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Author(s): Christopher Palenik, Ethan Groves, Skip Palenik

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Final Summary Report
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**Development of an Analytical System for the Forensic Comparison and
Identification of Fiber Dyes on Casework-Sized Fibers**

29 February 2016

Prepared by:
Christopher S. Palenik, Ethan Groves, Skip Palenik

Microtrace LLC

790 Fletcher Drive, Suite 106
Elgin, IL 60123-4755

847.742.9909 (p)
847.742.2160 (f)
www.microtracellc.com

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INTRODUCTION

Colorants (pigments and dyes) are everywhere. They impart color on virtually every object around us, from the paint on walls to the color in clothing, to the ink on printed media. There are several hundred commercially important dyestuffs in use,¹ with more being developed each year. Dyes are most often encountered in forensic evidence as the coloring component of textile fibers (whether exploited or not). The majority of manmade fibers are dyed post-production. The number, amount, and identity of the dyes present on a fiber are all factors that may permit it to be discriminated from other fibers with similar polymer chemistries and physical properties. In some cases, fibers sharing the same macroscopic color, in addition to polymer chemistry and physical characteristics, may be distinguished (possibly at the batch level) due to the use of different dyes in at varying concentrations. For example, is not unusual for textile companies to color fabrics to the same specified color using different combinations of dyes (based upon availability and pricing) to achieve a similar final color. In some cases additional dyes are added to correct or adjust a color.

In theory, colorants seem like an obvious material to exploit as forensic evidence; yet beyond the macroscopic color that a combination of colorants imparts to a material, the identity of individual colorants has been largely ignored in forensic casework, or limited to comparative analyses which are not specific to chemical or molecular identification. The primary reason for this is their presence at a relatively low concentration in a given product. For example, pigments are often present at levels near or below 1% in a finished material, and dyes can be present at an even lower concentration (*e.g.*, <0.025% in many fibers). There have been several various successful attempts to characterize dyes, often directly in finished products.^{2,3, 4, 5} However, these examples are generally limited in some way, either to a small subset of colorants (as a result of the wide variety of dye chemistries) or in their practicality as a forensic analytical method.

In addition to issues with the development of generalized approaches to dye identification, difficulties in obtaining colorant reference collections, significant in quantity and diversity, has also limited generalized dye characterizations.ⁱ

The research presented here aims to overcome these challenges by exploring a significant range of dyestuffs, spanning numerous major dye application classes, chemistries, chromophores, and fiber types utilizing techniques and methodologies currently available (or readily obtainable by) in most forensic laboratories; these include: thin layer chromatography (TLC), Raman microspectroscopy, Fourier-transform infrared microspectroscopy (micro-FTIR), Ultra-Violet/Visible spectrophotometry and microspectrophotometry (UV/VIS and MSP, respectively). The possibility to conduct this examination is based upon a colorant collection that Microtrace has sourced and compiled over several decades. This report provides a broad overview of the avenues that have been explored and the publications that have been submitted and are being prepared.

APPROACH AND RESULTS

Traditional dye analysis begins with extraction of the dye(s) from a matrix (*e.g.*, the fiber) followed by development on a TLC plate. The developed dye bands are then compared against a known material to evaluate a potential association. When used this way, forensic dye analysis is strictly comparative and does not provide the means for

ⁱ Reference collections of authenticated dyes have been traditionally maintained by major dye manufacturers, such as BASF, Ciba Geigy, Sandoz, *etc.*, but as dyestuffs have evolved into a commodity, manufacturing has shifted to Asia, where quality and R&D are often of lesser interest. To this end, curated reference collections have largely been abandoned.

dye identification. The incorporation of a database of analytical data for dyestuffs, including a) the solubility properties of a dye, b) the TLC developing characteristics, c) UV/VIS absorption profiles, and d) Raman and infrared spectra are anticipated to provide a generalized means to permit systematic characterization, comparison, and identification of dyes, and thus to elevate this technique from a strictly comparative method to an investigative one.

Given the number (thousands) and variety (in terms of chemistry) of dyes that have found use in commercial applications, a truly generalized approach is difficult; however, using a carefully selected subset of commercially relevant dyes, it is anticipated that this work will illustrate the value of a generalized approach to the topic of forensic dye identification by a) validating the approach, b) constructing a library of working reference dye data, c) building a framework within which other reference dyes can be incorporated, and d) developing a capability to place constraints on dye class and chemistry, even if the reference dye(s) is not in the collection. Multiple aspects of this project were presented at Inter/Micro 2013.⁶

SAMPLE SELECTION

The colorant industry is continually evolving and colorants are developed and replaced for a variety of reasons: the development of a “high-performance” analog, the removal of a heavy metal, or a change in the color preferences of a market. Some colorants which were popular a decade or two ago are now no longer in production and can be difficult to obtain, in their raw form, as a reference. The dyestuffs chosen here were selected based upon literature and data from colorant manufacturers. The details of the sample selection process have been written up and are included as part of a dyestuff extraction paper that has been submitted for publication (Groves et al., submitted).

Ultimately, a list of 300 dyestuffs was compiled by cross-referencing product offering from major dyestuff manufacturers and literature related to forensic dye analysis. The resulting list consists of commercially available and relevant dyestuffs that are predominately marketed toward textile applications in North America. Samples from the Microtrace colorant collectionⁱⁱ corresponding to this list were identified and are provided in Appendix A. This population was supplemented as necessary to ensure that as many application classes and chemical classes (as defined by the *Colour Index (C.I.)*) as possible were included.

It is important to note that only colorants which have been classified by the Colour Index were selected for this research. Information obtained from dye manufacturing companies suggests that only half of the current colorant market has a C.I. classification. The other half of the available products were either never submitted to the Colour Index (sometimes to protect proprietary dye chemistries) or are mixtures of multiple dyes listed under a trade name. Several companies have stopped submitting to the Colour Index in attempt to protect their products from competitors.

While dyestuffs without C.I. designations certainly cannot be ignored, the authors have decided that using a population of dyes from which chemical information is generally available provides the best possibility for developing a generalized approach to dyestuff characterization.

ⁱⁱ The Microtrace colorant collection consists of over 22,000 samples of raw colorants, colorant precursors and colored (*i.e.*, finished) products. This collection consists of multiple examples of dyestuffs from a variety of manufacturers in both their raw and textile (*i.e.*, finished) form.

DYESTUFF CHARACTERIZATION

Given the wide range of dye chemistries, no single approach or single analytical method can be expected to suffice for the extraction, separation and identification of every dyestuff. That said, the goal here was to develop an approach that would be generally applicable to the largest possible number of dyestuffs. Furthermore, the results of this research are also intended to provide information on the classes and dyes that are not compatible with the generalized methods selected here. In this way, it will be possible to extract at least some classifying information about the colorant system, even if the analytical techniques selected here are not sufficient to identify a particular dyestuff.

A variety of analytical methods were selected for characterization of reference dyes. The methods were chosen on a combination of availability in trace evidence laboratories, their robustness, their anticipated applicability to casework sized samples, their anticipated reproducibility, and their anticipated applicability to the task of dye characterization and identification. This list of techniques explored includes extraction, thin layer chromatography, Raman spectroscopy, infrared spectroscopy, UV-Visible spectroscopy, and alternative light sources.

The analytical classification research here focused on developing a reproducible approach as well as evaluating the potential discriminating power of each method as an independent technique for classifying, constraining and identifying dyestuffs. Ultimately, the benefit of these methods would be their strength in concert, when applied to casework sized samples. While this work is not of sufficient scope to conduct inter-laboratory comparisons, another goal of these analyses is to establish the robust analytical framework which would hold the potential for the inter-comparison of data across multiple laboratories.

THIN LAYER CHROMATOGRAPHY (TLC)

TLC is a cost effective, routine analytical tool which can provide forensic benefit through both separation and identification. Additionally, since this technique is already available and utilized in many forensic laboratories throughout the world, the potential for its adaptation to as a standardized approach in forensic dye analysis (so that reference data could be collected and inter-compared) is not unreasonable. Due to the present lack of a standardized approach for dye analysis, the use of TLC has typically been limited to either a comparative analysis conducted on a case-by-case basis or used with internally developed libraries of reference samples (such as the IRS and Secret Service ink libraries).

The research here focused on a) optimizing and fixing variables that would impact the reproducibility of results, b) collection of reference data, c) examination of sample-to-sample reproducibility of the same dye from different sources, d) the evaluation of dyestuffs on the developed TLC plate by color, fluorescence, number of bands, and retardation factor(s) (R_f) to evaluate the discriminating power of the method.

HPTLC Plate Selection

An evaluation of commercially available HPTLC plates was performed to compare the performance and degree of variation between manufacturers. This survey focused on evaluating the resolution of each plate using a mixture of dyes in which multiple bands share similar R_f values. While many of the plates share similar characteristics, the Merck EMD Millipore (13748) HPTLC plate displayed the best resolution (for this set of dyestuffs) over the other evaluated counterparts. While this survey was not exhaustive, it was sufficient to identify a plate that would provide the adequate resolution for dye analysis.

HPTLC Performance Evaluation

Constraining the use of TLC to a comparative method results from the range of variables that can impact a result. Some of these variable (among a larger list) include the age of an eluting solution, the number of plates developed in a solution, the length of time a tank is allowed to equilibrate, the method, and amount of analyte application to a plate, the storage conditions of the plates, and the activation procedure. In an attempt to achieve a stable state whereby TLC results could be inter-compared, numerous aspects of HPTLC analysis were explored: eluent stability, developing tank saturation, developing distance, pre-elution, concentration limitations. While many of these topics are discussed in the literature,^{7, 8, 9, 10, 11, 12, 13} the wide applicability of TLC results in a broad range of operating conditions not necessarily applicable to the analysis of dyestuffs. The results of this study were presented at Inter/Micro 2014.¹⁴

Standardization of Developing Conditions

Given the wide range of dye chemistries, no single approach or single analytical method can be expected to suffice for the identification of all dyes. That said, the goal here was to develop a method that would be generally applicable to the possible largest set of dyes. Based upon this goal, and taking into account the variables discussed above, a standardized process for developing dyes and monitoring the consistency of results (through the use of a standard dye solution) was formalized. This procedure will be submitted for publication.

HPTLC Data and Discriminating Power

TLC alone can provide a great deal of information about the colorant(s) in a sample. Information that can be obtained includes: the number of components, their color, and the relative concentration of each component with respect to the whole. In total, five characteristics per dyestuff were utilized in evaluating the discriminating power of HPTLC: Color, retardation, fluorescence, number of bands/components, and band shape. These results suggest that for the 300 dyes studied, color and R_f alone are sufficient to identify 20% specifically. Approximately 75% of the population of dyes studied can be narrowed to five possible dyes (which can then be further constrained by other analytical methods or characteristics of the dyestuff (*e.g.*, fluorescence)). The results of this evaluation were presented at Inter/Micro 2014¹⁴ and are also being compiled for publication.

INSTRUMENTAL CHARACTERIZATION

Raman

The development of Raman microspectroscopy has opened a new avenue for the possibility of identifying colorants in a consistent and reliable manner. A manual for practitioners regarding the systematic identification of a broad set of approximately 300 commercially important pigments was published by our laboratory in 2011.¹⁵ To a similar end, Raman spectra were collected from both the solid (raw) dyestuff and from the separated TLC bands of each raw dye (which often consisted of multiple bands for a given solid). Interpretation of this data is more complex than for the pigment project; however, certain trends in the Raman spectra have been identified that, in some cases, permit classification to a specific chemical group. The use of this data, combined with other available analytical data for the reference dyes, has been successfully applied to identify dyes in numerous blind trials and casework samples.

Micro-FTIR

Infrared spectra were collected from the raw dye samples in anticipation that this data could supplement HPTLC analyses to provide added discrimination capabilities. This data serves as a secondary chemical identification tool for dye analysis (in conjunction with Raman spectroscopy). However, the *in situ* FTIR spectrum of dyes in fibers are typically difficult if not impossible to observe due to their presence at concentrations typically <<1% and the strong

absorption bands from the fiber matrix. It was anticipated that FTIR spectra of extracted and separated dyes, which were subsequently eluted from the TLC plate could provide useful information. Unfortunately, surfactants native to the TLC plates evaluated provided a significant interferences with obtaining spectra of the dyestuff.

Visible Absorption Spectroscopy

UV-VIS and MSP spectra of the raw dyes (in solution) were collected. In addition, the MSP spectra of numerous fibers, dyed with known dyestuffs, were collected to evaluate the discrimination and classification potential of visible absorption spectra. This work led to the development of a capillary MSP method, whereby small volumes of fiber extract may be analyzed in a flat capillary cell to obtain data from fibers that are otherwise problematic. This work has been submitted for publication (Hargrave et al., submitted). Furthermore, the visible absorption reference spectra have been shown to be valuable in constraining the type of dye present on a minute length of fiber in casework.

EVALUATION OF DYED FIBERS

Historically forensic dye analysis has been a comparative analysis due to the difficulty in pursuing the identification of individual dye compounds. Here we explored the use of HPTLC as an investigative tool for the purpose of separating and purifying dyes for analysis and identification. Based upon the list of 300 reference dyes of commercial interest, approximately 200 fiber samples dyed with one of these known dyes was the dyes were characterized through a variety of analytical techniques beginning with TLC analysis. The 200 fiber samples were selected from a collection of manufacturer produced shade cards (fibers and fabrics of known type, dyed with known dyes, at specified concentrations) (listed in Appendix B).

In order to conduct this analysis, the standardized TLC approach was further expanded to include a generalized extraction approach (discussed further below). The results were evaluated to compare the consistency of reference data collected from the raw dyes with that obtained from known dyes on fibers.

EXTRACTION

The classical system for dye isolation (*i.e.*, extraction) was evaluated on nearly two-thirds of the studied dyestuffs. This evaluation demonstrates the applicability of pyridine:water as an extraction solvent for the isolation dyestuffs for subsequent analysis outside of their matrix. This generalized extraction approach and the results of its application to nearly two hundred reference fibers have been submitted for publication (Groves et al., submitted). These extracted dyestuffs were then analyzed using HPTLC in accordance with the constraints previously established. This served as a blind test for the feasibility of dye identification using HPTLC in conjunction with Raman spectroscopy. The results of this evaluation are being written up as the conclusive result of this project as a whole.

INTERPRETATION

Data collected from the dyed fibers, including: MSP spectra, solubility data, HPTLC retardation factor(s), color(s), and fluorescence properties have been compiled and compared to the database of reference data in order to

evaluate the applicability of these methods to the classification and identification of dyes by these methods. The results from this study are being prepared for publication.

PRACTICAL EXAMPLES

The information derived from the above methods (MSP, solubility, FTIR, Raman, HPTLC and fluorescence) have been utilized to study several unknown samples, both for comparative and identification purposes. In one case, the fibers from two sets of similarly colored yarns (one red and one blue) were processed by these methods. The results explore the comparative values of the various methods (comparison microscopy, MSP, and TLC) and demonstrate that TLC data can provide detailed discriminations based upon the relative amounts of multiple dyes from a fiber as well as provide confirmation and sometimes further discrimination of otherwise similarly colored fibers. The results also illustrate that TLC coupled with Raman spectroscopy can be used to classify and/or identify the dye (or in some cases dyes) in many of these unknown fibers. This latter result provides detailed chemical information about the sample which is otherwise unobtainable using the other analysis methods (*e.g.*, comparison microscopy, MSP). These results are being prepared for publication.

CASEWORK APPLICATIONS

Throughout the course of this research, the approaches and data developed here have been successfully applied to a number of cases to classify and/or identify dyes in fibers (as well as other matrices). The results of these limited studies show that a variety of analytical methods have been required for analysis, but in the end we have been able to utilize the reference data to identify the great majority of the dyes that have been encountered in casework (where we have attempted to pursue dye identification). A select population of these are being prepared as case studies for publication.

SUMMARY

Numerous aspects of dye analysis, from extraction through microanalytical characterization, classification, and finally identification have been explored for a set of 300 commercially prevalent dyestuffs. The goal of this work has been to develop generally applicable approaches to assist forensic laboratories in extending the use of colorant information (in the form of dyes) beyond simple comparison to classification and identification. The results show that dye identification can be conducted using techniques available in many labs. The submitted and in preparation publications will provide formalized approaches to obtaining this data, will illustrate the increased evidentiary significance of such data, and will provide examples of casework in which this approach coupled with the reference data we have accumulated has been applied to identify dyestuffs.

SCHOLARLY PRODUCTS

ARTICLES

Accepted – See Appendix C for Abstracts

Hargrave, K., et al. (accepted) “Capillary Microspectrophotometry,” *The Microscope*. Publication date: 2016.

Groves, E., Palenik CS, Palenik S. (accepted) “A Survey of Extraction Solvents in the Forensic Analysis of Textile Dyes,” *Forensic Science International*. Publication date: April 2016.

In Preparation

A formalized forensic approach to the classification and identification of commercial textile dyes in casework samples. (in preparation) for submission to *J. of Forensic Sciences*.

The relative discriminating power of various forensic methods or the comparison and identification of colorants. (in preparation) for submission to a journal to *Analytical Chemistry*.

Applications of dye identification to forensic trace evidence: Casework Examples (in preparation) for submission to the *J. of Forensic Sciences*.

PRESENTATIONS

Groves, E.G., Hargrave, K.H., Palenik, S.J., and Palenik, C.S. “A generalized approach to forensic dye identification” Submitted for presentation at Inter/Micro 2016, Chicago, IL.

Palenik, C.S., Groves, E.G., and Palenik, C.S. “Dye Identification in Casework: How far can you go?” Submitted for presentation at Inter/Micro 2016, Chicago, IL.

Palenik, C.S. (2015) Keynote Address. Microscopy and the lost art of observation. SCIX 2015, Providence Rhode Island.

Hargrave, K., Palenik, S.J., Beckert, J., Palenik, C.S. (2015) Characterization of Extracted Dyes by Capillary Microspectrophotometry: Proof of Concept. Inter/Micro 2015. Chicago, IL.

Palenik, C.S. (2014) Identification and Significance of Colorants in Forensic Casework. World Forensic Festival (IAFS 2014, AFSN 2014, APMLA 2014), Seoul, Korea.

Palenik, C.S. and Palenik, S.J. (2014) Seeing Color: Practical Methods in Pigment Microscopy. Inter/Micro 2014, Chicago, IL.

Hargrave, K., Beckert, J., Palenik, C.S., White, K., Sigman, M. (2014) The Comparison of Similarly Colored Fabrics and Yarns Using Comparison Microscopy and Microspectrophotometry. Inter/Micro 2014, Chicago, IL.

White, K.M., Palenik, C.S., Beckert, J.B., and Hargrave, K. (2014) Evaluating Different Methods of Comparison for Fibers with Subtle Variations in Dye Concentration. Inter/Micro 2014, Chicago, IL.

Palenik, C.S. "Practical colorant identification applied to forensic science." Invited presentation at SCIX 2013

Palenik, S. and Palenik, C. "Development of a Systematic Approach to Forensic Dye Identification." Inter/Micro, Chicago, IL. 2013.

Groves, E. and Palenik, C. "Colorant Basics: Chemical Organization of a Dye and Pigment Database." Inter/Micro, Chicago, IL. 2013.

Hargrave, K. and Palenik, S. "A New Technique for the Identification of Dyes Extracted from Single Fibers." Inter/Micro, Chicago, IL. 2013.

Groves, E. "The Discriminating Power of High Performance Thin Layer Chromatography (HPTLC) For Commercial Textile Dyestuffs." Inter/Micro, Chicago, IL. 2014

Hargrave, K. "Characterization of Extracted Dyes by Capillary Microspectrophotometry: Proof of Concept." Inter/Micro, Chicago, IL. 2015.

Beckert, J.C., Groves, E.G., and Palenik, C.S. (2016) Workshop to McCrone Research Institute Staff, scheduled for January 8, 2016. A one day class designed to provide them with advanced knowledge of colorant analysis for application to their mission of not for profit teaching.

IMPLICATIONS FOR CRIMINAL JUSTICE POLICY AND PRACTICE IN THE UNITED STATES

Despite their prevalence in nearly every commercially produced product and the wide range of variation that can exist in this category of evidence, dyes and pigments have remained as a largely unexploited component of evidence. This is due to the fact that they are difficult to analyze, which arises from their low concentration (typically less than 1-2% in a finished product), small size (often in the nanoparticle range), and the varied range of chemical compositions that they represent. Through this and prior NIJ funded research, we have developed systematic, generalized approaches to the identification of pigments and now dyes in forensic evidence such as paint, cosmetics, polymers, and fibers. This project has generalized and formalized the processes for classifying and identifying dyes in the examination of fiber evidence. The approach is based upon mature analytical methods (many of which are already available in forensic laboratories) that are applicable to casework sized samples. The approach has been validated through the analysis of reference samples and has been demonstrated to be effective in both blind and real casework samples. The results from this research have been, and continue to be, distributed to forensic trace evidence examiners through publications, presentations at meetings, workshops and classes. This research is anticipated to provide a basis from which laboratories can expand their approach from a generalized comparison of color between questioned and known materials to a specific identification of the colorants present in a sample. The specific identification of individual colorants in a sample holds the potential to increase the significance of results in the prosecution or defense of a case. These results also hold the potential to significantly improve the value of investigative leads in cases where a comparison sample does not exist by constraining or identifying specific dyes. In some cases, it is possible to determine the specific manufacturer of a dye and its concentration in the fiber, which leads to information about the amount produced and the types of products in which it is used. When coupled with other physical evidence, such as the polymer and cross section, a great deal of specificity may be achieved. In summary, the results of this work will help to expand the scope and significance of fiber examinations and trace evidence examinations, in general, by expanding their scope to include the identification of dyes in forensic casework.

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APPENDIX A – LIST OF DYES STUDIED IN THIS RESEARCH

C.I. Common Name	Chemical Class	C.I. Common Name	Chemical Class	C.I. Common Name	Chemical Class
C.I. Acid Black 1	Disazo	C.I. Acid Red 1	Monoazo	C.I. Basic Blue 3	Oxazine
C.I. Acid Black 24	Disazo	C.I. Acid Red 14	Monoazo	C.I. Basic Blue 9	Thiazine
C.I. Acid Black 52	Monoazo	C.I. Acid Red 18	Monoazo	C.I. Basic Blue 17	Thiazine
C.I. Acid Black 60	Monoazo	C.I. Acid Red 51	Xanthene	C.I. Basic Blue 26	Triarylmethane
C.I. Acid Black 63	Monoazo	C.I. Acid Red 52	Xanthene	C.I. Basic Blue 41	Monoazo
C.I. Acid Black 107	Monoazo	C.I. Acid Red 57	Monoazo	C.I. Basic Blue 45	Anthraquinone
C.I. Acid Black 132	Azo	C.I. Acid Red 87	Xanthene	C.I. Basic Blue 47	Anthraquinone
C.I. Acid Black 172	Monoazo	C.I. Acid Red 88	Monoazo	C.I. Basic Blue 54	Monoazo
C.I. Acid Black 194	Azo	C.I. Acid Red 92	Xanthene	C.I. Basic Brown 1	Disazo
C.I. Acid Black 210	Trisazo	C.I. Acid Red 119	Disazo	C.I. Basic Green 4	Triarylmethane
C.I. Acid Blue 9	Triarylmethane	C.I. Acid Red 127	Monoazo	C.I. Basic Green 5	Thiazine
C.I. Acid Blue 25	Anthraquinone	C.I. Acid Red 131	Monoazo	C.I. Basic Orange 1	Monoazo
C.I. Acid Blue 40	Anthraquinone	C.I. Acid Red 138	Monoazo	C.I. Basic Orange 21	Methine
C.I. Acid Blue 45	Anthraquinone	C.I. Acid Red 151	Disazo	C.I. Basic Red 1	Xanthene
C.I. Acid Blue 62	Anthraquinone	C.I. Acid Red 182	Monoazo	C.I. Basic Red 2	Azine
C.I. Acid Blue 74	Indigoid	C.I. Acid Red 249	Monoazo	C.I. Basic Red 5	Azine
C.I. Acid Blue 80	Anthraquinone	C.I. Acid Red 266	Monoazo	C.I. Basic Red 14	Methine
C.I. Acid Blue 92	Monoazo	C.I. Acid Red 299	Disazo	C.I. Basic Red 18	Monoazo
C.I. Acid Blue 93	Triarylmethane	C.I. Acid Red 315	Monoazo	C.I. Basic Red 46	Monoazo
C.I. Acid Blue 113	Disazo	C.I. Acid Red 337	Monoazo	C.I. Basic Violet 3	Triarylmethane
C.I. Acid Blue 129	Anthraquinone	C.I. Acid Red 360	Unknown	C.I. Basic Violet 10	Xanthene
C.I. Acid Blue 158	Monoazo	C.I. Acid Red 361	Monoazo	C.I. Basic Violet 14	Triarylmethane
C.I. Acid Blue 171	Monoazo	C.I. Acid Red 414	Azo	C.I. Basic Violet 16	Methine
C.I. Acid Blue 182	Monoazo	C.I. Acid Red 419	Monoazo	C.I. Basic Yellow 11	Methine
C.I. Acid Blue 193	Anthraquinone	C.I. Acid Red 447	Unknown	C.I. Basic Yellow 13	Methine
C.I. Acid Blue 204	Anthraquinone	C.I. Acid Violet 7	Monoazo	C.I. Basic Yellow 15	Monoazo
C.I. Acid Blue 205	Anthraquinone	C.I. Acid Violet 12	Monoazo	C.I. Basic Yellow 28	Methine
C.I. Acid Blue 221	Anthraquinone	C.I. Acid Violet 17	Triarylmethane	C.I. Basic Yellow 29	Methine
C.I. Acid Blue 260	Anthraquinone	C.I. Acid Violet 43	Anthraquinone	C.I. Basic Yellow 51	Methine
C.I. Acid Blue 264	Anthraquinone	C.I. Acid Violet 48	Anthraquinone	C.I. Direct Black 19	Trisazo
C.I. Acid Blue 277	Anthraquinone	C.I. Acid Violet 126	Anthraquinone	C.I. Direct Black 22	Trisazo
C.I. Acid Blue 281	Anthraquinone	C.I. Acid Yellow 1	Monoazo	C.I. Direct Black 80	Trisazo
C.I. Acid Blue 284	Monoazo	C.I. Acid Yellow 3	Quinoline	C.I. Direct Black 112	Trisazo
C.I. Acid Blue 290	Unknown	C.I. Acid Yellow 7	Aminoketone	C.I. Direct Black 170	Trisazo
C.I. Acid Blue 298	Unknown	C.I. Acid Yellow 11	Monoazo	C.I. Direct Blue 71	Trisazo
C.I. Acid Blue 324	Anthraquinone	C.I. Acid Yellow 17	Monoazo	C.I. Direct Blue 80	Disazo
C.I. Acid Brown 355	Monoazo	C.I. Acid Yellow 19	Monoazo	C.I. Direct Blue 85	Phthalocyanine
C.I. Acid Green 16	Triarylmethane	C.I. Acid Yellow 23	Monoazo	C.I. Direct Blue 86	Phthalocyanine
C.I. Acid Green 25	Anthraquinone	C.I. Acid Yellow 49	Monoazo	C.I. Direct Blue 90	Disazo
C.I. Acid Green 27	Anthraquinone	C.I. Acid Yellow 59	Monoazo	C.I. Direct Blue 106	Oxazine
C.I. Acid Green 28	Anthraquinone	C.I. Acid Yellow 127	Monoazo	C.I. Direct Blue 108	Oxazine
C.I. Acid Green 73	Monoazo	C.I. Acid Yellow 137	Monoazo	C.I. Direct Blue 199	Phthalocyanine
C.I. Acid Green 81	Anthraquinone	C.I. Acid Yellow 151	Monoazo	C.I. Direct Blue 200	Azo
C.I. Acid Orange 3	Nitro	C.I. Acid Yellow 159	Disazo	C.I. Direct Blue 218	Disazo
C.I. Acid Orange 7	Monoazo	C.I. Acid Yellow 194	Azo	C.I. Direct Blue 286	Unknown
C.I. Acid Orange 10	Monoazo	C.I. Acid Yellow 199	Monoazo	C.I. Direct Brown 1	Trisazo
C.I. Acid Orange 60	Monoazo	C.I. Acid Yellow 219	Disazo	C.I. Direct Green 26	Trisazo
C.I. Acid Orange 74	Monoazo	C.I. Acid Yellow 220	Monoazo	C.I. Direct Green 28	Monoazo
C.I. Acid Orange 116	Disazo	C.I. Acid Yellow 230	Unknown	C.I. Direct Orange 34	Stilbene
C.I. Acid Orange 156	Disazo	C.I. Acid Yellow 246	Monoazo	C.I. Direct Orange 37	Stilbene

List of dyes continued.

C.I. Common Name	Chemical Class	C.I. Common Name	Chemical Class	C.I. Common Name	Chemical Class
C.I. Direct Orange 39	Stilbene	C.I. Disperse Orange 29	Disazo	C.I. Reactive Blue 160	Monoazo
C.I. Direct Orange 72	Disazo	C.I. Disperse Orange 30	Monoazo	C.I. Reactive Blue 171	Disazo
C.I. Direct Orange 102	Disazo	C.I. Disperse Orange 37	Monoazo	C.I. Reactive Blue 198	Unknown
C.I. Direct Red 16	Disazo	C.I. Disperse Orange 41	Monoazo	C.I. Reactive Blue 203	Azo
C.I. Direct Red 23	Disazo	C.I. Disperse Orange 44	Monoazo	C.I. Reactive Blue 220	Formazan
C.I. Direct Red 24	Disazo	C.I. Disperse Orange 73	Monoazo	C.I. Reactive Blue 250	Azo
C.I. Direct Red 28	Disazo	C.I. Disperse Red 1	Monoazo	C.I. Reactive Green 12	Phthalocyanine
C.I. Direct Red 75	Disazo	C.I. Disperse Red 5	Monoazo	C.I. Reactive Green 19	Azo
C.I. Direct Red 79	Disazo	C.I. Disperse Red 11	Anthraquinone	C.I. Reactive Orange 16	Monoazo
C.I. Direct Red 80	Polyazo	C.I. Disperse Red 17	Monoazo	C.I. Reactive Orange 72	Monoazo
C.I. Direct Red 81	Disazo	C.I. Disperse Red 50	Monoazo	C.I. Reactive Orange 84	Disazo
C.I. Direct Red 83	Disazo	C.I. Disperse Red 54	Monoazo	C.I. Reactive Orange 122	Azo
C.I. Direct Red 83:1	Disazo	C.I. Disperse Red 60	Anthraquinone	C.I. Reactive Red 43	Monoazo
C.I. Direct Red 89	Disazo	C.I. Disperse Red 73	Monoazo	C.I. Reactive Red 49	Monoazo
C.I. Direct Red 95	Unknown	C.I. Disperse Red 82	Monoazo	C.I. Reactive Red 120	Disazo
C.I. Direct Red 212	Trisazo	C.I. Disperse Red 86	Anthraquinone	C.I. Reactive Red 141	Disazo
C.I. Direct Red 227	Disazo	C.I. Disperse Red 91	Anthraquinone	C.I. Reactive Red 158	Azo
C.I. Direct Red 239	Disazo	C.I. Disperse Red 92	Anthraquinone	C.I. Reactive Red 159	Unknown
C.I. Direct Red 243	Disazo	C.I. Disperse Red 145	Monoazo	C.I. Reactive Red 180	Monoazo
C.I. Direct Yellow 4	Disazo	C.I. Disperse Red 153	Monoazo	C.I. Reactive Red 194	Monoazo
C.I. Direct Yellow 11	Stilbene	C.I. Disperse Red 167:1	Monoazo	C.I. Reactive Red 198	Monoazo
C.I. Direct Yellow 12	Disazo	C.I. Disperse Red 177	Monoazo	C.I. Reactive Red 242	Unknown
C.I. Direct Yellow 44	Disazo	C.I. Disperse Red 277	Azine	C.I. Reactive Violet 5	Monoazo
C.I. Direct Yellow 86	Disazo	C.I. Disperse Red 311	Unknown	C.I. Reactive Yellow 15	Monoazo
C.I. Direct Yellow 106	Stilbene	C.I. Disperse Red 343	Unknown	C.I. Reactive Yellow 17	Monoazo
C.I. Direct Yellow 118	Disazo	C.I. Disperse Violet 1	Anthraquinone	C.I. Reactive Yellow 37	Monoazo
C.I. Direct Yellow 147	Disazo	C.I. Disperse Violet 26	Anthraquinone	C.I. Reactive Yellow 84	Disazo
C.I. Disperse Blue 3	Anthraquinone	C.I. Disperse Violet 28	Anthraquinone	C.I. Reactive Yellow 135	Disazo
C.I. Disperse Blue 26	Anthraquinone	C.I. Disperse Violet 33	Monoazo	C.I. Reactive Yellow 145	Monoazo
C.I. Disperse Blue 27	Anthraquinone	C.I. Disperse Yellow 1	Nitro	C.I. Reactive Yellow 160	Monoazo
C.I. Disperse Blue 56	Anthraquinone	C.I. Disperse Yellow 3	Monoazo	C.I. Reactive Yellow 176	Monoazo
C.I. Disperse Blue 60	Anthraquinone	C.I. Disperse Yellow 5	Monoazo	C.I. Reactive Yellow 186	Monoazo
C.I. Disperse Blue 73	Anthraquinone	C.I. Disperse Yellow 23	Disazo	C.I. Solvent Black 3	Disazo
C.I. Disperse Blue 77	Anthraquinone	C.I. Disperse Yellow 42	Monoazo	C.I. Solvent Black 5	Azine
C.I. Disperse Blue 79	Monoazo	C.I. Disperse Yellow 54	Quinoline	C.I. Solvent Blue 45	Anthraquinone
C.I. Disperse Blue 79:1	Monoazo	C.I. Disperse Yellow 64	Triarylmethane	C.I. Solvent Green 3	Anthraquinone
C.I. Disperse Blue 102	Monoazo	C.I. Disperse Yellow 71	Azo	C.I. Solvent Green 28	Anthraquinone
C.I. Disperse Blue 106	Monoazo	C.I. Disperse Yellow 82	Lactone	C.I. Solvent Orange 60	Aminoketone
C.I. Disperse Blue 124	Monoazo	C.I. Disperse Yellow 86	Monoazo	C.I. Solvent Orange 63	Anthraquinone
C.I. Disperse Blue 148	Monoazo	C.I. Disperse Yellow 114	Monoazo	C.I. Solvent Red 23	Disazo
C.I. Disperse Blue 165	Monoazo	C.I. Disperse Yellow 163	Monoazo	C.I. Solvent Red 24	Disazo
C.I. Disperse Blue 183	Monoazo	C.I. Disperse Yellow 198	Unknown	C.I. Solvent Red 111	Anthraquinone
C.I. Disperse Blue 281	Azo	C.I. Food Green 3	Triarylmethane	C.I. Solvent Violet 13	Anthraquinone
C.I. Disperse Blue 284	Monoazo	C.I. Food Red 17	Monoazo	C.I. Solvent Yellow 14	Monoazo
C.I. Disperse Blue 291	Monoazo	C.I. Food Yellow 3	Monoazo	C.I. Solvent Yellow 56	Monoazo
C.I. Disperse Blue 366	Monoazo	C.I. Ingrain Blue 1	Phthalocyanine	C.I. Solvent Yellow 93	Triarylmethane
C.I. Disperse Brown 1	Monoazo	C.I. Mordant Black 11	Monoazo	C.I. Vat Blue 1	Indigoid
C.I. Disperse Green 9	Monoazo	C.I. Reactive Black 5	Disazo	C.I. Vat Red 13	Anthraquinone
C.I. Disperse Orange 3	Monoazo	C.I. Reactive Black 31	Unknown	C.I. Vat Red 41	Indigoid
C.I. Disperse Orange 25	Monoazo	C.I. Reactive Blue 21	Phthalocyanine	C.I. Vat Violet 1	Anthraquinone

APPENDIX B – TABLE OF DYED FIBERS STUDIED

C.I. Common Name	Fiber Type	C.I. Common Name	Fiber Type	C.I. Common Name	Fiber Type
C.I. Acid Black 1	Wool	C.I. Acid Yellow 23	Nylon	C.I. Disperse Blue 79	Polyester
C.I. Acid Black 24	Nylon	C.I. Acid Yellow 49	Wool	C.I. Disperse Blue 102	Acetate
C.I. Acid Black 52	Rayon	C.I. Acid Yellow 127	Wool	C.I. Disperse Blue 165	Cotton
C.I. Acid Black 60	Nylon	C.I. Acid Yellow 219	Wool	C.I. Disperse Blue 183	Polyester
C.I. Acid Black 107	Nylon	C.I. Basic Blue 9	Rayon	C.I. Disperse Blue 284	Cotton
C.I. Acid Black 172	Nylon	C.I. Basic Green 4	Acrylic	C.I. Disperse Brown 1	Acetate
C.I. Acid Blue 9	Wool	C.I. Basic Orange 21	Acrylic	C.I. Disperse Green 9	Cotton
C.I. Acid Blue 25	Acrylic	C.I. Basic Red 1	Wool	C.I. Disperse Orange 3	Rayon
C.I. Acid Blue 40	Nylon	C.I. Basic Red 14	Acrylic	C.I. Disperse Orange 25	Polyester
C.I. Acid Blue 45	Nylon	C.I. Basic Violet 3	Rayon	C.I. Disperse Orange 29	Modacrylic
C.I. Acid Blue 62	Silk	C.I. Basic Violet 10	Wool	C.I. Disperse Orange 29	Polyester
C.I. Acid Blue 74	Wool	C.I. Basic Violet 16	Acrylic	C.I. Disperse Orange 29	Polyester
C.I. Acid Blue 80	Wool	C.I. Basic Yellow 11	Acrylic	C.I. Disperse Orange 29	Rayon
C.I. Acid Blue 92	Wool	C.I. Basic Yellow 13	Acrylic	C.I. Disperse Orange 29	Silk
C.I. Acid Blue 113	Wool	C.I. Basic Yellow 15	Nylon	C.I. Disperse Orange 29	Wool
C.I. Acid Blue 129	Silk	C.I. Direct Black 19	Cotton	C.I. Disperse Orange 30	Nylon
C.I. Acid Blue 158	Polyester	C.I. Direct Black 22	Cotton	C.I. Disperse Orange 37	Polyester
C.I. Acid Blue 298	Nylon	C.I. Direct Black 80	Cotton	C.I. Disperse Orange 44	Cotton
C.I. Acid Blue 324	Nylon	C.I. Direct Blue 71	Cotton	C.I. Disperse Red 1	Nylon
C.I. Acid Green 16	Wool	C.I. Direct Blue 80	Nylon	C.I. Disperse Red 1	Rayon
C.I. Acid Green 25	Nylon	C.I. Direct Blue 85	Cotton	C.I. Disperse Red 5	Acetate
C.I. Acid Orange 3	Wool	C.I. Direct Blue 86	Cotton	C.I. Disperse Red 11	Nylon
C.I. Acid Orange 7	Acrylic	C.I. Direct Brown 1	Cotton	C.I. Disperse Red 17	Rayon
C.I. Acid Orange 10	Silk	C.I. Direct Green 26	Rayon	C.I. Disperse Red 50	Polyester
C.I. Acid Orange 74	Acrylic	C.I. Direct Green 28	Rayon	C.I. Disperse Red 54	Polyester
C.I. Acid Orange 156	Wool	C.I. Direct Orange 34	Rayon	C.I. Disperse Red 60	Nylon
C.I. Acid Red 1	Silk	C.I. Direct Orange 37	Rayon	C.I. Disperse Red 73	Polyester
C.I. Acid Red 14	Nylon	C.I. Direct Orange 39	Rayon	C.I. Disperse Red 82	Polyester
C.I. Acid Red 18	Wool	C.I. Direct Orange 72	Rayon	C.I. Disperse Red 86	Nylon
C.I. Acid Red 51	Silk	C.I. Direct Orange 102	Nylon	C.I. Disperse Red 91	Polyester
C.I. Acid Red 52	Silk	C.I. Direct Red 16	Rayon	C.I. Disperse Red 92	Polyester
C.I. Acid Red 57	Silk	C.I. Direct Red 23	Rayon	C.I. Disperse Red 153	Polyester
C.I. Acid Red 87	Wool	C.I. Direct Red 24	Rayon	C.I. Disperse Red 167:1	Acrylic
C.I. Acid Red 88	Acetate	C.I. Direct Red 28	Cotton	C.I. Disperse Red 167:1	Modacrylic
C.I. Acid Red 92	Wool	C.I. Direct Red 75	Rayon	C.I. Disperse Red 167:1	Polyester
C.I. Acid Red 119	Nylon	C.I. Direct Red 79	Rayon	C.I. Disperse Red 167:1	Polyester
C.I. Acid Red 127	Acetate	C.I. Direct Red 80	Rayon	C.I. Disperse Red 167:1	Rayon
C.I. Acid Red 131	Nylon	C.I. Direct Red 81	Rayon	C.I. Disperse Red 167:1	Silk
C.I. Acid Red 151	Rayon	C.I. Direct Red 83	Cotton	C.I. Disperse Red 167:1	Wool
C.I. Acid Red 249	Silk	C.I. Direct Red 89	Cotton	C.I. Disperse Red 177	Cotton
C.I. Acid Red 266	Wool	C.I. Direct Yellow 11	Cotton	C.I. Disperse Red 277	Polyester
C.I. Acid Red 337	Wool	C.I. Direct Yellow 12	Cotton	C.I. Disperse Red 311	Cotton
C.I. Acid Violet 7	Nylon	C.I. Direct Yellow 44	Nylon	C.I. Disperse Red 343	Polyester
C.I. Acid Violet 12	Acrylic	C.I. Direct Yellow 106	Cotton	C.I. Disperse Violet 1	Rayon
C.I. Acid Violet 43	Modacrylic	C.I. Disperse Blue 3	Nylon	C.I. Disperse Violet 26	Nylon
C.I. Acid Yellow 1	Wool	C.I. Disperse Blue 3	Rayon	C.I. Disperse Violet 28	Polyester
C.I. Acid Yellow 3	Rayon	C.I. Disperse Blue 27	Silk	C.I. Disperse Violet 33	Polyester
C.I. Acid Yellow 7	Wool	C.I. Disperse Blue 56	Nylon	C.I. Disperse Yellow 1	Nylon
C.I. Acid Yellow 11	Modacrylic	C.I. Disperse Blue 60	Acetate	C.I. Disperse Yellow 3	Cotton
C.I. Acid Yellow 17	Polyester	C.I. Disperse Blue 73	Nylon	C.I. Disperse Yellow 3	Rayon

List of fibers continued.

C.I. Common Name	Fiber Type	C.I. Common Name	Fiber Type	C.I. Common Name	Fiber Type
C.I. Disperse Yellow 5	Rayon	C.I. Reactive Black 31	Rayon	C.I. Reactive Red 159	Cotton
C.I. Disperse Yellow 23	Polyester	C.I. Reactive Blue 21	Rayon	C.I. Reactive Red 180	Rayon
C.I. Disperse Yellow 42	Cotton	C.I. Reactive Blue 160	Cotton	C.I. Reactive Red 194	Cotton
C.I. Disperse Yellow 54	Nylon	C.I. Reactive Blue 171	Nylon	C.I. Reactive Red 198	Rayon
C.I. Disperse Yellow 64	Nylon	C.I. Reactive Blue 198	Cotton	C.I. Reactive Violet 5	Cotton
C.I. Disperse Yellow 82	Acetate	C.I. Reactive Green 19	Cotton	C.I. Reactive Yellow 15	Rayon
C.I. Disperse Yellow 114	Polyester	C.I. Reactive Orange 16	Cotton	C.I. Reactive Yellow 17	Cotton
C.I. Mordant Black 11	Acetate	C.I. Reactive Orange 84	Cotton	C.I. Reactive Yellow 37	Rayon
C.I. Mordant Black 11	Cotton	C.I. Reactive Red 43	Cotton	C.I. Reactive Yellow 84	Cotton
C.I. Mordant Black 11	Nylon	C.I. Reactive Red 43	Linen	C.I. Reactive Yellow 135	Cotton
C.I. Mordant Black 11	Rayon	C.I. Reactive Red 43	Nylon	C.I. Reactive Yellow 145	Cotton
C.I. Mordant Black 11	Silk	C.I. Reactive Red 49	Rayon	C.I. Reactive Yellow 160	Rayon
C.I. Mordant Black 11	Wool	C.I. Reactive Red 120	Cotton	C.I. Vat Red 13	Cotton
C.I. Reactive Black 5	Acetate	C.I. Reactive Red 141	Cotton	C.I. Vat Violet 1	Cotton
C.I. Reactive Black 5	Cotton	C.I. Reactive Red 158	Cotton	C.I. Vat Violet 1	Rayon

Groves, E., Palenik CS, Palenik S. (accepted) "A Survey of Extraction Solvents in the Forensic Analysis of Textile Dyes," Forensic Science International.

The characterization and identification of dyes in fibers can be used to provide investigative leads and strengthen associations between known and questioned items of evidence. The isolation of a dye from its matrix (*e.g.*, a textile fiber) permits detailed characterization, comparison and, in some cases, identification using methods such as thin layer chromatography in conjunction with infrared and Raman spectroscopy. A survey of dye extraction publications reveals that pyridine:water (4:3) is among the most commonly cited extraction solvent across a range of fiber and dye chemistries. Here, the efficacy of this solvent system has been evaluated for the extraction of dyes from 172 commercially prevalent North American textile dyes. The evaluated population represents seven dye application classes, eighteen chemical classes, and spans nine types of commercial textile fibers. The results of this survey indicate that ~82% of the dyestuffs studied are extractable using this solvent system. The results presented here summarize the extraction efficacy by class and fiber type and illustrate that this solvent system is applicable to a wider variety of classes and fibers than previously indicated in the literature. While there is no universal solvent for fiber extraction, these results demonstrate that pyridine:water represents an excellent first step for extracting unknown dyes from questioned fibers in forensic casework.

Hargrave, K., et al. (accepted) "Capillary Microspectrophotometry," The Microscope.

Colorants observed on fibers and hairs in trace evidence examinations are commonly compared via polarized light and comparison microscopy, microspectrophotometry, and in some cases, chromatography. Some of the most difficult samples to evaluate and compare by these methods include lightly dyed fibers (which result in a "noisy" and nearly featureless spectrum due to a low signal to noise ratio), heavily dyed fibers (resulting in the nearly complete absorption of the incident illumination), and heterogeneously dyed fibers (such as vegetable fibers, which provide a range of spectral intensities that complicate interpretation). Here we present a method for the extraction and subsequent analysis of dyes directly from fibers. This approach relies upon the use of a flat microcapillary cell (*i.e.*, a low volume, fixed path length cell) to hold the extract that can then be analyzed by a microspectrophotometer (MSP). This capillary microspectrophotometry (cMSP) method provides a means to obtain reproducible spectra of the colorants from many problematic fibers.