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13. ABSTRACT (Maximum 200 words) This report lists the approaches taken to improve the properties of color filter materials for Flat Panel Display applications. Significant developments include a high saturation blue dye with high temperature stability; a purification process for a black dye used to replace a combination of blue and brown dyes for a new black matrix material with increased optical density with specific application for the liquid crystal display industry; the development of a rapid thermal curing technique to allow for higher dye loaded materials; and the use of a higher transparency polymer in the blue formulation to improve peak transmission properties.				
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FINAL TECHNICAL REPORT

of

"Advanced Development of Polyimide Color Filters for
Liquid Crystal Display Applications"

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Task Objectives & Technical Problems

The task objectives for this contract are to:

Develop polyimide color filter products with more desirable color properties, greater peak transmissivity, and improved stability during the cure process.

Technical problems: Using the criteria of high solvent solubility and high purity of the colorant (no fillers or metal ion contaminants), an expanded dye search was undertaken to find new colorants that could be incorporated directly into the PiC (polyimide color filter) matrix, or modified via purification, chemical modification, or direct synthesis. The general class structures of dyes to be searched were azo, triarylmethane, xanthene, azine, oxazine, thiazine, and anthraquinone. The color goals were to find a series of colorants to provide a greater blue transmission ratio (T_{450}/T_{520}), and blue and yellow components to be combined into greater green transmission ratios (T_{535}/T_{500} and T_{535}/T_{590}). Red formulas are in general acceptable but have an undesirable transmission in the green range (T_{610}/T_{535}) in film thicknesses less than 2 microns. Black matrix formulas may always be improved by increased absorbance across the visible wavelength range.

Problems encountered during the cure process on a case-by-case basis include: interaction of the dye components between layers (staining or leaching), cracking or adhesion loss of films, and thermal degradation or color shift of dyes or polymers upon repeated bake cycles.

These problems are to be addressed in the following ways: expansion of the dye searches to include categories previously not considered (possibly requiring chemical modification or stabilization techniques to improve solubility or alter the color), purify the

existing dyes to improve color strength, develop higher clarity polyimides to allow for improved blue color filter stability and transmission, and develop new curing processes and systems to be less abusive to the dyes.

Improve Storage Stability/Product Reproducibility

Technical Problems: Stability problems may be lumped into two categories: film stability with respect to heat, light or chemical resistance, and solution stability with respect to either particle formation (usually via slow recrystallization in heavily dye loaded systems), or film thickness changes with time, related to viscosity changes of the polymer.

Dye-related improvement paths involve structural changes to improve the heat and light stability of certain compounds, and purification techniques to remove destabilizing filler materials that may promote crystal growth and hinder filtration. Polymer-related paths include steps to stabilize viscosity and improve repeatability via mole ratio adjustments, endcapping, further raw material characterization, and add-order studies that would not only be useful for existing color filters but for higher clarity derivatives as well.

Implement New Materials and Process for Advanced Polyimide Color Filter Production

The goal is to translate all of the improvements through scaleup procedures and into standard products. Technical problems will differ on a case-by-case basis.

Gain Compatibility with Advanced Coating Methods

We intend to investigate the possibility of applying the PiC materials by such methods as roller coating, meniscus coating, flexographic or other printing means, inkjet deposition or other novel methods.

Technical Problems: The viscosity of the PiC products and the drying rate of the solvents are not in the range usually needed for these methods (they are too thick and slow drying). We will determine if the existing materials can be modified to fit these systems, and if not, how the polymer and colorants can be made compatible with the solvent systems typical of each application method.

General Methodology

A review of the Color Index, contact with dye manufacturers and subsequent literature searches on color/structure relationships led to the following:

Given that the current PiC curing temperatures are generally 230°C for 1 hour in a convection oven we used this as a stability baseline. Since colorants must hold up to multiple bake steps to produce a LCD (due to downstream processing), thermal stability was given primary importance, along with the ability to match a desired spectrum in a reasonable concentration. In general, the metal azo dyes were found to be the most useful based on stability and solubility, and also on the range of available colors. Phthalocyanines, azines, and anthraquinones were judged to be the next most useful given their relative stability and blue transmission properties. Thiazines, oxazines, xanthenes and triarylmethanes were known to provide more strong and unique color properties, but were also likely to require some modification method to stabilize them against heat or light degradation.

From laboratory experiments and literature, it was also known that optical clarity of the PMDA/ODA system was not adequate to allow for improvement in blue transmission (brightness) regardless of colorant selection. Darkening over successive cure cycles was the main criterion for selecting clearer polyimide systems. In addition, a polyimide system that could be fully cured at a lower temperature, and/or possessing a dense enough structure to prevent next-step color leaching under the lower cure cycle, would open up possibilities for new colorants. This would also logically require lower temperature post-processing steps. Taking this into consideration, we chose 190°C as the target temperature for lower temperature cure cycles. Laboratory experiments in the past had also shown that a denser cure structure could be achieved by use of higher temperatures, and if such a cure cycle produced a film of sufficient leach resistance, it would be useful in allowing for thinner films of the same color property (and theoretically improved resolution), or more robust systems in general.

The method for determining useful dye colors was based on liquid absorbance in the formulation solvents (generally NMP), and computer modeling to determine if that color could be useful as a component in a color formulation. Water soluble dyes were similarly screened in water to see if they had color properties worthy of conversion to a solvent-soluble species. Second level testing was to cast these dyes in a cured polyimide film and retest the color properties, using computer modeling to determine the proper colorant loading to achieve the desired film thickness.

It was decided that solvent extraction would be the most practical method for off-line dye purification, and ion exchange methods for in-situ purification. TLC methods were used to determine effectiveness of purification of dye powders.

FTIR (Fourier Transform Infrared), DSC (Differential Scanning Calorimeter), GPC (Gel Permeation Chromatograph) and viscosity analysis were used to determine polymer improvements and to study cure-related properties of the polymers. Spectrophotometric measurements of the polymer films without dye were used for optical comparisons.

Interactions between color filters (leaching and staining) as a function of cure cycle and dye loading were tested by use of a "color wheel" - successive patterning of RGB colors on a 3" glass round, with large geometries that allow for spectrophotometric color measurement at each stage of the 3 color process. With at least one color set, downstream compatibility was tested by producing sample plates at typical resolution, then processed through a planarization and ITO deposition process to check for mechanical and adhesion problems. The materials passed this test.

Technical Results

Summary of Progress on Task Objectives

Expanded Dye Search/Purification of Dyes/Chemical Modification of Dyes

- A list of the dyes that were investigated follows, by color and dye classification, in Appendix A. By structure classification, the results were as follows.

Azo dyes - Most of the azo dye improvements were made via purification or chemical modification, to be used in red and black formulations. Many reds, oranges, and yellows were screened but suffered from solubility problems due to poor purity, weak color strength, and insufficient spectral matches. Yellow dye structures also tended to suffer from too high of a solubility in standard base developer solutions, which resulted in poorer lithography properties. One orange dye was identified that produced a red film of improved brightness when mixed with other red and yellow dyes. This particular dye combination has become a standard product with improved room temperature stability (Red 101) which is in customer use as of mid-1995. See Figure 1 for color comparison. A supply issue required us to develop a backup orange dye consisting of conversion/purification of a sodium salt orange dye to a solvent soluble form. This material was identified and verified, and is ready to be incorporated into Red 101 if supply issues cannot be resolved.

As supplied, all black azo dyes required some purification or modification due to their filler content. Successful modifications were made in converting a sodium salt to the purified acid form, resulting in a purer black dye that provides good absorbance from 400-650 nm when formulated as black matrix (transmission of less than 0.7% in a 2 micron cured film - see Figure 2). This OD and absorbance profile are suitable for many display applications. Thus

this material was turned into a black matrix product which is in customer evaluation as of mid-1995.

Purification of a brown azo dye used in the existing black matrix products resulted in improved solution stability via reduction of particle formation over time, but could not be done repeatably. This work was ongoing as of mid-1995.

One combination of a yellow azo and blue phthalocyanine dye exhibited unexpected absorbance in the near-IR upon cure, but this phenomena could not be repeated and was thus abandoned.

Triarylmethane (TAM) dyes - TAM dyes were studied extensively as they are known to possess the desired blue spectral properties. Among the triarylmethane dyes studied were Acid Violet 17, Basic Blue 7, Basic Blue 8, Basic Blue 55, and Acid Blue 83. The dyes passing initial solution color tests were dissolved in polyimide solution, cast as a film, and cured to get baseline thermal stability and potential cure temperature. Of these dyes, only a few survived beyond 130°C. Addition of Barium and Calcium salts to these solutions in an attempt to form an in-situ pigment were unsuccessful. Most attempts at chemical modification destroyed the chromophore. The most useful triarylmethane dye combination was a coprecipitation of Basic Blue 7, (TAM with chloride anion) and Acid Violet 17, (sodium salt) to produce a higher molecular weight, highly colored compound. This dye achieved thermal stability of 190°C in a typical oven cure, allowing for adequate cure of future generation polyimides, and was capable of withstanding 210°C for limited time hot plate curing cycles. See Figure 3.

A series of xanthene dyes were studied, primarily rhodamine derivatives as red dye additives. Solvent Reds 43, 48, and 49, as well as Acid Red 94, are examples of these dyes. While providing the sharpest slope possible between red and green transmission, all of the xanthene dyes suffered from either thermal instability, light instability, unwanted fluorescence, or incomplete absorbance in the

blue and green ranges in usable concentrations. We were unable to overcome this with any purification or stabilization technique. No useful xanthene dyes were found.

Azine/oxazine/thiazine dyes - Dyes in this category, primarily blues, did not have a sufficiently high transmission ratio and were not studied further. Thermal stability was also a problem in this class. However, it became necessary (due to supply problems) to synthesize a replacement dye for an azine dye in the existing blue formulas by converting an acid dye to a solvent soluble version. This replacement dye was identified and scaled up.

Anthraquinone dyes - Several acid blue anthraquinone dyes were studied, including Acid Blues 7, 25, 40, 41, 78, 102, and 145. As supplied these dyes did not have sufficient color purity or thermal stability. Chemical modifications of this dye class allowed for sufficient solubility but did not show sufficient color strength to provide an improvement. These paths did not produce any directly useful materials.

Phthalocyanine dyes - some chemical modifications were attempted on commercial phthalocyanine dyes, primarily focusing on solubility improvement. Reaction at the sulfonic acid sites with long chain fatty acids and similar materials improved solubility somewhat, but did so at the expense of color strength. These paths did not produce any directly useful materials.

Customer requests for a subtractive (cyan/magenta/yellow set) allowed for an expansion of the dye search. Suitable cyans and yellows already existed as components of the RGB set. Xanthene structures were investigated as the magenta component, but were too unstable with respect to heat and light. A commercial dye (Orasol Pink 5BLG) was found to have proper color and borderline temperature stability. This dye is under study for possible formulation into a magenta PiC.

In some cases where an interaction was thought to occur between polymer and dye, primarily in blue and brown dyes, attempts were made to modify the structure of the dye to neutralize the reactive site. Results were mixed. In one case, purification of a brown dye was achieved via alternate ion-exchange methods, resulting in a great increase in stability. See Figure 4 for aging data. As of mid-1995 we are testing the repeatability of these results.

Developing Higher Clarity Polyimide Matrices

The first polyimide matrix to be studied was 4APS-BTDA. This polymer was compared to the standard PMDA-ODA polyimide system in PiC color filters, and was also used as the vehicle to evaluate blue dyes (to eliminate polymer darkening variables). Under successive cure conditions designed to simulate LCD manufacture, the improvement in clarity was dramatic. This allowed rebalancing of blue dye ratios to occur and allowed us to eliminate the use of a dye that was under scrutiny for supply problems. See Figures 5 and 6 for spectral comparisons. This matrix proved to be very useful in assessing blue dye improvements, and is being incorporated into future generations of high saturation/high transmission blue formulas as of mid-1995. Several other high clarity polyimides are under investigation including p-phenylene diamine + BPDA, p-phenylene diamine + OPDA, 4,4' diaminobenzanilide + OPDA. While they show high clarity they have not all been evaluated in color filter formulations, and those that have been tested in blue formulations have shown leach characteristics.

Several commercially available polyimides were tested as potential matrices, including photoimageable materials from Nissan Chemical, and Dow Chemical. They were all either incompatible with a system component (solvent or dye), or did not have sufficient leach resistance as a cured film.

Developing Less Severe Cure Processes for Polyimide Color Filters

Use of hot plate curing processes were useful in limited cases, particularly for triarylmethane blue formulas. Stability of TAM formulas up to 210°C for a 15 minute cure cycle was shown under ambient hot plate curing conditions (as shown in Figure 3) but it is not yet proven how uniform this will be using typical LCD substrate sizes.

Rapid thermal curing of PMDA-ODA based formulas in a typical convection oven produced a more leach resistant film in a red color filter, presumably via producing a denser encapsulation structure. A Red 101 processing cycle of 280°C for 10 minutes (vs. 230°C for one hour) produced a dramatic change in leach properties. Work is underway as of mid-1995 to see if this technique can be translated to large scale batch processing, or whether in-line processing is required. This property did not translate to all colors, and was not fully evaluated in the high clarity polymer systems.

Microwave curing of the PiC colors and analogs were not investigated as it was unclear how this technique could be suitably applied to a production environment.

Improving Storage Stability/Product Reproducibility

Stability Improvements Related to the Polyimide Matrix

Endcapping of the PMDA/ODA polyimide system with phthalic anhydride (PA) in stoichiometric ratios did provide improvement in

base polymer stability (defined as viscosity changes over time). It was incorporated as a standard procedure for polyimide manufacture. These gains did not always translate into more stable PiC formulations, or repeatable viscosity stabilization.

Consistency was improved by modification of the addition rate of monomers, and better raw material control (quicker turnover and more detailed incoming inspection).

Stability Improvements Related to the Dye Components

The most important stability breakthroughs were described above, primarily with TAM dye stabilization and dye purification that minimized the need for potentially destructive ion-exchange procedures.

A path was investigated to reduce dye content and increase film thickness to achieve the same color, on the assumption that leach and stability issues were the result of dye loading. Customer feedback suggested they were willing to accept thicker films for suitable stability and color tradeoffs. This approach was successful in some colors but not in others. Current blue colorants were already too weak to allow for this, but this approach worked well for some of the purified systems (particularly the coprecipitated TAM blue dye). At thicknesses approaching 2 micron, cracking problems and resolution/lithography issues began to surface.

Addition of small amounts of acid (usually acetic) slowed aging (via crystal formation over time in solution) and improved filterability in some heavily dye loaded blue systems by helping to solvate one of the dye components. This procedure has been incorporated into some of the standard blue products.

Implementation of New Materials and Process for Advanced Polyimide Color Filter Production

The following new materials and processes have been incorporated.

- A coprecipitated blue dye for high saturation blue products
- Two purified dyes to replace existing dyes that had supply and purity problems
- One high clarity polyimide system that has produced brighter and more robust blue products
- One purified black dye that cut the transmission of a black matrix in half at the same cured film thickness.
- Newer polyimide manufacturing techniques resulting in repeatability improvements
- A rapid thermal curing process that improves leach resistance in high colorant-loaded systems

Gaining Compatibility with Advanced Coating Methods

Adaptation of Polyimide Color Filters to New Coating Methods

Dilutions of existing PiC color filters in their host solvents (NMP, cyclohexanone, diglyme) were made to produce a lower viscosity material suitable for roller and meniscus coating. These systems were tested and were capable of being coated but did not have suitable drying characteristics. As of mid-1995 work has begun on matching colorants and polyimide systems to drying solvents more suited to roller coating. Other coating methods included investigation of flexographic printing (in cooperation with RIT), and inkjet printing (with BCB replacing polyimide as the carrier). Flexography does not appear capable of the desired resolution. Inkjet research is still in progress as of mid-1995.

Important Findings and Conclusions

- Purification of dyes did lead to increased solution stability in some systems, and also enabled brighter colors in thinner films.
- Chemical modification to improve solubility and thermal stability of the dyes led to one unique color with improved thermal stability and several solution stability improvements, but was not applicable across all colors.
- Dyes and high clarity polymers were identified to produce higher saturation blue and red materials. These products are in transition to be used as standard products. Some questions still remain about stability of these systems with improved colors.
- Rapid thermal curing processes can be used in heavily dye loaded systems to produce greater leach resistance in heavily dye loaded systems. It is not yet proven how effective this will be in full scale production processes.
- A potential CMY set was identified and will be further studied if customer demand warrants it.
- A color wheel method was developed to more accurately test the interaction and effects of colors in a production environment.
- New, higher optical density black materials, and more stable existing formulas were made possible via dye purification.
- Alternate coating methods have some promise but are in the initial stages of feasibility. It is likely that solvent adjustments will need to be made.

Significant Hardware Development

The focus of this program was on materials development. As such, hardware development was not one of the program objectives. Had there been more promise shown by the alternatives to spin coating such as flexographic printing or meniscus coating, it may have been worthwhile to divert some funds to this area.

Additionally had rapid thermal curing or microwave curing been pursued further, they too may have warranted some hardware development.

Special Comments

When this program was conceived in 1991, there was a good deal of activity in the US centered around flat panel displays. Color filter technology for these displays was almost exclusively the domain of the Japanese. Brewer Science offered the only alternative outside of the US. Several US companies were manufacturing liquid crystal displays albeit on a small scale. The US Government was very active in supporting these efforts in order to maintain an indigenous US industry for supplying state of the art displays to the Military.

In the ensuing years, a change in both Government's philosophy coupled with a shrinking of the activities of the domestic display makers meant there was no volume market in the US for a domestic manufacturer of color filter materials or filters themselves. Thus, although fairly successful from a technical standpoint, the program failed to establish Brewer Science, Inc. as a major supplier of color filter materials

Implications for Further Research

There are several areas where further improvements in the performance of color filter materials is warranted.

- Improved overall transmission with more saturated color
- Increased thermal stability
- Increased light stability
- Less material wastage
- A single step process (.i.e. make the color filter resins themselves photo-sensitive)

The dominant technologies in the market place are photosensitive pigmented materials. At the beginning of this contract, pigmented materials had one major drawback. The light scattering caused by the pigment particles results in a depolarization of the transmitted light and a resulting decrease in display contrast. This is the main reason that Brewer Science pursued dyes as the colorants of choice for this technology. During the ensuing years, these manufacturers succeeded in reducing the particle size of the pigments used in their products to the point where diminution of contrast is no longer an issue.

Pigments have advantages over dyes in terms of heat and light stability. Also comparing available dyes and pigments for the manufacture of blue color filter materials, pigments offer a superior color. The interaction of photo-initiator systems with the remainder of the components in color filter materials is better understood and controlled in pigmented systems than in dyed systems. The flat panel display industry has now matured to the point where price rather than performance is the dominant driver. Thus ways to manufacture

color filters more cheaply are paramount. A major cost saving is the single step process. The materials cost of photo-resist is saved and throughput and yield are increased due to the reduction in process steps.

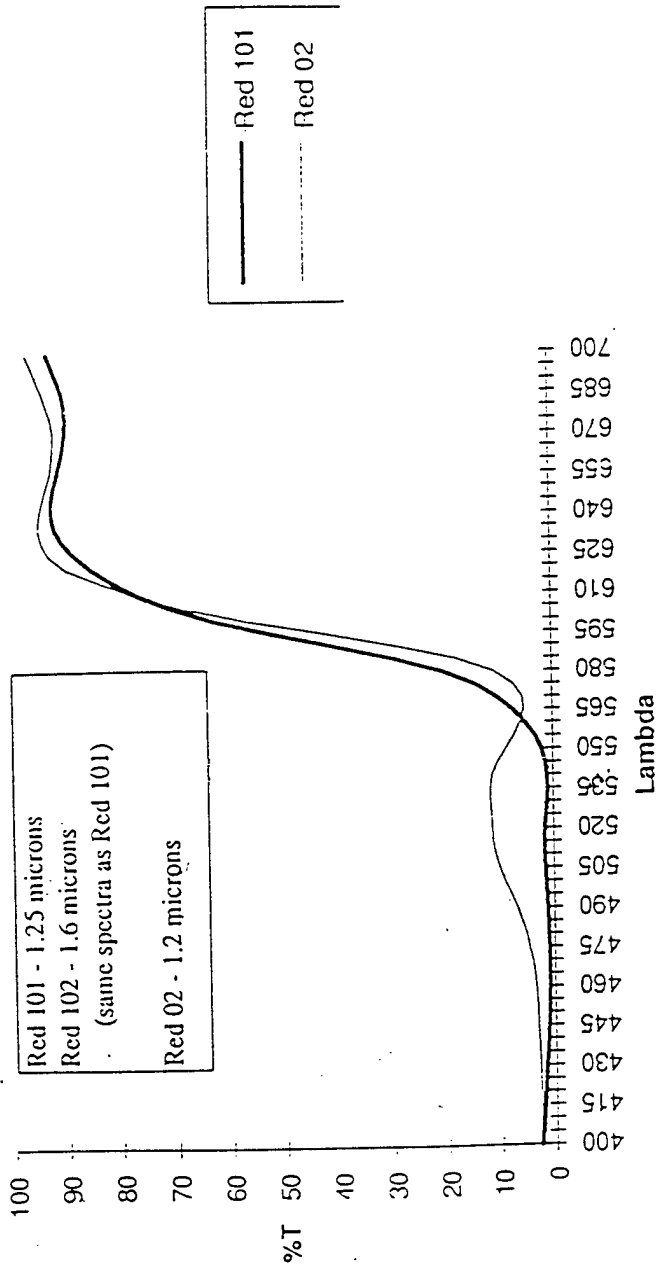
For the above reasons, more recent developments at Brewer Science have been targeted at pigmented photo-sensitive systems. However, we have been approached by one major manufacturer of color filter materials in Japan and several smaller interested parties because of our dye expertise. There is a perception in the industry, that the performance of pigment based systems has been pushed to its limit and that the next big breakthrough will come via dye technology.

An obvious way of reducing color filter cost is to reduce material usage. DARPA funded a program at Brewer Science to develop inkjet technology for this purpose. This program was terminated at the end of its second year. Meantime Canon have begun manufacturing color filter plates using ink jet technology. It is too early to say whether this technology will dominate the market.

Given the current situation, if a major breakthrough is to come about in the price and performance of color filter materials, it may well be the development of dye based photo-sensitive systems.

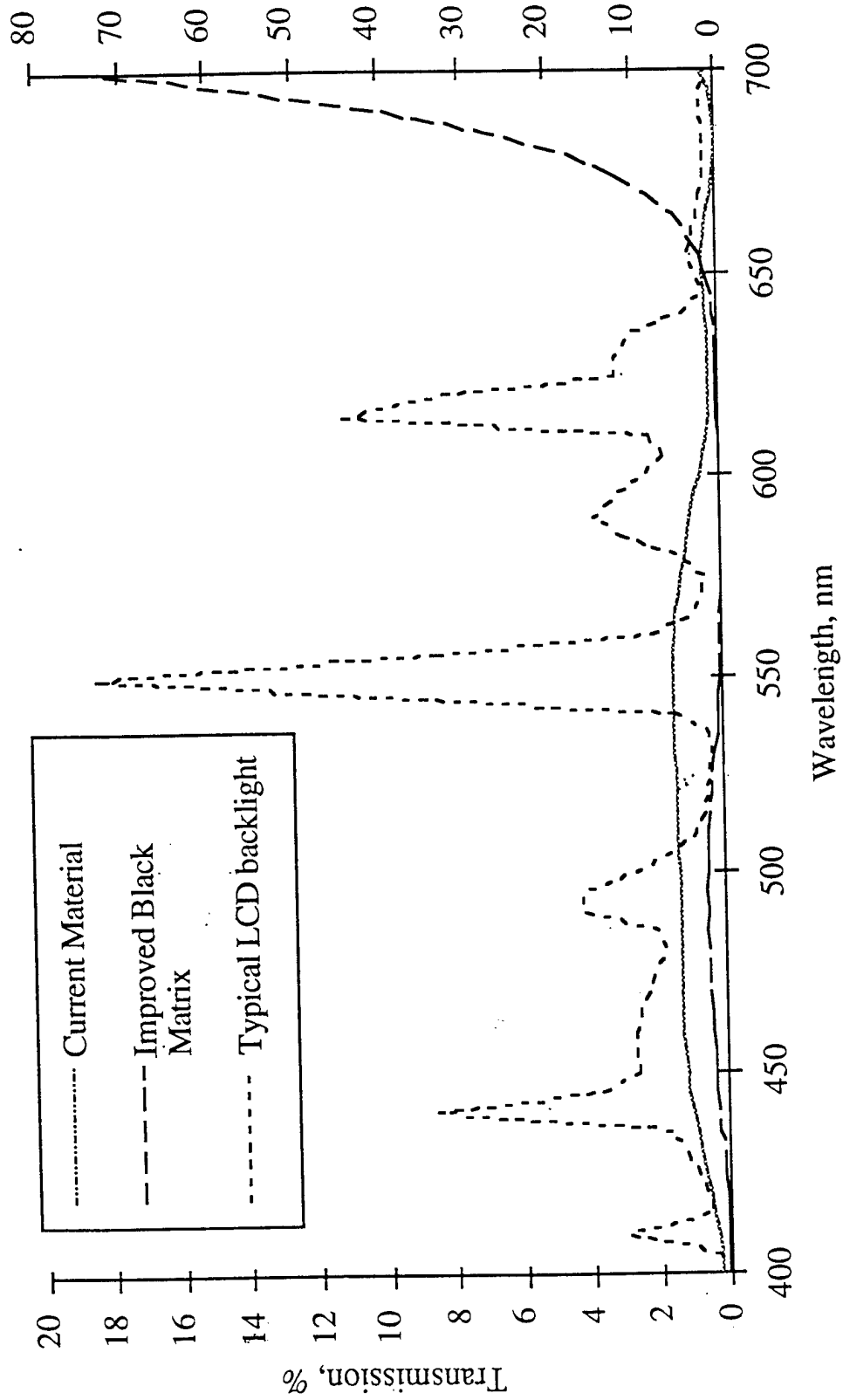
Figure 1. Red Color Improvement

Red 101/102 vs. Red 02



Brewer Science, Inc.

Figure 2. Improvement in Black Matrix Material



Brewer Science, Inc.

Figure 3. THERMAL STABILITY TEST OF DYE

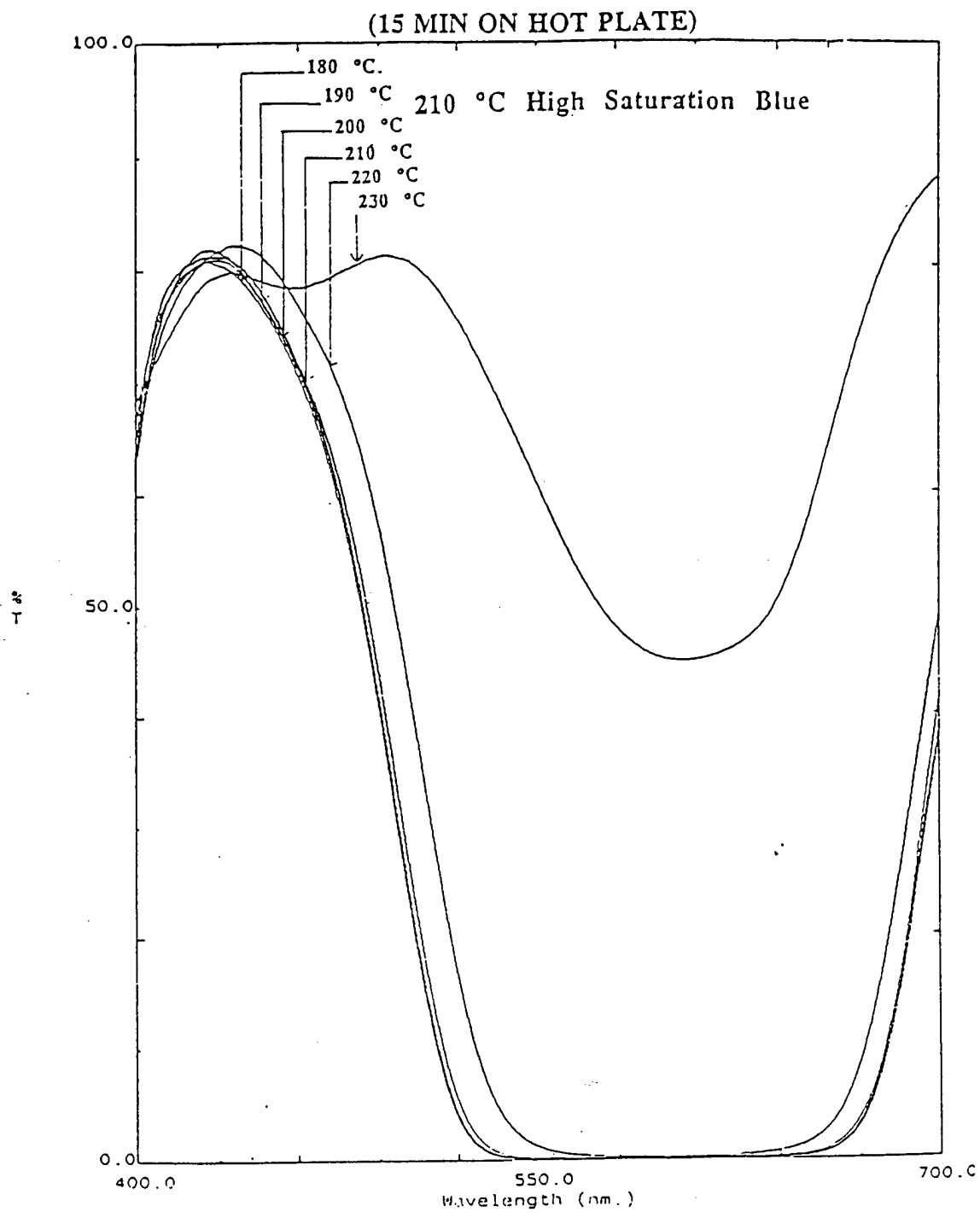
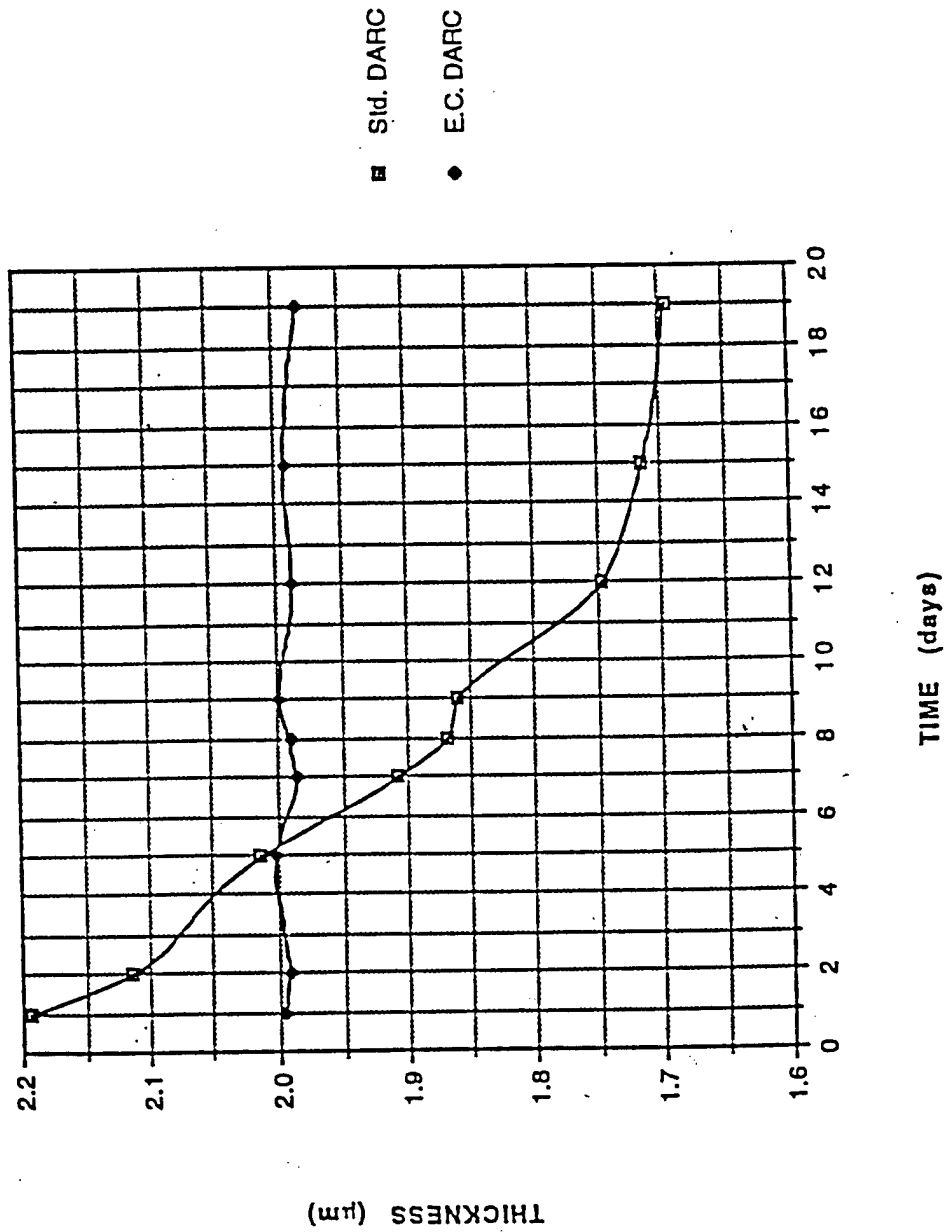


Figure 4. Shelf Life Improvement Through Dye Modification

Standard Orasol Brown 6RL vs Encapped Orasol Brown 6RL



Brewer Science, Inc.

Figure 5.
HIGH CLARITY POLYMERS FOR HIGH SATURATION BLUE

Conventional vs. Clear polymers
 Typical Spectra at Same Thickness

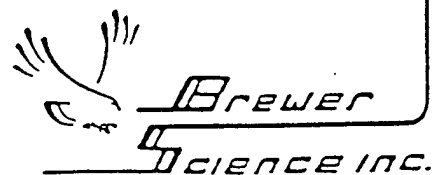
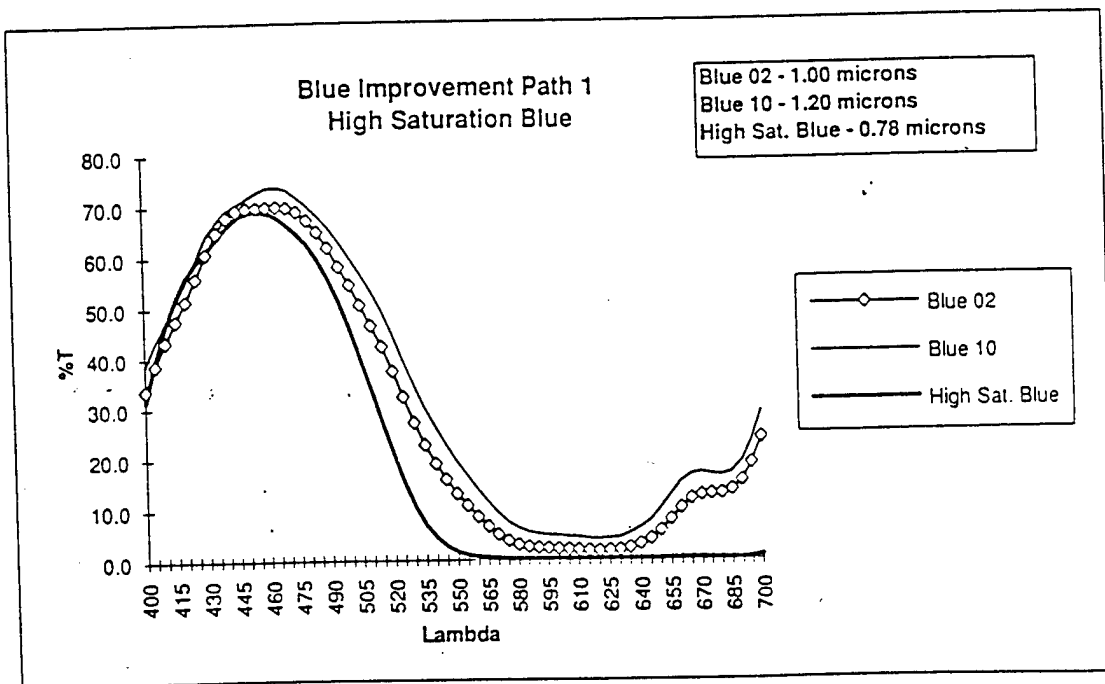
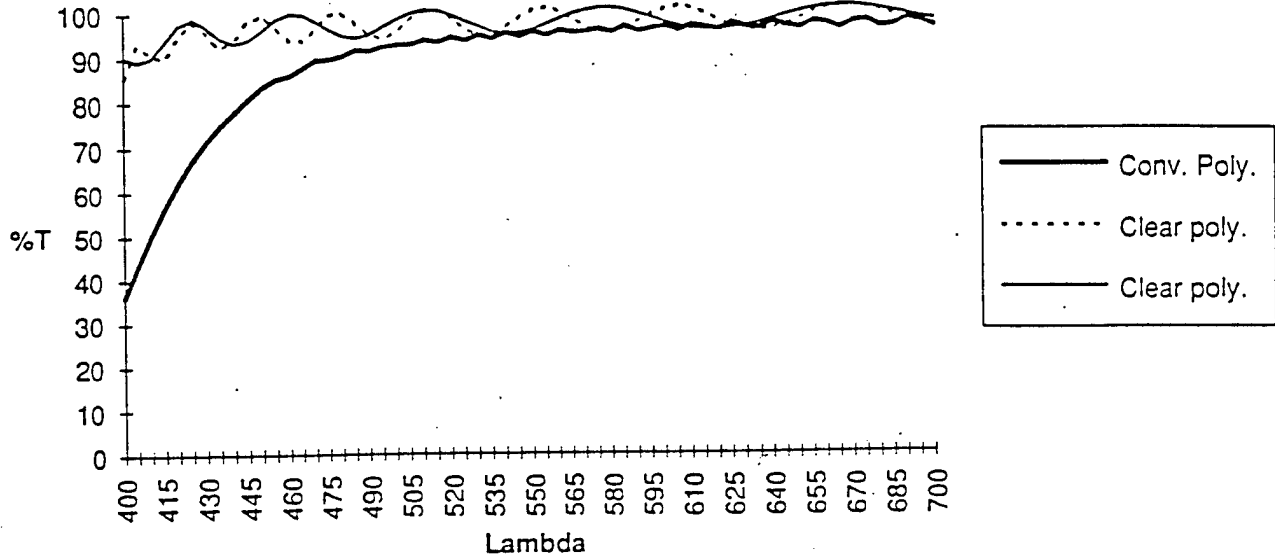
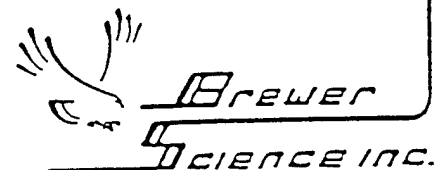
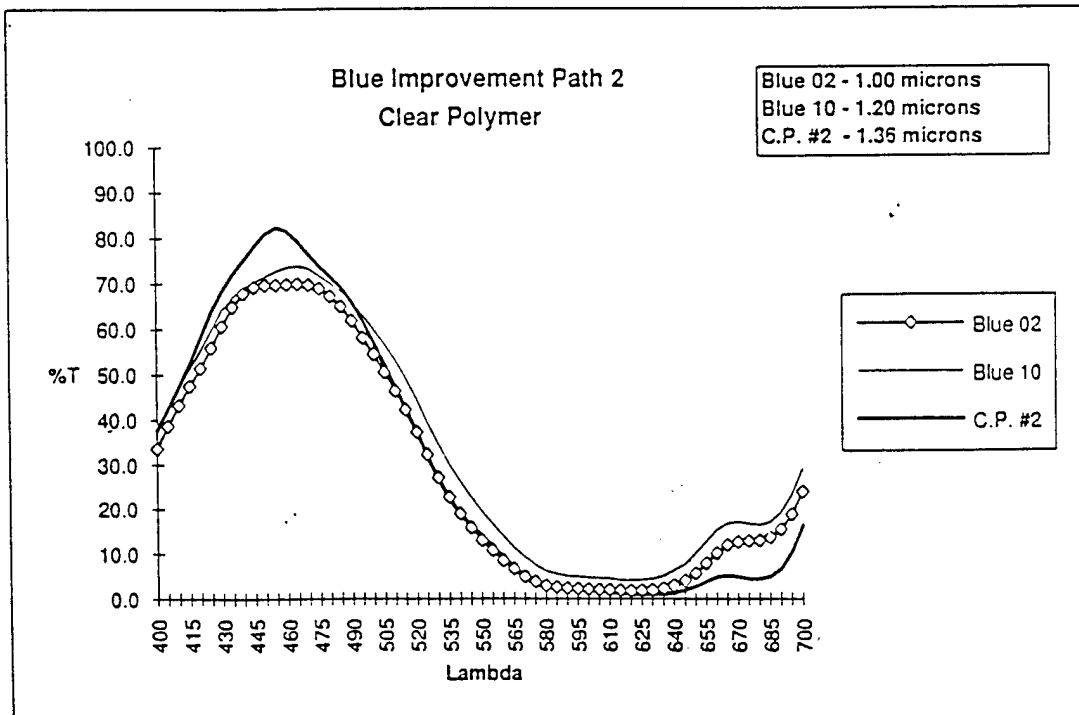
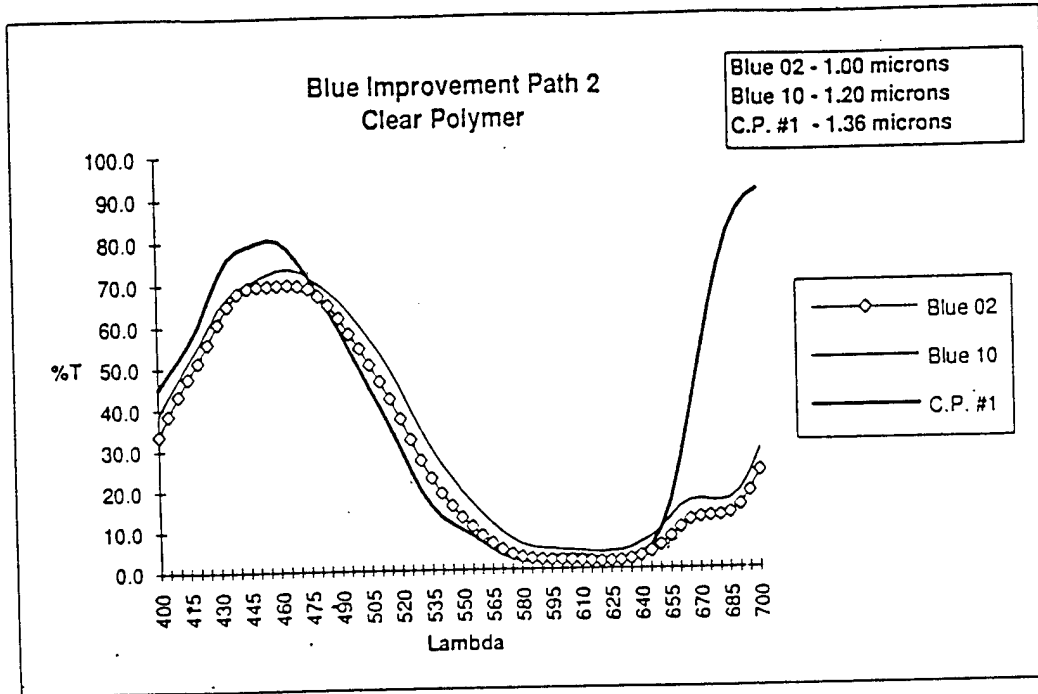


Figure 6.

HIGH CLARITY POLYMERS FOR NEW BLUE COLOR FILTERS



Appendix A

Dyes Studied Under this Contract

Blues

Acid Blue 1, 7, 9, 25, 40, 41, 80, 102, 145, 155, 158, 288

Basic Blue 7, 8, 9, 12, 16, 17

Direct Blue 14, 86, 108

Disperse Blue 1, 56

Food Blue 2

Mordant Blue 14

Pigment Blue 1

Reactive Blue 4, 5, 21

Solvent Blue 4, 5, 11, 14, 18, 21, 23, 25, 35, 36, 37, 38, 43, 44, 45, 48, 49, 51, 58, 59, 64, 67, 70, 81, 97, 98, 99, 101, 102, 104, 122, 128, 129, 132

Vat Blue 1

Violets

Acid Violet 17, 43, 49

Basic Violet 1, 3

Pigment Violet 23

Solvent Violet 2, 8, 13, 14, 23, 24, 36

Greens

Acid Green 12, 45

Solvent Green 3

Yellows

Acid Yellow 1, 3, 73

Basic Yellow 1, 2, 11, 13, 21, 37

Disperse Yellow 54, 64, 77, 93, 184, 201

Food Yellow 3, 4, 13

Pigment Yellow 12, 14, 17, 34, 147

Natural Yellow

Reactive Yellow 15, 42, 84

Solvent Yellow 13, 14, 16, 19, 21, 25, 29, 30, 33, 42, 43, 44, 56, 62, 72, 77, 79, 82, 83:1, 88, 89, 93, 98, 109, 138, 145, 146, 148, 160, 162, 163

Vat Yellow 33, 46

Oranges

Disperse Orange 47

Natural Orange 4

Solvent Orange 1, 3, 7, 11, 20, 23, 27, 41, 54, 56, 59, 60, 62, 63, 86

Reds

Acid Red 1, 2, 85, 94, 359

Disperse Red 159, 343

Food Red 14, 17

Natural Red 28

Solvent Red 8, 18, 19, 24, 43, 48, 49, 52, 68, 72, 74, 90, 91, 92, 106, 109, 111, 118, 119, 122, 124, 125, 127, 132, 138, 168, 195, 203, 204, 206, 207

Appendix B

Bibliography of Contract-Related Publications

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