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Introduction

This is a practical guide to the selection and straightforward quality assurance of phthalogen blue fabrics for attracting tsetse and biting flies. This information should be used in conjunction with the links at nzitrap.com below, and the fabric spectra presented in a companion document.

Nzitrap.com contains exhaustive details and references on appropriate products; only some of this is repeated here. I have also assumed a basic knowledge of the issues of interest.

Fabrics - General	http://www.nzitrap.com/Nzi_trap/Fabrics/Fabrics.htm
Blue Fabrics	http://www.nzitrap.com/Nzi_trap/Fabrics/Blues.htm
Blue Dyes	http://www.nzitrap.com/Nzi_trap/Dyes/Blue%20Dyes.htm
Fading / Weathering	http://www.nzitrap.com/Nzi_trap/Fading/Fading.htm
Dyeing Quality	http://www.nzitrap.com/Nzi_trap/Weaves/Weaves.htm
Netting	http://www.nzitrap.com/Nzi_trap/Fabrics/Netting.htm
Mesh Sizes	http://www.nzitrap.com/Nzi_trap/Fabrics/Mesh_Sizes.htm
Visual Ecology	http://www.nzitrap.com/Nzi_trap/Fabrics/Visual_Ecology.htm
Traps in Nature	http://www.nzitrap.com/Nzi_trap/Fabrics/Nature.htm
Exptl Fabrics	http://www.nzitrap.com/Nzi_trap/Fabrics/Experimental.htm
Links / Resources	http://www.nzitrap.com/Nzi_trap/Literature/Literat.htm

This guide is a personal perspective. I hope it will be of value to those facing the same technical issues I have faced over more than a decade in purchasing fabrics from an “opaque” textile industry. The industry rarely responds to requests for technical information, so it is typically impossible to obtain key facts about the nature of most retail fabrics. The industry is also so diverse that it is only possible to scratch the surface of what is available. Hence, the main purpose of this guide is to simply facilitate knowledgeable decisions, e.g. by showing where information can be found, or by showing how certain facts can be deduced from basic principles. To truly assure the quality of fabrics, one must know what questions to ask. Unfortunately, in a highly competitive market, informative answers from industry are rarely forthcoming.

The need for this guide came about as a result of many years of conducting standardized experiments with fabrics. This involved extensive collaboration with researchers on how tsetse and biting fly traps are being made around the world. Early on, it became obvious that there was little in common among disciplines/areas, and that there was minimal knowledge of why things were being done the way they were. As results accumulated, it became obvious that only genuine phthalogen blue fabrics were truly “safe” choices for nearly all situations. The biological reasons for this finding remain obscure, and are the subject of ongoing research (Mihok *et al.*, 2006). For some groups of flies, for some areas, or for some applications, fabric and colour selection may not be critical, e.g. for riverine tsetse (Okello-Onen *et al.*, 2004). However, the patterns of responses among species and groups are far from simple, and still require research.

Getting Started - A Basic Toolkit

To interact effectively with the textile industry, one must understand the materials, dyes and processes used to produce fabrics, as well as the way colours are described in the language of "colorimetry" (Nassau, 1998). As a minimum, one needs to read at least one introductory book that covers basic topics (Ingamells, 1993). To understand dyeing processes and options for cellulosics (e.g. cotton), I strongly recommend a more sophisticated treatment (Shore, 1995). The diverse methods for synthetic fabrics are also covered in a promising new textbook, which I have yet to see (Hawkyard, 2004). There are many other similar reference books (Aspland, 1997), but these are often too technical for an introduction to this topic. Unfortunately, useful books, and other resources, are difficult to find in academic libraries. To know what is available in the way of both books and journals, or to purchase useful materials, consult the web site of the Society of Dyers and Colourists (SDC) in the United Kingdom (http://www.sdc.org.uk/).

To make informed technical decisions, one should also have access to the Colour Index. This is the industry point of reference for the characteristics of fabric dyes. This multi-volume "encyclopedia" is now an electronic publication. It is available only by subscription from the SDC at a hefty price (450£ for comprehensive access for a single user for a one year licence). The historical, consolidated paper editions are now available on DVD, but also at a hefty price (500£). The older paper editions are held by relatively few libraries. In Canada, there are only two complete sets accessible to the public, and only in person. The Colour Index is supposedly up-to-date, but in reality, it lags far behind industry. New products may not appear for many years, and in some cases, technical details may never be disclosed. When industry discloses useful information, it is also often buried in obscure references, or is only mentioned in passing. A good example is the information on the nature of the Bayer "phthalogen blue" dyes for cotton. Outside of a brief summary in the Colour Index, the true details of the phthalogen dyes are only presented in one publication (Vollmann, 1971); this information is not available in any textbook.

Part of any basic toolkit is also the internet. There is a wealth of useful information on textiles and dyes on the web as industry does wish to sell and promote its products. Much of this is indexed at nzitrap.com. When library resources are scarce, it is also possible to learn a fair bit from general references, e.g. the Kirk-Othmer Encylopedia of Chemical Technology.

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Phthalogen Blue IF3GM Cotton

What is it?

The colour “phthalogen blue” is a generic term for the near “pure blue” that is produced by the chromophore copper phthalocyanine (CuPc) or Pigment Blue 15. The code “IF3GM” is the original Bayer abbreviation for a specific method for producing this colour. In the Colour Index, this method is referred to as Ingrain Blue 2:1. The nature of the chemical product (i.e. CuPc) is coded as C.I. 74160.

A “pure blue” is one that resembles our perception of blue in a “tristimulus” model of human visual perception (red-green-blue primary colours). The term “phthalogen blue”, “phthalo blue” or “thalo blue” should be a specific reference to Pigment Blue 15, but these terms are not always used properly. For the perfectionist, there is also no such thing as a phthalogen blue “dye”; the size and insolubility in water of CuPc itself precludes its use to “dye” fabric; CuPc is a pigment and not a dye. The historical Phthalogen Blue “dyes” from Bayer are properly referred to as “dye developers” or “ingrain dyes”. The Bayer method was a substantial innovation in its time; it involved the assembly of precursors of CuPc directly in the fabric. The method embeds layers of insoluble pigment in the fibre; this accounts for the superb brilliance and colour-fastness of the technique.

To make CuPc soluble for conventional applications, e.g. in solvent inks or in reactive or direct dyes for cotton/paper, the chromophore is typically modified by sulphonation; this changes the colour from blue to blue-green or turquoise/cyan. For example, the cyan component of the ink used in computer inkjet printers is typically a variant of CuPc. The large size and stability of the CuPc molecule also precludes its use in disperse dyes for polyester, which are dyed at high temperatures. However, CuPc itself can be used to directly colour some synthetics in “solution dyeing”. This is how phthalogen blue plastics, nylon, acrylic, etc. can be produced.

Who Makes the Dye?

The “phthalogen blue” methods for cotton were developed by Bayer (Farbenfabriken Bayer AG Leverkusen) and are described in Vollmann (1971). The technique, which uses special auxiliaries, is still maintained at Dystar in Germany (Textilfarben GmBH & Co. Deutschland KG in Frankfurt am Main, http://www1.dystar.com/index_textile.cfm). The phthalogen dyestuffs are still in commercial production, but are mainly used for tropical prints, or for exhaust dyeing of yarns or thread. Despite the brilliance and light-fastness of the phthalogen dyes, solid-shade dyeing of plain cotton was never a commercial success. Use of the technique died out after the 1970s as the global industry shifted to synthetics / blends, mostly produced in Asia.

In 1964, Dr. K.H. Gharda and Mr. R.M. Kawasmaneck reproduced the sophisticated chemistry required in India, and began manufacturing phthalogen blue dyestuffs for the retail market. Gharda Chemicals in Mumbai provided the phthalogen dyestuff used by Mount Kenya Textiles in Nanyuki, Kenya in the 1990s. Today, there are many sources of phthalogen dyes in India, and likely elsewhere in Asia. Two of the Indian manufacturers have been responsive to email requests and have sent me samples of their products.

Manibhadra Enterprise http://www.manibhadradyes.com/
Sitaram Chemicals http://www.sitaramchemicals.com/

Who Used to Make the Cloth?

The history of the phthalogen blue fabrics used by tsetse researchers in Africa is fairly straightforward, but is not clearly documented in the literature. In East Africa, three textile mills in Kenya in the 1980's and 1990's (Kicomi, Rivatex, Mount Kenya Textiles) were the source of most, if not all local fabrics. Except for myself, I am unaware of anyone pursuing the technical details of what was done. Hence, the only reliable point of reference is the use of dyestuff from India in the late 1990's by the only mill operational at that time (Mount Kenya Textiles). In central / southern Africa, Bonar Industries in Harare, Zimbabwe supplied fabrics to tsetse researchers. Bayer dyes were used initially in the 1980's, and researchers were aware of the textile mill involved (Glyn Vale, personal communication). In later years, Bonar provided "appropriate" fabrics. From spectra of samples from the 1990s, and from published spectra, the fabrics from Kenya and Zimbabwe were likely all genuine phthalogen blue IF3GM fabrics.

In West Africa, both phthalogen blue cotton and cotton/polyester fabrics were used in the 1980's and afterwards by researchers at organizations such as CIRDES and CIRAD-EMVT. Researchers used fabrics from a well-established textile mill, Ets. Gonfreville in Bouaké, Ivory Coast. It is probably safe to assume that French technical experts were involved in dyeing, and that Bayer dyestuffs from Germany were probably used, but this appears to have never been documented. As with fabrics from other areas in Africa, spectra from a few West African fabrics and from publications all appear to be genuine phthalogen blue. Up till recently, this factory has continued to produce a close match to phthalogen blue in polyester/cotton ("Santiago").

Who Still Makes the Cloth?

My practical knowledge of the current use of phthalogen blue dyestuffs by textile mills is limited to what I have been able to learn from personal inquiries to researchers, and from the few companies that have been kind enough to answer requests for information.

The good news is that the manufacturer of phthalogen blue dyestuffs in Germany still has one customer with practical experience in the use of Phthalogen Blue IF3GM for solid shade dyeing. TDV Industries in Laval France (http://www.tdv-industries.fr/en/produits/) can dye cloth in various weights/weaves for custom orders, with a minimum order of 1,000 m (1.5 m width of cloth). A large sample from a previous order was provided in May 2005 under the colour name of "Azur". The contact at that time was…

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Since Dystar still makes the dye, it is clearly the best source for current information on its use by industry. Dystar was very helpful in providing detailed technical information and fabric reference samples. The technical marketing manager of the phthalogen dyestuffs in May 2005 was Eilders Henning, eilders.henning@dystar.com, telephone 069 2109 25 96.

The many Indian sources of phthalogen dyestuffs indicate that there is a continuing market in the tropics where brilliant, light-fast blue cottons are still popular (e.g. for school uniforms in Africa). Unfortunately, the two dye manufacturers that have responded with fabric samples have not provided information on their export markets. This should be in their interest, but information has not been forthcoming. Nevertheless, I expect that any serious enquiries for purchasing large quantities of phthalogen blue fabrics would generate a useful response.

The bad news is that traditional sources of phthalogen blue cloth in Africa seem to have ceased operations. Some examples are:

In Kenya in 2005, a review (http://www.epzakenya.com/news.php?type=press&itemno=45) of the textile industry stated that none of the three textile mills in Kenya were operational, although a small-scale industry is still very active. I have obtained spectra from a few recent samples of blue cloth in Kenya; these samples have not been genuine phthalogen blue.

In Zimbabwe in 2003, Bonar Industries provided a "peacock blue" substitute for phthalogen blue cloth that was not genuine phthalogen blue. The person formerly in charge of supplying tsetse researchers with the proper cloth had retired and the new person in charge was either not well informed, or simply unable to provide the exact material required. From the state of industry in Zimbabwe, it is unlikely that any local phthalogen blue cloth is being produced.

In the Ivory Coast, due to civil unrest, French expatriates have left Ets. Gonfreville and the factory is now essentially not operating (Claude Laveissière, personal communication, 2006).

What are the Alternatives?

The brilliant blue of the phthalogen blue IF3GM dyestuff on cotton can be imitated in some fabrics. Hence there are retail alternatives that may be suitable substitutes. However, there is considerable risk in substituting other fabrics, especially for biting flies. Texture differences (shininess, polarized light) and ultraviolet absorbance properties can greatly affect how some species react to fabrics (Mihok *et al.*, 2006).

The best alternative to phthalogen blue cotton appears to be Sunbrella Pacific Blue awning fabric, a heavy-weight acrylic canvas that is almost surely solution-dyed with CuPc. This material is expensive, but it is worth the cost in terms of colour-fastness and durability. Its utility as a generic substitute will require confirmation against many species in many areas; so far it has only been tested on a few species in North America (Mihok *et al.*, 2006).

With the use of combinations of direct and/or reactive dyes based on anthraquinone and sulphonated CuPc, or the use of resin-bonded CuPc pigment, it is possible to obtain a visible colour match to phthalogen blue on cotton. Sulphonated CuPc reactive dyes only produce a deep greenish-blue or turquoise colour, which also appears to be suitable for biting flies, and possibly also for tsetse (Mihok *et al.*, 2006).

The nominal "phthalogen blue" fabric sold to many tsetse researchers by Awassa Textiles in Ethiopia in the 1990's is one example of the undocumented use of other processes to produce a colour nearly indistinguishable from phthalogen blue IF3GM. I have never been successful in

obtaining technical details from the textile mill on their past or current operations. The main disadvantage of dye substitutions is in terms of reduced light fastness. Imitation phthalogen blue fabrics produced with other methods will look fine when new, but may lose their saturated, brilliant colour within a few months of exposure outdoors.

A reasonable compromise is to use fabric blends where the cotton is dyed with a phthalogen dyestuff and the polyester is dyed with an anthraquinone or mono or diazo disperse dye to adjust the colour. This is likely what Ets. Gonfreville in the Ivory Coast did for many years to produce the standard West African tsetse trap and target fabric "Santiago". Use of phthalogen dyestuff in this blend results in excellent light fastness. TDV Industries produces a similar colour (Azur) in polyester/cotton. It is a stock item in various fabric weights/weaves with many optional finishes. It has good, but not excellent, light fastness. TDV has not disclosed whether it is using phthalogen blue IF3GM in this blend. Along with the colour "New Azzurro" from by Klopman International in Italy (http://www.klopman.com/pages/home.asp), there are many similar (slightly expensive) products for the uniform market that may be suitable substitutes for phthalogen blue cotton. On the North American market, similar fabrics seem to never be produced in a brilliant blue. Instead, darker royal blue fabrics are readily available. Some of these retail products may be useful for attracting tsetse and biting flies, but have yet to be tested against proper standards. From unpublished sticky trap experiments with candidate Klopman fabrics in Ethiopia, I think that the risks of using a good colour match to phthalogen blue in a well-textured (non-shiny) polyester / cotton are not large.

A risky alternative for many species is the use of pure synthetic fabrics, e.g. nylon or polyester, which are shiny, even if the yarns have been "texturized". This is well-documented in carefully-controlled comparisons of similar fabrics in Kenya and in Canada. It is a potentially serious problem for the blue polyester fabrics from Vestergaard Frandsen (VF) in Denmark (Mihok, 2002; Mihok *et al.*, 2006). Until similar research is done by others, it is hard to know how general these insights might be. In extensive unpublished comparisons of phthalogen blue cotton, and a now discontinued "pongee 2" polyester fabric from VF, polyester Nzi traps performed poorly on numerous occasions for biting flies and for savannah tsetse. Catches of riverine tsetse were not affected. This may be why fabric selection for *G. fuscipes* in Uganda does not seem to be a critical issue in a recent comparison to a polyester standard (Okello-Onen *et al.*, 2004). Unfortunately, no phthalogen blue control was included in the Ugandan study. For other species, predicting the effects of using synthetic fabrics is more problematic as results can differ among areas as well as among species. This is a major puzzle that remains unresolved from extensive unpublished research on the behaviour of *G. pallidipes* in Kenya and Ethiopia.

Identifying Genuine Fabrics

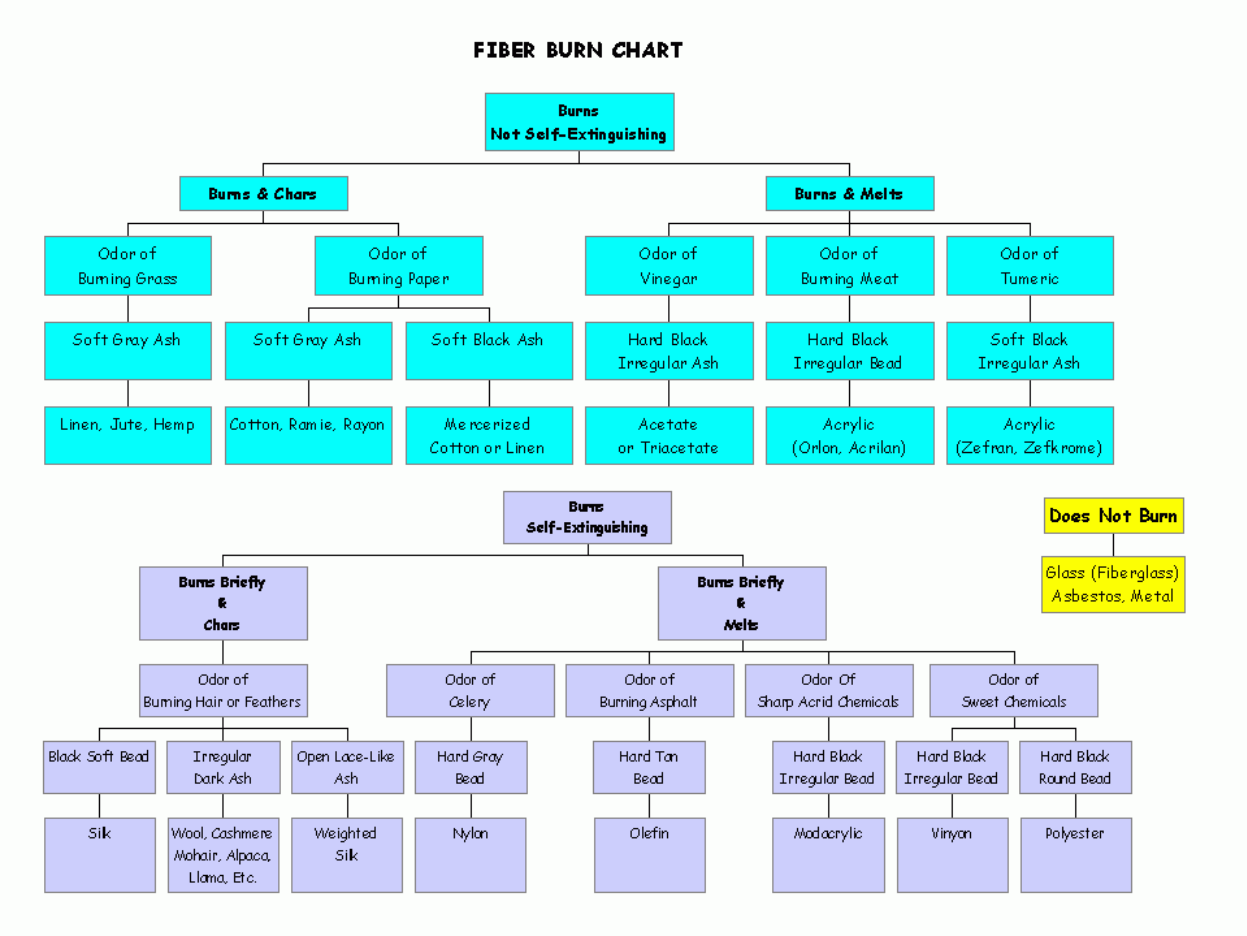
The Basics

To identify genuine phthalogen blue IF3GM cotton, it is always best to have a reference sample for a direct visual comparison. It is very difficult to discern the nuances of "brilliance" and "hue" without side-by-side comparisons; these should be done under bright, natural light. Discrimination under tungsten or fluorescent light is poor. It is not as easy as one would think to match phthalogen blue by eye. I know this from several attempts by myself and others to exactly match genuine phthalogen blue cotton on the retail market.

In the absence of a reference sample for direct visual inspection, use the many examples and colour comparisons provided at nzitrap.com for reference.

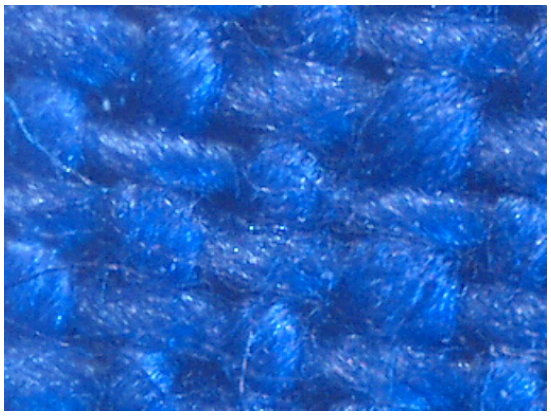
If there is a good colour match, confirm that the material is 100% cotton, and not a blend. Cotton will burn rapidly and cleanly, leaving only a small amount of grey ash. Synthetics and blends will burn or melt, usually with dark smoke, obvious odours and dark residue. There are many simple guides to burning properties; one from the web is reproduced below:

http://www.southwestohiocrochetguild.com/FiberBurnChart.htm

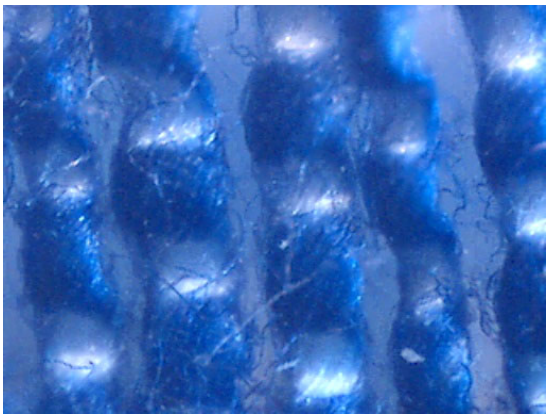


Given the right fabric in the right colour, the next step is to exclude poor quality imitations, e.g. fabrics with a resin-bonded CuPc pigment, or simply poor dyeing. Although there are many detailed chemical tests [see Reference Materials, (Salvin, 1968)], it is usually sufficient to examine the fabric under high magnification using a stereomicroscope or a good quality hand lens (http://www.nzitrap.com/Nzi_trap/Weaves/Weaves.htm). Simply tease out a few bundles of fibres of the warp and the weft. Look for weak surface colouration and uneven colour, e.g. conspicuous white spots where the fibres cross. These features indicate that you probably do not have genuine phthalogen blue IF3GM cotton. Unfortunately, good dyeing at this level of examination does not necessarily confirm that phthalogen blue IF3GM was used. High-quality, evenly-dyed colours can be produced with other dyes/methods.

Below is an extreme example of poor quality cloth at about 60x magnification from the local market in Tanzania (Fabric Code #191). It was used by the Tropical Pesticides Research Institute in Arusha for biconical traps in the mid 1990s. It's reflectance spectrum was "royal blue" and did not match genuine phthalogen blue. The quality of the dyeing left much to be desired.



The fabric looks reasonable only if one crudely examines surface features.



After the fibres are teased out, poor dyeing is obvious where the fibres cross.

Genuine phthalogen blue cotton should look like the sample on the right with very even dyeing of all fibres

Given that the fabric passes all of the above simple tests, there are two practical methods for obtaining a reasonable level of confidence that one has the genuine material.

The first method is easy, but very slow. Since phthalogen blue IF3GM has the highest light fastness rating of any similar blue dye for cotton (8 on an 8-point scale), it will not fade after considerable time outdoors. In bright sunlight in temperate environments, poor imitations will become obvious in 1-2 months; good imitations may only fade noticeably after about three months of exposure. Sunbrella Pacific Blue awning fabric can be used as a convenient light-fast control for this test.

A second method is perhaps less certain but more convenient. It is based on fundamental dye chemistry. The test follows from the limited number of possibilities whereby one can produce a close colour match to phthalogen blue on cotton, using various dyes. Even if this method is not definitive for all the new developments in dyes, it should work as a general screening tool to exclude most imitations of dubious value.

From the table of chemical reactions of dye classes on the next page [see Reference Materials, (Salvin, 1968)], vat and pigment dye classes are the only ones that do not strip after a few minutes in concentrated hypochlorite bleach (2% available chlorine; fresh household bleach is typically 5-6%). Since there are few, if any, modern vat dyes that use CuPc as a chromophore (SDC & AATCC, 1971), it should be sufficient to confirm the presence of CuPc pigment by its bleach resistance. If in doubt about the use of an ingrain dye, other simple chemical tests could be done to exclude use of a vat dye. *All other dyes readily strip.* Hence, imitations produced with CuPc derivatives, e.g. in mixtures with anthraquinone or some other chromophore using direct or reactive dyes should be obvious in terms of poor bleach fastness.

Table II

SUMMARY OF CLASS REACTIONS OF SOME DYES

	REAGENTS					
DYE	REDUCTION	ACETIC ACID	OXIDATION	DMF	HYPOCHLORITE	ASH
Pigment	N C[a, b]	N C	N C	Bleeds	N C	Negative[c]
Azoic	Strips	N A[d]	N A	Bleeds	Strips	Negative
Vat	Reduces	Leuco	Original Shade	Bleeds	N C	Negative
Sulfur	Reduces	Leuco	Original Shade	Bleeds	Strips	Negative
Fiber Reactive	Strips	N A	N A	No Bleed	Strips	Negative
Direct	Strips	N A	N A	Bleeds[e]	Strips	Negative
Diazotized and Developed	Strips	N A	N A	Bleeds[e]	Strips	Negative
Aniline Black	Reduces	Leuco	Original Shade	Bleeds	Brown	Negative
Chrome	Reduces	Leuco	Original Shade	Bleeds	Strips	Positive

[a] N C—no change.

[b] Inorganic pigments are affected by the reduction reagent. They are not completely restored by oxidation. Their presence should be confirmed by microscopic examination.

[c] Inorganic pigments and lakes of azoic reds are exceptions.

[d] N A—not applicable.

[e] Also bleed in 1:1 DMF:Water.

There are many national and international standard tests for fastness to hypochlorite bleach which I have not explored. Below I present results from an arbitrary test of bleaching behaviour at 1% sodium hypochlorite in hot tap water (about 50-55°C, e.g. a 20% dilution of household bleach). Except for the use of hot water to accelerate differentiation, this test is very similar to a British Standard, where the test is done at room temperature for one hour, but with more attention to detail (buffers, final neutralization of the chlorine, etc.) (SDC, 1978). I did this test to understand the nature of the phthalogen cloth sold by Awassa Textiles in Ethiopia relative to phthalogen blue IF3GM cotton from Kenya. The Ethiopian cloth was clearly dyed through another process based on its reflectance spectrum and microscopic examination of the fibres. The two materials are extremely close in colour, and were both used extensively in East Africa in the 1990s for tsetse traps. For contrast, I included a reactive and a direct dye based on sulphonated CuPc. The results were straightforward and are shown below.

The genuine phthalogen blue IF3GM cloth was highly resistant to bleach and lost only some colour after 15 minutes of exposure. The reactive dye stripped gradually and lost a moderate amount of colour. The direct dye stripped completely and rapidly.

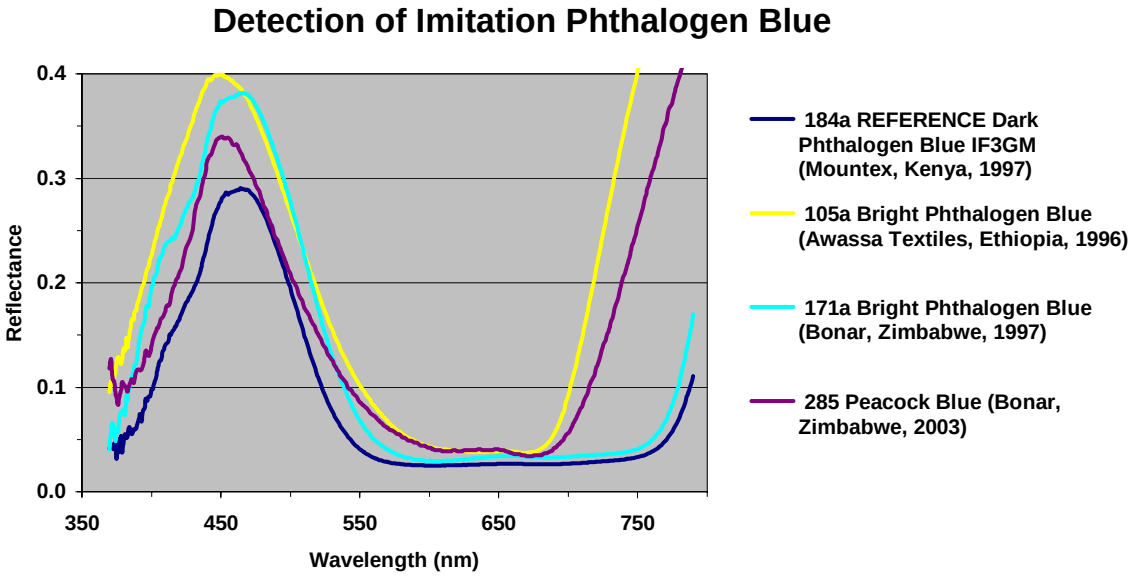
The unknown Ethiopian cloth stripped to white within a few minutes. Although I cannot be sure of the underlying process or processes, the Ethiopian cloth was clearly not dyed with phthalogen blue IF3GM. Its lack of resistance to bleach was consistent with poor light fastness in outdoor tests, which was puzzling at the time of purchase, but clear now. Knowing the results of this simple test would have been useful in terms of understanding the various retail options for phthalogen blue cloth at that time (late 1990's).

Original material	Hot bleach 15 minutes	Notes
		**Phthalogen Blue IF3GM - Mount Kenya Textiles, Kenya** HIGHLY resistant, almost no change for first 10 minutes Colour starts to fade only towards very end of test
		**Unknown Phthalogen Blue - Awassa Textiles, Ethiopia** Hardly any resistance to bleach, very rapid loss of colour Completely stripped in 2-3 minutes
		**Procion Turquoise M-G (5% Reactive Blue 140)** Moderately resistant, Gradual fading with noticeable loss of colour after 10 minutes
		**Direct Blue 86 (7%)** **Barely resistant to bleach with rapid loss of colour** Completely stripped in 5-6 minutes

Spectra

The companion document goes into exhaustive detail on the characteristics of diverse fabrics that have the general appearance of "phthalogen blue". For polyester/cotton blends, it is impossible to be sure if CuPc has been used to dye the cotton. This is because the spectrum of CuPc is blended into the profile of the other dye(s). Hence, similar blue polyester/cotton blends from textile manufacturers like TDV Industries (France), Klopman International (Italy), and Ets. Gonfreville (Ivory Coast) will all look very similar in terms of reflectance and one cannot tell (except by more sophisticated forensics or weathering experiments) whether CuPc is present.

For 100% cottons, identification of genuine phthalogen blue is straightforward, so long as a sophisticated spectrophotometer is used. This is illustrated below with a practical example of some contrasting African fabrics with similar colours.



Phthalogen Blue IF3GM, like the samples above from 1997 in Kenya (genuine reference) and Zimbabwe (assumed), has a unique set of characteristics. At a gross level, note the simplicity and symmetry of the single broad peak at about 465 nm, with little reflectance outside of the blue region. In brighter samples (e.g. #171a from Zimbabwe) a high-quality scan may also reveal two useful features, but only if equipment has fine wavelength resolution and a very stable baseline. These subtle features are best seen by examining the raw spectra in the EXCEL file SPECTRA REFERENCE (many examples). The first unique feature is an inflection point or shoulder in the slope of the declining blue peak between about 410-420 nm. A second, more ephemeral feature is a very slight bump in the flat spectrum at about 650-670 nm.

When the colour phthalogen blue has been imitated with other dyes, it is impossible to exactly reproduce the spectrum. This is shown in the samples from Ethiopia (1997) and Zimbabwe (2003). Typically, there is a striking difference between genuine and imitation materials in the infrared profile (not always measured by researchers). These examples have sharply rising

reflectance above 700 nm, genuine phthalogen blue fabrics has rising reflectance above about 750 nm. Brilliant blues can be produced with many chromophores, so it is impossible to specify all possible diagnostic differences. In these two examples, the imitations differ in terms of a lower wavelength peak, and a different shape of the blue peak. With some dye creativity, a very good match to phthalogen blue can be obtained using 100% cotton. However, specific features of the spectrum, poor light fastness, and poor bleach fastness all help to differentiate imitations from genuine phthalogen blue IF3GM.

Forensics

There are many sophisticated methods for the identification of materials and dyes, but these are of limited practical value to entomologists, because of requirements for sophisticated equipment and expertise, not to mention the need for comprehensive standards. Unfortunately, the blues produced by CuPc and its derivatives are among the most difficult ones to identify with "simple" procedures. A practical introduction to this topic is available in an eminently readable textbook (Robertson & Grieve, 1999). Some basic chemical tests are nevertheless still useful for the novice, and can be carried out in a basic laboratory (e.g. simple chemical reactions, thin layer chromatography). Since the now outdated treatise of Salvin (1968), a standard system is the one used by the FBI in the USA. It is available on the web in a major 1999 handbook on "Forensic Fiber Examination Guidelines" published in *Forensic Science Communications*:

http://www.fbi.gov/hq/lab/fsc/backissu/april1999/houcktoc.htm

An example from this reference for cotton and viscose is reproduced below -

Stage 1
Glacial acetic acid, 100°C, 20 minutes
Good extraction ≈ **AZOIC Dye**
Little or no extraction, Go to Stage 2

Stage 2
Pyridine/Water (4:3), 100°C, 20 minutes
Good extraction ≈ **DIRECT Dye**
Little or no extraction, Go to Stage 3

Stage 3
Dithionite/polyvinylpyrrolidone, (see endnote 1 of this appendix) 100°C, 20 minutes (see endnote 1)
Apply extract to TLC plate; check color of spot
Fiber color changed ≈ **REACTIVE Dye**
(No colored spot or spot not original fiber color)
Fiber color unchanged ≈ **INGRAIN Dye**
(No colored spot or spot not original fiber color)
Fiber color changed, Go to Stage 4
(original colored spot)

Stage 4
10-14% Sodium hydroxide, 100°C, 10 minutes (new fiber)
Fiber color changed ≈ **SULFUR Dye**
Fiber color unchanged ≈ VAT Dye

Endnote

1. Dithionite/polyvinlypyrrolidone—Use immediately and discard excess:
80 mg sodium dithionite
30 mg polyvinylpyrrolidone
450 mL 10% sodium hydroxide
9 mL water.

In the paint industry, there is an American Society for Testing and Materials (ASTM) standard for the identification of both blue and green pigments based on phthalocyanine. It uses the extraction of pigment with sulphuric acid and some simple characteristics of the extracted pigment. This test may be useful in the identification of fabrics produced with ingrain dyes such as phthalogen blue IF3GM, or with fabrics prepared with resin-bonded pigment (ASTM, 1999).

Forensic identification of dyes using increasingly sophisticated techniques is a very active topic (Huang, Yinon & Sigman, 2004). It is currently the subject of a major research program funded by the FBI at the University of Southern Carolina, led by Dr. Stephen Morgan (http://www.chem.sc.edu/faculty/morgan/).

Assuring Quality

With a basic knowledge of the textile industry, its jargon, and how different blues are produced on different types of fibres, it is should be straightforward to obtain "the right fabric" by custom ordering material that has been produced using a specific process (e.g. heavy-weight 100% cotton twill dyed with 4-5% phthalogen blue IF3GM as in the typical tsetse trap fabric). Unfortunately, this is difficult to do in a limited market, as the minimum custom order of a textile mill is often several thousands of metres. Hence, one is usually limited to purchasing finished products made for other purposes (e.g. Sunbrella Pacific Blue awning fabric).

For sportswear, workwear, etc., retail suppliers are typically unable to answer technical questions about dyes, light-fastness, etc. Even when information is potentially available from manufacturers, the competitive nature of the textile business severely limits the availability of useful technical information. The only practical solution is to be knowledgeable of what can be done, and be aware of the basic characteristics of various dyes and fabrics.

Asking the Right Questions

Regardless of the type of fabric, the ultimate source should be requested to provide a report on key fabric characteristics in terms of ISO or other defined test standards, especially in terms of light fastness and/or weathering (Pugh & Guthrie, 2001). Vague, generic answers to specific technical questions, or a reluctance to provide standard test data should raise a red flag.

For cotton - The only technical question that matters is whether the material was dyed with phthalogen blue IF3GM. If this cannot be confirmed in proper technical jargon, then one should examine samples as outlined earlier, e.g. for the quality of dyeing, and for light fastness. A good quality reflectance spectrum can also settle whether the ingrain dyeing process was used. The bleach test is also useful for quickly screening dubious fabrics. Given these procedures, it should always be possible to identify genuine phthalogen blue IF3GM cotton, even if suppliers are unable to provide the required information. At the retail level, this may be the only way to be sure of purchasing the right material.

If an "equivalent" unknown fabric is all that is available, then one should ideally obtain technical production details. This is the only way to interpret the effects of a substitution in terms of the inherent properties of various processes and/or dyes. As this information is often difficult to obtain, one may have to simply rely on generic test results (i.e. if the supplier has the details). The most common problem with retail fabrics will be poor light fastness, as this not a priority outside of the tropics, and has a definite cost associated with it.

A xenon arc light fastness rating of 6-7 (Pugh *et al.*, 2001), versus 8 for phthalogen blue IF3GM, is the minimum rating one should accept in terms of outdoor use.

Many retail fabrics are treated with resins for "easy-care" consumer use, and hence, it is also important to ask the supplier about the presence of any treatments or additives. These may affect fly behaviour in unpredictable ways, e.g. through changes to ultraviolet reflectance or the addition of a "sheen" to the fabric. When ordering direct from textile mills, one can specify the production of plain fabric.

Choosing Synthetics and Blends

The same "buyer beware" principles of quality assurance for cotton apply to all other fabrics, but there are few clear-cut rules to avoid purchasing inappropriate fabrics, other than to avoid distinctly shiny materials. Fabrics that look reasonable in terms of colour and texture to the human eye may not be appropriate for attracting all tsetse and biting flies. The root causes are a function of substantial differences between human and fly visual perception, but with many idiosyncrasies (Mihok *et al.*, 2006). Of the few synthetic fabrics tested, the only potentially "safe" substitute for phthalogen blue cotton appears to be Sunbrella Pacific Blue awning fabric. Fortunately, this is a major consumer product in a popular colour, readily-available worldwide.

It is critical to emphasize that even a good colour match to phthalogen blue in a light-fast synthetic or blend may not be suitable in terms of biological efficacy. This is most likely a result of the inherent shininess of synthetic fibres. It may also be related to the difficulty of reproducing the unique shape of the phthalogen blue spectrum, e.g. in terms of a simple, symmetrical "pure blue" peak with no colour "shoulders" and very low ultraviolet reflectance. Hence, the best one can do is to choose durable, light-fast fabrics intended for outdoor use based on manufacturer's specifications. Suitable fabrics should then be tested empirically for attractiveness to the species of interest. For some groups, these issues may not be critical (riverine tsetse?). Hence, workwear or sportswear fabrics with the general characteristics of phthalogen blue cotton may be suitable.

Unlike cottons, there are an extremely large number of blue dyes that can be used with synthetic fibres, so it is impractical to attempt to deduce dyes and processes for specific products, especially given the ephemeral nature of colours used for clothing. To my knowledge, none of the dye formulations of the blue synthetics or blends used by entomologists have been disclosed, even for Vestergaard Frandsen "tsetse fly" phthalogen polyesters. One is therefore dependent on manufacturers for quality assurance, and ethical behaviour in producing consistent products.

Matching Colours

In the absence of disclosure by the manufacturer of technical information, the suitability of an uncharacterized fabric should ultimately be assessed by comparing the uv-vis reflectance spectrum (e.g. ~350-700 nm, 1 nm intervals if possible) to that of cotton dyed with phthalogen blue IF3GM. An extended spectrum in the near infrared (700 - 800+ nm) is also informative in terms of differentiating dyes and processes. Useful quality control data can also be obtained at 5 nm intervals, but this wide wavelength interval will limit interpretation. Since flies are highly sensitive to ultraviolet light to about 350 nm, it is essential to obtain the ultraviolet spectrum.

With "high-end" spectrophotometers and appropriate integrating spheres, or through the use of a separate glossmeter, it is also useful to compare fabrics in terms of "appearance" as well a "colour". This has yet to be done for fabrics used in entomology, likely accounting for some of the current ambiguities in interpretation of fabric effects. To understand the implications of using smooth, shiny fabrics, equipment should be flexible enough to allow for quantification of diffuse versus specular reflectance. "Low-end" instruments often measure only total reflectance.

Routine colour matching of textiles is often simply done with "colorimeters"; these have only some of the features of more sophisticated spectrophotometers. Colorimeters provide colour characteristics and colour difference indices that reflect human visual perception, given various

illuminating conditions (daylight, artificial light, etc.). These statistics are usually based on 5 or 10 nm wavelength intervals in the visible range only (400-700 nm). Colorimeters are too crude for the needs of entomologists. The main useful output from this type of instrument is a colour difference index relative to a standard fabric (e.g. a common index is CMC(2:1) ΔE). In textile quality control, fabrics are matched to a ΔE of at most just a few units. Unfortunately, as noted above, fabrics that match very well to the human eye may differ substantially in other spectral features that are important in terms of insect vision, especially in the ultraviolet. Hence, colour statistics based on human perception add little to quality assurance efforts for entomological fabrics, relative to what an experienced observer can discern from simple visual inspection.

The results of a spectral colour comparison and any quantitative measures of gloss will need to be interpreted based on the research results obtained to date on the finer points of how flies react to blue fabrics (Mihok et al., 2006). A statistical interpretation of the exact features of the ultraviolet and blue-green spectrum that are key to attracting tsetse and biting flies is still in progress. There are fundamental questions to be answered as to why intuitively small differences in colour can sometimes result in large differences in attractiveness.

Equipment Options

In the tropics, light fastness testing is practical outdoors throughout the year and is the preferred method to monitor the weathering behaviour of fabrics (Wypych, 2003). For other environments, one could purchase a suitable testing device from a source such as ATLAS Material Testing. Sophisticated devices are expensive. An affordable alternative for a small laboratory is the *SUNTEST CPS/CPS+ Tabletop Xenon Exposure System:*

http://www.atlas-mts.com/cgi-atlas/show.cgi?productid=7&from=xenon

It is difficult to recommend an economical spectrophotometer for assessing the colour and appearance of entomological fabrics. Issues of interest are diverse and do not overlap well with those of the textile or paint industry, which is the main market for these devices. Hence, one has to make compromises in purchasing a device that is affordable. It is therefore best to adapt a research instrument for use with textiles by purchasing accessories for an existing laboratory spectrophotometer. This is the best option if a high performance uv-vis + near infrared device (e.g. Varian, Beckman, Perkin-Elmer, etc.) is available in a laboratory with staff experienced in optical measurements. For several thousand dollars, integrating sphere(s) and adaptors and standards can be purchased for use with modern spectrophotometers. Detailed information is available at manufacturer's web sites.

If one can justify purchasing a more user-friendly, stand-alone spectrophotometer designed for the QA/QC of materials, then an investment of about US$25,000 is needed to purchase a flexible, high-quality instrument. Two examples of sophisticated devices with 5 nm reporting intervals from reputable companies are the Datacolor 650 (http://www.datacolor.com/) and the HunterLab UltraScan Pro (http://www.hunterlab.com/). The HunterLab UltraScan Pro has an extended wavelength range that is useful for identifying dyes and underlying processes (350-1050 nm versus 360-700 nm for the Datacolor 650). Infra-red capability is costly so it is not clear how HunterLab has managed to price its product in this range.

Simpler devices are available from Datacolor, HunterLab and many other companies at much less cost, but with progressive sacrifices in key capabilities, e.g. reporting at 10 rather than 5 nm intervals, coverage of only the visible wavelength range, inability to measure transparency as well as reflectance, inability to differentiate diffuse and specular reflectance, inability to measure fluorescence, poorer measurement repeatability, fewer options in viewing area or angles, etc. As this is a competitive market, there are many products at similar prices.

A last option to consider is a different measurement system from a company such as Ocean Optics (http://www.oceanoptics.com/products/usb2000uvvis.asp). Ocean Optics makes several fibre optic instruments, including PC-compatible devices with an ideal wavelength range (200-850 nm). Their instruments can be accessorized with an appropriate integrating sphere(s), light source(s), etc. at modest cost (e.g. US$5,000 for components with features suitable for evaluating entomological fabrics). A potential problem with this cost-saving strategy is that this type of flexibility in hardware requires a knowledgeable operator who can configure and use the equipment properly. This should not be attempted by a novice with minimal experience in colour theory and measurement.

Reference Materials

Simple Analytical Methods - (Salvin, 1968)

The American Association of Textile Chemists and Colorists produced a practical guide to the identification of dye classes on fibres many years ago (Salvin, 1968). This reference is clearly out of date, but should still be useful for differentiating phthalogen blue dyes or pigments from other classes of dyes. Unlike modern methods, these chemical tests are very simple to carry out.

an affinity for cotton are first applied and then treated with a solution of stabilized diazonium salts, whereupon a dyestuff is formed *in situ*. Neither of the two parts entering into the formation of the dyestuff possesses water-solubilizing groups and the dyestuff formed is water-insoluble.

In this systematized scheme of identification, these two classes of dyes are placed at the very end of the procedure, when all other possible classes of dyes have been eliminated, and the problem is narrowed down to the question of differentiating between them.

Naphthols and Insoluble Azo Dyes

The most characteristic property of this class is bleeding in pyridine. Place a 20-50 mg. dyed sample in a 10 to 15 ml. test tube, add one to two ml. of pyridine, and boil the sample. All naphthol dyeings bleed to some extent. Due to their water insolubility, naphthols reduce much slower in caustic soda and hydrosulfite than any other class of dyes in Group III.

Confirmatory Test. Place 100-200 mg. of dyed sample in a 10 to 15 ml. test tube, add two ml. of 10% caustic soda solution and five ml. of alcohol. Boil and add five ml. of water and 40-50 mg. of sodium hydrosulfite. Boil again and after the color is reduced, cool and filter. To the filtrate, add 10-20 mg. of white cotton and 20-30 mg. of salt. Boil one to two minutes, cool and remove the cotton. A yellow dyeing which fluoresces in ultraviolet light confirms that the original was dyed with a naphthol or printed with an insoluble azo dye.

Diazotized and Developed Dyes

These dyes are identified by exclusion of all other classes of this group. Diazotized and developed dyeings do not bleed in pyridine and they are reduced readily in boiling caustic soda and sodium hydrosulfite. Some of these dyes might tint pyridine, and for reassurance, the test may be repeated on the same sample with two to three fresh portions of pyridine. The tinting by some of these developed dyes decreases rapidly and in most cases terminates completely. The bleeding in the case of naphthol dyeings continues at the same rate, since solubility is an inherent property of all naphthol dyeings.

Occasionally, part of the dye will be poorly coupled and bleed in water-ammonia in the tests of Group I. This bleeding, however, is only slight and of a different shade than the original dyeing. Redyeing

on white cotton is impossible or too poor to warrant placing in the class of straight direct dyes.

Group IV

Pigments

When prior tests for other dye classes on cellulose show negative results, the presence of pigments or fiber reactive dyes should be suspected. Microscopic examination, solvent bleeding, and chemical tests can yield information about the presence and the type of resin bonded pigment.

In mass pigmented viscose rayon, pigment particles show uniform distribution throughout the fiber under the microscope. Resin bonded pigments show surface coloration. The surface coloration is obvious in deep shades but not readily recognized in light shades.

Extraction of the sample with a boiling solvent such as dimethylformamide can be useful in distinguishing between some classes of dyes and as a preliminary test for pigments. Resin bonded pigments may belong to the azoic, phthalocyanine or vat classes but may also be inorganic substances. The water-in-oil type of resin bonded pigments, especially the phthalocyanine blue and phthalocyanine green pigments, do not bleed in dimethylformamide whereas the oil-in-water formulation does bleed.

Microscopic Examination. Samples of fiber may have a starch or resin type finish which should be removed so that they do not interfere with the identification. An enzyme scour with 2% enzyme and 0.25% detergent at 160°F for 30 minutes will remove normal starch finishes. Stripping of the resin may be carried out by treatment with 1% hydrochloric acid for 5 minutes at the boil, followed by a rinse. A microscopic test may then be carried out by gently removing a few fibers from the sample and untwisting. The fibers are mounted in a drop of ethyl salicylate and covered with a coverslip. A granular appearance of the fiber surface is characteristic of resin bonded pigments.

Dimethylformamide Extraction. Place the fiber in 5 ml. of a 1:1 solution of dimethylformamide and water and bring to a boil. Remove from the heat and observe staining of the solvent. Place another sample of the fiber in 5 ml. of 100% dimethylformamide and repeat the procedure. The degree of solvent staining serves to distinguish between pigments and fiber reactive dyes. If the sample dyed with fiber re-

active dyes is not washed adequately, a slight bleeding will be shown in the water-dimethylformamide solution. The solvent staining behaviors of various types of dyes are summarized in Table I.

Table I

DIMETHYLFORMAMIDE STAINING TEST FOR DYES

1:1 DMF:Water	100% DMF
Stained by:	*Stained by:*
All Direct	Vats
Diazotized and Developed	Leuco Vats
Some Basic	Naphthols
Some Mordants	Sulfurs
	Pigments
Not Stained by:	Some Basic
Fiber Reactive	Some Mordants
Leuco Vats	
Naphthols	*Not Stained by:*
Pigments	Fiber Reactive
Some Basic	
Some Mordants	

Reactive Dyes

Reactive dyes are characterized by a relatively stable chemical combination with the cellulose fiber. This linkage gives the dyeing excellent insolubility in solvents and water. There is no specific unambiguous test for reactive dyes. It is necessary to eliminate the possibilities of other dyes which are fast to water bleeding e.g. azoic, sulfur, and vat dyes before seeking to establish the color as a reactive dye. It should be noted that pigments resemble reactive dyes in several of the identification tests and that solvent insolubility and behavior with reducing and oxidizing agents are key tests in proving the presence of reactive dyes.

Table II summarizes some of the tests already discussed which may be used to distinguish fiber reactive dyes from some of the other dye classes which have been mentioned.

REAGENTS. Reduction—5% sodium hydroxide and 5% sodium hydrosulfite in water. Acetic acid—5% in water. Oxidation—5% hydrogen peroxide or 10% sodium perborate in water. DMF—100% dimethylformamide. Hypochlorite-sodium hypochlorite bleach containing 2% available chlorine.

PROCEDURE. Place the sample in slow boiling reduction reagent and

Table II

SUMMARY OF CLASS REACTIONS OF SOME DYES

	REAGENTS					
DYE	REDUCTION	ACETIC ACID	OXIDATION	DMF	HYPOCHLORITE	ASH
Pigment	N C[a, b]	N C	N C	Bleeds	N C	Negative[c]
Azoic	Strips	N A[d]	N A	Bleeds	Strips	Negative
Vat	Reduces	Leuco	Original Shade	Bleeds	N C	Negative
Sulfur	Reduces	Leuco	Original Shade	Bleeds	Strips	Negative
Fiber Reactive	Strips	N A	N A	No Bleed	Strips	Negative
Direct	Strips	N A	N A	Bleeds[e]	Strips	Negative
Diazotized and Developed	Strips	N A	N A	Bleeds[e]	Strips	Negative
Aniline Black	Reduces	Leuco	Original Shade	Bleeds	Brown	Negative
Chrome	Reduces	Leuco	Original Shade	Bleeds	Strips	Positive

[a] N C—no change.

[b] Inorganic pigments are affected by the reduction reagent. They are not completely restored by oxidation. Their presence should be confirmed by microscopic examination.

[c] Inorganic pigments and lakes of azoic reds are exceptions.

[d] N A—not applicable.

[e] Also bleed in 1:1 DMF:Water.

observe the reaction and time of reaction. Azoic direct and diazotized and developed dyes decolorize rapidly. Vat, sulfur and aniline black dyes are rapidly converted to the alkaline leuco form. Some red pigments are stripped by this reduction, but the reaction is slow and is easily distinguished from the more rapid cases cited above.

Some fiber reactive dyes containing anthraquinone chromophores will be reduced by the reagent, but will seldom be converted to the original shade by oxidation. Others containing an azo chromophore will be decolorized by the reducing reagent and by hypochlorite.

If the test sample is converted to the alkaline leuco form in the reduction reagent, the sample is removed and placed in the acetic acid solution. Vat, sulfur, aniline black and chrome dyes will form an acid leuco compound differing in color from both the original sample and the alkaline leuco compound.

The acid leuco sample may be re-reduced and then placed in the oxidizing reagent. The original shade of the sample should be restored.

The effect of the hypochlorite on the dyed sample is observed after immersing a fresh sample in the reagent for a few minutes, blotting between paper towels and drying.

Reactive dyes have many kinds of structure, but for reference purposes they may be classified by the kind of group which attaches the dye to the fiber molecule, as in Table III. Within any series, the dyes may contain different dye chromophores. For example, bright blues can have phthalocyanine or anthraquinone structures. Azo structures with and without metal complexes are common in this group.

Table III

CLASSIFICATION OF FIBER REACTIVE DYES

Trade Name	Manufacturer	Chemical Nature of Attaching Group
Remazol	Hoechst	Vinylsulfone
Primazin	BASF	Acrylamide
Procion	ICI	Mono-and dichlorotriazine
Drimarene	Sandoz	Chloropyrimidine
Reactone	Geigy	Chloropyrimidine
Levafix	Verona	Sulfatoethylsulfonamides
Levafix E	Verona	Dichloroquinoxaline
Levafix P	Verona	Methylsulfonepyridine
Cibacron	Ciba	Monochlorotriazine
Calcobond	Calco	Dye & reactive intermediate
Basosol	BASF	Dye & reactive intermediate

Although reactive dyes are attached to the cellulose by primary valencies, certain types e.g. Remazols and Primazins, will hydrolyze when boiled under alkaline conditions (5% caustic). The bleeding into the solution may indicate direct dyes but the hydrolyzed dye will not redye cotton as direct dyes will.

Reduction of fiber reactive dyes can give shade changes similar to those experienced with vat dyes. Phthalocyanine and anthraquinone structures in the fiber reactive dyes are reducible to leuco compounds which will return to the original shade on oxidation.

The procedure for identification of reactive dyes should first eliminate the possibility of vat, sulfur, and azoic dyes. Tests already described and summarized in Table II should be used after removing any resin from the sample by boiling in a dilute acid solution.

Specific tests for azoic, vat, and sulfurs should be carried out. Azoic dyes are soluble in cold chloroform and give a colored solution when a sample is shaken with this solvent in a test tube. Bright and strongly colored samples of vat dyes suggestive of fiber reactive dyes will bleed in the reduction reagent. Similarly, a dull yellow or orange-colored sample with some color bleeding into the solution indicates a sulfur dye. In both cases, if the reduced sample is washed and exposed to the air, it will return to its original shade. Some reactive blues will give strongly colored reduction products, although the original shade returns much more slowly. These blues can be distinguished from vat dyes since they are decolorized in cold weak sodium hypochlorite, whereas vat dyes are not.

Reactive Phthalocyanine and Anthraquinone Dyes. Phthalocyanine and anthraquinone compounds are reducible to leuco compounds, which in turn can be reconverted to the initial compounds by reoxidation. The reduction-oxidation process should be obvious and reversible, that is, the action of the reducing agent must produce a clearly visible shade change and on oxidation the original shade must be restored. The optimum conditions for this procedure consist of reduction of the specimen for 30 seconds at room temperature in a water solution containing 20 gm./l. of sodium hydrosulfite and 2.5 gm./l. of sodium hydroxide. Rinse 2 minutes in cold water, 2 minutes in hot, 2 minutes in cold water.

Generally, phthalocyanines regain their original shade on rinsing, while anthraquinones do not. Oxidation at 90-95°C (194-201°F) for 2½ minutes will restore the original shade of the anthraquinones.

Reactive Azo Dyes. Dyeings of reactive azo dyes can be reduced to primary amines and decomposed so that the reactive fragment remains linked to the fiber while the amine fragment or fragments can be washed off. Often the primary aromatic amino group of the resulting fiber-linked compound can be diazotized and the diazonium compound combined with a suitable coupling component to give a new reactive dyeing. To carry out this method for demonstrating the presence of a reactive azo dye, the amine bound to the fiber must be diazotizable. A number of cleavage products may be obtained which react with nitrous acid in ways other than the formation of a diazonium compound. The success of this method is therefore contingent upon carrying out the diazotization and coupling under suitable conditions and therefore requires some knowledge of azo dye synthesis.

The presence of reactive dyes may be confirmed by their resistance to bleeding when they are boiled with solvents. A summary of this behavior is included in Table II. Direct dyes can be stripped with mixed solvents such as 57% pyridine-43% water and 50% dimethylformamide-50% water. Azoic dyes will bleed into pyridine and into dimethylformamide. Vat dyes, with some exceptions, are partially stripped by 100% pyridine or dimethylformamide.

With the exception of a few Levafix dyes and certain turquoise blues, reactive dyeings which have been freed of unfixed dye show practically no bleeding when they are treated at the boil for 5 minutes with solvents.

More detailed procedures for identification of reactive dyes can be obtained from references 7-9.

DYES ON ANIMAL FIBERS

A burning test serves to identify wool or silk. These fibers are also differentiated from others by their solubility in boiling 5% caustic soda solution. The following dyestuff classes are normally used in the dyeing of wool or silk: Basic, Direct (cotton), Acid, Soluble metallized (acid dyeing), Chrome (wool only), Dispersed metallized (neutral dyeing), Vat, Leuco ester and Naphthols.

Basic Dyes

Place a 100-300 mg. sample of dyeing in a 35 ml. test tube, boil in 10 ml. alcohol for a few minutes and remove. Evaporate the alcohol solution almost to dryness, add 5 ml. of water, and boil the mixture to

remove the alcohol. Add one-fourth to one-half ml. of 10% sodium hydroxide and cool the solution. Add five milliliters of ether and shake the tube to extract the basic dye. Allow the layers to separate, decant the ether layer into a 10 ml. test tube and add a few drops of 10% acetic acid with shaking. The salt form of the basic dye which results should have the original color of the dyed sample.

Additional proof of the presence of basic dyes may be obtained by placing a sample of mordanted cotton or cationic dyeable acrylic fiber in the test tube with the alcohol extract, adding the sodium hydroxide and transferring the dye to these new fibers.

Direct Cotton Dyes

Place a 100-300 mg. sample of dyeing in a 35 ml. test tube, add 5 ml. of water and 1 ml. of concentrated ammonia and boil the sample in this solution for 1-2 minutes. Remove the dye sample and add 30 mg. of salt and 10-30 mg. of white cotton. Continue boiling for 1-2 minutes, remove the cotton and rinse.

If a direct dye is present the cotton will be dyed a deep shade. Some acid dyes may stain cotton, but never to a deep shade.

Acid Dyes

The procedure to be used has been covered in the previous method, but instead of adding salt, neutralize the ammonia with a 10% sulfuric acid solution, and add a few drops of acid in excess. Add a 20-40 mg. piece of wool and boil the mixture for 1-2 minutes.

Redyeing of the extracted dye on wool from this acid bath is evidence of the presence of acid dyes.

This test is significant only if the previous test on redyeing of cotton and the following test for soluble metallized dyes have been carried out.

Positive evidence of direct cotton dyes and acid dyes indicates a union dyeing on a blend of wool and cotton.

Soluble Metallized Dyes ("Acid Dyeing")

The procedure to be used is identical with the method given above for Acid dyes. Soluble metallized dyes are distinguished by the presence of chromium in the ash.

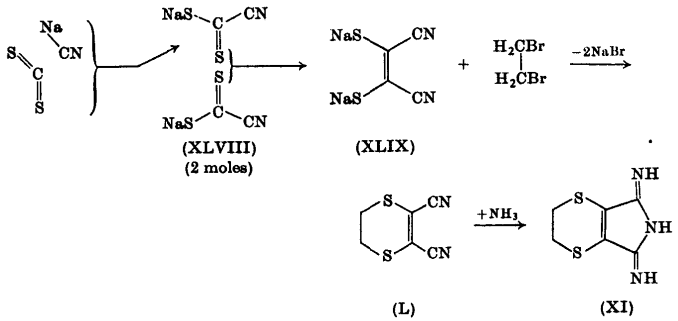
The procedure for the determination of metals is described in the section dealing with direct dyes on cellulosic fibers.

decomposition,[31] pure yield about 60%. The reaction takes place via the 1-chloroisoindolenone (XLIV), which can, however, only be isolated in low yield. Although (X) is obtained in good yield,[32] when (XLV) is treated with ammonia, this method is not suitable for commercial production.

While heptachloroisoindolenine (XLVI) is accessible from tetrachlorophthalimide and somewhat more than 2 moles of phosphorus pentachloride in almost quantitative yield and can also be easily reacted with ammonia to form tetrachlorodiiminoisoindoline (XLVII), the hope that it might be possible to develop from this product on the fiber the well-known green hexadecachloro-CuPc failed because of its difficult solubility.

4. *Production of 3,6-Dithiacyclohexene-1,2-dinitrile (L) and of 1,3-diimino-4,7-dithia-4,5,6,7-tetrahydroisoindoline (XI)*

A structural analog of phthalonitrile is the 1,3-diimino-4,7-dithia-4,5,6,7-tetrahydroisoindoline (XI), which has already been mentioned in Section II,A and which will be briefly referred to in the following as *dithia developer*. The production of this product is based on papers by Bähr *et al.*,[33] who showed that the disodium salt of dimercaptomaleic



dinitrile (XLIX) is obtained from carbon disulfide and alkali cyanide in dimethylformamide via alkali cyanodithioformate (XLVIII) as an intermediate and with splitting off of sulfur from two molecules of this

[31] H. Vollmann, F. Baumann, B. Bienert, and FBy, *DBP* 904,287.
[32] *Idem. DBP* 906,935.
[33] G. Bähr, G. Schleitzer, and H. Bieling, *Chemische Technik*. 8, 597 (1956).

intermediate. According to Degener and Petersen[34] (XLIX) easily yields 3,6-dithiacyclohexene-1,2-dicarbonitrile (L) by reaction with 1,2-dibromoethane. And, finally, it is possible according to Wolf *et al.*,[35] to produce from (L) in good yield the "dithia developer." The same methods are suitable for this purpose as have been described above for the conversion of phthalonitrile into 1,3-diiminoisoindoline (X).

In contrast to the colorless (X), the dithia developer (XI) shows a deep yellow inherent color, gives red salts with acids, and shows in concentrated sulfuric acid a deep violet color.

Mixtures of the dithia developer with diiminoisoindoline (X) yield[36] on the fiber, developed with a copper donor by the Phthalogen process, with increasing content of dithia developer increasingly *redder* and deeper shades up to dark navy blue—and when nickel donors are used even black shades. Mixtures of the kind mentioned here are commercial products of the Phthalogen range (see Table I).

III. Commercial Products of the Phthalogen Class, Including the Auxiliaries

The commercial range at present comprises 15 metal phthalocyanine developers which are broken down in Table I (first vertical column) into Groups 1, 2, and 2a in accordance with Sections B and C of this chapter:

Group 1 comprises metal complexes with a preformed DehydroPc ring.

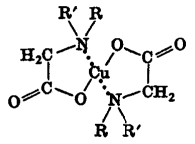
Group 2 comprises simple 1,3-diiminoisoindolines.

Group 2a comprises the same diiminoisoindolines, but homogeneously mixed with the metal donors required for dyestuff development, in the form of Phthalogen K or Phthalogen Ni. The Phthalogen dyestuff developers Nos. 12–15 (vertical column 2) also contain quantities, increasing in accordance with the sequence given below, of the dithia developer (XI) (see Section II,C,2).

Group 3 in Table I comprises the following auxiliaries.

Phthalogen K and Phthalogen Ni are copper and nickel complexes of the structure (LI) in which R is a methyl group and R′ a β-hydroxyethyl group. These are thus *N*-substitution products of the well-known glycocoll copper (LI; R and R′ = H). Also very suitable are analogously designed complexes of the structure (LI) in which both R and R′ are β-hydroxyethyl groups or carboxymethyl groups, $-CH_2-COOH$. The latter are accessible from nitrilotriacetic acid with copper salts.

[34] E. Degener, S. Petersen, and FBy, *BeP* 565,804.
[35] W. Wolf, H. Klappert, E. Degener, and FBy, *BeP* 565,803.
[36] W. Wolf, E. Degener, and S. Petersen, *Angew. Chem.* 72, 963 (1960).



(LI)

Phthalogen K and Ni are also offered in pure form as auxiliaries for Phthalogen dyeing (Nos. 16 and 17 of the table).

Phthalogen P is a further copper donor which only recently has been developed *especially* for Phthalogen dyeing of polyamide fibers.

Phthalotrop B is a resisting agent for printing with Phthalogen dyestuffs of Groups 2 and 2a by the preprint resist process. It is chemically a difficultly volatile, easily water-soluble di-primary amine of the aliphatic series.

Phthalofix FN is a premordant for yarn dyeing with Phthalogen Brilliant Blue IF3GK.

The Levasol brands P, PO, D, Tr, and F are difficultly volatile, water-soluble organic solvents which are indispensable for the development of the metal phthalocyanines from the Phthalogens on the fiber. There is only one exception from this rule: Phthalogen Blue IBN, described below.

Levasols P and PO are mixtures which, beside polyalcohols, also contain trialkanolamines and in many cases lead to optimum coloristic results. Levasols D (=diethylene glycol), Tr (=triethylene glycol), and F (=formamide) are technically pure, virtually uniform solvents which are used partly alone and partly in mixture with Levasols P or PO.

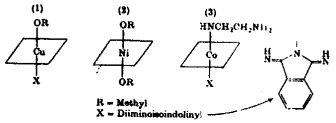
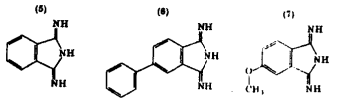
A new Phthalogen dyestuff developer which, strictly speaking, does not belong to any of the groups mentioned above is *Phthalogen Blue IBN*, which will now be briefly described. In accordance with our findings, this product is a complex derivative of the trivalent cobalt phthalocyanine and consequently shows, both in substance as well as in solution—for example, in 3–5% aqueous acetic acid—a deep greenish blue color. The agent required for reduction is already contained in it, built into the molecule. In Table I Phthalogen Blue IBN follows the metal DehydroPc complexes in Group 1 as No. 4.

The suffixes to the names of the dyestuff developers have the following meanings:

I	Indanthrene fastness of the dyeings and printings achievable therewith.
F and FF	(Rising) brilliance of the color shades.
G and GG B and 2B R and RR	(Rising) yellow, blue, or red cast of the shades.
M	Mixtures of diiminoisoindolines with the metal donors Phthalogen K or Ni (Group 2a).
K	A preformed complex (DehydroPc metal complex) is involved.
N	A new brand.

TABLE I

COMMERCIAL PRODUCTS OF THE PHTHALOGEN CLASS

Group	*No.*	*Phthalogen*		*Composition*
1	1	Brilliant Blue	IF3GK	
	2	Turquoise	IFBK	
	3	Blue I	IB	
	4	Blue	IBN	
2	5	Brilliant Blue	IF3G	
	6	Brilliant Green	IFFB	
	7	Brilliant Green	IF2B	
2a	8	Brilliant Blue	IF3GM	Base No. 5 + Phthalogen K
	9	Turquoise	IFBM	Base No. 5 + Phthalogen Ni
	10	Brilliant Green	IFFBM	Base No. 6 + Phthalogen K
	11	Brilliant Green	IF2BM	Base No. 7 + Phthalogen K
	12	Brilliant Blue	IFGM	Mixtures of base No. 5 with dithia developer (XI) and Phthalogen K or —for No. 15—with Phthalogen Ni
	13	Blue	IRM	
	14	Marine Blue	IRRM	
	15	Blue Black	IVM	
3	16	Phthalogen K		Copper or nickel complex from hydroxyethyl sarcosine (LI), $R = CH_3$; $R_1 = CH_2 \cdot CH_2 \cdot OH$
	17	Phthalogen Ni		
	18	Phthalogen P		
	19	Phthalotrop B		For application see Section II,C,2
	20	Phthalofix FN		

IV. Technique and Reaction Mechanisms of the Application of Phthalogen Dyestuffs

The application methods of the Phthalogens usually correspond to those of the customary practice in pad dyeing and machine or screen printing. When preparing the dye liquors or the printing inks, it is *necessary* to add, in addition to the customary additives—such as emulsifiers and thickening agents—also certain quantities of difficultly volatile, water-miscible solvents and, as far as these solvents (like some Levasols) do not already have sufficient reducing power, also additional substances having a reducing action. Exception: for Phthalogen Blue IBN, see preceding section.

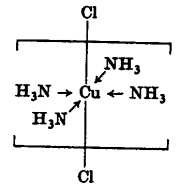
For Phthalogen dyestuffs of *Group 1* (metal DehydroPc complexes) or of *Group 2a* (mixtures of diiminoisoindolines with metal donors), the dye liquors or printing inks are ready for use. If Phthalogen dyestuffs of *Group 2* (metal-free diiminoisoindolines) are employed, an optimum quantity of a copper or nickel salt or complex salt must additionally be incorporated, in accordance with the instructions.

The padded or printed goods are then warmed, with the water rapidly evaporating, promoted by the large surface of the textile material, and the solvents becoming effective. Depending on the kind of the Phthalogen dyestuffs used, various reactions then take place on and in the fiber:

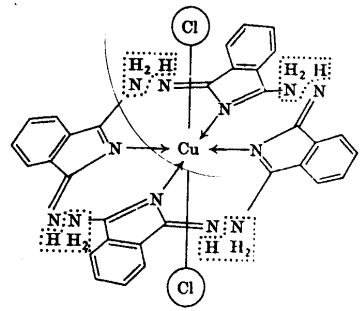
(1) Phthalogen types of Group 1 dissolve in the solvent and largely penetrate into the fiber as a solution. On higher heating, dependent upon the solvent and the reducing agent, reduction to the metal phthalocyanine then takes place rapidly, with the dyestuff being fixed. For formulation see Section II.

(2) Phthalogen types of Groups 2 or 2a, in which the required heavy metal donor—in this stage—is present, are primarily converted on drying in dissolved, now almost anhydrous form (at 60–70°) into diiminoisoindoline heavy metal complexes showing the same character as the coordinately hexavalent metal–DehydroPc complexes described in Section II,B. The "dyeings" obtained after careful drying show in this stage, if copper donors are used, washed-out yellowish to olive-colored shades. However, on spotting with a freshly prepared solution of sodium dithionite in dilute sodium hydroxide solution they *instantaneously* change to the deep, not hydrolyzable blue of the CuPc. The second step of the reaction, the reduction, and thus the fixation, then takes place at higher temperature, as under (1).

Today we think that we can best interpret our view regarding the course of the reaction of the *total synthesis* of the copper phthalocyanine *on the fiber* in the following manner:



(LII)



(LIII)

Cupric chloride is known to give with 4 moles of ammonia the complex salt copper tetrammine dichloride, for which it is customary to write $[Cu(NH_3)_4]Cl_2$. If we write it in accordance with formula (LII) (without implying any meaning other than that of the classical formulation), it becomes apparent that, if the ammonia is replaced by 4 moles of 1-amino-3-iminoisoindolenine, a precomplex of the structure (LIII) may be obtained whose steric structure *greatly favors ring closure* with elimination of 4 moles of ammonia. The reduction of the dichloro-DehydroPc–copper complex to CuPc takes place, as we know from experiments, with extreme ease. In the practice of Phthalogen dyeing, there will, however, be involved, as the final precomplexes of CuPc formation, instead of the dichloro complex [from (LIII) minus 4 NH_3]—which we selected more for didactic reasons—mostly analogously structured chlorine-free complexes, whether instead of cupric chloride Phtalogen K is used, as recommended, or from the beginning such complexes have been used [for example, as Phthalogen Brilliant Blue IF3GK of the structure (XVI)], or the primarily obtained dichloro complex is converted under application conditions (presence of excess diiminoisoindoline = Phthalogen Brilliant Blue IF3G and of difficultly volatile alcohols) into chlorine-free complexes.

Bayer Phthalogen Dyestuff Manual (German)

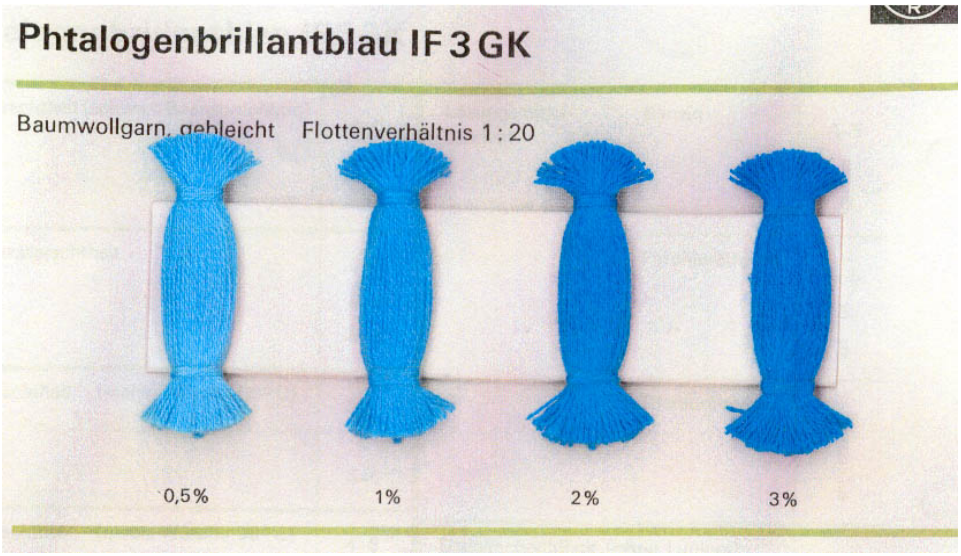
Fabriken Bayer AG Leverkusen (undated) Phtalogen-Farbstoffe in der Fäberei. Le 1095, Fa. Printed in Germany. 66 pages.

The following pages contain selected portions of a technical manual provided by Dystar in 2005 on phthalogen dyestuffs. These pages outline the properties of Phthalogen Blue IF3GM. Type IF3GM and a similar methodological variation (IF3G - just a separate copper complex) are the specific methods used for solid-shade dyeing of cotton in a permanent, deep, brilliant blue. Key details of the chemistry of these dyestuffs are in Vollmann (1971). The Bayer manual is mainly useful in terms of the technical details on how to use the products, and the many colour figures of dyestuffs on contrasting fabrics / yarns (Type IF3GK is shown below). Zoom in on the scanned pages if the print is too small. You will note the reason why there are various spellings of phthalogen; the original German term for "Phthalogen Brilliant Blue" has only one "h".

The first few pages refer to the German standards (DIN, Deutsches Institut fur Normung) that were used to characterize dyeing properties in terms of reactions to chemicals or treatments. Only some of this information is reproduced in the Colour Index.

The key feature of IF3GM is a light fastness rating of 8 (top of the scale) at a normal depth of shade (1/1). This is on the page with the colour swatches going from 0.5% to 5% dye owg (on weight of goods). Fabrics used by entomlogists are typically in the range of 4-5%.

I think the DIN test results on the last page are all on a scale of 5, and hence ratings of 4-5 are also excellent. Reactions to hypochlorite bleach are listed as 3G to 4-5 to 5 (not certain of the exact meanings here for multiple entries). These ratings are listed in the Colour Index under "hypochlorite" as simply 3-4. Dystar mentioned that the excellent light fastness of IF3GM was countered by less than perfect resistance to bleaching agents. Note that resistance to "peroxide bleaching" and "soda boil" are both 5.



Bemerkungen zu den Echtheitsangaben

Die Echtheitseigenschaften wurden geprüft und bewertet nach den aufgeführten Normen der Deutschen Echtheitskommission (DEK), herausgegeben vom Deutschen Normenausschuß (DNA) in Übereinstimmung mit der Europäischen Echtheitskonvention (ECE) und unter Berücksichtigung der Empfehlungen der International Organization for Standardization (ISO).

Die Lichtechtheit der Phtalogen-Farbstoffe wurde in folgender Weise geprüft: **Phtalogen M-Farbstoffe** auf merc. gebleichtem Baumwollpopelin in $^1/_{12}$, $^1/_6$, $^1/_3$, $^1/_1$, $^2/_1$ Richttyptiefe.

Phtalogen K-Farbstoffe auf gebleichtem Baumwollgarn in $^1/_{25}$, $^1/_{12}$, $^1/_6$, $^1/_3$ Richttyptiefe.

Auf Naturseide in $^1/_6$, $^1/_3$ Richttyptiefe.

Auf merc. gebleichtem Baumwollpopelin (Äthylglykol-Verfahren) in $^1/_{12}$ Richttyptiefe.

Die übrigen Echtheitsangaben beziehen sich auf Färbungen in $^1/_1$ Richttyptiefe auf merc. gebl. Baumwollpopelin bei den Phtalogen M-Farbstoffen,

$^1/_3$ Richttyptiefe auf gebl. Baumwollgarn bzw. Naturseide bei den Phtalogen K-Farbstoffen,

$^1/_{12}$ Richttyptiefe auf merc. gebl. Baumwollpopelin bei den Phtalogen K-Farbstoffen (Äthylglykol-Verfahren).

Lichtechtheit (Tageslicht)	DIN 54003
Lichtechtheit (künstliches Licht)	DIN 54004
Wasserechtheit (schwere Beanspruchung)	DIN 54006
Waschechtheit (mech. Wäsche 40°C)	DIN 54014
Waschechtheit (mech. Wäsche 60°C)	DIN 54010
Waschechtheit (mech. Wäsche 95°C)	DIN 54011
Meerwasserechtheit	DIN 54007
Chlorbadewasserechtheit	DIN 54019
Schweißechtheit	DIN 54020
Peroxid-Waschechtheit	DIN 54015
Sodakochechtheit, ohne Ludigol	DIN 54031
Lösungsmittelechtheit	DIN 54023
Bügelechtheit	DIN 54022
Säureechtheit	DIN 54028

Alkaliechtheit	DIN 54030
Peroxid-Bleichechtheit	DIN 54033
Hypochlorit-Bleichechtheit (leichte Beanspruchung)	DIN 54034
Hypochlorit-Bleichechtheit (schwere Beanspruchung)	DIN 54035
Chlorit-Bleichechtheit (leichte Beanspruchung)	DIN 54036
Chlorit-Bleichechtheit (schwere Beanspruchung)	DIN 54037
Schwefelechtheit	DIN 54038
Mercerisierechtheit	DIN 54039
Entbastungsechtheit	DIN 54055

Die Bewertung der Lichtechtheit im Fade-o-meter in SFH gibt an, wieviele Standard Fading Hours (SFH) erforderlich sind, bis eine Farbänderung eingetreten ist, die der Stufe 4 des Graumaßstabes »Änderung der Farbe« entspricht (lt. Standard Test Method AATCC 16A – 1964, 7. 4.).

Die Zahlenwerte bedeuten in senkrechter Reihenfolge:

Bei der Wasserechtheit, Waschechtheit 40°C und 60°C, Meerwasserechtheit, Schweißechtheit, Lösungsmittelechtheit und Entbastungsechtheit:

Farbtonänderung bzw. Änderung der Farbstärke
Antönen weißer Baumwolle bzw. Naturseide
Antönen weißer Wolle bzw. Baumwolle

Bei der Waschechtheit 95°C, Peroxid-Waschechtheit und Peroxid-Bleichechtheit:

Farbtonänderung bzw. Änderung der Farbstärke
Antönen weißer Baumwolle
Antönen weißer Zellwolle

Bei der Chlorbadewasserechtheit, Säureechtheit, Alkaliechtheit, Mercerisierechtheit und Schwefelechtheit wird nur die Farbtonänderung bzw. Änderung der Farbstärke angegeben.

Bei der Sodakochechtheit, Hypochlorit- und Chlorit-Bleichechtheit:

Farbtonänderung bzw. Änderung der Farbstärke
Antönen weißer Baumwolle

Bei der Bügelechtheit:

Farbtonänderung, sofort
Farbtonänderung nach 4 Stunden
Anbluten von weißer Baumwolle
(100% Feuchtigkeit)

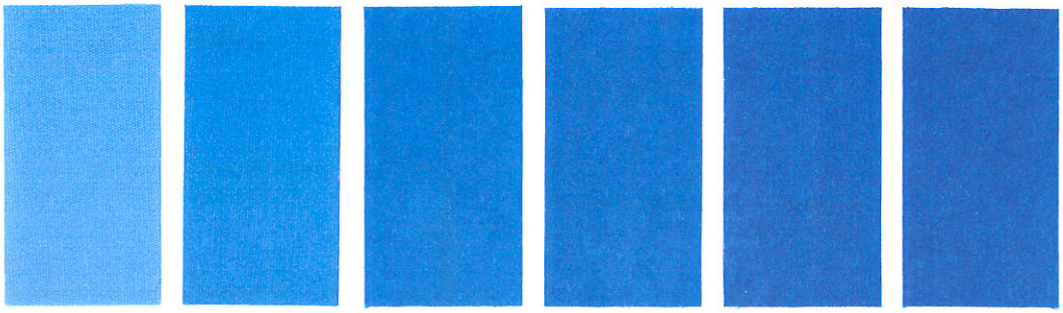
32

Mit unseren Angaben, die auf sehr sorgfältigen Ausarbeitungen beruhen, wollen wir Sie beraten. Eine Garantie für Ihre Ausrüstungen können wir nicht übernehmen. Wir garantieren jedoch eine gleichmäßige Qualität unserer Produkte.

Farbenfabriken Bayer AG Leverkusen

Phtalogenbrillantblau IF 3 GM

Klotzfärbung auf merc. gebl. Baumwolle



5 g/kg | 10 g/kg | 20 g/kg | 30 g/kg | 40 g/kg | 50 g/kg

Lichtechtheit

Richttyptiefe	Tageslicht	Xenotest	Fade-o-meter
1/12	7	6–7	240 SFH
1/6	7	6–7	320 SFH
1/3	7–8	7	480 SFH
1/1	8	8	640 SFH
2/1	8	8	640 SFH

Kennzeichnung mit dem Warenzeichen INDANTHREN

Allgemein	geeignet	
Vorhang	geeignet	
Wasch	geeignet	
Markisen	geeignet	(Mindesttiefe beachten)

Bemerkungen

Phtalogenbrillantblau IF3GM entspricht Phtalogenbrillantblau IF3G + Phtalogen K im Verhältnis 100:30.
Nur für Klotzfärbungen auf Baumwolle und Zellwolle geeignet.
Eine Farbstärke von 5–50 g Farbstoff pro kg Färbegut soll nicht unter- bzw. überschritten werden.
Phtalogenbrillantblau IF3GM kann kombiniert werden mit
Phtalogenbrillantgrün IF2BM und
Phtalogenbrillantgrün IFFBM

Phtalogenbrillantblau IF 3 GM

Wasserechtheit (schwere Beanspruchung)		5 5 5
Meerwasserechtheit		5 5 5
Waschechtheit (mech. Wäsche 95° C)		4–5 5 5
Peroxid–Waschechtheit		4–5 5 5
Chlorbadewasserechtheit		5
Schweißechtheit	alkalisch	5 5 5
	sauer	5 5 5
Bügelechtheit		
	trocken sofort	4 G
	nach 4 Stunden	5
	feucht Bluten	5
Lösungsmittel-echtheit	Benzin	5 4–5 4–5
	Perchloräthylen	5 4–5 4–5
	Trichloräthylen	5 4–5 4–5
Sodakochechtheit (ohne Ludigol)		5 5
Peroxid-Bleichechtheit		5 5
Säureechtheit	Essigsäure	5
	Weinsäure	5
Alkaliechtheit		5
Hypochlorit-Bleich-echtheit	leichte Beanspruchung	4 4–5
	schwere Beanspruchung	3 G 4–5
Chlorit-Bleich-echtheit	leichte Beanspruchung	4–5 5
	schwere Beanspruchung	4–5 5
Mercerisierechtheit*		5
Knitterfestausrüstung		geeignet

* Zunahme der Farbtiefe, keine Änderung des Farbtones und der Reinheit

Dyeing Methods - Manibhadra Enterprise (English)

Indian Chemical Industries provides a brief summary of phthalogen dyeing methods on the web at http://www.icidyes.com/fast_salt_ingrain_dyes_textile.htm. Similar information is reproduced below from the company brochure of Manibhadra Enterprise in India.

PHTHALOGEN BLUE DYES (INGRAIN DYES)

Colour Shade	Product Name	C. I. Hue Name	C. I. No.
	Phthalogen Blue IBN	Ingrain Blue 5	74161:2
	Phthalogen Blue 3GM	Ingrain Blue 2:1	74160
	Phthalogen Blue 3G	Ingrain Blue 2:2	74160
	Phthalogen Blue 3GK	Ingrain Blue 13	74161

PHTHALOGEN BLUE IBN (INGRAIN BLUE 5)

Application Printing :

Cellulose and silk. Dissolve in an organic acid and thickaning and a spl. auxiliary agent having reducing properties. Print, dry develop with netural or acid steam or by backing treat in a hot acid bath. Rinse and soap.

Can be printed alongside azoic, reactive and vat dye and pigment and on grounds of azoic coupling components.

PHTHALOGEN BLUE 3GM (INGRAIN BLUE 2:1)

Ingrain Blue 2:1 Pdr. (Mixed with Copper Complex) Dyeing for Fabrics

Padding Process :

6	Parts Ingrain Blue 2:1 Pdr.
8	Parts Solvent cum Emulsifier
10	Parts Urea
25	Parts Followed by addition of
51	Parts Ice Cold Water
100	Parts

Pad and dry the material at about 80-90° C.

Cure at 150°C for about 5to6 minutes.

After Treatment : The material is rinse in cold water & is treated with Sodium nitrite 4 gms/litre & 5 gms/litre HCL at boil for 20 minutes. wash with soap & soda ash at boil and rinse.

Ingrain Blue 2:1 Pdr. Printing for Fabrics

Padding Process :

6	Parts Ingrain Blue 2:1 Pdr.
10	Parts Urea
26	Parts Water
58	Parts Gum Thickening
100	Parts

Print on screen and dry in air. Steam for one hour wash with cold water and dry.

PHTHALOGEN BLUE 3G (INGRAIN BLUE 2:2)

Ingrain Blue 2:2 Pdr. Dyeing for Fabrics

Padding Process :

4	Parts Ingrain Blue 2:2 Pdr.
8	Parts Solvent Cum Emulsifier
10	Parts Urea
20	Parts Water followed by addition of
50	Parts Ice Cold Water & Add
8	Parts Copper Complex
100	Parts

Pad and dry the material at about 80-90°C.

Cure at 150°C for about 5 to 6 minutes.

After Treatment : The material is rinse in cold water & is treated with Sodium nitrite 4 gms/litre & 5 gms/litre HCL at boil for 20 minutes. Wash with Soap and Soda ash at boil and rinse.

Ingrain Blue 2:2 Pdr. Printing for Fabrics

Padding Process :

4	Parts Ingrain Blue 2:2 Pdr.
10	Parts Urea
20	Parts Water
8	Parts Copper Complex after Stirring add
58	Parts Gum Thickening
100	Parts

Print on screen and dry in air. Steam for one hour wash with cold water

PHTHALOGEN BLUE 3GK (INGRAIN BLUE 13)

Ingrain Blue 13 Pdr. for Yarn Dyeing

MORDANTING (For 5 gm, Yarn)

The Yarn is soaped with Soda Ash for 10-15 min. at boil then rinse will.

200 mgm	Mordant B.
150 CC.	Water at 70°C. Yarn is entered in Mordanting bath After 10 minutes add.
1.5 gm.	Glauber Salt keep the yarn for 20 minutes in this bath then after squeezing take it to the dye bath.

DYE BATH (BEAKER SHOULD BE ABSOLUTELY DRY)

200 MGM.	Ingrain Blue 13.
400 MGM.	Diethylene Glycol.
1 GM.	Acetic Acid Glacial after keeping the above for 20 minutes in cold bath it is to be poured into
150 CC.	Ice Cold Water enter the Mordanted yarn into this dye bath and keep it for 25 minutes. The dyed yarn is taken out and developed in.
500 MGM.	Caustic Soda add.
300 MGM.	Sodium Hydrosulphite
100 CC.	Water at 60°C for 10 minutes the yarn is stirred and then treated with.
1 CC.	HCL
500 MGM.	Sodium Nitrite.
100 CC.	Water at 60°C the yarn is kept for treatment for 10 minutes.

The yarn rinsed in cold water and washed with soap & soda ash at boil.

References Cited

Aspland, J.R. (1997) *Textile dyeing and coloration*, Research Triangle Park, New Jersey: American Association of Textile Chemists and Colorists.

ASTM (1999) *Standard test methods for chemical anlaysis of phthalocyanine blue and green pigments, D 3256 - 86*, West Conshohocken, Pennsylvania, USA: American Society for Testing and Materials.

Hawkyard, C. (2004) *Synthetic fibre dyeing*, Bradford, UK: Society of Dyers and Colourists.

Huang, M., Yinon, J. & Sigman, M.E. (2004) Forensic identification of dyes extracted from textile fibers by liquid chromatography mass spectrometry (LC-MS). *Journal of Forensic Sciences*, **49**, 1-12.

Ingamells, W. (1993) *Colour for textiles. A user's handbook*, Bradford, UK: Society of Dyers and Colourists.

Mihok, S. (2002) The development of a multipurpose trap (the Nzi) for tsetse and other biting flies. *Bulletin of Entomological Research*, **92**, 385-403.

Mihok, S., Carlson, D.A., Krafsur, E.S. & Foil, L.D. (2006) Performance of the Nzi and other traps for biting flies in North America. *Bulletin of Entomological Research*, (in press).

Nassau, K. (1998) *Color for science, art and technology*, Amsterdam: Elsevier.

Okello-Onen, J., Okoth, J.O., Abila, P.P., Matete, G.O., Wamwiri, F. & Politzar, H. (2004) Effectiveness of monoscreen traps for tsetse fly control. *Uganda Journal of Agricultural Sciences*, **9**, 667-671.

Pugh, S.L. & Guthrie, J.T. (2001) The development of light fastness testing and light fastness standards. *Review of Progress in Coloration*, **31**, 42-56.

Robertson, J. & Grieve, M. (1999) *Forensic examination of fibres*, London: Taylor & Francis.

Salvin, V.S. (1968) Identification of dye classes on fibers. pp. 187-218 in AATCC. (Ed.) *Analytical methods for a textile laboratory, second edition, AATCC Monograph Number 3*. Research Triangle Park, North Carolina, American Association of Textile Chemists and Colorists.

SDC (1978) Standard BS 1006:1978, N01:1978 Colour fastness to bleaching: hypochlorite. *Standard methods for the determination of the colour fastness of textiles and leather, fourth edition. Society of Dyers and Colourists*.

SDC & AATCC (1971) *Colour Index, Third edition*, Bradford, UK: Society of Dyers and Colourists and the American Association of Textile Chemists and Colorists.

Shore, J. (1995) *Cellulosics dyeing*, Bradford, UK: Society of Dyers and Colourists.

Vollmann, H. (1971) Phthalogen dyestuffs, Chapter V. pp. 283-311 in Venkataraman, K. (Ed.) *The chemistry of synthetic dyes, Volume V*. New York, Academic Press.

Wypych, G. (2003) *Handbook of material weathering, 3rd edition*, Toronto: ChemTec Publishing.