote by the AATCC Colorfastness to Washing Committee (5) reverses this decision, calling for a direct substitution of synthetic detergent for neutral soap in two of their recommended washfastness tests.

In current research Professor Rene Freytag (6) of France is acting as co-ordinator for an international project to evaluate 22 washfastness test methods. His representatives are studying washfastness on 51 fabrics covering many dye classes and fibers.

There are many problems in washfastness testing, and it is doubtful that one test could ever be devised to provide the needed information of fastness properties for the innumerable end uses and fabrics. Complicating the problem is that reproducibility between laboratories is difficult. Washfastness testing can present difficulties in any of a number of operations from dyeing through washing and evaluation.

#### Statement of the Problem

In a general sense the washfastness properties of disperse dyes are proportional to molecular complexity. Predictably, dye complexity increases problems of the reverse sort by decreasing dyeing and building-up qualities (7).

In the dyeing process disperse dyes utilize a very small, but finite, solubility to slip into the narrow fiber pores and attach them-

selves with non-polar forces (8). Molecular complexity certainly has a role in this process, but other important factors have been attributed to the dyeing process. Similar to molecular complexity, these factors might be expanded to explanations of fastness.

Bird (9) conducted research which showed the importance of dispersants in the application of disperse dyes. During dyeing a very dilute but saturated solution is in equilibrium with the surface of the fibers. As long as the dye aggregates dissolve rapidly to maintain the solution concentration, the surface of the fiber will be saturated. Dispersing agents act to increase the solution of dye aggregates but reduce the exhaustion at equilibrium. The increased solubility enhances the dyeing rate for many dyes, particularly those with low solubility or high rates of sorption by the fiber. For some dyes the dispersant has a significant retarding effect resulting from the shift in equilibrium. When dispersant is added the equilibrium exhaustion drops and the aggregated dye molecules are dissolved quicker. Once the dye is dissolved and diffuses into the fiber, the solution will not be saturated. Under this condition the surface of fiber cannot be saturated, resulting in a lower diffusion rate of the dye through the fiber. In a washing system the situation is similar with the surfactants shifting equilibrium and the bath being unsaturated.

Determining the relative detergent or cleansing action of the various wash conditions is outside the purpose of this research. Such investigations are available in the literature (10). These investigations point out that the factors of temperature, surfactant type and concentration, time, additives and pH are significant with respect to the

detergent process. Within limits detergency increases with time and temperature. The critical micelle concentration acts as a limiting and optimum condition for detergency (11).

Harris (12) points out that the effect of builders can be pronounced in their effects on surface tension, critical conductivity, wetting speed, and detergency. "Builder addition to anionic agents to produce a given result can decrease the quantity of surface active agents from two to ten fold." Addition of cationic electrolytes as calcium or aluminum chloride to cationic surfactants exhibit much smaller effects. In both areas the effects appear to be valence dependent.

#### Colorfastness Measurement

The two basic methods for determining the amount of dye on a fiber are dye extraction and reflectance colorimetry (13). For many purposes dye extraction is the preferable method, but in an evaluation of fading this is doubtful. A percentage concentration change for a blue dye when compared to the same percentage concentration change of a yellow dye is likely to be much more apparent when viewed by a normal observer (14).

In the AATCC Test Method 36-1969 and other tests to characterize the fastness of textile colorants, the gray scale is widely used as the standard for evaluating color differences and fastness properties. As recommended by the ISO, the gray scale consists of several sets of neutral gray chips which the observer uses to subjectively correlate to his standard and tested specimen. Typically there are five sets of chips with the first set being a reference chip and the next four sets consisting of a reference chip paired with successively lighter gray chips.

The gray chips represent various color difference units determined by the Adam's chromatic value formula (5).

Table 1. The ISO Gray Scale

Fastness Class	Qualitative Description of Fastness	Color Difference Units by Adams' Formulation
5	negligible or no change	0.0
4	slightly changed	1.5
3	noticeably changed	3.0
2	considerably changed	6.0
1	much change	12.0

In research evaluating the color differences of chromatic chips with identical hue and chroma but different lightness, significant differences were found between the gray-scale evaluated color differences and the Adams' chromatic value formula calculated color differences. Suga (15) suggests that the gray scale is a practical answer for evaluating color differences when the original and tested samples are achromatic, but that the extent to which the evaluations can be extended to colored samples is questionable.

Many systems exist designed to determine color differences more precisely than the gray scale. The Munsell System (16) describes colors on the basis of three factors: Munsell Hue, Munsell Value, and Munsell Chroma. Associated with the system is a series of colored chips with equal apparent color differences between adjacent chips. Other systems



as the Ostwald System also have physical standards directly associated with them.

One of the most important methods for defining color, independent of physical samples, is the CIE system (Commission International de l'Eclairage). In the CIE system color is defined in terms of a standard light source, a normal observer, and three tristimulus values. The advantage of the CIE system is that the tristimulus values can be determined through instrumentation. The tristimulus values X, Y, Z are defined as (17):

$$\begin{aligned} X &= \int ER\bar{x} \, d\lambda \\ Y &= \int ER\bar{y} \, d\lambda \\ Z &= \int ER\bar{z} \, d\lambda \end{aligned}$$

where E and R are functions of the spectral distributions of the light source and the object respectively. The functions  $\bar{x}$ ,  $\bar{y}$ ,  $\bar{z}$  are approximated by functions describing the sensitivities of the eye to red, green, and blue, respectively.

Many other systems exist, systems that are transformations of the CIE system and systems which redefine the basic factors. Richter (18) developed a color difference formula based on the three tristimulus factors Hue (T), Saturation Degree (S), and Darkness Degree (D) in his DIN system. Earlier, Adams put forth a color formula based on Adams Chromatic Value Space. Other systems and formulas that have been suggested for color difference are the Cube-Root formula, and Godlove formula, and Judd's Uniform Chromaticity Scale.

For the quantitative evaluation of fastness each of the mentioned

systems has advantages and drawbacks. As Vickerstaff and Tough (19) suggest, one factor in choosing a system for evaluating fastness properties should be the availability of a color difference unit which is just perceptible to the eye. Ideally large color differences could be split up and numerically evaluated in terms of just perceivable units. No available system meets the requirements of this suggestion, but the CIE-recommended Friele-MacAdam-Chickering (FMC) color difference formula is among the best approximations (20). The results of the FMC color difference calculations are in terms of approximately just-perceivable MacAdam's units (Table 4).

Several other formulas are based on units of uniform perceptibility, but as McLaren (21) indicates, comparisons for other than rough estimates between systems are not justified. Compared to the Adams Chromatic Value units or the National Bureau of Standards unit (NBS), the MacAdam's unit varies between one-fourth to one-third the size depending on the location in color space (16). The calculation of MacAdam's units using the FMC formula requires a knowledge of the X, Y, Z CIE tristimulus values. These can be calculated if the reflectance characteristics of the specimen are known. For many specimens the reflectances can be determined directly by colorimetry, but for a specimen whose reflectance is a function of the color of the background on which it is mounted, a further calculation is required to determine its true reflectance. The instrumental reflectance of the specimen can be measured by mounting it alternately on white and black backgrounds. Assuming the black plate approaches true non-specular characteristics, with a knowledge of the spectral character of the white plate, the true

reflectance of the sample (R) can be determined from the following formula (22):

$$R = a - (a^2 - 1)^{1/2}$$

where

$$a = R_b/2 + (R_w - R_b + W)/(2R_w W)$$

The values  $R_w$  and  $R_b$  are the decimal reflectances of the specimen mounted on a white plate and black plate respectively, and  $W$  is the reflectance of the white plate when compared to a reference white standard (23).

When using the FMC formula for color difference, care should be taken to avoid the effects of fluorescence and optical brighteners. Current color difference formulas do not account for light being re-emitted at other wavelengths. The total reflectance ( $R_t$ ) of a specimen is the sum of its surface reflectance ( $R_s$ ), body reflectance ( $R_b$ ), and reflectance due to fluorescence ( $R_f$ )

$$R_t = R_s + R_b + R_f$$

For the FMC formula to be valid  $R_f$  must be zero (24).

In a paper by Billmeyer (25) some of the cautions to be observed when instrumentation is used to determine color differences are explained. Absolute color measurement can be particularly hard to reproduce especially over a period of time. Also, color differences may lose significance when moving from one color space to another.

### Approach

There are three recognized general types of surface active agents (26). The anionics are surfactants in which the negatively charged portion of the molecule is the more highly active part. Examples include soaps, and sulfated or sulfonated products. The cationic surfactants have highly active positively-charged parts and include the quarternaries and pyridinium halides. The nonionics do not ionize appreciably and include the polyglycols and esters.

One surfactant from each class was chosen and compared with a neutral soap and a common heavy-duty laundry detergent in wash tests. The tests were made on Helanca nylon with three disperse dyes individually applied to the fabric. Temperature, surfactant concentration, additive concentration, and washing time were controlled in a systematic manner for the series of tests. Specimens were evaluated for fastness with a reflectance spectrophotometer.

## CHAPTER II

## MATERIALS, CHEMICALS, AND INSTRUMENTATION

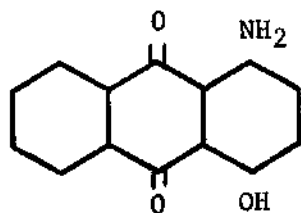
Fabric

The fabric chosen for all tests was a Helanca double-knit. The fabric was constructed with a density of 0.17 g./in.<sup>2</sup>. Helanca is the registered trademark nylon processed according to the patent held by the Heberlein Patent Corporation.

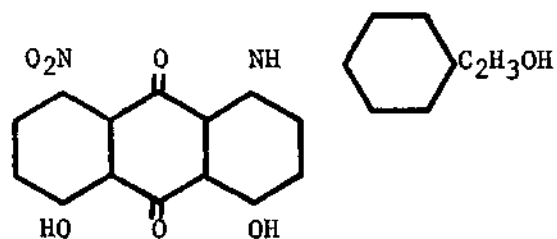
Dyes

Three dyes of varying washfastness characteristics were used in the experiments. C. I. Disperse Red 15 (C. I. 60710) is an anthraquinone derivative with a washfastness rating on nylon 1-2. The dye used was marketed by Ciba as "Cibacete Red 3B". C. I. Disperse Blue 27 (C. I. 60767) is another anthraquinone derivative but with an excellent washfastness rating. The sample used was marketed by Eastman as Eastman Fast Blue GLF. C. I. Disperse Yellow 3 (C. I. 11855) is an azo dye with a gray scale washfastness rating 3. The sample was another Eastman dye marketed as Eastone Yellow GN.

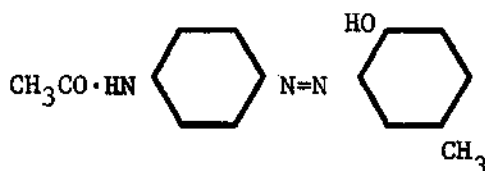
All dyestuffs were checked visually under ultraviolet light and found to be non-fluorescent.



C. I. Disperse Red 15



C. I. Disperse Blue 27



C. I. Disperse Yellow 3

Figure 1. Dye Structures

### Surfactants

Five surfactants were used to scour the samples in this research. As recommended by the AATCC for characterizing textile colorants for colorfastness to washing (27), one of the surfactants utilized in the experiments was a standard neutral chip soap lacking optical brighteners. The specifications require that the minimum anhydrous soap content be 85.0 per cent. The soap used was "Saratoga Chips" made by Proctor and Gamble.

A current heavy duty laundry detergent with phosphate builder was represented by a formulation provided by Colgate-Palmolive. This was a standard alkyl aryl sulfonate formulation, especially prepared without

optical brighteners. The detailed composition will be found in Table 7.

One of the first synthetic detergents to gain widespread acceptance was a sulfonated substituted amide, Igepon T (26). Igepon T is a multi-purpose anionic surfactant used in production situations and as a component for industrial cleansers. It is available as a powder, liquid, flakes, or gel. The starting material used in this research was the 28 per cent powder.

The nonionic surfactant used in this research comes in liquid 100 per cent form. This multi-purpose surfactant, Triton X-100, is an octyl phenoxy polyethoxy ethanol from Rohm and Haas.

For a cationic surfactant, an experimental sample from Atlas Chemical Industries was used. The agent was a 35 per cent aqueous solution of N-cetyl N-ethyl morpholinium ethosulfate.



Figure 2. N-cetyl N-ethyl Morpholinium Ethosulfate

### Builders

Three reagent chemicals were used as additives for the surfactants in the experiments. They were trisodium phosphate, calcium chloride, and sodium carbonate.

#### pH Meter

Hydrogen ion concentrations were measured with a Corning Model 10 pH Meter. The electrodes were a triple purpose Ag-AgCl electrode and a calomel electrode.

#### Launder-Ometer

All samples were washed in an Atlas Launder-Ometer in pint glass-top jars. The Launder-Ometer was equipped with a thermostatically controlled water bath, pre-conditioning table, and driving mechanism. The jars were secured to the rotor and driven through the temperature bath at forty-two revolutions per minute.

#### Reflectance Spectrophotometer

Reflectance measurements were made using a large sphere Color-Eye, Signature Model, manufactured by Instrument Development Laboratories. The Color-Eye is an abridged spectrophotometer-colorimeter of double-beam construction. It is used to determine the reflectance of directional materials under a choice of light sources. Samples are viewed from a plane eight degrees from normal. The model used in this research was equipped with an 18-inch integrating sphere coated with barium sulfate. Standardized white Vitrolite plates were used as reference standards.



## CHAPTER III

## PROCEDURES

Dyeing

Considerable care was taken to insure reproducible dyeings on a day-to-day basis. All dye solutions were prepared immediately prior to use.

The nylon fabric was cut into six inch by three inch rectangles. Sixteen of these three gram swatches were counted out and smaller pieces of fabric added to make the total fiber weight 50.00 grams. The fabric was then wet out in distilled water at 90°C.

A one gram per liter dye solution was prepared and a 500 ml aliquot added to 2.50 liters of distilled water equilibrated at 90°C. After the system had again reached 90°C, the wet-out fabric was drained, added to the flask, and dyed for 75 minutes.

For reproducibility the dyeings were made in a closed system. The temperature was controlled to  $\pm 0.5^{\circ}\text{C}$  with a rheostatically controlled immersion heater in a bath of Carbowax 400. A triple-neck, five-liter flask was used as the dyeing vessel. The flask was fitted with a reflux condenser and an electric stirrer. All dyeings were made with the individual dyestuffs at the previously described 1.0 per cent owf (on weight fiber) and 60:1 liquor ratio.

After the dyeing was completed the fabric was rinsed in 600 ml of water. To remove any surface dye and to prevent diffusion to the fiber

surface, the second rinse was made for sixty seconds in a 300 ml. ice and 300 ml. methanol mixture. The fabric was rinsed with water again, centrifuged to dryness, and carefully stored. To minimize gas and light fading the samples were wrapped in cotton cloth and stored in a dark drawer.

### Washing

The washings were made in a Launder-Ometer with pint jars containing 200 ml. total liquor and ten one-quarter inch steel balls. Systematically, each of the three different colored fabrics, was agitated for times varying from 22.5 minutes to 3 hours with the various surfactants. The effect of temperature was studied at 80°F, 110°F, 140°F, and 160°F. Surfactant concentrations were measured as per cent ows (on weight solution) of the producer's form of the surfactant. The concentrations ranged from 0.0 per cent to 1.00 per cent. When builders were added to the surfactant, the surfactant concentration was maintained at 0.25 per cent ows. The electrolyte concentration was varied between 0.1 and 1.0 per cent ows. All tests included a control containing 200 ml of distilled water and a dyed specimen which was agitated for 45 minutes. This specimen was used as the standard from which the other swatches were rated for fastness. The number of specimens washed during a test ranged from two to ten. The swatches used in each test were 3.0 inches square with small pieces added to make the weight of fabric 1.500 grams.

After the pH of the washing solution had been determined, the jars containing the solution were pre-conditioned to temperature in a water bath for a minimum of 45 minutes. Once conditioned the fabric

specimens were added to the jars and placed on the Launder-Ometer rotor. Washing time was counted as actual minutes of agitation. The specimens were removed, rinsed thoroughly with warm water, and centrifuged to dryness. To prevent wrinkling, which would cause deviations in color measurement, the swatches were placed open width on numbered watchglasses. The watchglasses were set in a dark drawer overnight in preparation for color measurement.

To promote reproducibility the many handling operations of washing were performed to a set routine. The operations of adding the fabric specimens to the washing jars, securing the lids, securing the jars to the rotor, unlocking the lids, and removing the fabric specimens from the jars were always performed in the same order according to specimen identification number.

#### Color Measurement

Reflectance measurements were taken of the fabric specimens with a Color-Eye abridged spectrophotometer colorimeter. The Color-Eye was calibrated according to the manufacturer's procedure. All reflectances were taken on high sensitivity, using the small two-inch sample port. Non-specular inserts were set in the integrating sphere to reduce surface gloss effects. The specimens were always oriented with the face toward the detector and the lengthwise portion of the wales perpendicular to the floor.

The working standard used for determining the absolute reflectance of the samples was a pair of Vitrolite white plates with known characteristics for conversion to CIE values. The true reflectance of each washed swatch was obtained for the four Color-Eye colorimetric filters

x, X, Y, and Z. Table 2 contains the equations used to convert the decimal reflectances to CIE values for the Vitrolite standard used in this research.

Table 2. Equations for Converting Reflectances to CIE Values

---

$$X_{\text{CIE}} = .1765 (x) + .6865$$

$$Y_{\text{CIE}} = .885 (Y)$$

$$Z_{\text{CIE}} = 1.0429 (Z)$$

---

Preliminary study indicated that samples much larger than 3" x 3" were unsuitable for washing, but the reflectance of such samples was background dependent when viewed by the Color-Eye. To compensate for this lack of opaqueness and determine the true reflectance of the fabric, two readings were taken for each specimen with each of the four colorimetric filters. One reading was taken with a non-specular insert behind the fabric and another with a Vitrolite plate behind the fabric. As explained in Chapter I, knowing the spectral characteristics of the Vitrolite plate, and knowing the reflectances of the specimen with the white and black backgrounds placed behind it, the true reflectance of the specimen can be calculated. The spectral characteristics of the Vitrolite backgrounds were learned by comparing it with a barium sulfate standard.

Table 3. Reflectances of the Vitrolite Plate Compared to Barium Sulfate White

---

$$x = .9230$$

$$X = .8997$$

$$Y = .9065$$

$$Z = .9213$$

---

For the convenience of the reader a computer program to determine MacAdam's color differences written for a Fortran V compiler is included in Table 4. This program is similar to the one used for this research. Particular care should be noted that lines 13-16 and 33-38 are white standard dependent.

## CHAPTER IV

## DISCUSSION OF RESULTS

Washfastness quality was determined by calculation of MacAdam's color difference units ( $\Delta E$ ). A control specimen, which was agitated for 45 minutes in distilled water with each test, was used as the standard for determining the extent of fading caused by the influence of the various surfactants and washing conditions.

Effects of Surfactant Type

The choice of surfactant was a significant factor in the outcome of the washfastness tests. In all tests the fabrics dyed with C. I. Disperse Red 15 showed considerably more fading than the fabrics dyed with C. I. Disperse Yellow 3 or C. I. Disperse Blue 27 when washed under similar conditions. The differences in fastness as influenced by surfactant choice were most evident in the tests involving the red fabric but were reflected similarly in the tests with the blue and yellow fabrics. The standard surfactant for washfastness testing for many years has been neutral soap. The experiments on all dyes indicate that of the five surfactants tested, neutral soap is one of the most rigorous choices ranking along with Triton X-100 in severity. The surfactant displaying the mildest effect was the heavy duty laundry detergent with the cationic, N-cetyl N-ethyl morpholinium ethosulfate, and Igepon T being intermediate in severity.

### Effects of Surfactant Concentration

Over the range of concentrations studied, 0-1.0 per cent ows, fastness decreased with increasing surfactant concentration. On the more fast C. I. Disperse Yellow 3 and C. I. Disperse Blue 27 dyed samples, the loss of fastness was moderate with there being one or two color units difference between the samples washed at highest concentration and the samples washed at intermediate concentrations. The samples dyed with C. I. Disperse Red 15 showed marked decreases in fastness with increased surfactant concentrations. Differences of 10 to 15 MacAdam's color units were common between the swatches washed at 1.0 per cent ows and those washed at lower concentrations. The greatest change of fastness occurred between 0.25 and 0.50 per cent ows surfactant concentrations. As indicated in Figures 8 and 9, the fastness/concentration slopes appear to be tending toward maxima at points slightly higher than 1 per cent ows. It should be remembered that detergency also increases with surfactant concentration up to maximum activity at the critical micelle concentration (11).

### Effects of Time, Temperature, and pH

The wash tests performed to show the effects of time and temperature on washfastness continued to exhibit the poor fastness of C. I. Disperse Red 15 relative to the blue and yellow dyes.

Over the range of times of wash cycles studied, 22.5 to 180 minutes, only a slight trend was noticeable toward decreasing washfastness with increasing cycle time. Shifts of three to four MacAdam's color units were noted in the tests showing the different effects of various surfactants between the 90 and 180 minute cycles on C. I.

Disperse Yellow 3, but as indicated in Figures 15-18, the solutions containing the C. I. Disperse Red 15 and the C. I. Disperse Blue 27 dyed fabrics were approaching equilibrium after 45 minutes of washing time.

Increasing the temperature of the wash bath produced larger color changes. Particularly with neutral soap and Triton X-100, the fading increased with increasing temperature. The largest effects were noted between 140°F and 160°F.

With the exception of the instance in which calcium chloride was added to the cationic surfactant, fading tended to increase as the pH of the surfactant solutions differed from neutral pH 7. Of the surfactants tested the two most harsh in terms of fastness properties, neutral soap and Triton X-100, represent the extremes in pH. At 1.0 per cent ows, Triton X-100 has pH 3.97 while neutral soap exhibits pH 10.16 at 0.5 per cent ows. With increasing calcium chloride concentration, the cationic surfactant solution became less acidic, but calcium chloride is more alkaline than N-cetyl N-ethyl morpholinium ethosulfate.

#### Effects of Electrolyte Addition

The addition of electrolytes as builders for surfactant solutions resulted in some washfastness effects similar to those encountered from increasing surfactant concentrations. The C. I. Disperse Red 15 dyed fabrics exhibited more fading than the C. I. Disperse Blue 27 and C. I. Disperse Yellow 3 dyed swatches. Fastness generally decreased and tended toward limiting values with increasing electrolyte concentration.

Two electrolytes, trisodium phosphate and sodium carbonate, were added to a nonionic surfactant, Triton X-100, and an anionic surfactant, neutral soap. At corresponding electrolyte concentrations, washfastness



was slightly better on the neutral soap-electrolyte washed fabric than on the ones washed in Triton X-100 electrolyte. Divalent sodium carbonate additions resulted in better fastness qualities than the equivalent concentration addition of trivalent sodium phosphate. The addition of calcium chloride to the cationic solution of N-cetyl N-ethyl morpholinium ethosulfate resulted in fastness trends similar to, but less pronounced than, increases in concentration of the cationic surfactant.

A significant shift in the color of the wash liquor was observed when the red and yellow samples were washed in sodium carbonate or trisodium phosphate containing solutions. The wash liquors containing the yellow samples shifted to red-orange, and the liquors containing the red sample shifted to a deep red-violet. As indicated by the experiments performed over a variety of wash cycle times, most wash solutions were approaching an equilibrium between the unsaturated aqueous and fiber phases after 45 minutes of washing. Combined with the results of the tests on addition of surfactants and builders, this research indicates that the presence of such additives has a significant effect on the equilibrium state in a wash bath. Other changes in the system as a shift in temperature also have an effect on washfastness, not only by changing the equilibrium state, but also by altering the diffusion of the dye through the fiber and the rate at which equilibrium is approached.

## CHAPTER V

## CONCLUSIONS

This research established that the choice of surfactant and washing conditions has a pronounced effect on the washfastness qualities of disperse-dyed nylon fabric. It was found that the standard test surfactant for many years, neutral chip soap, and the synthetic nonionic, Triton X-100, provided the most rigorous conditions for washfastness testing of the surfactants tested. In many instances when washing under similar conditions but changing surfactants, the fastness quality was shifted significantly toward more acceptable levels. Temperature also plays an important part in the degree of fastness.

As Bird suggested (9) the presence of an electrolyte can increase the solubility of disperse dyes in water. When electrolytes were added to the surfactants as builders, the fastness qualities of the samples decreased, indicating that more disperse dye was remaining in the wash liquor. This change in solubility could be related to the intense color changes noted on addition of sodium carbonate or trisodium phosphate to the wash bath with the C. I. Disperse Yellow 3 and C. I. Disperse Red 15 dyed fabric.

The indications from this research are that the controls over washfastness are similar to those in a dyeing system. The presence of a surfactant or electrolyte or other changes in the washing system which influence the solubility of the dye in the aqueous and fiber phases or

the diffusion of the dye through the fiber affect washfastness.

As tools for fastness evaluation, spectrophotometry and colorimetry require close scrutiny. Maintaining consistency in readings over a period of time is difficult. The reproducibility of the wash test must be reckoned with when measuring small color differences, and the available equations for evaluating color differences have shortcomings when measuring large color differences.

## CHAPTER VI

## RECOMMENDATIONS

This work represents only a small portion of the research which could be done to help determine washfastness effects of surfactants. Dyes taken in combination, other dye classes, and different substrates would exhibit different fastness qualities from those studied. With the interest of detergent manufacturers shifting to non-phosphate builders, much information could be learned about their relative detergency and their effects on fastness. In relation to disperse dyes, data on the changes of dye solubility as a function of the builders would be useful.

In the area of fastness evaluation, further research in color theory and instrumentation will be welcomed. Determining fastness by comparison to physical standards has its drawbacks. With improvements in instrument sensitivity and methods of relating the results to color difference, instrumental fastness determinations would be more meaningful.

APPENDIX

Table 4. Friele-MacAdam-Chickering Color Difference Formula

The FMC formula (20) for color difference calculations to MacAdam's units ( $\Delta E$ ) is:

$$\Delta E = \left[ (\Delta C)^2 + (\Delta L)^2 \right]^{1/2}$$

where

$$\Delta C = K_1 \Delta C_1 \text{ and } \Delta L = K_2 \Delta L_2,$$

$$\Delta C_1 = \left[ (\Delta C_{rg} / a)^2 + (\Delta C_{yb} / b)^2 \right]^{1/2}$$

$$\Delta L_1 = (P \Delta P + Q \Delta Q) / (P^2 + Q^2)^{1/2}$$

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

$$\Delta C_{yb} = S \Delta L_1 / (P^2 + Q^2)^{1/2} - \Delta S$$

$$\Delta L_2 = 0.279 \Delta L_1 / a$$

$$a^2 = 1.73 \times 10^{-5} (P^2 + Q^2) / \left[ 1 + 2.73 \frac{P^2 Q^2}{(P^4 + Q^4)} \right]$$

$$b^2 = 3.098 \times 10^{-4} (S^2 + 0.2015Y^2),$$

where P, Q, S are, values of the standard and

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

$$Q = 0.48X + 1.37Y + 0.1276Z$$

Table 4. Friele-MacAdam-Chickering Color Difference  
Formula (Continued)

---


$$S = 0.686Z,$$

and, X, Y, Z are the tristimulus CIE values. Also,

$$\Delta P = P_{\text{sample}} - P_{\text{standard}}$$

$$\Delta Q = Q_{\text{sample}} - Q_{\text{standard}}$$

$$\Delta S = S_{\text{sample}} - S_{\text{standard}}$$

and

$$K_1 = 0.55669 + 0.049434Y - 8.2575 \times 10^{-4}Y^2 \\ + 7.9172 \times 10^{-6}Y^3 - 3.0087 \times 10^{-8}Y^4$$

$$K_2 = 0.17548 + 0.027556Y - 5.7262 \times 10^{-4}Y^2 \\ + 6.3893 \times 10^{-6}Y^3 - 2.6731 \times 10^{-8}Y^4$$

where Y is the CIE value of the standard.

---

Table 5. Program for Computing MacAdam's Color Differences

C	DATA FORMAT	
C	COLS 1 - 4	XPRIME BLACK
C	COLS 5 - 8	X BLACK
C	COLS 9 - 12	Y BLACK
C	COLS 13 - 16	Z BLACK
C	COLS 17 - 26	SURFACTANT
C	COLS 27 - 31	PH
C	COLS 32 - 34	TEMP
C	COLS 35 - 40	CONC
C	COLS 41 - 43	TEST NUMBER
C	COLS 44 - 50	DATE -IN LAUNDRER-OMETER
C	COLS 51 - 54	TIME
C	COLS 54 - 62	COMMENTS
C	COLS 63 - 66	XPRIME WHITE
C	COLS 67 - 70	X WHITE
C	COLS 71 - 74	Y WHITE
C	COLS 75 - 78	Z WHITE
C	1ST CARD N -	2DIGITS FOR NO. OF SAMPLES
C	M -	2DIGITS FOR NO. OF STANDARDS
C	COLS 5-20	FOR DYE NAME
	REAL K1,K2	1
	DIMENSION DY(4),SAM(30,8),SOA(30,2), PH(30), TEMP(30),CON(30),	2
	ITEST(30), DAT(30,2), COM(30,3), STD(4),S(4), TIM(30)	3
	1 READ(5,10) N,M,(DY(I), I=1,4)	4
10	FORMAT(2I2,4A4)	5
	IF(N) 20,20,30	6
30	CONTINUE	7
	DO 40 I=1,N	8
	READ(5,50) (SAM(I,J),J=5,8), (SOA(I,J), J=1,2), (PH(I)), TEMP(I),	9
	ICON(I), TEST(I), (DAT(I,J), J=1,2), (TIM(I)), (COM(I,J), J=1,3),	10
	2(SAM(I,J), J=1,4)	11
50	FORMAT(4F4.4, 2A5, A5, A3, A6, A3, A4, A3, A3, 3A3, 4F4.4)	12
	AXP=SAM(I,1)/2.0+(SAM(I,5)-SAM(I,1)+.923)/(2.0*SAM(I,5)+.923)	13
	AX =SAM(I,2)/2.0+(SAM(I,6)-SAM(I,2)+.8997)/(2.0*SAM(I,6)+.8997)	14
	AY =SAM(I,3)/2.0+(SAM(I,7)-SAM(I,3)+.9065)/(2.0*SAM(I,7)+.9065)	15
	AZ =SAM(I,4)/2.0+(SAM(I,8)-SAM(I,4)+.9213)/(2.0*SAM(I,8)+.9213)	16
	SAM(I,1) = AXP - SQRT(AXP*AXP -1)	17
	SAM(I,2) = AX - SQRT(AX*AX -1)	18
	SAM(I,3) = AY - SQRT(AY*AY -1)	19
	SAM(I,4) = AZ - SQRT(AZ*AZ -1)	20
40	CONTINUE	21
	DO 60 I=1,M	22
	DO 70 L=1,4	23
	STD(L)=SAM(I,L)	24
70	CONTINUE	25
80	WRITE(6,90) (DY(J), J=1,4), (SOA(I,J), J=1,2)	26
90	FORMAT(1X,/, ' THE STANDARD IS ',4A4, ' WASHED WITH ', 2A5)	27
	KA=I+1	28
	DO 100 K=KA,N	29
	DO 110 L=1,4	30
	S(L)=SAM(K,L)	31
110	CONTINUE	32
	X =.1765*STD(1)+.6865*STD(2)	33
	X2=.1765*S(1) +.6865*S(2)	34
	Y =.885*STD(3)	35
	Y2=.885*S(3)	36
	Z =1.0429*STD(4)	37



Table 5. Program for Computing MacAdam's Color Differences (Continued)

Z2=1.0429*(4)	38
P =.724*X +.382*Y -.098*Z	39
P2=.724*X2+.382*Y2-.098*Z2	40
Q =-.40*X +1.37*Y +.1276*Z	41
Q2=-.40*X2+1.37*Y2+.1276*Z2	42
S1=.686*Z	43
S2=.686*Z2	44
P2A02= P*P+Q*Q	45
P2G2=P*P+Q*Q	46
P2A04=P**4+Q**4	47
G=SQRT(3.098E-04*(S1+S1+.2015*Y*Y))	48
A=SQRT(1.73E-05*P2A02/(1.0+2.71*P2G2/P2A04))	49
K1=.50E6+.049434*Y-.82575E-03 * Y*Y + .79172E-05*Y**3	50
L=.30007E-07*Y**4	51
K2=.1754E+.027556*Y-5.7262E-04*Y*Y+6.3993E-06*Y**3-2.7631E-08*Y**4	52
D=P2-P	53
D=C2-C	54
DS=S2-S1	55
DL1=(P*DP+Q*DG)/(SQRT(P2A02))	56
D.L2=(.279*DL1/A)	57
DC16=(C1*DL)/(SQRT(P2A02))-DS	58
DC16=(C1*DP-P*DC)/(SQRT(P2A02))	59
D.L1 = SQRT((DC16/A)**2 + (DC16/P)**2)	60
DC=K1*C1	61
DL=K2*L2	62
DE=SQRT(DC*DC + DL*DL)	63
I=(K-1) 120,130,120	64
130 WRITE(6,140)	65
140 FORMAT (' CYE LANG',6X,'SIRFRACTANT TIME PH TEMP',	66
1* CMC TEST DATE DELTAC DELTAL DELTAE %	67
2*COMPUTED REFLECTANCES,6X,COMMENTS)	68
3* ITE(6,150) (QY(J),J=1,4),(SOA(I,J),J=1,2), YIX(I), PH(I),	69
1T MP(I),CON(I), TEST(I), (DAT(I,J), J=1,2), (STD(J), J=1,4),	70
2(CON(I,J),J=1,3)	71
150 FORMAT(1X,4A4,2X, 2A5, 2X, A5, 2X, A5, 2X, A4, 2X, A6, 2V, A3,	72
12*, A4,A3, 26X,4F6.4,3X,3A3)	73
120 CONTINUE	74
WRITE(6,160) (Y(J),J=1,4),(SOA(K,J), J=1,2), TIM(K), PH(K),	75
1TEP(K), CON(K), TEST(V), (DAT(K,J), J=1,2), DC,DL,DE,	76
2(S(J), J=1,4),(CON(K,J), J=1,3)	77
160 FORMAT(1X,4A4,2X, 2A5, 2V, A3, 2X, A5, 2X, A4, 2X, A6, 2V, A3,	78
12*, A4,A3,3F8.4,2X,4F6.4,3X,3A3)	79
100 CONTINUE	80
60 CONTINUE	81
GO TO 1	82
20 END	83

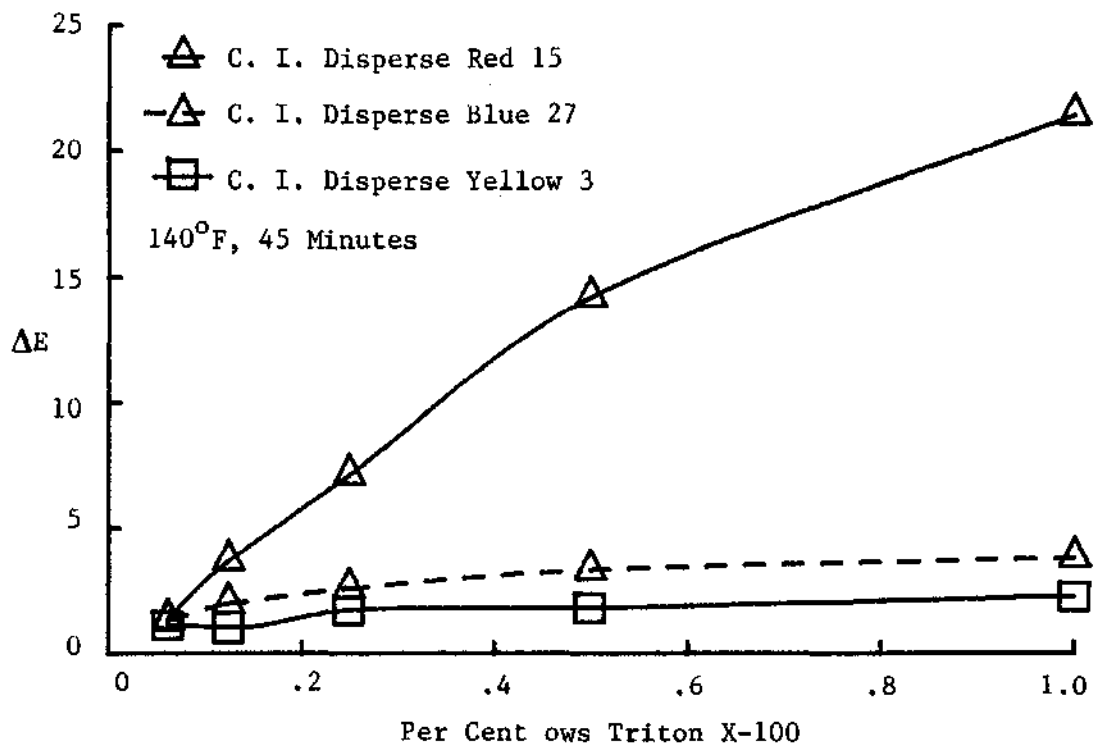


Figure 3. Effect of Triton X-100 Concentration on Washfastness.

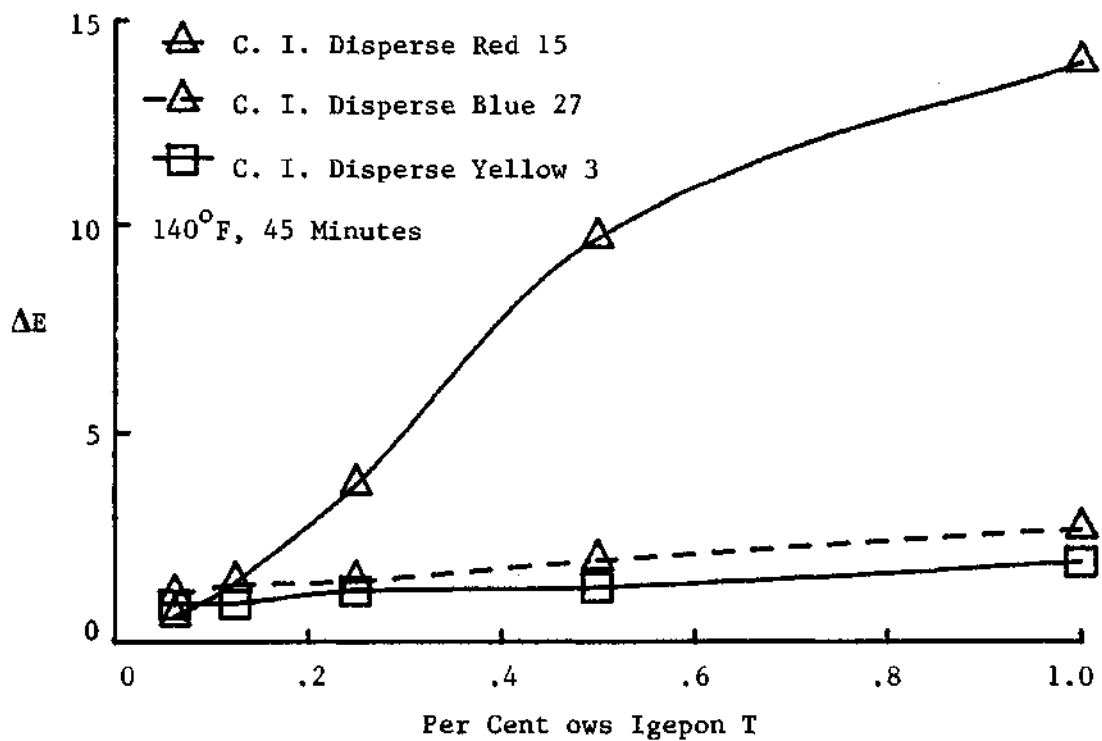


Figure 4. Effect of Igepon T Concentration on Washfastness.

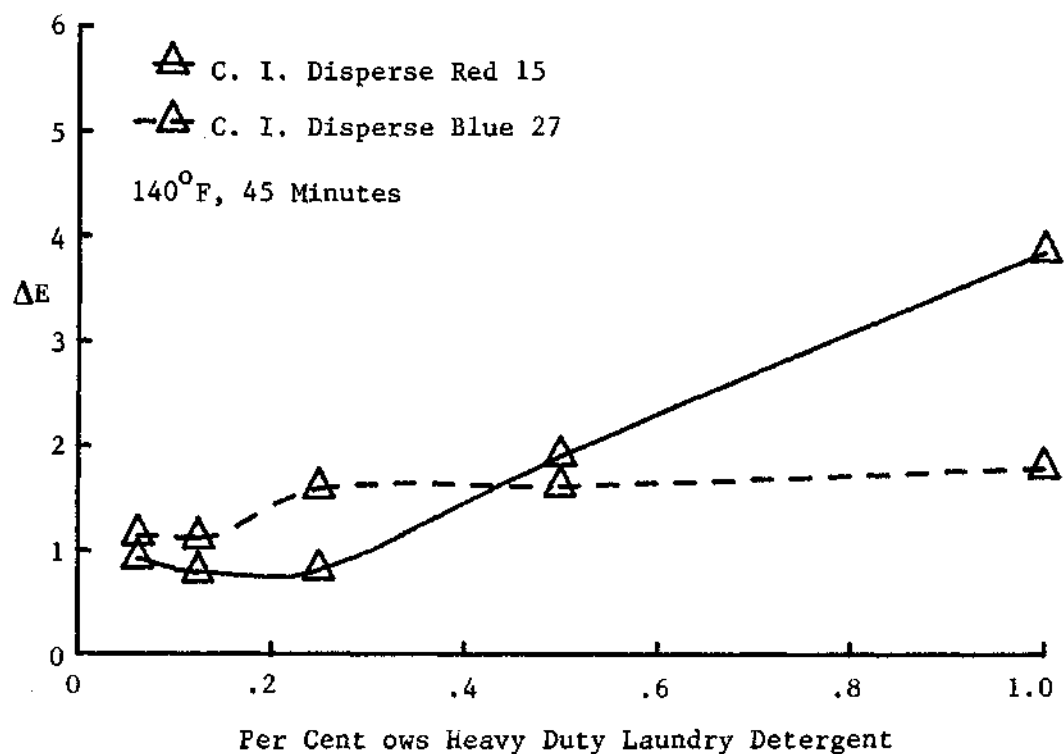


Figure 5. Effect of a heavy Duty Laundry Detergent on Washfastness.

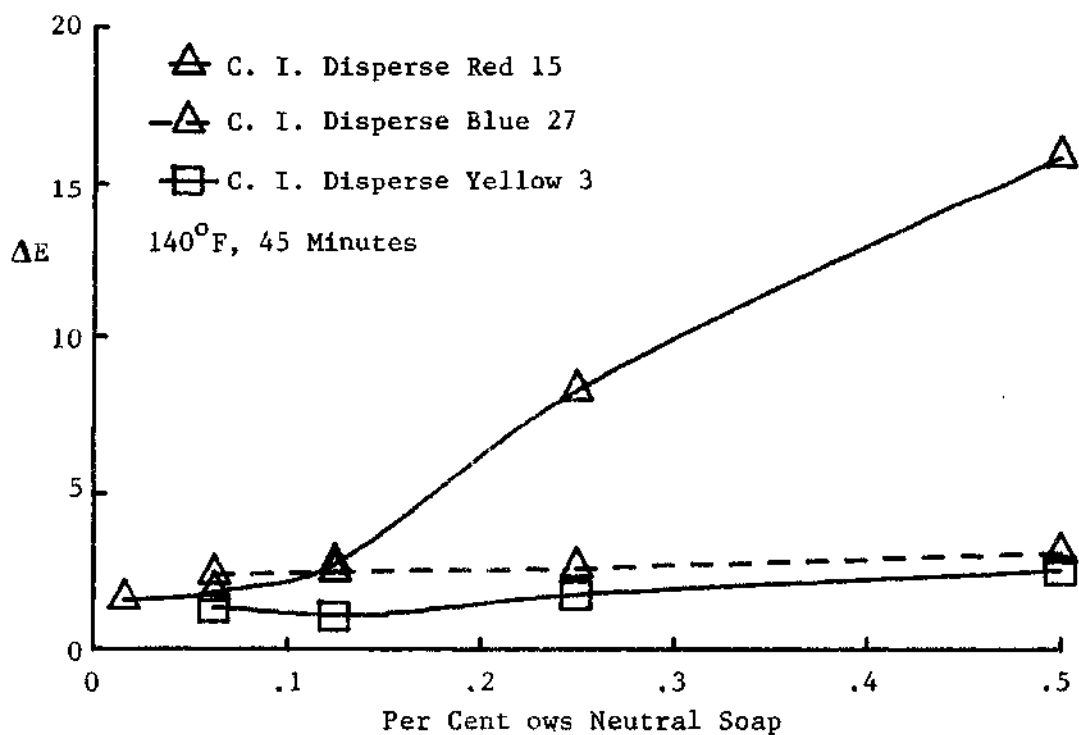


Figure 6. Effect of Neutral Soap Concentration on Washfastness.

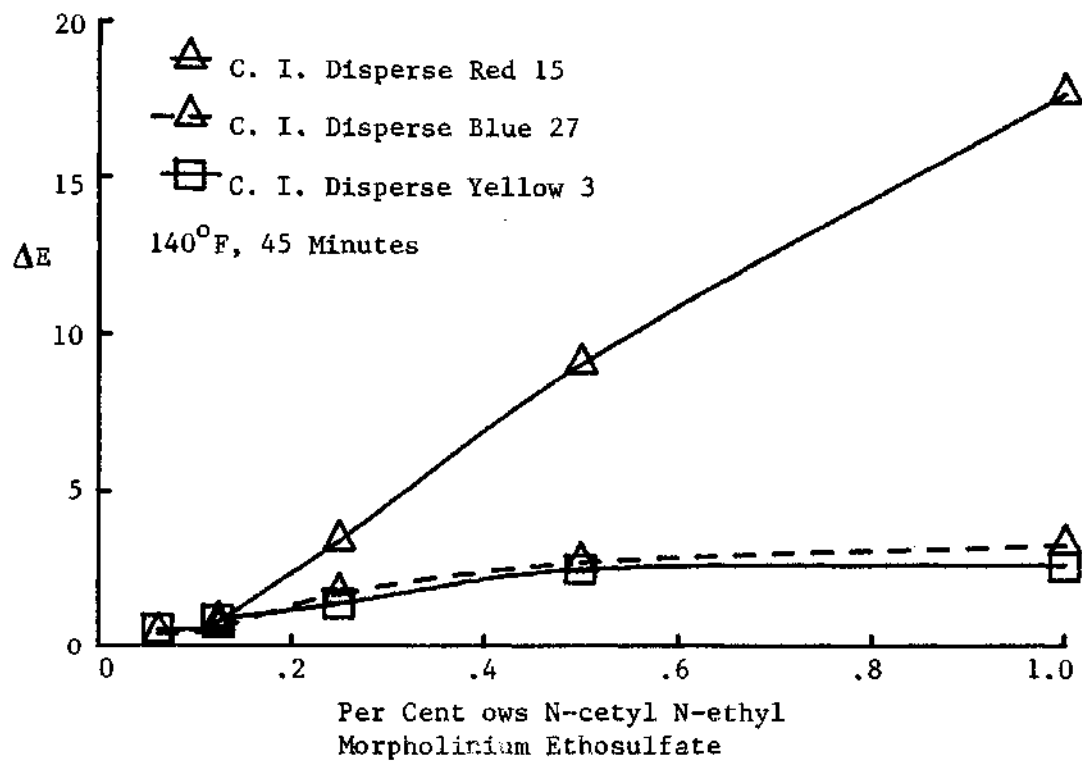


Figure 7. Effect of N-cetyl N-ethyl Morpholinium Ethosulfate Concentration on Washfastness.

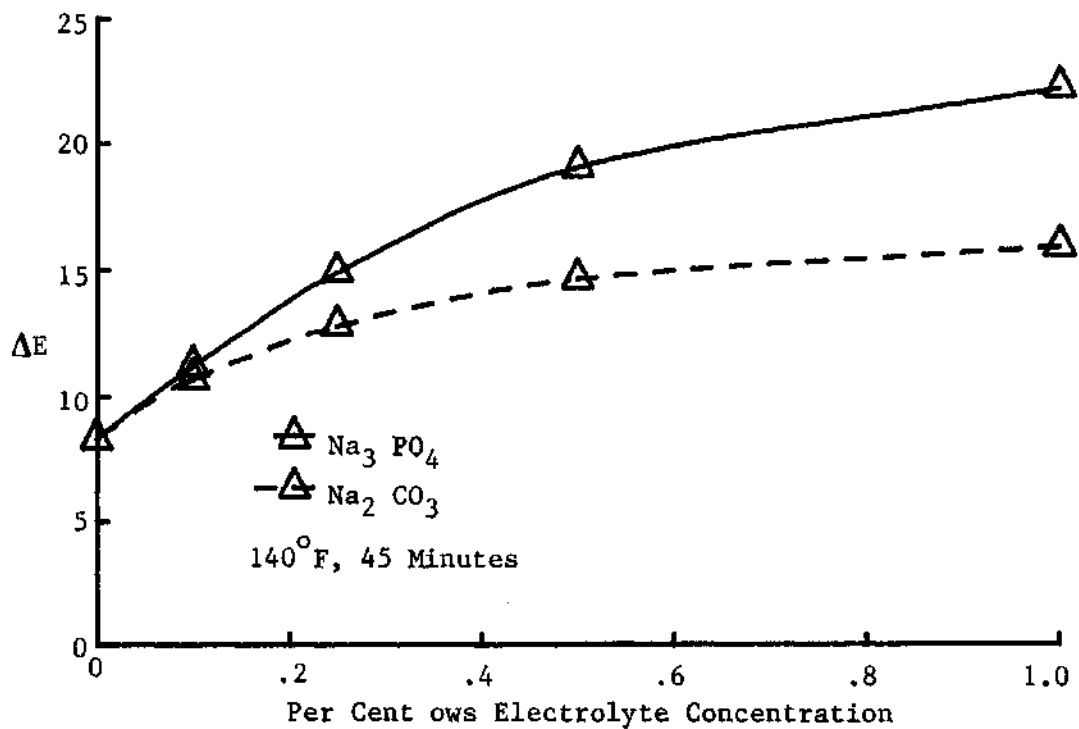


Figure 8. Washfastness of C. I. Disperse Red 15 to 0.25 Per Cent ews Neutral Soap and Electrolyte Concentration.

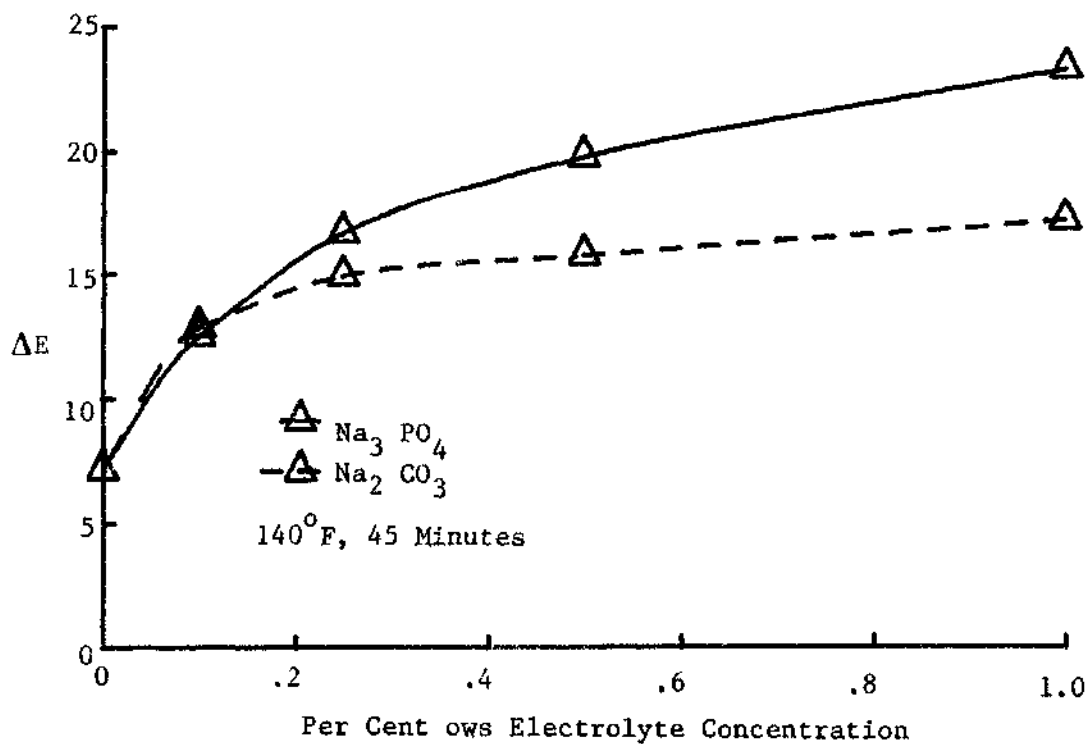


Figure 9. Washfastness of C. I. Disperse Red 15 to 0.25 Per Cent ews Triton X-100 and Electrolyte Concentration.

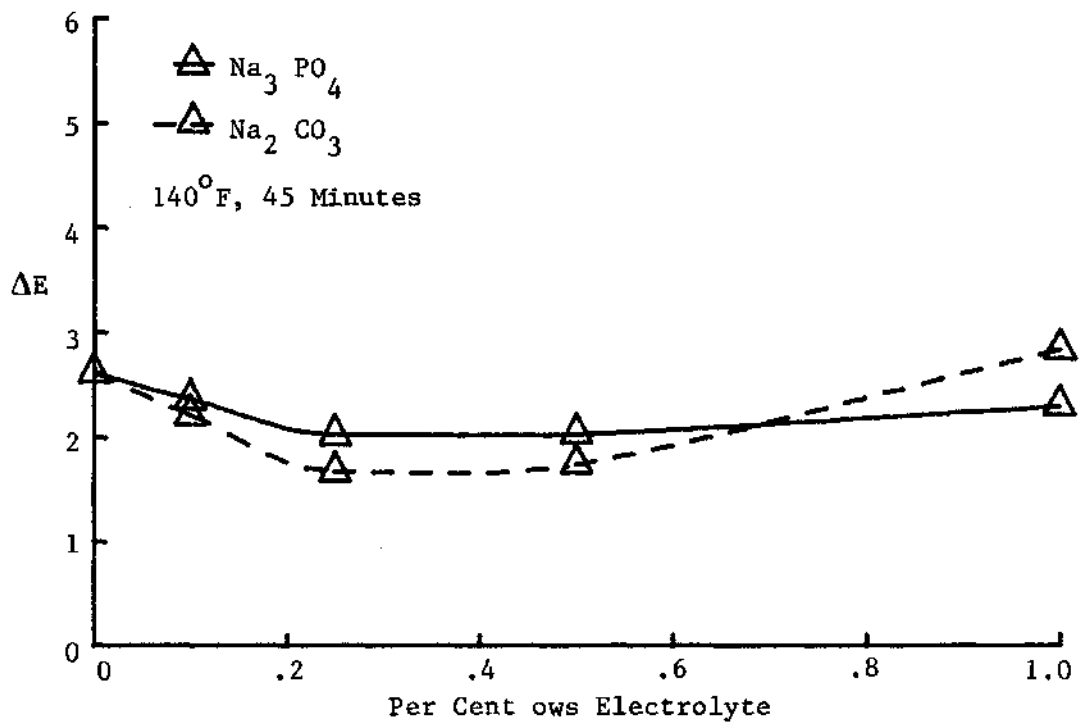


Figure 10. Washfastness of C. I. Disperse Blue 27 to 0.25 Per Cent Triton X-100 and Electrolyte Concentration.

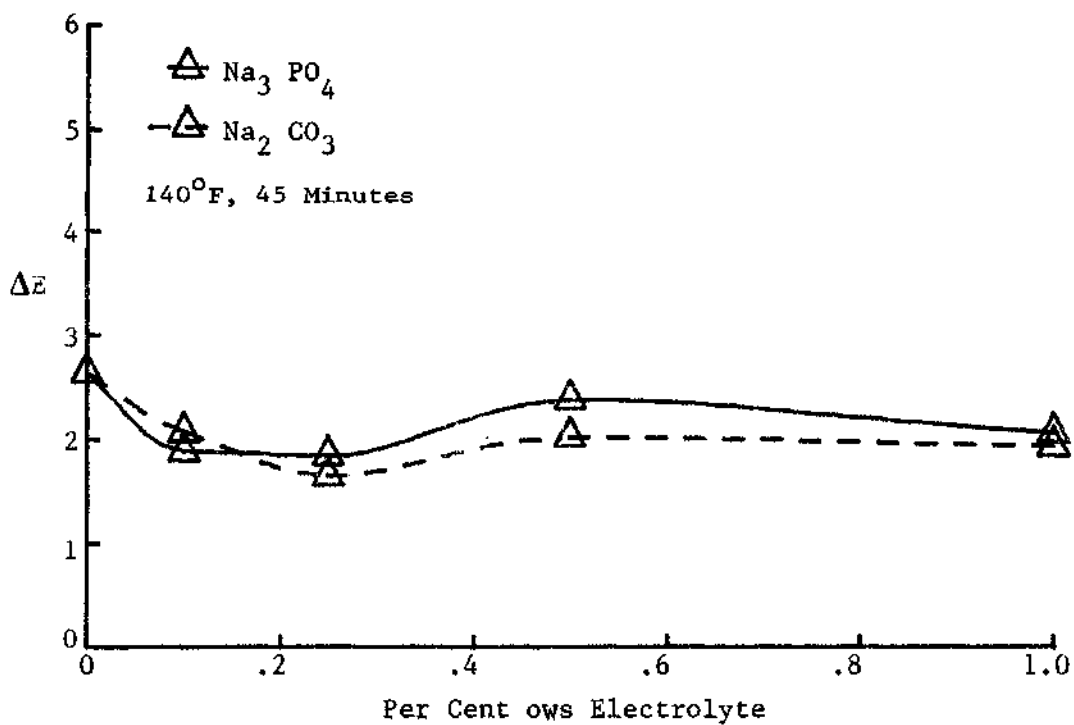


Figure 11. Washfastness of C. I. Disperse Blue 27 to 0.25 Per Cent Neutral Soap and Electrolyte Concentration.

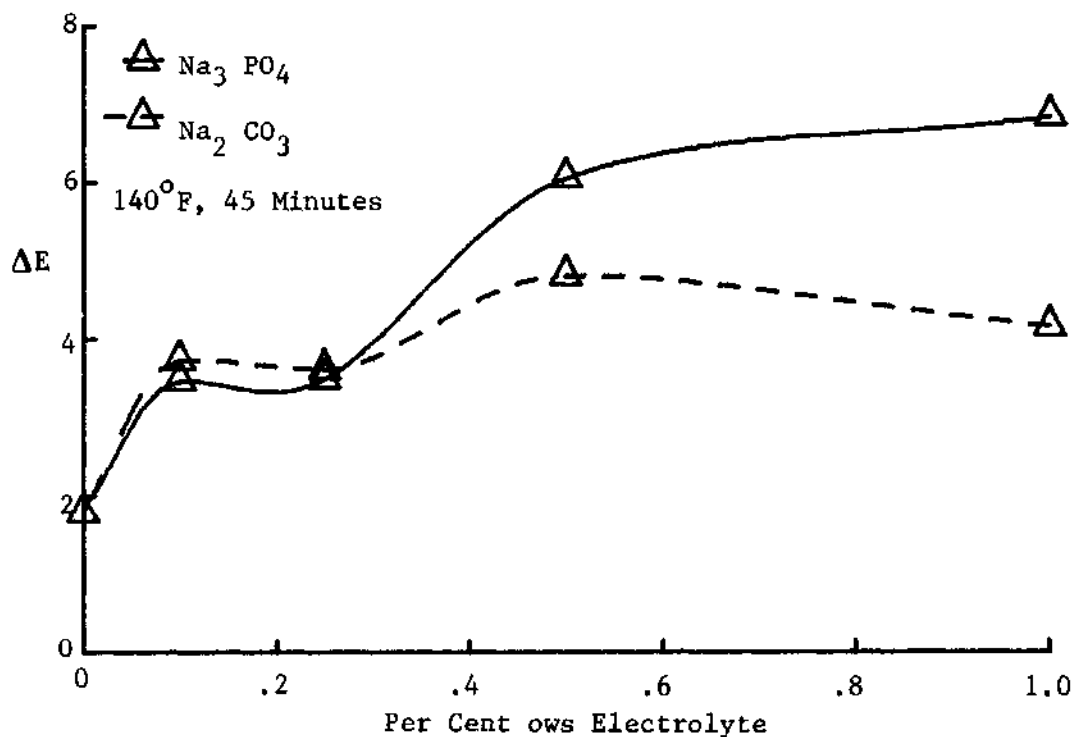


Figure 12. Washfastness of C. I. Disperse Yellow 3 to 0.25 Per Cent Triton X-100 and Electrolyte Concentration.

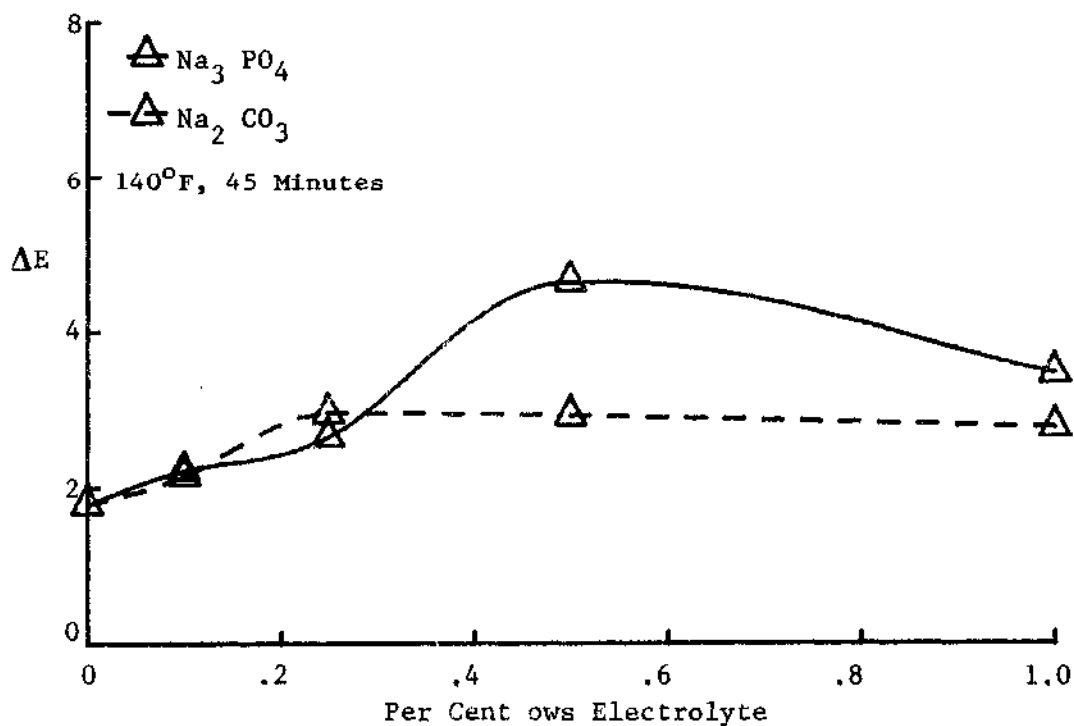


Figure 13. Washfastness of C. I. Disperse Yellow 3 to 0.25 Per Cent Neutral Soap and Electrolyte Concentration.

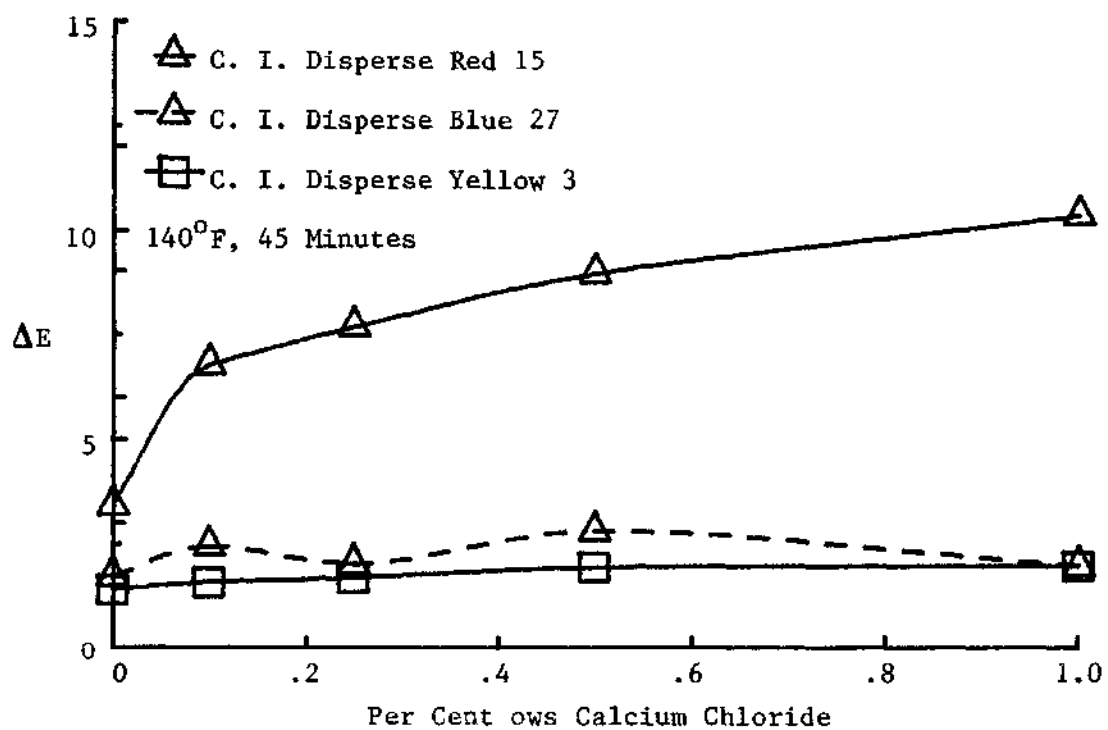


Figure 14. Washfastness to 0.25 Per Cent N-cetyl N-ethyl Morpholinium Ethosulfate and Calcium Chloride Concentration.



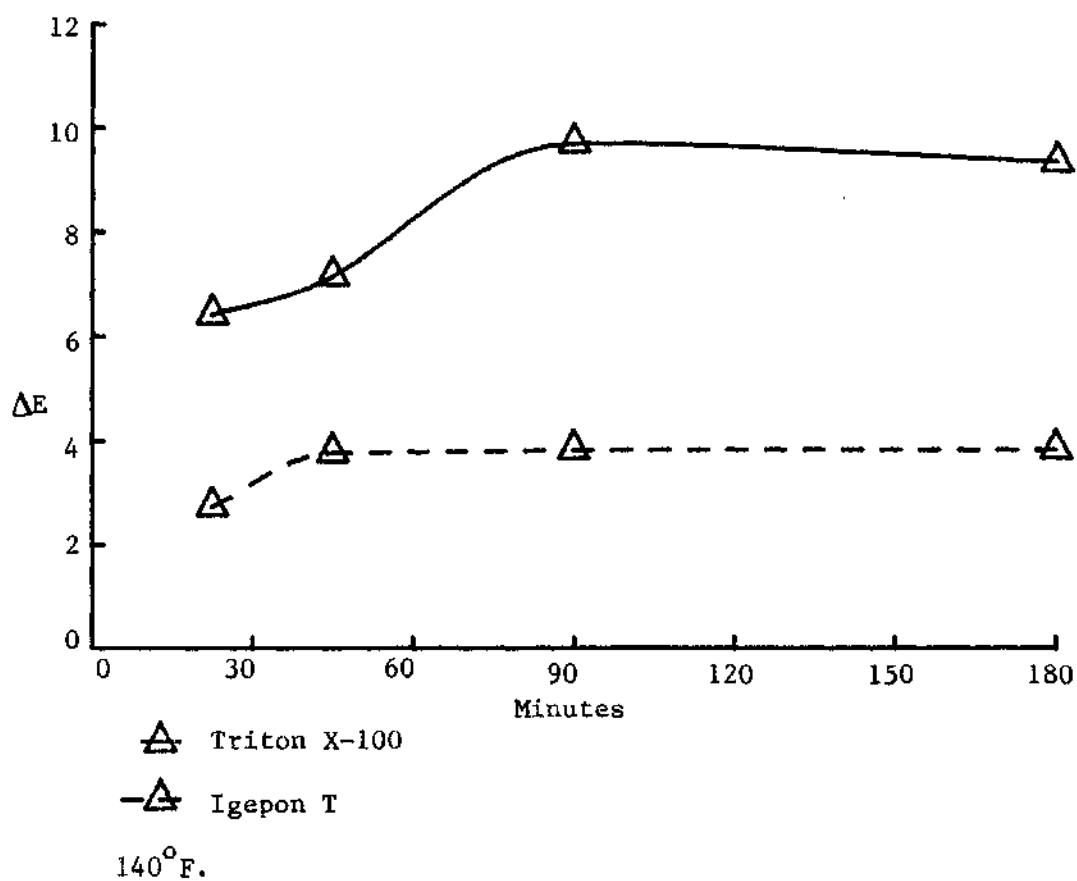


Figure 15. Washfastness of C. I. Disperse Red 15 to 0.25 Per Cent Surfactant Concentration and Time-1.

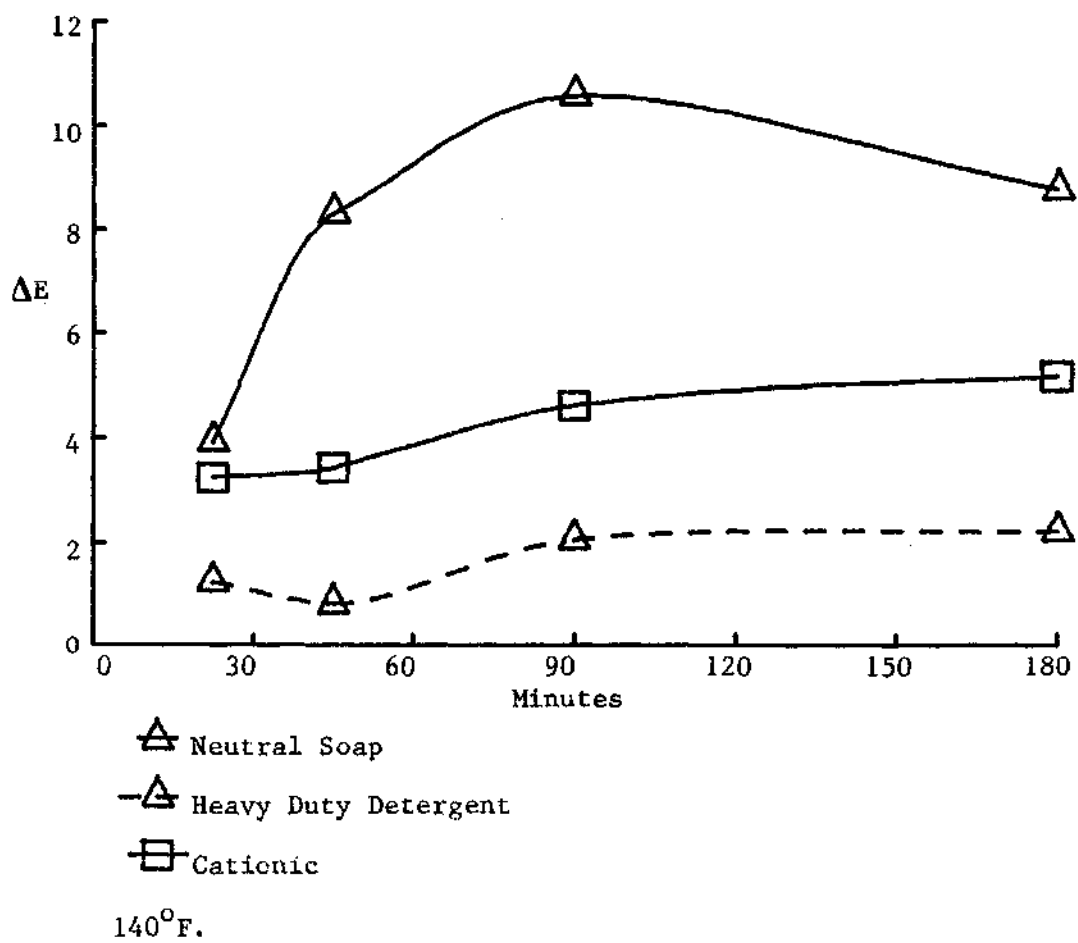


Figure 16. Washfastness of C. I. Disperse Red 15 to 0.25 Per Cent Surfactant Concentration and Time-2.

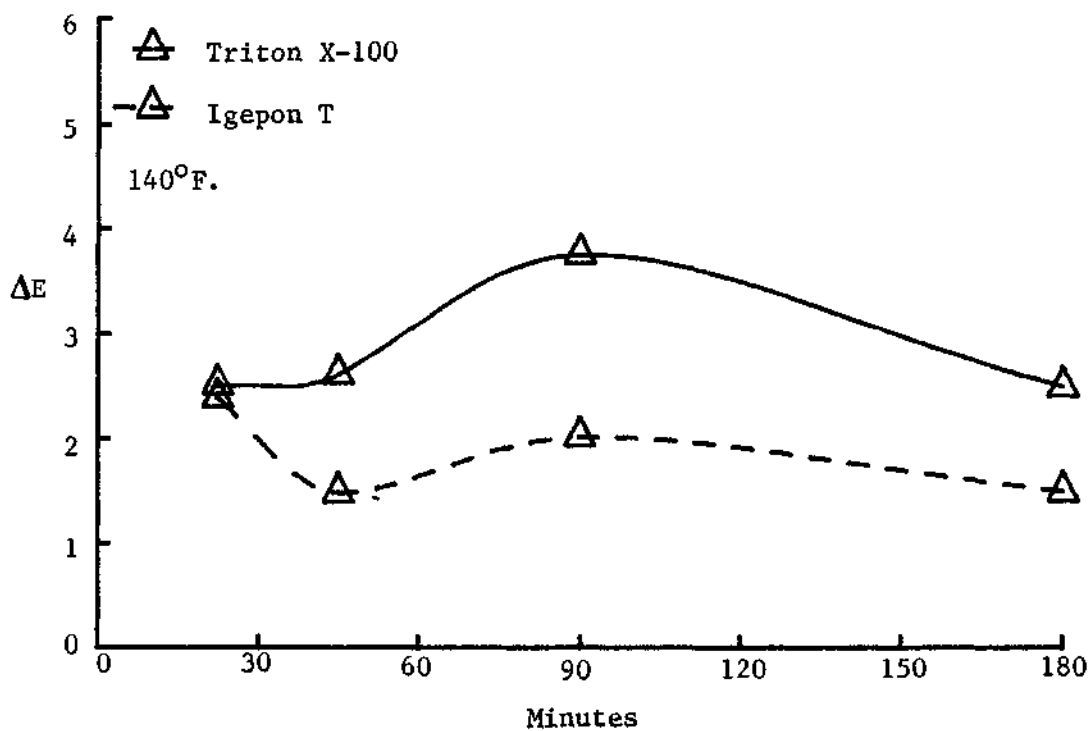


Figure 17. Washfastness of C. I. Disperse Blue 27 to 0.25 Per Cent Surfactant Concentration and Time-1.

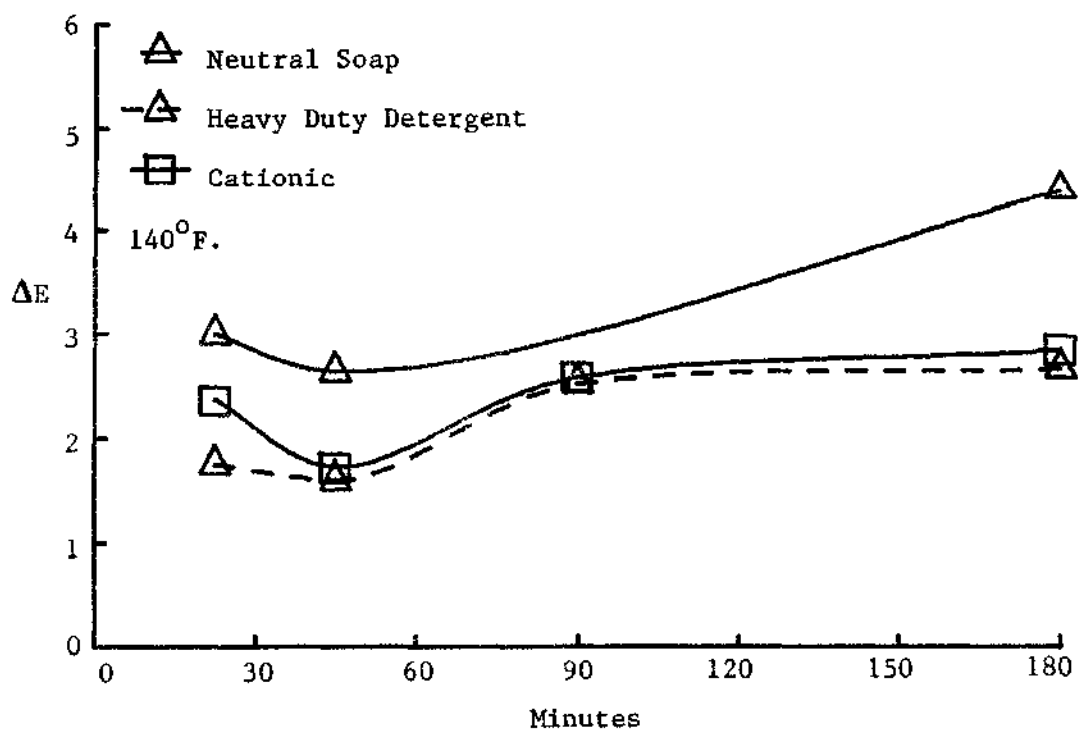


Figure 18. Washfastness of C. I. Disperse Blue 27 to 0.25 Per Cent Surfactant Concentration and Time-2.

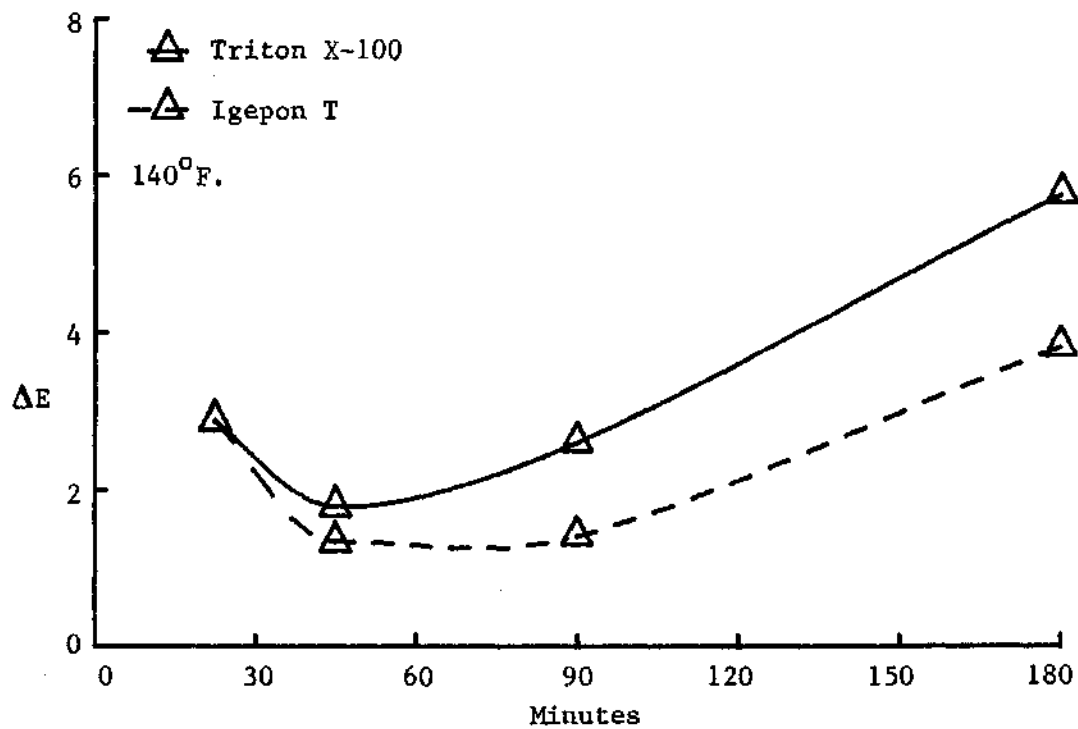


Figure 19. Washfastness of C. I. Disperse Yellow 3 to 0.25 Per Cent Surfactant Concentration and Time-1.

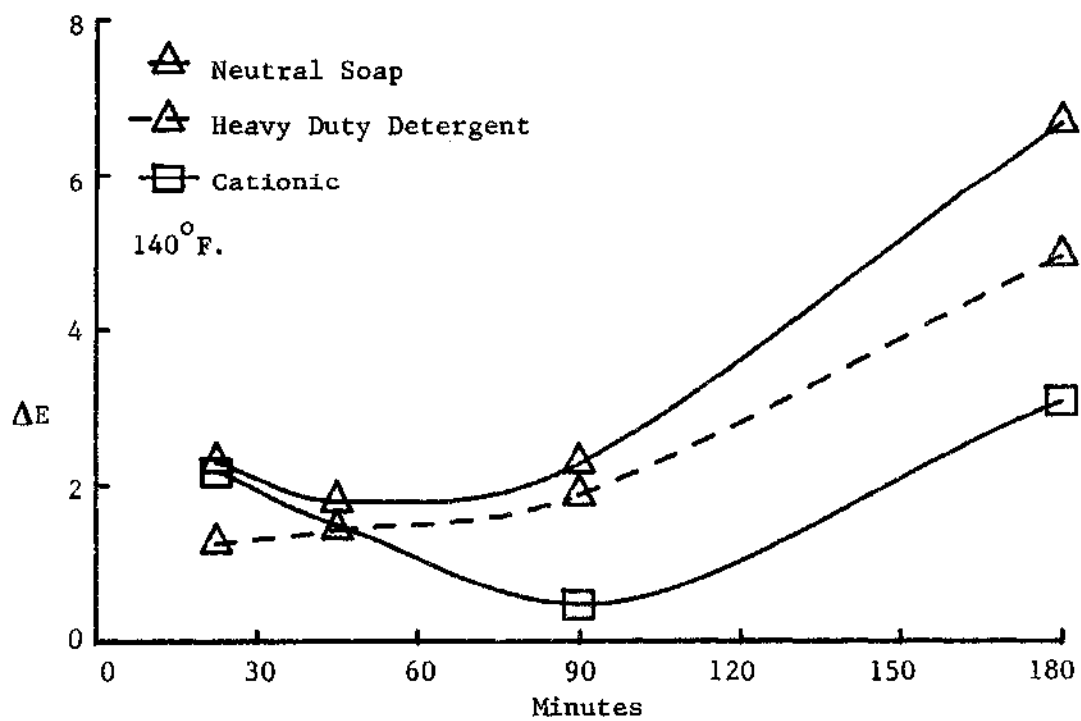


Figure 20. Washfastness of C. I. Disperse Yellow 3 to 0.25 Per Cent Surfactant Concentration and Time-2.

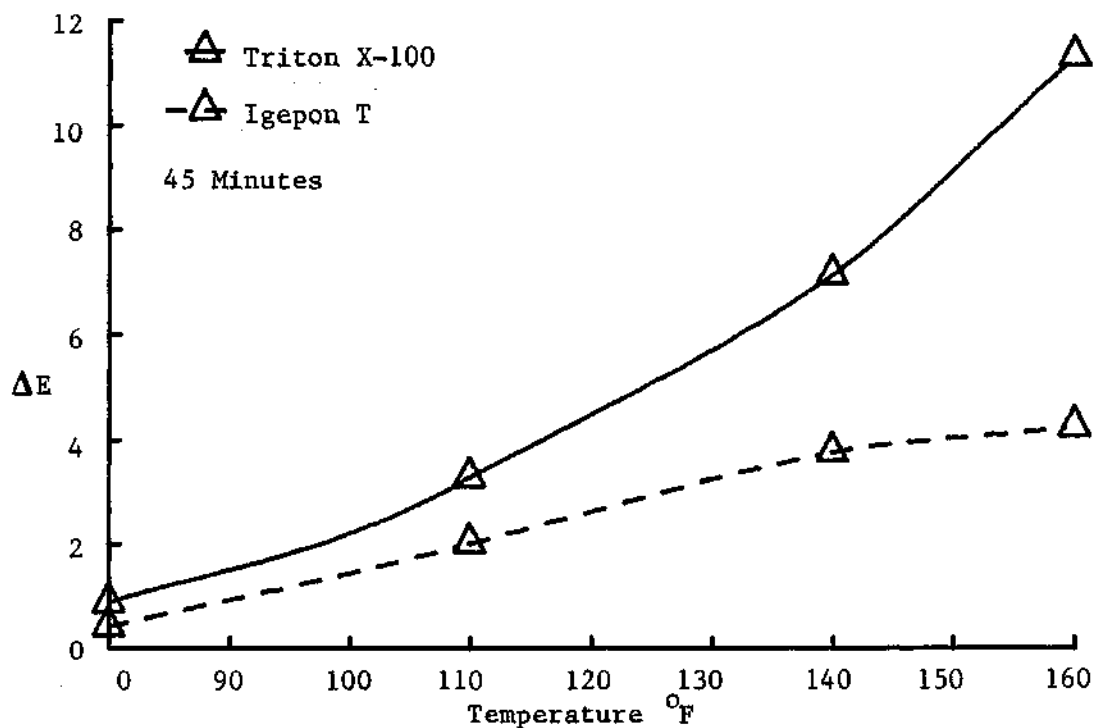


Figure 21. Washfastness of C. I. Disperse Red 15 to 0.25 Per Cent Surfactant Concentration and Temperature-1.

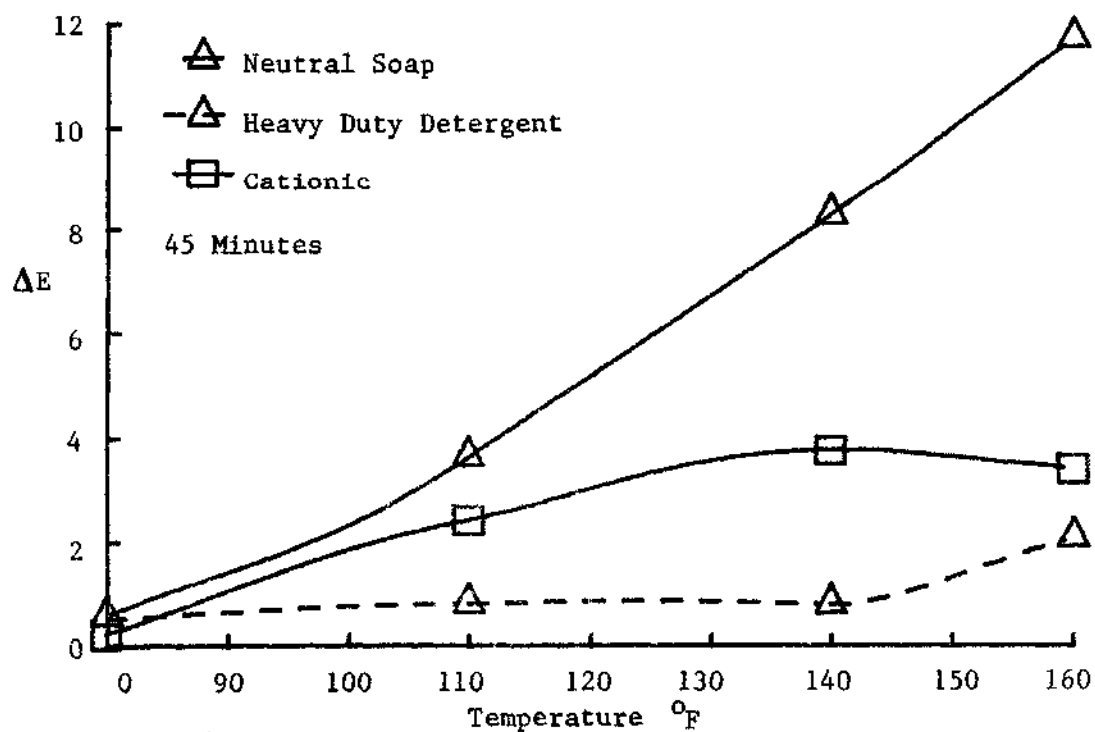


Figure 22. Washfastness of C. I. Disperse Red 15 to 0.25 Per Cent Surfactant Concentration and Temperature-2.

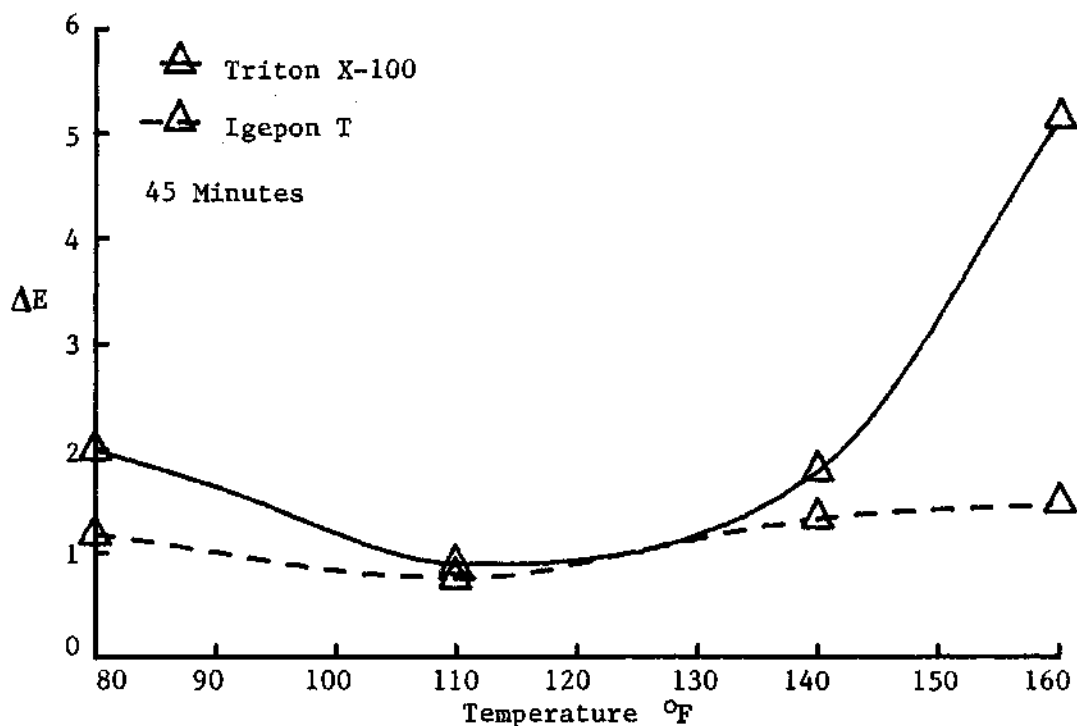


Figure 23. Washfastness of C. I. Disperse Yellow 3 to 0.25 Per Cent Surfactant Concentration and Temperature-1.

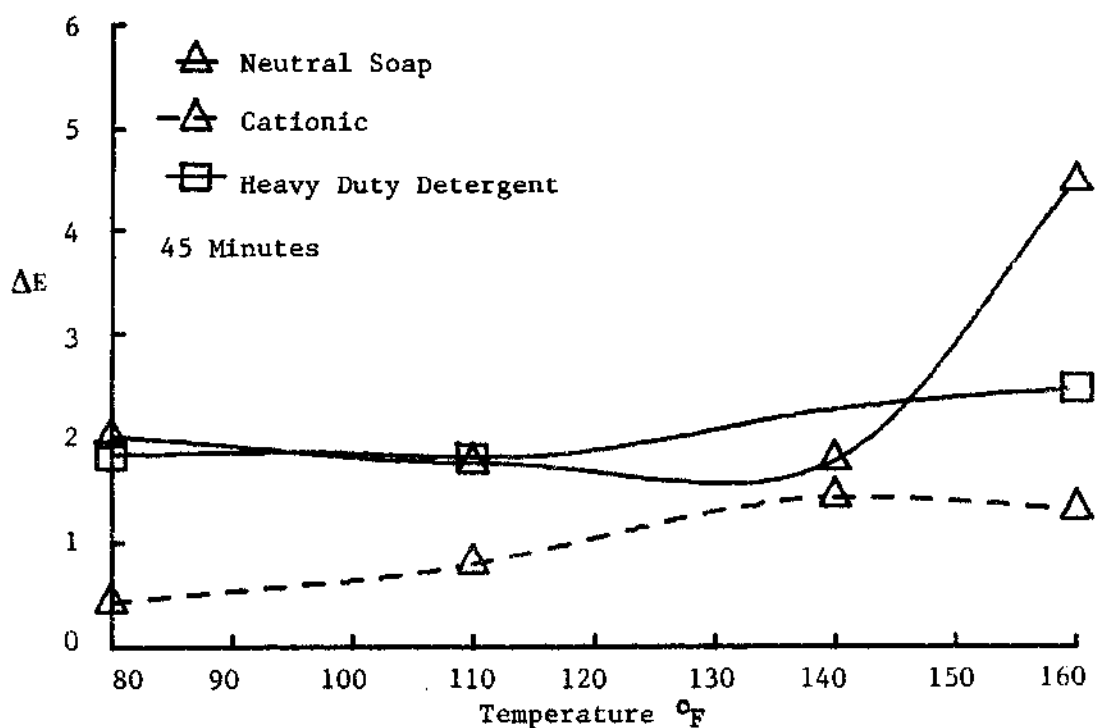


Figure 24. Washfastness of C. I. Disperse Yellow 3 to 0.25 Per Cent Surfactant Concentration and Temperature-2.

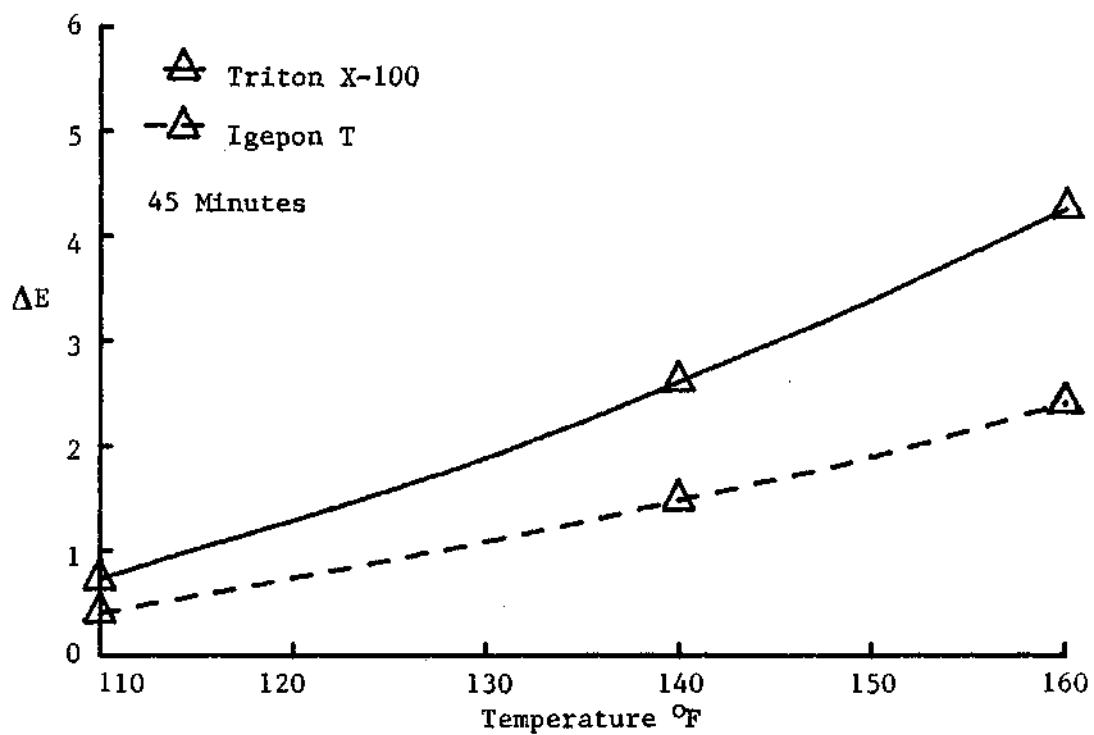


Figure 25. Washfastness of C. I. Disperse Blue 27 to 0.25 Per Cent Surfactant Concentration and Temperature-1.

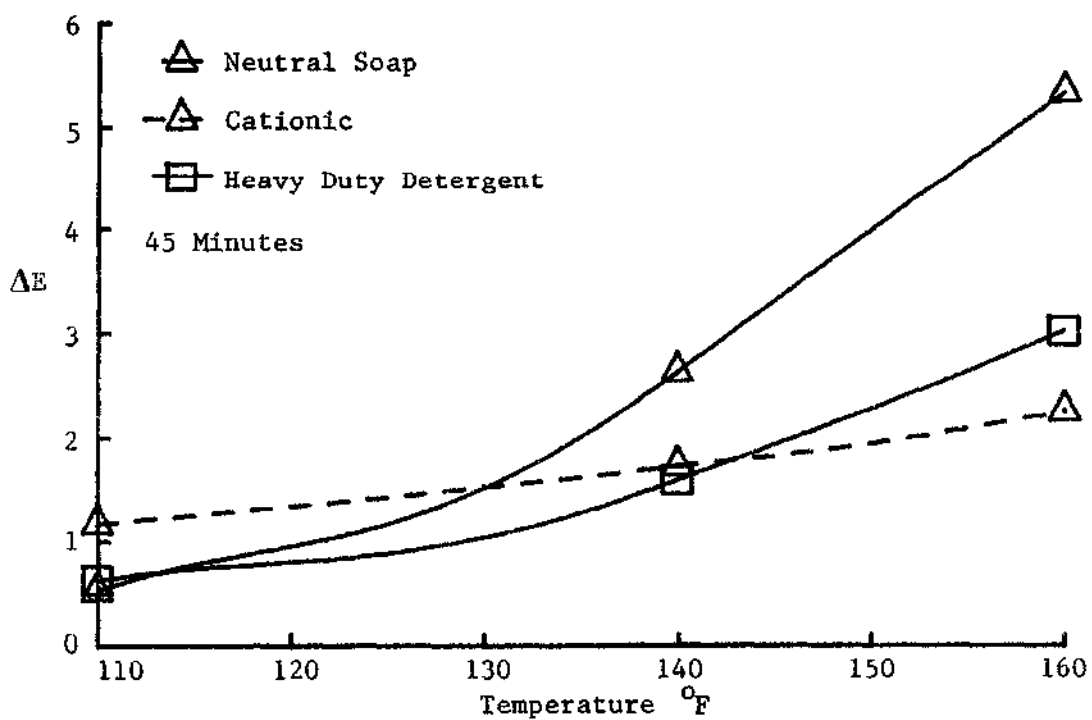


Figure 26. Washfastness of C. I. Disperse Blue 27 to 0.25 Per Cent Surfactant Concentration and Temperature-2.

Table 6. pH of Surfactant Solutions Measured at Room Temperature

Description	Concentration Per Cent ows					
	.0625	.10	.125	.25	.5	1.0
Neutral Soap	9.39	9.57	9.50	10.16		
Igepon T	6.78	6.68	6.69	6.67	6.55	
N-cetyl N-ethyl Morpholinium Ethosulfate	4.98	4.69	4.45	4.36	4.31	
Triton X-100	4.96	4.68	4.43	4.19	3.97	
Heavy Duty Detergent	9.90	9.95	10.10	10.10	10.14	
0.25 Per Cent ows Triton X-100 and Na <sub>3</sub> PO <sub>4</sub>		11.45	11.73	11.95	12.14	
0.25 Per Cent ows Triton X-100 and Na <sub>2</sub> CO <sub>3</sub>		10.61	10.80	10.89	10.95	
0.25 Per Cent ows Neutral Soap and Na <sub>3</sub> PO <sub>4</sub>		11.08	11.42	11.80	11.94	
0.25 Per Cent ows Neutral Soap and Na <sub>2</sub> CO <sub>3</sub>		10.56	10.88	10.97	11.25	
0.25 Per Cent Cationic and CaCl <sub>2</sub>		4.72	5.13	5.30	5.97	
Na <sub>3</sub> PO <sub>4</sub>			11.72			
Na <sub>2</sub> CO <sub>3</sub>			11.15			
CaCl <sub>2</sub>			6.60			



Table 7. Composition of Heavy Duty Laundry Detergent

Description	Per Cent Composition
Linear Tridecyl Benzene Sulfonate	10.1
Neodol 45-11	2.0
Soap (80/20 Tallow/Coco)	2.0
Carboxy Methyl Cellulose	.5
Sodium Tripolyphosphate	34.3
Polyvinyl Alcohol	.2
Sodium Silicate	7.1
Sodium Sulfate	33.8
Water	10.1
	<u>100.1</u>

Table 8. MacAdam's Color Differences of Fabric Washed With 0.25 Per Cent of Selected Electrolytes for 45 Minutes at 140°F.

Electrolyte	MacAdam's Color Difference		
	C. I. Disperse Red 15	C. I. Disperse Blue 27	C. I. Disperse Yellow 3
Trisodium Phosphate	11.80	1.13	2.31
Sodium Carbonate	8.35	1.70	2.18
Calcium Chloride	3.78	0.44	0.39

Table 9. Fastness of Specimens Washed for 45 Minutes at 140°F. in Distilled Water Compared to the Unwashed Fabric.

Sample	MacAdam's Color Difference		
	C. I. Disperse Red 15	C. I. Disperse Blue 27	C. I. Disperse Yellow 3
1	2.94	0.78	0.37
2	2.68	0.67	2.29
3	2.66	0.22	0.74
4	3.88	0.90	0.30
5	2.88	0.26	0.94
6	2.52	0.80	1.11
7	3.66	0.42	0.50
8	2.47	0.46	1.01
9	2.75	0.19	0.96
10	2.01	0.68	1.05
$\bar{X}$	2.85	0.54	0.93
S	0.53	0.25	0.53

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