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Taught Postgraduate Programmes
MPhil Textile Conservation

2014-2015

Student's Name: Gennifer Majors

Dissertation Title: Sublime Applications: Creating an Efficient Cyclododecane Barrier on Textiles

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Sublime Applications:
Creating an Efficient Cyclododecane Barrier on Textiles

A Master's Thesis by Gennifer Majors
The Centre for Textile Conservation
University of Glasgow
2015

Abstract

Dye bleed caused by wet cleaning is a common problem during conservation treatments. A protective hydrophobic barrier is sometimes created over such water-sensitive dyes using the wax-like material cyclododecane. However, little research has been done on how to create the most efficient cyclododecane barrier on textiles. Seven different application methods were devised and tested for effectiveness. Samples were soaked in water to find which application method best protected against dye bleed. Gravimetric and visual analysis were used to track any changes in the applied cyclododecane. ATR-FTIR and DRIFTS infrared spectroscopy were performed to discover how well the cyclododecane had coated the sample fibers. The data revealed that the most effective methods involved applying cyclododecane to one side of a piece of fabric while the fabric was at room temperature while applying it to the opposite side while the fabric was heated. This heating allows the cyclododecane to become well-embedded within the fabric where it coats and protects the fibers. The paintbrush and *kistka* application tools were also compared during the experiment with the result that the brush applied more cyclododecane but cyclododecane applied with a *kistka* was more effective against dye bleed. The results were used to create instructions for applying cyclododecane to textiles which conservators can use during treatments.

Acknowledgements

I would like to thank:

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And the Textile Conservation Centre Foundation for years of financial support without which none of this would have been possible.

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Chapter One: Introduction

Introduction

Cyclododecane was originally used in the production of flame retardants and detergents.[1] However, in the early 1990s conservators began to use it for their treatments because of its ability to completely sublime after a period of time, leaving no traces on the objects.[2] The material is chemically simple, containing only 12 carbons and 24 hydrogens combined into the cyclic structure seen in *figure 1.* At room temperature cyclododecane is a white, wax-like solid but it melts thoroughly at the relatively low temperature of 60°C.[3] However, its most important feature is its ability to safely sublimate at room temperature.[4]

Cyclododecane is now commonly used in a variety of conservation fields for a multitude of purposes. These purposes include: temporary consolidant; temporary protective hydrophobic barrier; temporary hydrophobic consolidant; release layer in mould-making; temporary support or physical barrier; and temporary adhesive. As such, ample research has been done on the performance of cyclododecane in these fields. However, the use of cyclododecane for textile conservation is relatively new and so there is little research so far on how to best utilize it for textile conservation purposes. Its common uses in textile conservation include: functioning as a temporary protective hydrophobic barrier for soluble dyes or paints during aqueous treatments; and working as a temporary consolidant for broken or flaking coatings, such as paint, in order to prevent further damage to these coatings during treatment. This experiment focuses on the use of cyclododecane as a hydrophobic barrier for dyes and was inspired by a previous study by A. Bruselius Scharff and I. Nielsen in which they compared 13 application methods of cyclododecane to textiles and paper.[5] Refining the creation of this hydrophobic barrier would allow cyclododecane to be more effectively used and could prevent dye bleed that can irreparably alter or even damage an object. Using the results of this study it is hoped that a precise set of steps for the application of melted cyclododecane to textiles can be generated for textile conservators to use during their treatments.

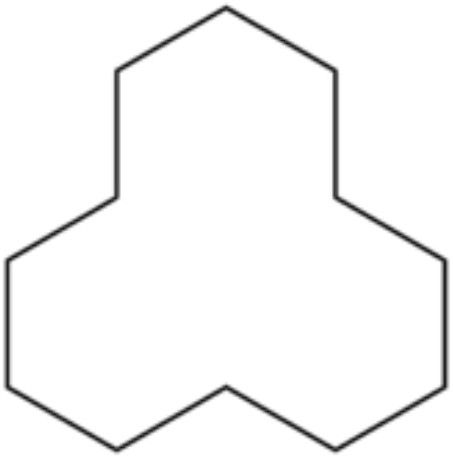

Figure 1: The cyclic structure of cyclododecane, containing 12 carbons that form the main structure and 24 hydrogens attached to the carbons.

[1] Rowe, Sophie, and Christina Rozeik, "The Uses of Cyclododecane in Conservation," *Studies in Conservation* 54, no. 1 (2009): 17.
[2] Rowe, 17.
[3] PubChem, "CYCLODODECANE," *CYCLODODECANE*, accessed August 1, 2015, http://pubchem.ncbi.nlm.nih.gov/compound/cyclododecane#section=Top

[4] Rowe, 17.
[5] Bruselius Scharff, A., and I. Nielsen, "The use of cyclododecane for the fixation of bleeding dyes on paper and textiles: a critical evaluation of application methods," *Dyes in history and archaeology* 19 (2003): 149.

Supportive Research

A variety of literature on the application methodologies and experimental analysis of cyclododecane was reviewed in order to fully develop a rigorous and satisfactory experiment. Because the material is commonly used in conservation, ample research has been done on the performance of cyclododecane in these fields. However, the use of cyclododecane specifically for textile conservation is relatively new and so there is little research so far on how to best utilize it for textile conservation purposes. Its common uses in textile conservation include: functioning as a temporary protective hydrophobic barrier for soluble dyes or paints during aqueous treatments; and working as a temporary consolidant for broken or flaking coatings, such as paint, in order to prevent further damage to these coatings during treatment. Fortunately, due to previous research on cyclododecane, there exists a number of works on the material's chemistry, health risks, application to objects like paper that are somewhat similar to textiles, and the use of analytical methods for the evaluation of cyclododecane.

Application Methods

The inspiration for this study was the experiment performed by Bruselius Scharff and Nielsen [2] in which they tested 13 methods of applying cyclododecane to textile and paper substrates to form a hydrophobic barrier. They applied the cyclododecane using a kistka, a wax-melting pen, and found that the most effective method was to apply cyclododecane to the front of a textile substrate when it was cold and then apply cyclododecane to the back of the textile substrate when it was warmed with a heated spatula. However, there was only one textile sample created and tested and so the results may not be fully representative. Brückle et al. [1] also applied melted cyclododecane to a heated substrate. This allowed the cyclododecane to flow easier, forming a more even layer with better penetrative abilities.

Watters [8] used a brush to apply melted cyclododecane but notes that creating an even, thin layer is "impossible" as it solidifies too quickly. However, S. Confer [3] had great success creating thin and even layers of cyclododecane with a brush on textile substrates. This may have been aided by her use of a double-boiler to keep the cyclododecane heated to 5° C above its melting temperature.

Munoz-Vinas [5] and the other authors in this section all noted that the efficacy of a hydrophobic cyclododecane layer is improved by the penetration of the material into the substrate, and that heating the substrate increases this penetration.

The study by Confer [3] compared cyclododecane sublimation from silk, wool, cotton, and linen in order to discover whether the fiber type had any effect on the sublimation. The effect the fibers seemed to have was that dimensional fibers like wool and cotton slowed sublimation by possibly physically restraining the cyclododecane.

The literature review by Rowe [6] was an invaluable source as compiled chemical, health and safety, and application method information about cyclododecane. This work was especially written for conservators and even has a section on the uses of cyclododecane for textile conservation.

Analytical Methods

Confer [3] used a combination of gravimetric, visual, and FTIR analysis in order to study to sublimation rates of cyclododecane from a variety of textile substrates. Scharff [2] tested their

samples by subjecting them to various dye bleed tests. Watters [8] generated detailed gravimetric data during analysis of the sublimation rates of various amounts of cyclododecane.

Smith [7] and Derrick [4] provide two books on infrared spectroscopy, including FTIR analysis. Both include a wealth of information and yet are not outside of the realm of a conservator's knowledge. One [4] is even written specifically for generating and analyzing infrared spectra for conservation purposes.

Reviewing the literature clarified the substrates, application methods, application tools, and analytical methods that could be used in this study. Cotton and silk were chosen to be the substrates based on the physical differences of the fibers and because they are commonly treated in textile conservation. A *kistka* and a brush would be used to apply cyclododecane to the substrates via methods where the temperature of the substrate and the cyclododecane were variated. After application, dye bleed testing, gravimetric analysis, visual analysis, and infrared spectroscopy would be completed in order to evaluate the efficacy of these cyclododecane applications.

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2. Bruselius Scharff, A., and I. Nielsen. "The use of cyclododecane for the fixation of bleeding dyes on paper and textiles: a critical evaluation of application methods." *Dyes in history and archaeology* 19 (2003): 149-154.

3. Confer, S. "Cyclododecane: an examination of the rate of sublimation from various textile substrates." Master's thesis, Queens University, Kingston, Ontario, 2006.

4. Derrick, Michelle R. et al. Infrared Spectroscopy in Conservation Science. Los Angeles: Getty Conservation Institute, 1999.

5. Muñoz-Viñas, Salvador. "A Dual-Layer Technique for the Application of a Fixative on Water-Sensitive Media on Paper." *Restaurator* 28, no. 2 (2007).

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7. Smith, Brian. *Infrared Spectral Interpretation: A Systematic Approach*. Boca Raton, London, New York, Washington, DC: CRC Press, 1999.

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Aims and Objectives

Aim

- Create cyclododecane methods that can be easily and safely used in real-world treatments.

Objectives

- Test the methods on silk and cotton, two fibers commonly treated with cyclododecane.
- Use a paintbrush and a *kistka,* tools commonly used to apply melted cyclododecane to objects.
- Use other tools such as pipettes and heated spatulas that are commonly used in conservation and are generally safe for objects.
- Any aspect of the application method should be easy to replicate in a conservation setting and generally safe for objects.

Aim

- Find a method that is effective as a hydrophobic barrier.

Objectives

- Test the bleeding of a dye that is highly mobile in water and that provides bleeding that is visible both in the bath water and on the sample.
- Complete a rigorous wash bath in order to test for dye bleeding.
- Complete visual analysis of the samples in order to find signs of dye bleeding.

Aim

- Discover why that method is more effective than others.

Objectives

- Complete gravimetric analysis of the samples in order to find any relation between the application methods and the retention of cyclododecane, as measured by weighing the mass of the cyclododecane present throughout sublimation.
- Complete visual analysis of the samples in order to find any relation between the application methods and the visible signs of the presence of cyclododecane throughout sublimation.
- Complete infrared spectroscopy of the samples in order to find any relation between the application methods and the retention of cyclododecane after sublimation.

Aim

- Provide a set of recommended application steps for use in future conservation treatments.

Objectives

- Compile the steps from the effective method and give brief explanations for why these are effective.
- Provide suggestions that may further improve this method.
- Provide expectations for how the treated area will act during application and sublimation.

Chapter Two: Methodology, Creating the Samples

Methodology: Creating the Samples

For the purposes of this experiment, it was calculated that 112 samples were needed. Two substrate fabrics were chosen for testing, and on each substrate seven different application methods (discussed below) were used, each executed using both a paintbrush and an electric wax-melting pen called a *kistka.* Four samples would be created for each application method using each tool, on each of the two substrate fabrics, since creating multiples of any sample in an experiment ensures that the resulting data will be of a higher quality and consistency. To further ensure the quality of the data, all of the samples were created to be as similar and consistent as possible. Each sample consisted of a fabric substrate that was stamped with a water-soluble dye and then coated with a layer of cyclododecane.

Making the Sample Substrates

In order to determine which substrates to use for the experiment, research was done to determine which fabrics would best represent real-world applications of cyclododecane in textile conservation. Objects made with cotton and silk are commonly treated with aqueous cleaning methods in textile conservation and sometimes contain water-sensitive elements. Based on the study by Confer cotton and silk are, in a sense, opposites since cotton fibers are highly shaped and cellulosic and silk fibers are very featureless and proteinaceous. In the same experiment linen was tested as a featureless cellulosic, while wool acted as the highly shaped protein fiber. However, there was little to no difference in results noticed between the proteinaceous and cellulosic fibers that shared similar physical features (i.e. linen and silk), although some differences in the results were theorized as having been caused by the highly shaped fibers physically holding onto the cyclododecane more firmly than the featureless fibers, causing increased sublimation times.[6] Because of this difference caused by the fibers' physical shape and because the two materials are often treated in textile conservation, cotton and silk were chosen as the two substrates for this experiment. Another reason was that sourcing a cotton and a silk fabric with similar thread counts and densities was much easier than sourcing a similar wool and silk fabric. Although not crucial to the experiment, using two substrates that were as similar as possible would allow the results to be easier to manipulate during the following analysis and future evaluation by readers.

Because physical characteristics of the fiber and the weave might affect the results of the experiment, it was decided that any substrate chosen should be a balanced plain-weave with warps and wefts that were very similar. Initial testing was done on various cottons and silks to determine how successful the application methods would be on these substrates. This testing showed that thicker substrates would be required than initially anticipated because the melted cyclododecane immediately and thoroughly saturated the yarns of the thinner substrates, an action that would have made it impossible to determine whether one application method caused the cyclododecane to better penetrate the fibers than another application method. A suitable cotton was quickly found. However, procuring a silk fabric with a dense, balanced plain-weave made of thick warps and wefts proved difficult. The silk substrate that was eventually chosen was thick enough that it was not immediately saturated with melted cyclododecane and its warps and wefts were very similar, but the weave was an unbalanced plain-weave. This difference should

[6] Confer, S. "Cyclododecane: an examination of the rate of sublimation from various textile substrates." Master's thesis, Queens University, Kingston, Ontario, 2006.

be noted as it may have caused slight differences between the analysis results of the cotton and the silk substrate samples.

The cotton that was chosen as a substrate was 0.316 mm thick, had a thread count of 100 ends per cm x 100 picks per cm, and a density of 0.4604 g/cm3. The silk substrate was 0.175 mm thick, had a thread count of 100 ends per cm x 70 picks per cm, and a density of 0.5013 g/cm3. Squares approximately 10x10cm were cut from both fabrics. The size of each square varied slightly, but the mass of each was 1.20±0.005g when cotton and 0.75±0.005g when silk. Ensuring the consistency of mass for each square was important so that this would not cause false results during later gravimetric analysis, which will be further discussed later.

The substrate squares were stamped with a water-soluble dye so that the samples could be tested for dye bleeding, which will be further discussed later. Indigo carmine was chosen as the dye as it was readily available, safe to use, and preliminary testing showed that it dyed and bled favorably. The dye did not spread incredibly quickly through the fibers when applied to the substrates and so was easier to control. Also, when applied to blotting paper and submerged in water, the indigo carmine bled not only into the water but onto the surrounding area of paper so that evidence of the dye bleeding was preserved. The indigo carmine also did not separate into a variety of component colors like other water-soluble colorants may. 0.50g of pure powdered indigo carmine was dissolved in 50mL of water as per the solubility instructions on the compound's MSDS sheet.[7]

A stamp was created from a 1.7x1.7cm2 piece of latex make-up sponge as the material was readily available and worked well to soak up an appropriate amount of indigo carmine for applying to the substrates. Placing the fabric onto a piece of blotting paper to absorb any excess dye, the center of each substrate square was stamped with indigo carmine. This area of stamped dye, which will be referred to as the dye mark, was then outlined on one face of the fabric using a black water-proof ballpoint pen so that any dye movement during the dye bleed testing would be more easily distinguished. The face with this outline was designated as the front of each sample.

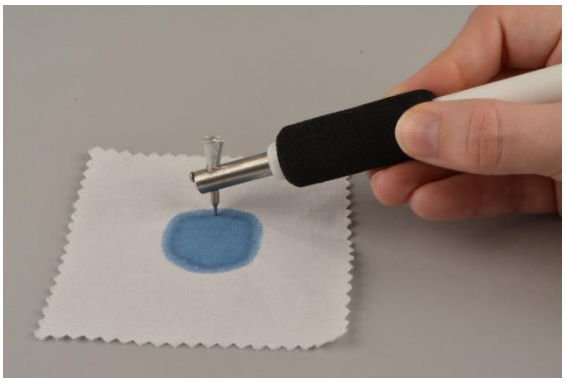
The cotton and the silk substrate squares were then randomly divided in half, and then into seven sets of four. This was because there were to be seven *kistka* application methods tested four times each and seven brush application methods tested four times each, using both the cotton and the silk substrates. The substrate squares were then labelled with uppercase letter A-G to indicate which of the seven application methods was used, then number 1-4 to indicate which repeat the sample was, and then lowercase b or k to indicate whether the tool used was a brush or a *kistka.* When necessary, the letter B or F was added to the end of this label in order to indicate whether the back or the front of the sample was studied.

Using the Tools

[7] ScienceLab, "Material Safety Data Sheet: FD&C Blue #2," Last modified 21, 5, 2013, www.sciencelab.com/msds.php?msdsId=9924015

Two tools were used to apply cyclododecane to the sample substrates, and seven different application methods were executed using each tool. A flat, *hake* style brush was used to apply melted cyclododecane to half of the cotton and silk sample substrates. The cyclododecane was melted at approximately 70-80° Celsius using a beaker placed inside an electric baby bottle and food warmer. This warmer was very convenient as it was easy to use and kept the cyclododecane in its liquid state, which can be difficult as the cyclododecane begins to solidify immediately when removed from heat. In a study on using cyclododecane as a stone consolidant by Stein et al a specified volume of cyclododecane was applied to each test piece. It was noted that the accuracy of their results would have been improved if instead a consistent mass of cyclododecane had been used for each piece.[8] However, it was difficult to ensure that consistent amounts of cyclododecane were being applied to each sample. In order to make sure that the amounts of cyclododecane applied were as similar as possible, for each sample the brush was saturated with the melted cyclododecane then wiped three to five times on the wall of the beaker to remove any excess. The loaded brush was placed just to the left of the dye mark and then quickly drawn to the right to cover the dye mark with only one pass. The loaded brush was first placed to the side of the dye mark as initially pressing the brush to the substrate deposited more cyclododecane than the brushing motion itself, which would have caused false and inconsistent results. The brushing motion had to be completed quickly as the cyclododecane began to solidify on the brush as soon as it was removed from the bottle warmer, which at times caused the brush to stick to the substrate.

Creating a Sample using a Paintbrush



Creating a sample using a kistka

An electric wax-melting pen called a *kistka* was used to apply cyclododecane to the remaining sample substrates. The *kistka* is shaped like a writing utensil which can be loaded with any wax-like substance via a metal reservoir. The loaded substance melts as the metal is heated to approximately 93° Celsius and then runs out of a fine tip at the bottom of the reservoir. Starting at a random point on the dye mark, the tip of the *kistka* was applied to the substrate and melted cyclododecane was drawn from the tip into the fibers. This fine tip created a narrow line of melted cyclododecane and so required many passes in order to completely cover the dye mark, unlike the brush that covered the dye mark in one pass. As a result, it took at least six times longer to create samples with a *kistka* than it had using a brush and small areas could have easily been missed, creating gaps in the cyclododecane layer. Also unlike the brush, the cyclododecane applied with the *kistka* had no time to cool and solidify before reaching the substrate. The

[8] Stein, Renee, Jocelyn Kimmel, Michele Marincola, and Friederike Klemm, "Observations on Cyclododecane as a Temporary Consolidant for Stone," *Journal of the American Institute for Conservation* 39 (2000): 366.

reservoir was very small and so inserting the unevenly shaped chunks of cyclododecane was difficult and resulted in uneven melting. Instead, narrow plastic pipettes were filled with melted cyclododecane, cooled, and then cut open in order to create "sticks" of cyclododecane that easily slotted into the reservoir and melted evenly. Applying consistent amounts of cyclododecane was easier than with the brush as the "sticks" could be weighed before being inserted into the reservoir. Preliminary testing showed that 0.2g of "stick" would cover one face of the dye mark on cotton, and 0.1g of "stick" for silk.

Applying the Cyclododecane

It was initially theorized that heating the cyclododecane and the substrate in some manner would promote the penetration of the cyclododecane into the fibers, which would allow for a more effective hydrophobic barrier. Seven different methods were used to apply the cyclododecane to the sample substrates using both the brush and the *kistka.* These methods were labelled A through G and had changes in temperature as a variable. In every case the cyclododecane was applied to the designated front of the sample substrate, allowed to solidify, and then applied to the back. All seven methods were designed so as to be easily replicated for actual object treatments without causing damage to the object. An electrically heated spatula was used for application during methods B, C, D, and G. A small electric room heater was used for methods E and F to heat the metal surface and increase the room temperature.

Table 1.1: Terms and Meanings in Table 1.2	
"cold"	Substrate is left at room temperature (approximately 23° Celsius except in cases of "WE")
"hot"	Substrate is heated with a spatula (approximately 60-65° Celsius) for approximately 20 seconds
"WE"	Warmed Environment The cyclododecane was applied while the substrates were on a heated metal surface in a warmed room. The metal surface and substrates were heated to approximately 35° Celsius, while the room temperature was heated to approximately 30° Celsius.
"PAS"	Post-Application Spatula After it has solidified, cyclododecane applied to substrate is heated with a spatula (approximately 60-65° Celsius) for approximately 20 seconds

Label	**Table 1.2: Application Method**
A	Cyclododecane applied to the sample front and then the back while both faces were "cold".
B	Cyclododecane applied to the sample front while "cold", then to the back while "hot".
C	Cyclododecane applied to the sample front while "hot", then to the back while "cold".
D	Cyclododecane applied to the sample front and then back while both were "hot".
E	"WE" Cyclododecane applied to the sample front and then the back while both faces were "cold". The samples were then cooled to approximately 23° Celsius.
F	"WE" Cyclododecane applied to the sample front and then the back while both faces were "cold". The samples were then cooled to approximately 10° Celsius.
G	Cyclododecane applied to the sample front and then the back while both faces were "cold" and then the samples were given "PAS".

Notes on Application

There were some small, unexpected things that were noticed during the application process which are important as they may have had an impact on the final results of this study and they may inform future applications of cyclododecane during treatments.

Regardless of tool or application method, the cyclododecane applied to the front of the sample sometimes seeped through to the sample back. When the back was then coated in cyclododecane, it resulted in a back layer that was much thicker and more uneven than that on the front. The front layer was instead very thin as the cyclododecane had soaked into the fibers. This effect occurred much more commonly on the cotton samples than on the silk; more while using the *kistka* than the brush; and more during the C and D application methods than any other. At times, the cyclododecane that soaked through to the back would solidify, adhering the sample to the work surface and causing a rough surface on the sample back when peeled from the work surface. When cyclododecane was applied to the back over this rough surface it sometimes resulted in uncoated gaps and uneven application. It is not surprising that this effect occurred more commonly when using the *kistka* as the tool kept the cyclododecane melted and hot until it was applied directly to the fibers and, since the tool was itself heated and in direct contact with the surface of the sample, it continuously heated the surface and the cyclododecane, pushing the chemical further into the fibers.

Another application anomaly occurred during the creation of the E group of samples. It was noted during the *kistka* applications that the specified masses of cyclododecane "stick" did not seem to cover as much of the dye mark as they had during the other application methods. This likely occurred since the cyclododecane was weighed at room temperature and then kept within the heated environment during the entire application of the E group samples. During the F group *kistka* application, the cyclododecane was measured out at room temperature throughout the application process and so was not exposed to the warmed environment for as long as the cyclododecane used in the E group application process. Since heat encourages the cyclododecane to sublimate, it is likely that the warm environment caused the cyclododecane to sublimate slightly during the E group sample creation. The noticeable effects of this will be discussed again in the "Dye Bleed Test" section of the "Interpreting the Data" chapter.

Chapter Three: Methodology, Analyzing the Samples

Methodology: Analyzing the Samples

Once the samples were created, four different analytical methods were used to study the cyclododecane layers of each sample. A dye bleed test and a continual visual analysis provided qualitative data for the samples, while gravimetric analysis and infrared spectroscopy provided quantitative data. Gathering both qualitative and quantitative results allowed for a more thorough and holistic view not only of the efficacy of the application methods, but also of the nature of the cyclododecane layers throughout the experiment. All of the tests were chosen also because completing any one of them would not interfere with the ability to complete the others or affect the data gathered in any way. Initially it was planned that resin-embedded cross sections would be taken of the samples so that the amount of cyclododecane embedded within the fibers could be microscopically analyzed. However, it was realized that the general methods of creating a cross section would destroy the sample and the data it held: Because the cyclododecane is hard and wax-like, it cracks when cut even with a sharp scalpel. A heated scalpel or cutting tool could not be used as it would possibly melt the cyclododecane further into the fibers and give a false result. A cold microtome could have been used successfully to cut thin layers of the samples in a way that would not melt the cyclododecane, but sourcing one to use required an amount of difficulty and time that would have negatively affected the experiment as a whole. Also, preventing the resultant fine layers of cyclododecane from quickly sublimating during the cutting and resin-embedding process would have been difficult, if not impossible. Instead, the gravimetric analysis and visual analysis using a stereomicroscope were used to provide information about the amounts of cyclododecane embedded within the fibers.

Dye Bleed Test

As this experiment had its inspiration from the study by Scharff in which a dye bleed test was the method used to analysis the efficacy of different cyclododecane application methods, it was appropriate that a dye bleed test was also completed in this experiment.[9] The test is also a very direct method of testing the real-world application of cyclododecane in textile conservation as the ability of the material to safely prevent dye bleeding during wet cleaning is the sole objective in these cases.

For the dye bleed test, each sample was submerged in water for approximately 10 minutes. The water used was soft tap water that was slightly cooler than room temperature, which is approximately 23° Celsius. Small containers large enough to one sample each were filled with enough of the tap water to fully submerge the sample. The samples were submerged individually so that the dye bleed from one did not contaminate another, and also so that any dye bleeding from a sample into the water could be observed. Any dye bleed was observed during this time, noting whether the dye bleed occurred immediately when the sample was submerged, whether there was a high or low amount of dye released, whether the dye bled onto the sample substrate or only into the water, or whether there was no noticeable dye bleed occurring at all.

After the samples were submerged for 10 minutes, they were removed from the containers and placed on blotting paper to dry. Pressure could not be applied to the samples to remove moisture by blotting as this might have cracked the cyclododecane layers, and so some distortion of the sample substrates occurred during drying. However, this distortion occurred outside of the areas of cyclododecane and so it is not believed to have affected the results during the other tests. The samples were then left at room temperature in order to let them sublimate for further analysis. At the end of the experiment after the layers of cyclododecane had completely, or nearly completely, sublimated the samples were visually assessed for any signs of dye bleed remaining on the substrate. This included areas of discernible lightening

[9] Scharff, 150.

within the dye mark and dye that had moved outside of the dye mark. The results of the dye bleed testing are given in Appendices D.1 and E and the data will be further analyzed in the next chapter.

Table 2.1: Amount of Dye Bleed		
"H"	high levels of dye bleed	Dye bled profusely onto the sample substrate and/or into the water when the sample was submerged.
"L"	low levels of dye bleed	Dye bled somewhat onto the sample substrate and/or into the water when the sample was submerged.
"N"	no bleed	Dye never bled during the time when the sample was submerged.
Table 2.2: Characteristic of Dye Bleed		
"I"	immediate bleed	Dye began to bleed immediately when the sample was submerged.
"W"	water-only bleed	Dye bled only into the water, not onto the sample substrate, when the sample was submerged.

Table 2.3: Levels of Dye Bleeding

Sample label	Dye bleed types	Sample label	Dye bleed types	Sample label	Dye bleed types	Sample label	Dye bleed types
cA1b	I, L	sA1b	N	cA1k	L	sA1k	N
cA2b	I, H	sA2b	N	cA2k	L	sA2k	L
cA3b	N	sA3b	N	cA3k	H, W	sA3k	L
cA4b	I, H, W	sA4b	N	cA4k	L	sA4k	N
cB1b	L	sB1b	N	cB1k	L, W	sB1k	N
cB2b	L	sB2b	N	cB2k	L, W	sB2k	N
cB3b	L	sB3b	N	cB3k	L, W	sB3k	N
cB4b	L	sB4b	N	cB4k	L, W	sB4k	N
cC1b	I, L	sC1b	N	cC1k	I, H, W	sC1k	N
cC2b	I, H	sC2b	N	cC2k	L, W	sC2k	N
cC3b	L	sC3b	N	cC3k	I, H, W	sC3k	N
cC4b	I, L, W	sC4b	N	cC4k	L, W	sC4k	N
cD1b	I, H	sD1b	N	cD1k	L, W	sD1k	L
cD2b	H	sD2b	N	cD2k	L, W	sD2k	N
cD3b	L	sD3b	N	cD3k	I, L, W	sD3k	N
cD4b	I, H, W	sD4b	N	cD4k	L, W	sD4k	N
cE1b	I, H, W	sE1b	L, W	cE1k	I, H, W	sE1k	I, L, W
cE2b	I, H, W	sE2b	I, L, W	cE2k	I, H, W	sE2k	L, W
cE3b	I, L, W	sE3b	N	cE3k	I, H, W	sE3k	I, L, W
cE4b	I, L, W	sE4b	L, W	cE4k	I, H, W	sE4k	L, W
cF1b	I, H, W	sF1b	L, W	cF1k	I, L	sF1k	L
cF2b	I, L, W	sF2b	L, W	cF2k	I, H	sF2k	I, L
cF3b	I, H, W	sF3b	N	cF3k	I, L	sF3k	N
cF4b	I, L, W	sF4b	N	cF4k	H	sF4k	N
cG1b	L	sG1b	N	cG1k	I, L	sG1k	I, L
cG2b	L	sG2b	N	cG2k	L	sG2k	N
cG3b	I, L	sG3b	N	cG3k	I, L	sG3k	N
cG4b	I, L	sG4b	N	cG4k	I, H, W	sG4k	N

Gravimetric Analysis

The samples were weighed throughout the experiment in order to observe any patterns in the changes in mass during the sublimation process. Like all materials, cyclododecane has a consistent and predictable mass of its own and a density of 790 kg/m³.[10] When the cyclododecane was applied to the sample substrates, the mass of the sample was more than it had been before the cyclododecane was applied. Recording the masses of the samples and then subtracting the weight of the sample substrate fabric gave the mass of the cyclododecane that remained on the sample. This calculation was made even easier since the sample substrates were cut to consistent weights when they were created. Observing the mass of the samples during the sublimation process gave a clear picture of how much cyclododecane remained on each, and charting these changes over the entire sublimation process revealed the rates of sublimation for the cyclododecane on each sample. In short, samples that lost mass faster were losing cyclododecane faster, and because cyclododecane sublimates more quickly when exposed to the air, the cyclododecane on these samples must be more exposed to the air than the cyclododecane on samples that lost weight more slowly.[11] Losing weight quickly was then taken as a sign that the cyclododecane sat on the surface of the fibers where it was exposed to the air, rather than being deeply embedded within the fibers.

A scale that recorded up to three decimal places was used at this was enough to record changes in mass from one measuring period to the next. The initial weights were taken before the dye bleed testing was done as the layers of cyclododecane would begin sublimating during the drying process. The samples were then left at room temperature in order to dry and sublimate further after the dye bleed test. Masses were subsequently recorded up to twice a day during weekdays while the samples were left at room temperature to sublimate. Since the face of the sample that was laying against the worktop sublimated at a slower rate due to less exposure to air the samples were turned over once a day during the weekdays. The gravimetric analysis overlapped with the infrared spectroscopy since the spectroscopy required only partial sublimation of the cyclododecane layers while the gravimetric analysis was continued until there was complete sublimation. Because of this, all of the samples were placed into refrigerated storage for the two-week period during which the infrared spectroscopy was being completed simply because it was better for the experiment to not try completing two time-consuming analyses simultaneously. This hiatus left a gap in the data that is clearly visible, but that does not affect the data negatively. Approximately halfway through the weighing process, it was realized that the ability of the fibers to gain weight by absorbing moisture from the air meant that daily environmental changes had to be accounted for. At that point an untreated piece of the cotton and silk substrate, cut to weigh the same as the sample substrates, were placed with the samples as they sublimated in order to act as constants throughout any environmental changes. The masses of the samples over the course of the experiment are given in Appendix D.2 and the data will be further analyzed in the next chapter.

Visual Analysis

In order to take note of how the cyclododecane on the samples was sublimating, a visual analysis of the cyclododecane layers on the front and back of each sample was made each weekday, generally at the same time as the masses of the samples were recorded. This was done because often the cyclododecane was very visible on the surface of the sample and so it was easy to determine how much cyclododecane remained on the sample, and easy to note when visible traces of cyclododecane disappeared. Also, when compared to the mass of the sample over time, the visible signs of cyclododecane could be used to indicate

[10] PubChem
[11] PubChem

whether a significant portion of cyclododecane was embedded within the fibers instead of sitting on the substrate surface.

Five categories of layer characterization were created, as described below, and these were used to assess both the front and back of each sample over the course of its sublimation process. The amount of cyclododecane present was only noted when it was located on the dye mark. Also, the "partial layers" were very different between the *kistka*-created and brush-created samples: Cyclododecane applied with a brush sublimated very evenly while cyclododecane applied using a *kistka* sublimated in an uneven way that left large mounds of the substance almost like islands separated by areas that were severely sublimated.

Table 3: Categories of Visible Sublimation	
"Complete"	Complete layer of cyclododecane.
"Partial"	Partial layer of cyclododecane.
"Embedded"	Cyclododecane embedded within the fibers but not laying on the substrate surface.
"Surface"	Cyclododecane layered on the substrate surface but not embedded within the fibers.
"None"	No visible cyclododecane remaining.

These categories were generally simple to distinguish, especially as the "embedded", "surface", and "none" categories were not applicable until far along in the sublimation process. At first, the "surface" type of cyclododecane layer caused some confusion. It appeared almost as a fuzzy, mold-like white layer. However, upon closer inspection using a stereomicroscope and ATR-FTIR analysis, it was found that the substance was only cyclododecane that appeared white because it had separated from the substrate fibers below. The results can be found in Appendix D.3 and the data will be further analyzed in the next chapter.

IR spectroscopy

Infrared spectroscopy was the final analysis made on the samples. This analytical method uses infrared light to measure the vibrations of molecules present within an unknown sample, allowing for the identification of these molecules.[12] In this study infrared spectroscopy was used to identify whether or not cyclododecane remained on the samples after a period of sublimation. It was also able to generate qualitative information about how much cyclododecane remained, although it unfortunately could not provide quantitative data for this information. The spectroscopy was completed using both ATR-FTIR (attenuated total reflectance Fourier transform infrared spectroscopy) and DRIFTS (diffuse reflectance infrared Fourier transform spectroscopy), both of which were able to complete analysis without damaging or destroying the samples in the process.

ATR-FTIR was the main method of spectroscopy used because the analytical machine was easily accessible, relatively easy to use, and preliminary testing proved that it would provide the needed information for the vast majority of the samples. There were some initial worries that ATR-FTIR would not be strong enough, nor penetrate the sample deeply enough, causing the results to be highly affected by even very small amounts of any molecule on the sample surface. Instead, testing showed that in the

[12] Derrick, Michelle R. et al, *Infrared Spectroscopy in Conservation Science*, Los Angeles: Getty Conservation Institute, 1999, 27.

majority of cases the ATR-FTIR was capable of finding the presence not only of cyclododecane but also of the substrate below the cyclododecane. These were optimum results since they provided the qualitative information about how much cyclododecane remained on the sample. The most consistent and average samples from each group was chosen to be scanned since all of the samples could not be scanned due to practicality issues. The front and the back of the samples were scanned three times in order to generate an overall view of the state of the cyclododecane layer. At times this was difficult, but not impossible, as the cyclododecane layer sublimated unevenly, especially on *kistka* samples. The ATR-FTIR machine used was a PerkinElmer® Spectrum One FT-IR Spectrometer with an ATR top plate. The machine utilized PerkinElmer® Spectrum version 5.0.1 and all readings were done using the default settings in order to generate absorption spectra of the samples.

Although similar, DRIFTS analysis is much more sensitive and can penetrate the sample further than ATR-FTIR.[13] Because of this it was chosen as the method to analyze samples that had so little cyclododecane the ATR-FTIR could not analyze them. DRIFTS was only used to analyze these samples, instead of all of the samples, because the machine was difficult to access. Also, the resulting data was much more detailed and so required more assistance to properly interpret. The samples chosen for DRIFTS analysis were scanned a total of five times in order to generate an overall view of the cyclododecane layer. Although both tools required 30 seconds to 1 minute to complete a scan, more scans could be done using DRIFTS than ATR-FTIR since there were far fewer samples being tested. The DRIFTS machine used was an Agilent Technologies 4100 ExoScan Series FTIR handheld tool with a diffuse reflectance sampling interface. The tool utilized MicroLab FTIR software using the default settings in order to generate absorption spectra of the samples.

In both the ATR-FTIR and DRIFTS analysis, control spectra were generated from readings of unused cyclododecane as well as dye-marked cotton and silk. These readings were used as the standards against which the samples were compared in order to find and compare notable peaks from the cyclododecane and substrate. These notable peaks for cyclododecane are listed below in Table 4 as are the molecular structures that cause the peaks. The control spectra were overlapped with spectra from the samples so that visual comparisons could be made between their peaks, which is the recommended analysis method for infrared spectra.[14] This can be seen in Appendix D.4 and will be analyzed in the next chapter.

Table 4: Cyclododecane Peaks

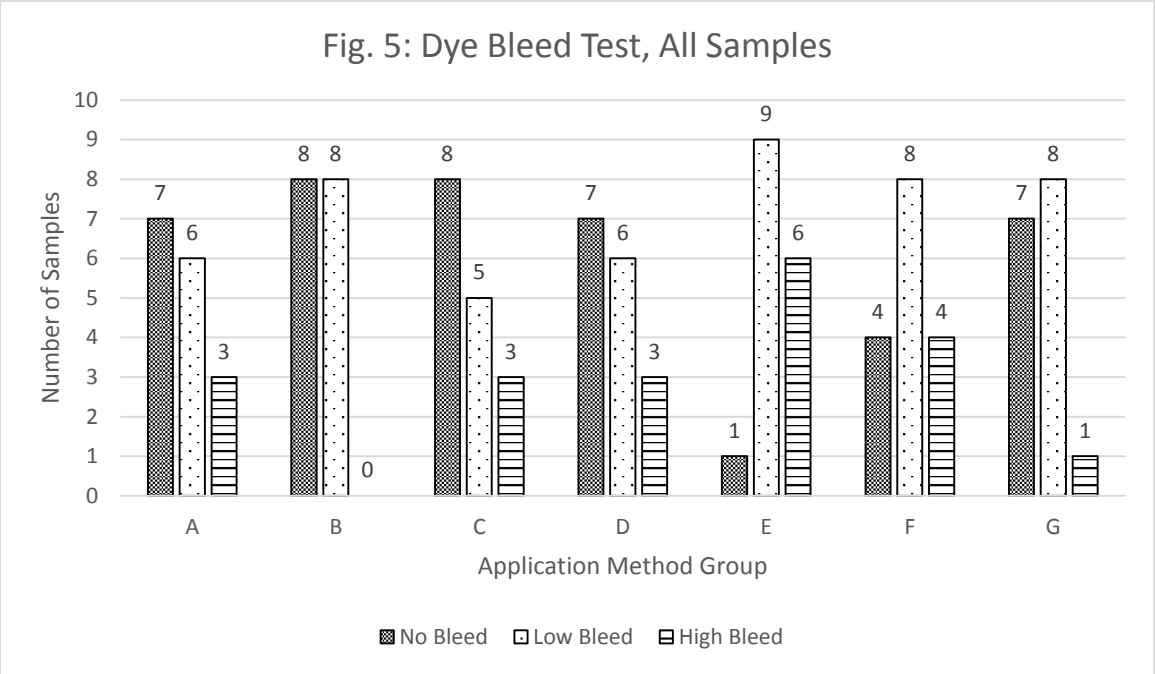
Frequency	Peak Strength	Bond Causing Peak
~3000-2850 cm^{-1}	Medium	C-H stretching in alkane functional groups
~1470-1450 cm^{-1}	Medium	C-H bending in alkane functional groups
~1370-1350 cm^{-1}	Medium	C-H rocking in alkane functional groups
~725-720 cm^{-1}	Medium	C-H rocking in alkane functional groups

[13] Smith, Brian*, Infrared Spectral Interpretation: A Systematic Approach,* Boca Raton, London, New York, Washington, DC: CRC Press, 1999, 117.
[14] Smith, 26.

Chapter Four: Interpreting the Data

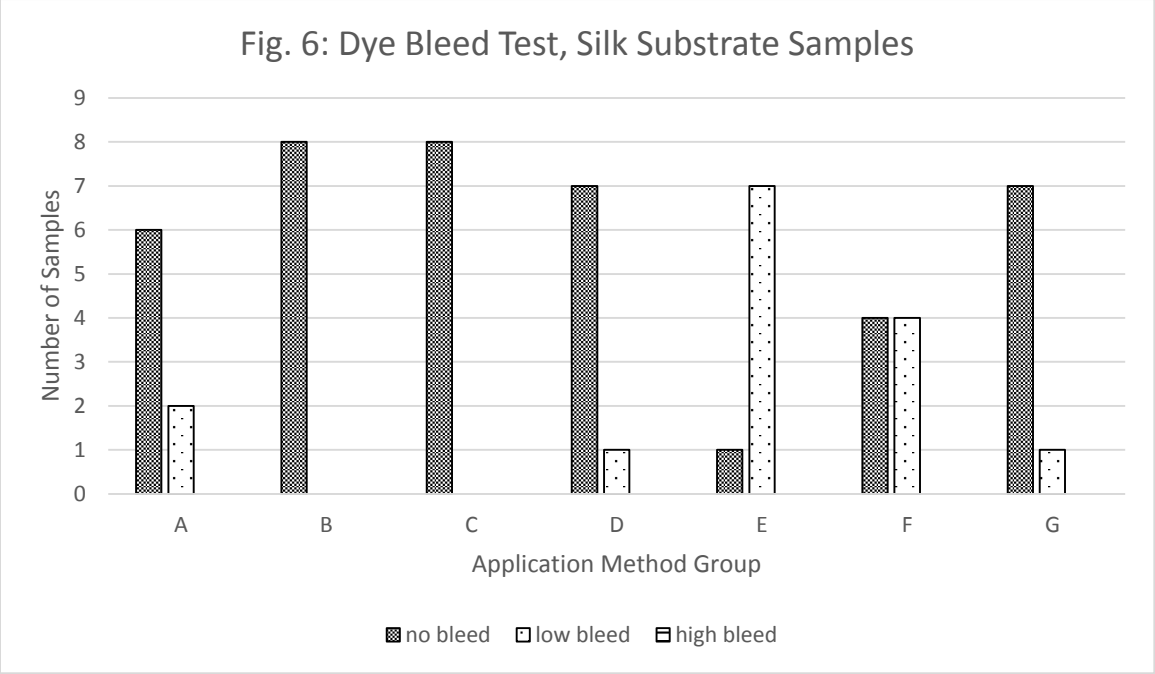
Interpreting the Data

Dye Bleed Test

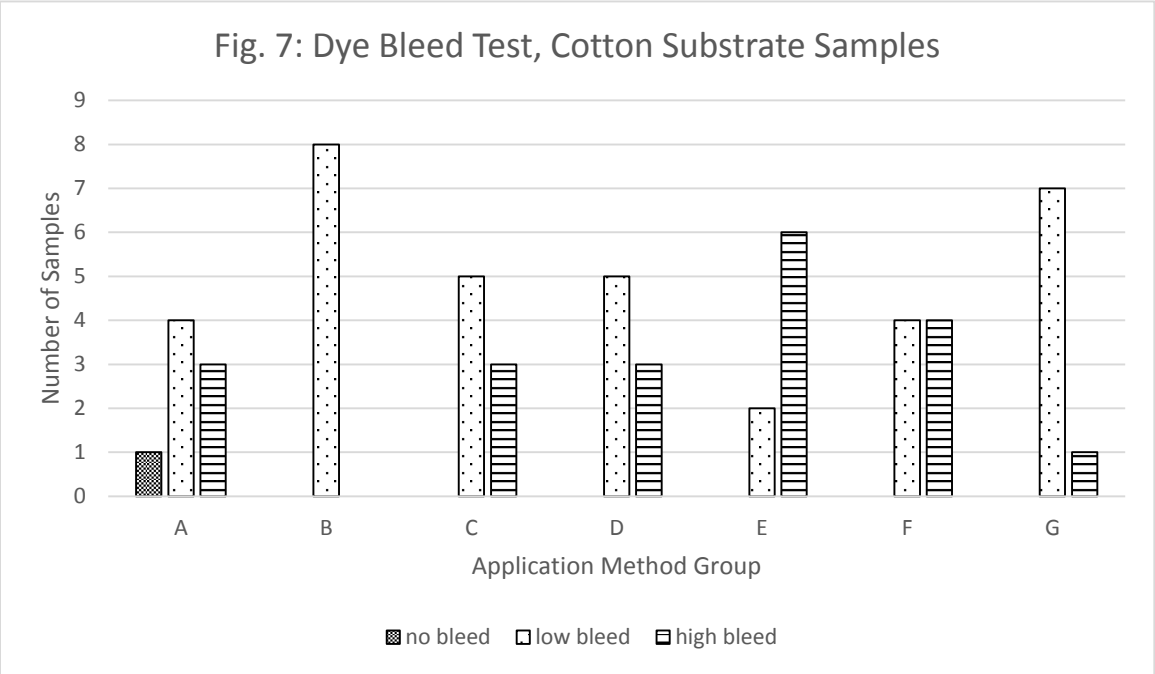


The amount of dye bleeding that occurred during the dye bleed test was categorized as either "none", "a low amount of bleeding", or "a high amount of bleeding". The characteristics of the dye bleed labelled "immediate" or "water-only" as discussed in the previous chapter will not be included in this statistical analysis of the test results since they only affect what the dye bleeding was like, not how much dye bleeding occurred.

When comparing the levels of dye bleed that occurred between the different application methods tested in Figure 5, it is very apparent that the B group of samples showed the best results during the dye bleed test since it had the highest level of samples that showed "no bleeding" combined with the lowest level of samples that showed "high bleeding". The G group results were very similar to that of B, with a combination of the second-highest amount of samples with "no bleed" and the second-lowest amount of samples with "high bleed". The C group can be ranked as the third best as it had the same amount of samples with "no bleeding" as the B group, and the third lowest amount of samples with "high bleeding". The E group fared the worst during the dye bleed test with only one sample with "no bleeding" and the highest amount of samples with "high bleeding". Although the application method was almost identical to that of the F group, E and F acted very differently during the dye bleed test.

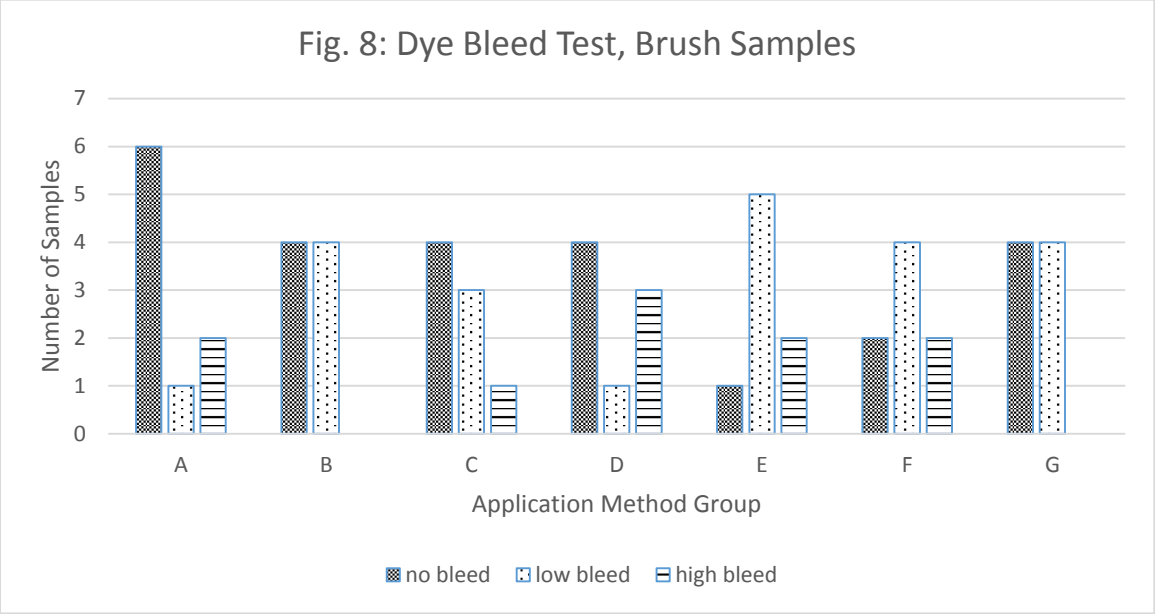


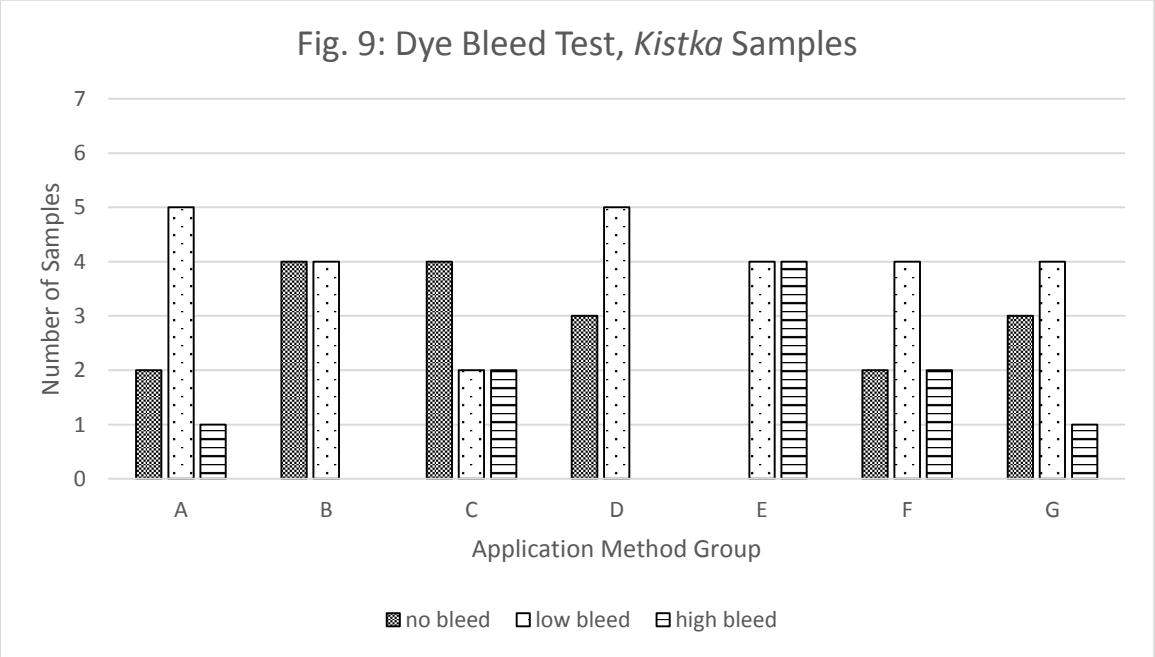
In Figure 6, when comparing only the silk substrate samples, as these tended to resist dye bleeding much more than the cotton, the B and C groups tied for the best performing with all of their samples showing no signs of bleeding. The D and G groups tied for the second best with 7 samples with "no bleeding" and only one each with "low bleeding". The E group showed the worst results with 7 of its samples having "low bleeding" and only one with "no bleeding".



Comparing the cotton substrate samples, the B group again showed the best results with all of the samples showing only "low bleeding". The G group showed the second-best results with 7 samples with "low bleeding" and only one showing "high bleeding". The E group again performed the worst with 6 samples showing "high bleeding".

Although a difference was expected, the scale of the consistent difference between the E and F group was unusual. The possible cause of this disparity was discussed further in the "Notes on Application" section of the "Sample Creation Methodology" chapter, and is likely an effect of the cyclododecane being stored in different ways during the creation of the E and F group samples.





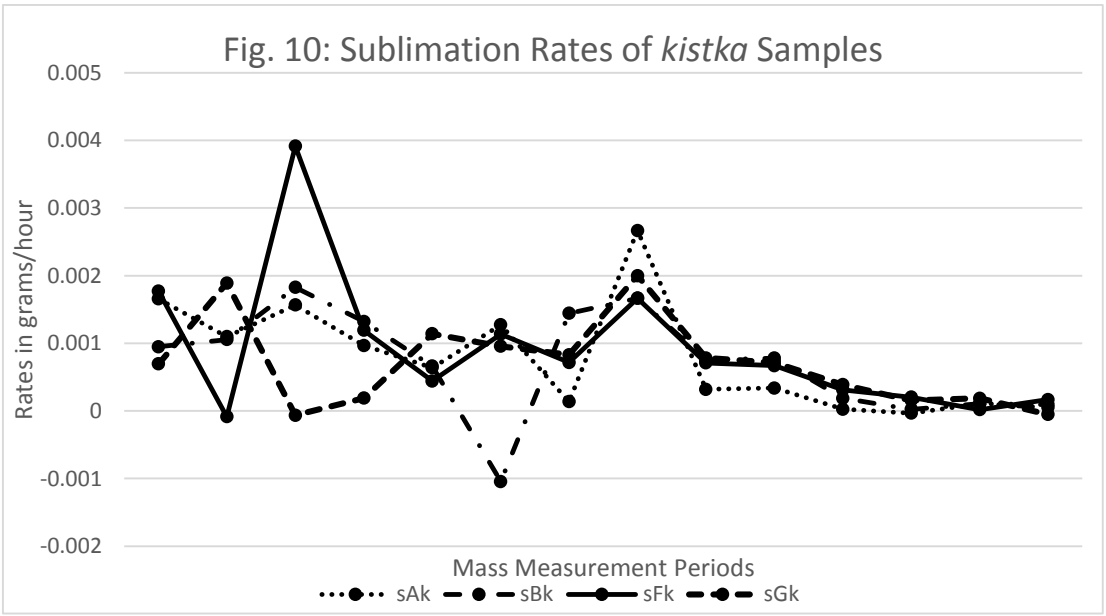
When comparing the samples created using a brush in Figure 8 to those created with a *kistka* in Figure 9*,* it can be seen that the *kistka* samples consistently performed more poorly than the brush samples except in the case of the F and B group samples, which performed exactly the same. Within the brush samples, the B and G group tied for second-best with four "no bleeding" samples and four "low bleeding" samples each. The A group performed best of the brush samples. Of the samples created with a *kistka,* the B group performed the best with its number of "no bleed" and "low bleed" samples unchanged from the brush-created samples. The C and G groups have been considered as tied for second-best since the C group

had more “no bleed” samples but the G group had fewer “high bleed “samples. The *kistka*-created A group samples performed very poorly in comparison to the other groups.

The samples were compared after the dye bleeding test was completed and the cyclododecane layers had sublimated in order to see the full extent of any dye bleed. Photographs of these samples are included in Appendix D.1. The bleeding was defined as either spotty, spreading, or faded. Spotty bleed is defined as small faded spots within the dye mark. Spreading bleed is defined as dye that spread onto the substrate beyond the defined border of the dye mark. Fading is a lightening of the overall color of the dye mark. The silk substrate samples showed no signs of dye bleeding except in the E group where some possible fading was noted. The A and G group brush-created samples with cotton substrates fared the worst with high levels of spotty bleed, spreading bleed, and fading. The B and C brush-created samples with cotton substrates fared the best, although the samples from all groups except A and G were very similar. The *kista*-created cotton samples from all groups were all very similar with slight spotty bleed and some fading. The groups that performed the best repeatedly throughout the dye bleed test were then compared with the samples that had the best results when examined after sublimation. This led to the conclusion that, overall, the B and C groups showed the absolute best performance for dye bleed.

Gravimetric Analysis

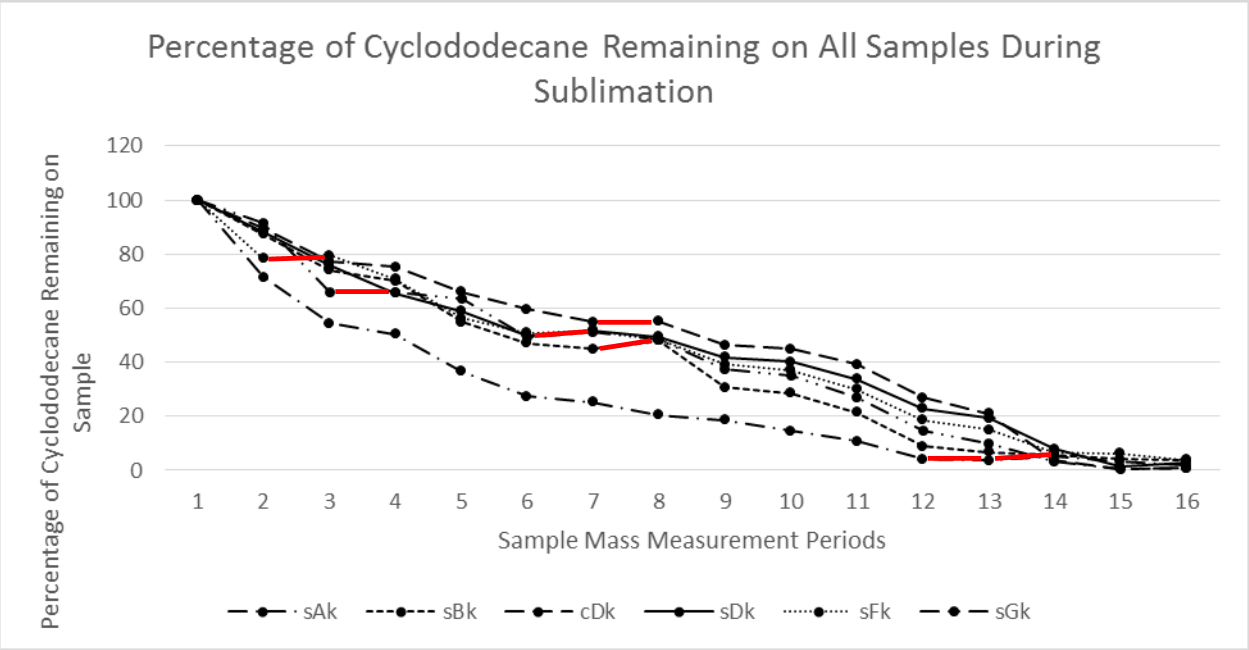
As described in the previous chapter, the masses of the samples were recorded over the course of the experiment in order to calculate how much cyclododecane remained on each sample. Initially, the rates of sublimation were calculated for each sample. This was done by dividing the change in mass that occurred between two measurement periods by the number of hours that passed between these two measurements, resulting in a sublimation rate of grams per hour. This was done since cyclododecane that is less exposed to air has a slower rate of sublimation[15], a fact that was combined with the assumption that cyclododecane which was embedded within the sample substrate fibers would be less exposed to the air than cyclododecane that sat on top of the fibers. However, the calculated sublimation rates were difficult to compare when plotted onto a graph, as can be seen below in Figure 10 where only four of the 28 tested method and tool combinations were plotted.



[15] PubChem

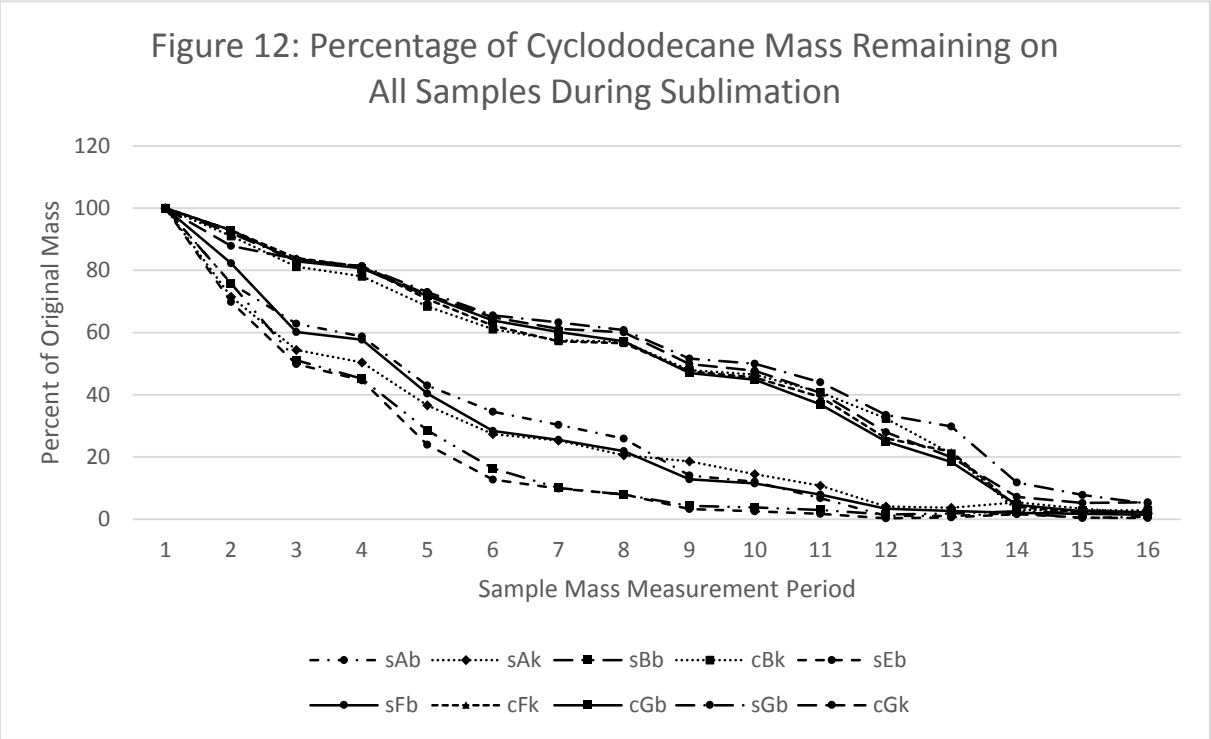
Instead, calculations were made in order to find what percentage of the applied cyclododecane mass remained during the course of the sublimation process. The mass of each sample was recorded and then divided by the initial mass of that sample in order to calculate this percentage, and the percentages were then plotted on a graph to show how they changed over the course of the sublimation process. The resulting graphs were much easier to interpret since the percentage of cyclododecane remaining could only reduce over time, unlike the rates of sublimation which could either increase or decrease, causing a confusing overlap of peaks and troughs when graphed.

Figure 11



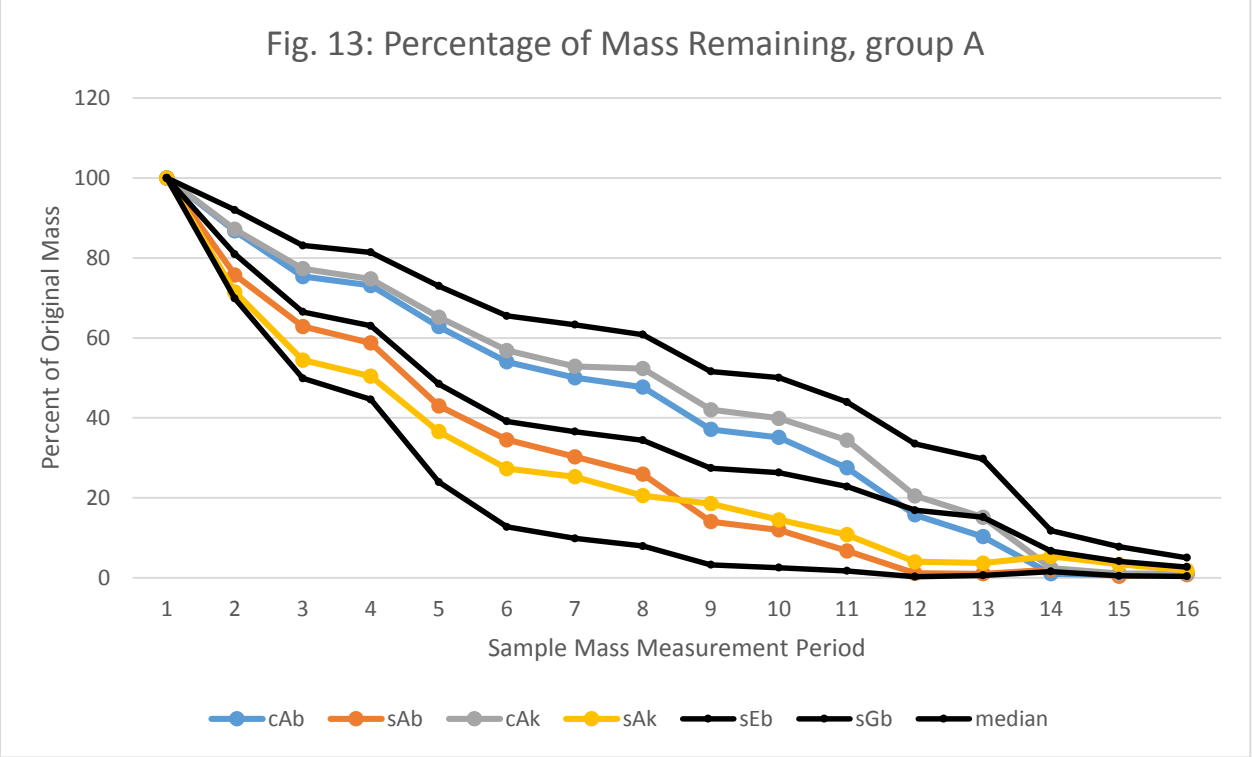
There are a few anomalous points which can be seen as the red bars in Figure 11 where the percentage of cyclododecane remaining on a sample increased. However, many of these occurred either during or immediately after the first six measurement periods where the exact mass of the cotton and silk sample substrates were unknown. In order to calculate the percentages during these six periods, an average mass was calculated for the silk and for the cotton from all of the known masses recorded for each. The cyclododecane mass for the initial measurement period, where no cyclododecane had yet sublimated, used this averaged substrate mass. This initial mass measurement had 100% cyclododecane present and was the mass by which all of the following percentages were calculated. As a result, all of the calculated percentages may not be exact, causing some of the anomalies seen in the graphs. The measured masses of the cotton substrates varied by up to 0.019g, causing the actual value of the cotton sample percentages could have varied by up to 5.313%. The silk substrate masses varied by up to 0.009g, so the percentages of cyclododecane on these samples could have varied by up to 6.0716%. Other anomalous points on the percentage graphs appeared towards the later measurement periods, 13 to 16, where the percentages were very close to 0%. Since all of the samples had very similar values in this area and were difficult to distinguish, the possible deviations in actual percentage values were much larger than 0%, and the remaining areas of the graphs held sufficient information for analysis, this section of the graphs has largely been ignored. Also, only samples that showed a majority of either maximum or minimum percentages were evaluated since

these represented, respectively, the “best” and “worst” cyclododecane retention. This means that samples with a majority of median percentages, and any anomalies present on their graphs, were not evaluated.

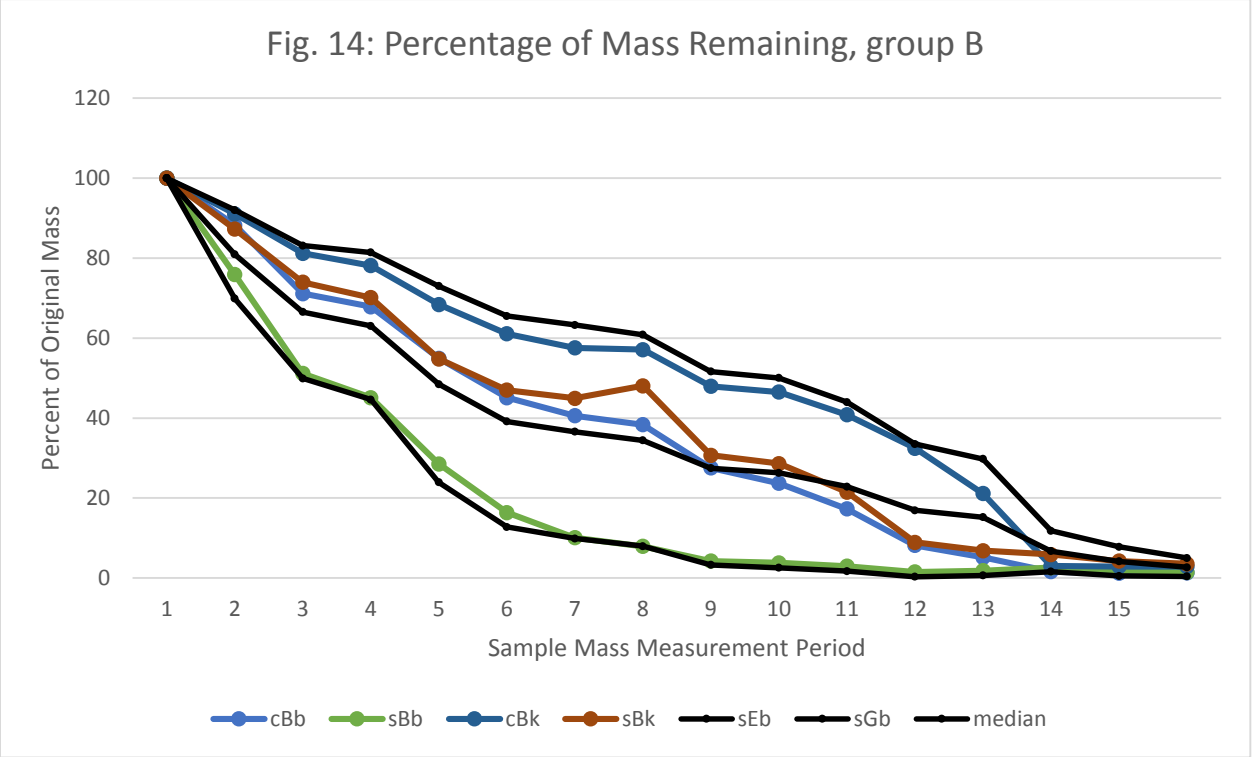


As stated earlier, the percentage graphs can be divided into “best”, “worst”, and “median”. The best had a majority of maximum percentages, while the worst had a majority minimum percentages. The median graphs, with a majority of median percentage values, were not evaluated and so were eliminated from the graphs shown here. When comparing the percentage graphs of all of the samples sGb, cBk, cGk, cGb, and cFk had the best results, while sEb, sBb, sAk, sFb, and sAb had the worst. Further refining the graph, it can be seen that sGb and cGk had the absolute best results, while sBb and sEb had the absolute worst.

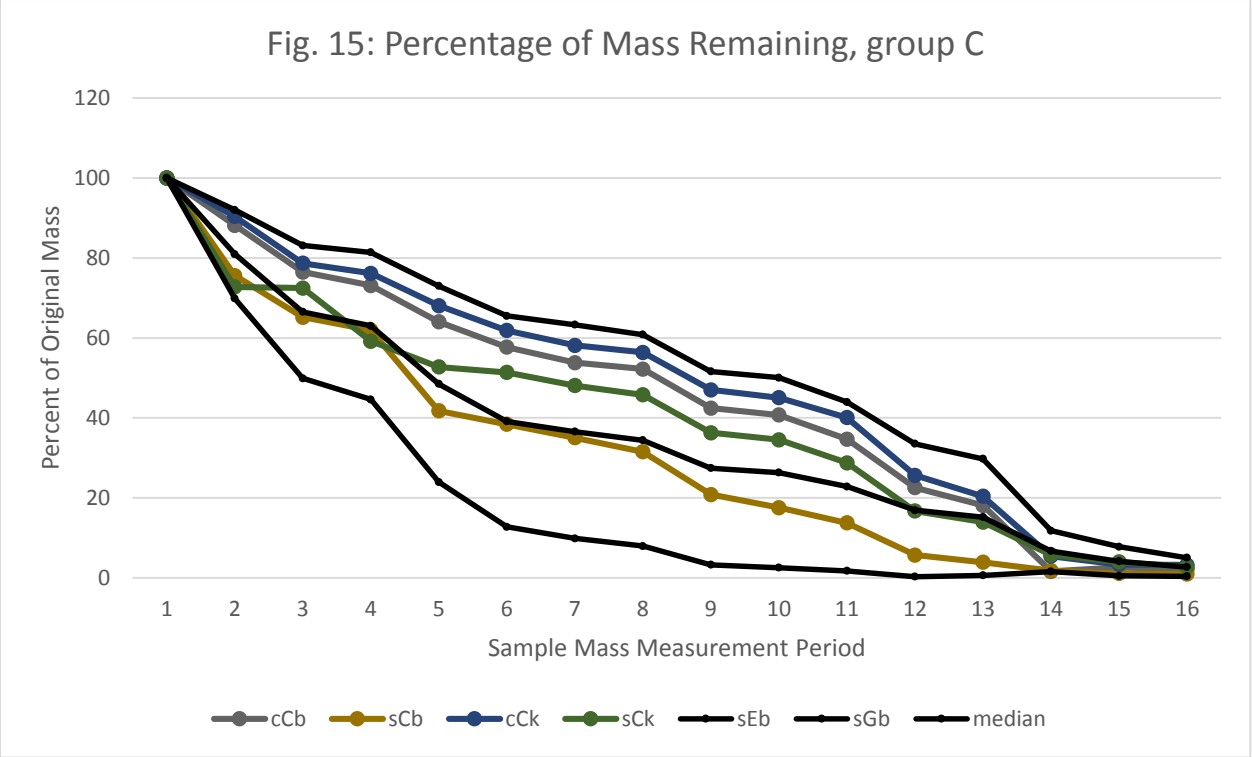
The graphs were then divided into the silk, cotton, brush, and *kistka* for comparison. Within the both brush and silk samples, sGb again performed the best. The cGk group performed the best within the cotton and *kistka* samples. The sEb and sBb also performed the worst within the brush and the silk samples. The worst for cotton samples was cEb and cBb, while sAk was the worst for the *kistka* group. When looking at all of these results together, it is seen that the G group has repeatedly shown the best cyclododecane retention across all substrate and tool types. Likewise, the E and B brush groups typically had the worst cyclododecane retention. However, the results become slightly complicated when the samples are divided into their groups and looked at individually in comparison to the maximum (absolute best, sGb), the minimum (absolute worst, sEb), and a median line created between the two.



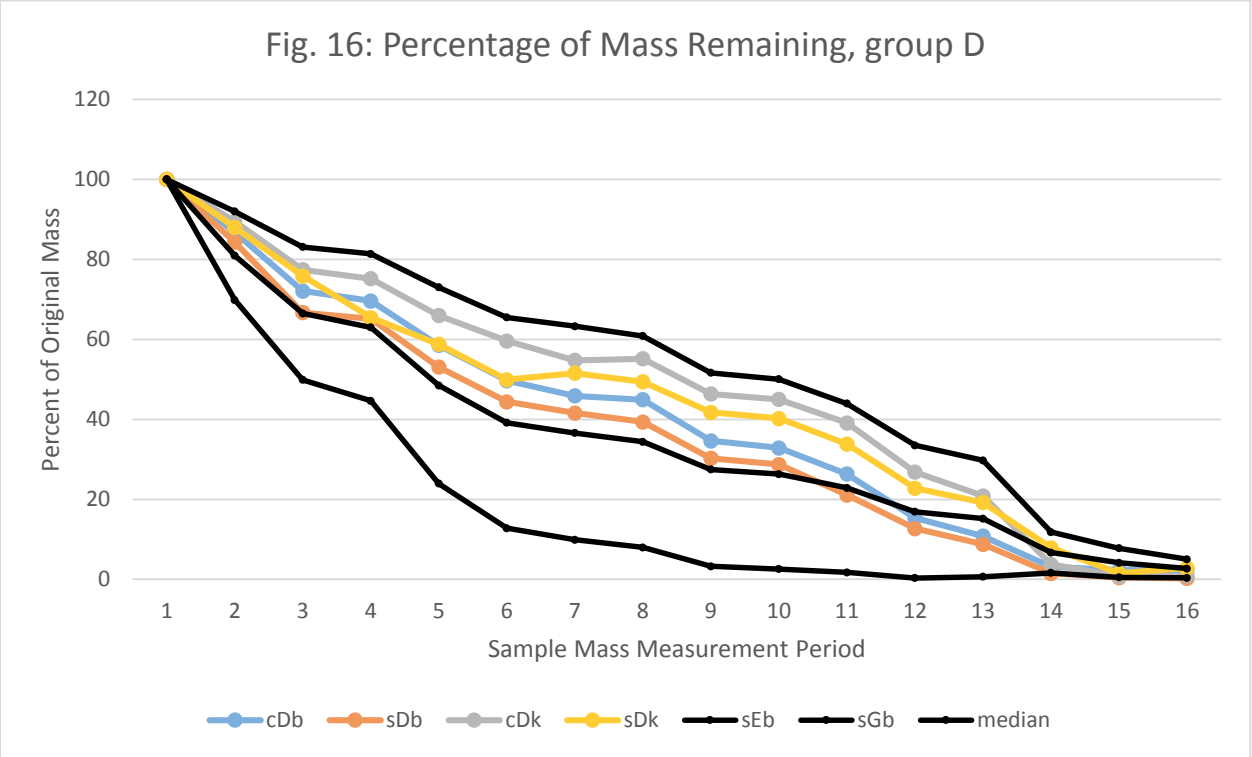
Although sAk performed worst within the *kistka* samples, cAk performed very highly. The poor performance in the A group seems to instead be linked to the silk substrate fabric, since both cotton A samples performed above the median line.



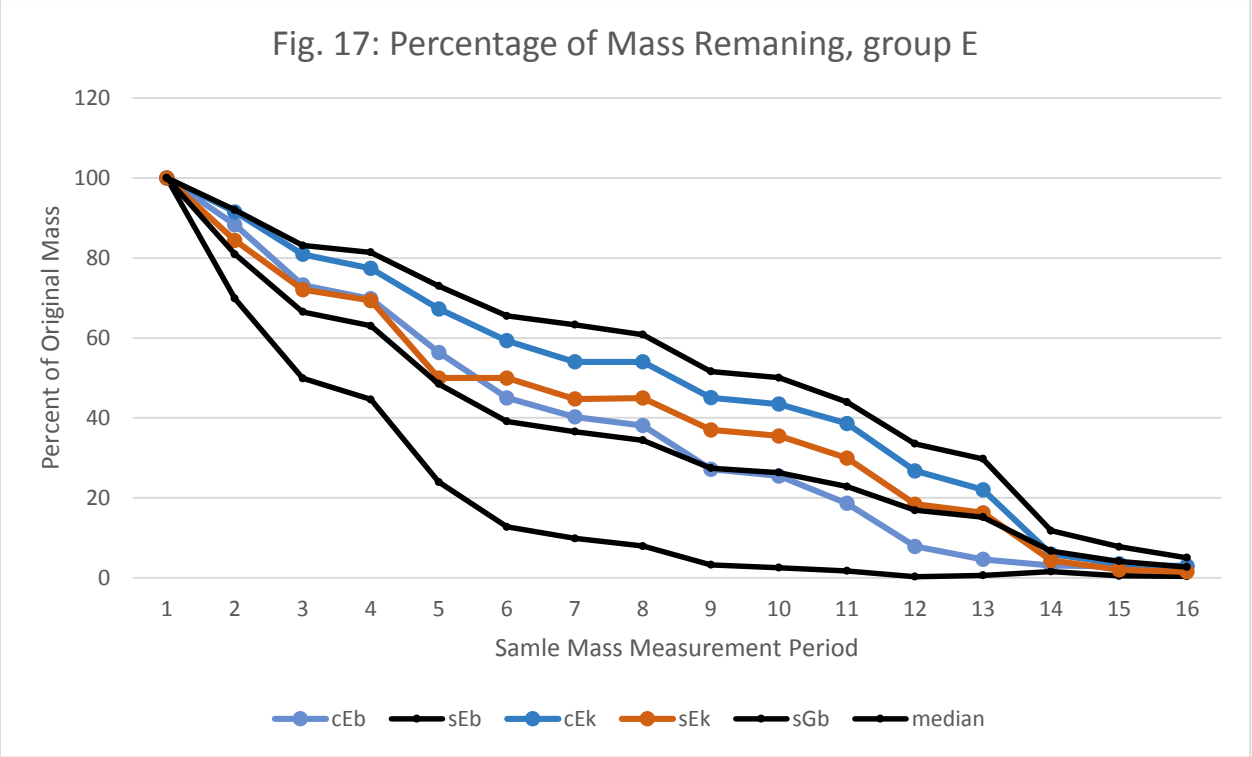
Interestingly, and except for sBb, all of the B group regardless of tool or substrate performed above the median line. sBk and cBb performed very closely to the median line.



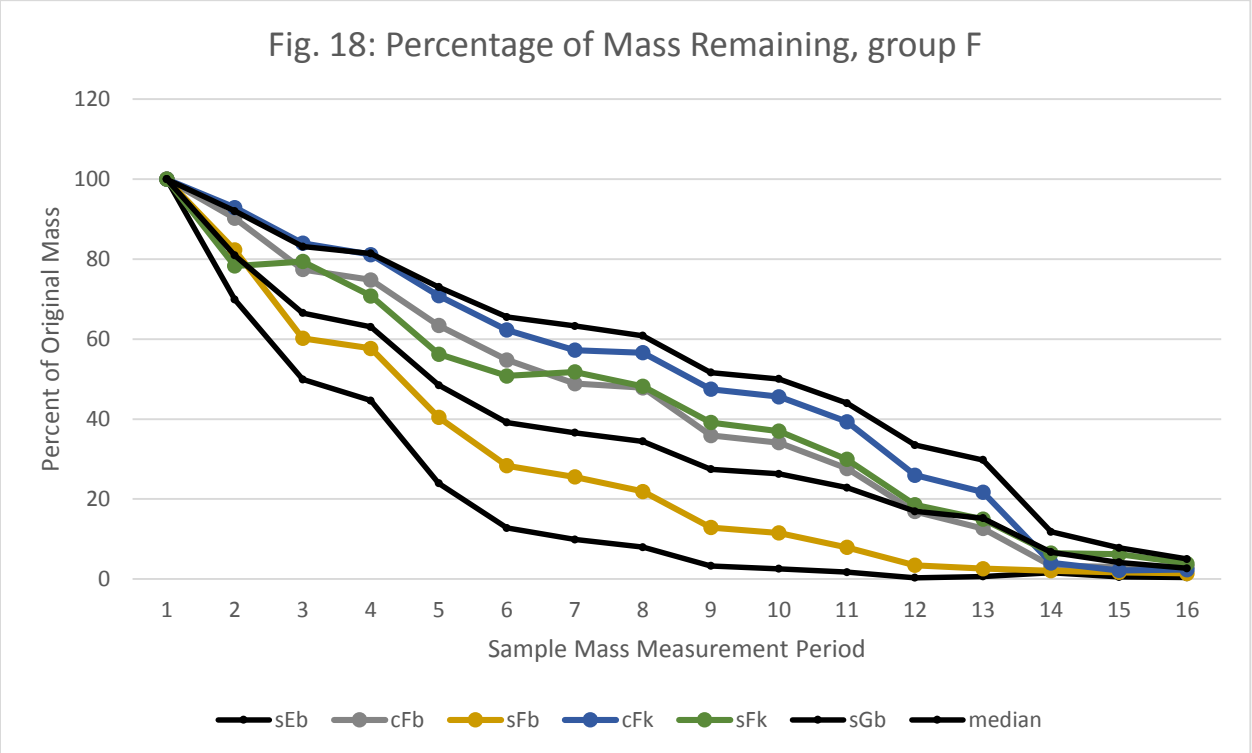
sCb performed just below the median line while sCk performed just above it. The cotton C group samples performed closer to the maximum line.



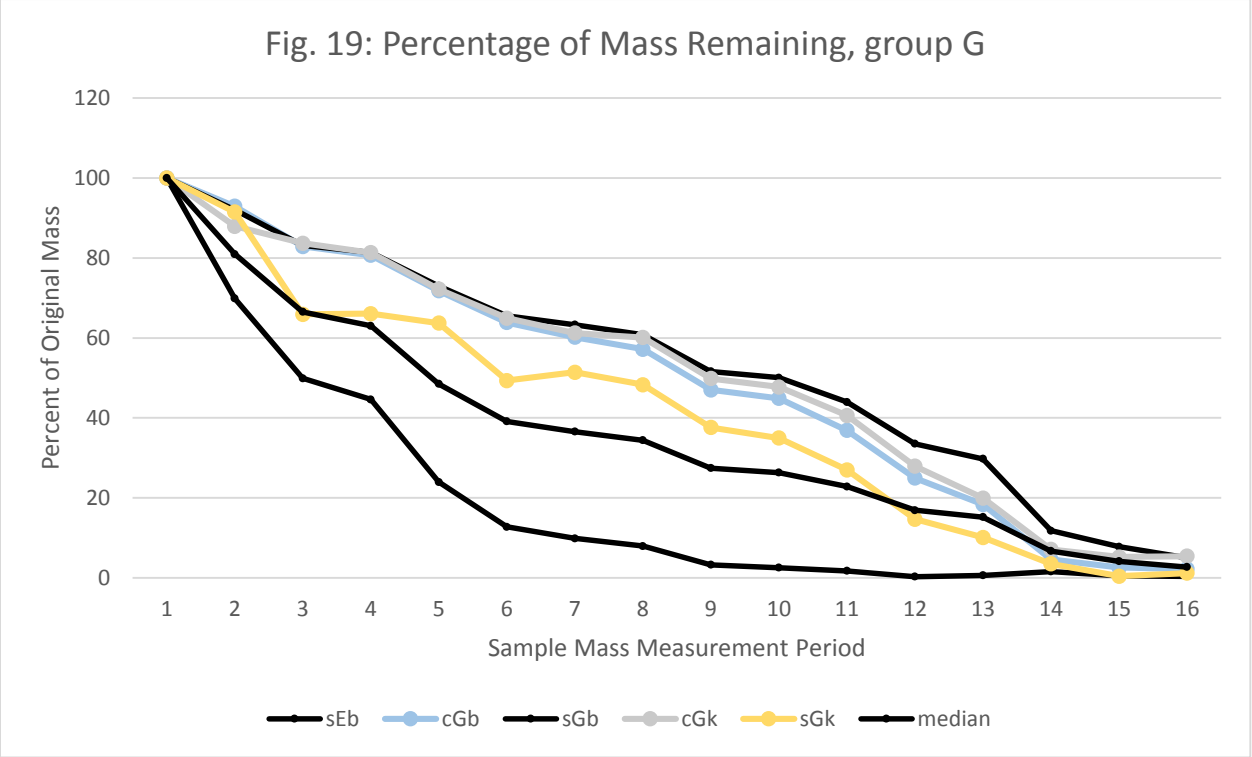
The silk samples in the D group, as well as the brush cotton sample, performed closer to the median line, while the *kistka* cotton sample was closer to the maximum. The silk *kistka* sample also performed better than the two brush samples.



Like the B group, all of the E group except for sEb performed above the median line regardless of substrate or tool.



The brush silk F group sample performed below the median line, while sFk and cFb were very similarly above the line. cFk performed the best near to sGb.



All of the G group performed very well with all graphs being above the median and cGb and especially cGk being very near the maximum sGb.

The generally consistent features of all of the application method groups is that the samples with cotton substrates that were created with a *kistka* performed the best when it came to retaining the cyclododecane during the sublimation process, while the silk samples made with a brush performed the worst. The apparent difference between substrate types is possibly due to the difference in fabric density. The only group that did not fit this trend was the G group, as sGb performed the absolute best of any of the samples, whereas the silk brush samples of the other groups performed the worst. This is made more interesting by the fact that the other three samples in the G group followed the trend set by the other groups. Unfortunately, it is unknown why sGb was such an outlier.

Groups G and D had the best overall results for mass retention with all four sample types for each being above the median line. Groups C and F tied for second with three of their four sample types being above the median line. Groups B and E tied for third with each having three of the four sample types being above the median line while the fourth was or was close to the minimum. Group A performed the worst with only two sample types above the median line and two below.

Visual Analysis

The visual characteristics of the cyclododecane layers on the front and the back of each sample were noted at almost every mass measurement made for the gravimetric analysis. Five categories of layer characteristics were created and are described below.

Table 5.1 and 5.2: Categories of Visible Sublimation Characteristics		
○	"Complete"	Complete layer of cyclododecane.
Δ	"Partial"	Partial layer of cyclododecane.
◊	"Embedded"	Cyclododecane embedded within the fibers but not laying on the substrate surface.
∩	"Surface"	Cyclododecane layered on the substrate surface but not embedded within the fibers.
□	"None"	No visible cyclododecane remaining.

front	6/14/201	6/15/201	6/17/201	6/19/201	7/6/2015	7/7/2015	7/8/2015	7/9/2015	7/10/201	7/13/201	7/14/201	7/15/201
sG2k	○	○	○	○	○	Δ	Δ	Δ	Δ	Δ	□	□
sG1k	○	○	○	○	○	Δ	Δ	Δ	Δ	□	□	□
sG4k	○	○	○	○	○	Δ	Δ	Δ	◊	□	□	□
sD4k	○	○	○	○	Δ	Δ	Δ	Δ	Δ	Δ	◊	□
sD2k	○	○	○	○	Δ	Δ	Δ	Δ	Δ	□	□	□
sA1k	○	Δ	Δ	Δ	Δ	Δ	Δ	Δ	Δ	□	□	□
sB1k	○	Δ	Δ	Δ	Δ	Δ	Δ	Δ	Δ	□	□	□
sB2k	○	Δ	Δ	Δ	Δ	Δ	Δ	Δ	Δ	□	□	□
sB4k	○	Δ	Δ	Δ	Δ	Δ	Δ	Δ	Δ	□	□	□
sC1k	○	Δ	Δ	Δ	Δ	Δ	Δ	Δ	Δ	□	□	□
sC3k	○	Δ	Δ	Δ	Δ	Δ	Δ	Δ	Δ	□	□	□
sD3k	○	Δ	Δ	Δ	Δ	Δ	Δ	Δ	Δ	□	□	□
sE2k	○	Δ	Δ	Δ	Δ	Δ	Δ	Δ	Δ	□	□	□
sE3k	○	Δ	Δ	Δ	Δ	Δ	Δ	Δ	Δ	□	□	□
sF3k	○	Δ	Δ	Δ	Δ	Δ	Δ	Δ	Δ	□	□	□
sA3k	○	Δ	Δ	Δ	Δ	Δ	Δ	Δ	□	□	□	□
sB3k	○	Δ	Δ	Δ	Δ	Δ	Δ	Δ	□	□	□	□
sF2k	○	Δ	Δ	Δ	Δ	Δ	Δ	Δ	□	□	□	□
sA4k	○	Δ	Δ	Δ	Δ	Δ	Δ	□	□	□	□	□
sC4k	○	Δ	Δ	Δ	Δ	Δ	Δ	□	□	□	□	□
sC2k	○	Δ	Δ	Δ	Δ	Δ	□	□	□	□	□	□
sE4k	○	Δ	Δ	Δ	Δ	Δ	□	□	□	□	□	□
sF1k	○	Δ	Δ	Δ	Δ	Δ	□	□	□	□	□	□

The samples were then divided into cotton-brush, silk-brush, cotton-*kistka,* and silk-*kistka* types and organized from the longest retention of complete and partial layers to the shortest retention. The samples had to be so divided since the data in each set could not be directly compared to that of another set due to

the differences in the amount of applied cyclododecane for each tool and substrate. The ten samples with the longest retention and the ten with the shortest retention were then found for each of substrate-tool divisions. Once the best and worst for each group were found, they were compared in order to notice any patterns resulting from a possible relation between cyclododecane retention and application method.

Table 6: Top/Bottom 10 Samples							
Top 10 Samples	A	B	C	D	E	F	G
Front Cotton Brush	30%	30%	0%	10%	0%	0%	30%
Front Silk Brush	20%	0%	10%	30%	0%	0%	40%
Front Cotton *Kistka*	40%	30%	10%	20%	0%	0%	0%
Front Silk *Kistka*	10%	30%	10%	20%	0%	0%	30%
Average Percentage	25%	22.5%	7.5%	20%	0%	0%	25%
Top 10 Samples	A	B	C	D	E	F	G
Back Cotton Brush	10%	0%	20%	10%	10%	30%	20%
Back Silk Brush	20%	0%	30%	20%	0%	0%	30%
Back Cotton *Kistka*	0%	10%	0%	10%	30%	30%	20%
Back Silk *Kiskta*	0%	0%	0%	30%	30%	20%	20%
Average Percentage	7.5%	2.5%	12.5%	17.5%	17.5%	20%	22.5%
Bottom 10 Samples	A	B	C	D	E	F	G
Front Cotton Brush	0%	0%	40%	30%	10%	20%	0%
Front Silk Brush	0%	20%	20%	0%	40%	20%	0%
Front Cotton Kistka	0%	0%	10%	10%	20%	20%	40%
Front Silk Kistka	20%	10%	20%	0%	20%	30%	0%
Average Percentage	5%	7.5%	22.5%	10%	22.5%	22.5%	10%
Bottom 10 Samples	A	B	C	D	E	F	G
Back Cotton Brush	20%	30%	10%	20%	10%	0%	10%
Back Silk Brush	10%	40%	0%	0%	30%	20%	0%
Back Cotton Kistka	30%	20%	30%	20%	0%	0%	0%
Back Silk Kistka	40%	20%	20%	0%	0%	0%	20%
Average Percentage	25%	27.5%	15%	10%	10%	5%	7.5%

When comparing the visual characteristics of the cyclododecane layers on the front and back of all of the layers, the front and back had very different results. The groups that generally performed the best on the front of all of the different sample types were A and G. The G group also performed the best on the cyclododecane layers on the backs of the samples. Groups C, E, and F performed the worst on the front cyclododecane layers, while B performed the worst on the back of all of the sample types. Also, 9 of 16 anomalous “surface” layers appeared on G samples and another 5 appeared on A samples. These were at first thought to be some type of residue or deposit left by the sublimation due to their white color and sudden appearance. However, ATR-FTIR and stereoscopic visual analysis showed these to only be thin cyclododecane layers that sat on the uppermost surface of the substrate, almost like a thin sheet of ice.

Tables 7.1 and 7.2

	Brush	*Kistka*
Top Front Silk	50%	50%
Top Front Cotton	60%	40%
Top Back Silk	30%	70%
Top Back Cotton	70%	30%
Average	52.5%	47.5%

	Brush	Kistka
Bottom Front Silk	100%	0%
Bottom Front Cotton	100%	0%
Bottom Back Silk	30%	70%
Bottom Back Cotton	20%	80%
Average	62.5%	37.5%

The visual characteristics were then analyzed in Tables 7.1 and 7.2 to determine what percentages of the top and bottom performing samples were created with a brush or with a *kistka.* On average, 5% more of the top-performing samples were made with a brush. However, 25% more of the worst-performing samples were created with a brush, indicating that overall the *kistka* samples had somewhat better results when they were visually analyzed.

FTIR Analysis

The infrared spectrum of cyclododecane has peaks at:

Frequency	**Peak Strength**	**Bond Causing Peak**
~3000-2850 cm^{-1}	Medium	C-H stretching in alkane functional groups
~1470-1450 cm^{-1}	Medium	C-H bending in alkane functional groups
~1370-1350 cm^{-1}	Medium	C-H rocking in alkane functional groups
~725-720 cm^{-1}	Medium	C-H rocking in alkane functional groups

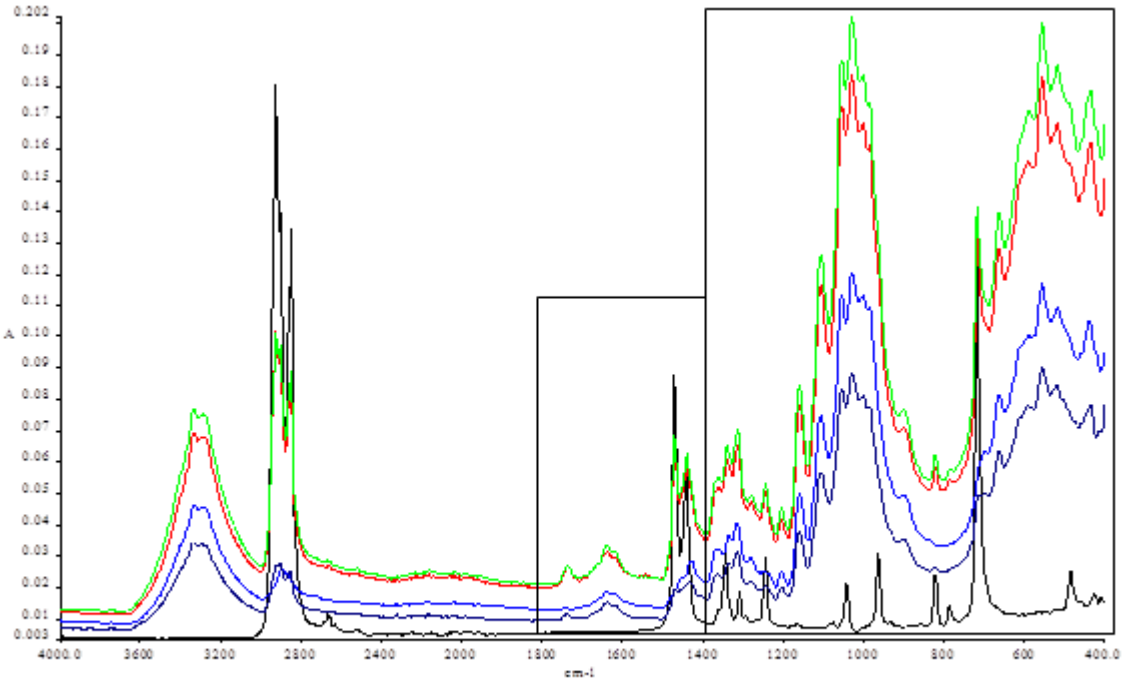
When comparing the absorption spectra of the samples gathered both by ATR-FTIR and DRIFTS methods the intensity, shape, and frequency of the sample peaks were visually compared for similarities to those of cyclododecane and the appropriate substrate fabric.[16] The peaks that were generally used for comparison existed within the "fingerprint zone", approximately 1600-500 cm^{-1}.[17] There are many peaks within this zone which can be very unique to particular and indicative molecular bonds. Another benefit of analyzing peaks mainly within the "fingerprint zone" is that it eliminates interfering peaks that result from CO_2 in the environment or moisture within the analyzed materials.[18]

The analyzed peaks from the samples had a range of similarity to the cyclododecane and substrate spectra. When the sample spectrum peaks were the same intensity, shape, and frequency as both the cyclododecane and the substrate spectra, then the sample contained what was labelled a "medium" amount of cyclododecane. If some of the sample spectrum peaks matched those of cyclododecane but there were no peaks that matched those of the substrate, or these peaks had diminished intensities or shapes, then the sample contained a "high" amount of cyclododecane. Similarly, if the sample spectrum peaks matched those of the substrate but the peaks similar to cyclododecane were diminished, then the sample had a "low"

[16] Derrick, 82.
Smith, 26-28.
[17] Derrick, 94.
Smith, 25.
[18] Derrick, 93, 95, 116.

amount of cyclododecane. In either of these two latter cases, the peaks of one material were being attenuated by the overwhelming peaks of the other material.[19] Initially all of the sample spectra from ATR-FTIR analysis were compared to those of cyclododecane and the substrates and rated whether there were "none", "very low", "low", "medium", "high", or "very high" levels of cyclododecane present. When it was necessary to distinguish between the "low" and "none" levels of cyclododecane, a derivative function was applied to the spectra graphs, which amplified any differences between the substrate spectra and the sample spectra that may have been caused by the presence of a very small amount of cyclododecane. If, after applying the derivative, an amount of cyclododecane was found it was labelled as "very low". Those that still showed absolutely no distinguishable signs of cyclododecane were then analyzed using the DRIFTS technique and the comparison process was repeated for these samples.

Figure 8: Infrared Absorption Spectra of the back of cA2b



CDD (black) cotton (blue) cA2bB3 (red) cA2bB2 (green) cA2bB1 (navy)

After the spectra were labelled with how much cyclododecane they presented, they were analyzed for any patterns relating the amount of cyclododecane to the application method. Unlike the analysis for the dye bleed test and the visual analysis, the total number of examined samples was relatively small. Because of this, the samples could not be separated into subgroups based on substrate and application tool while still maintaining enough data to present a pattern. Instead, the total number of each level of cyclododecane level was calculated for each application method, for example the results for the front of the A group samples, including brush and *kistka* samples on both substrate fabrics, had a total of five "high" level spectra readings. This data is listed in Table 9.

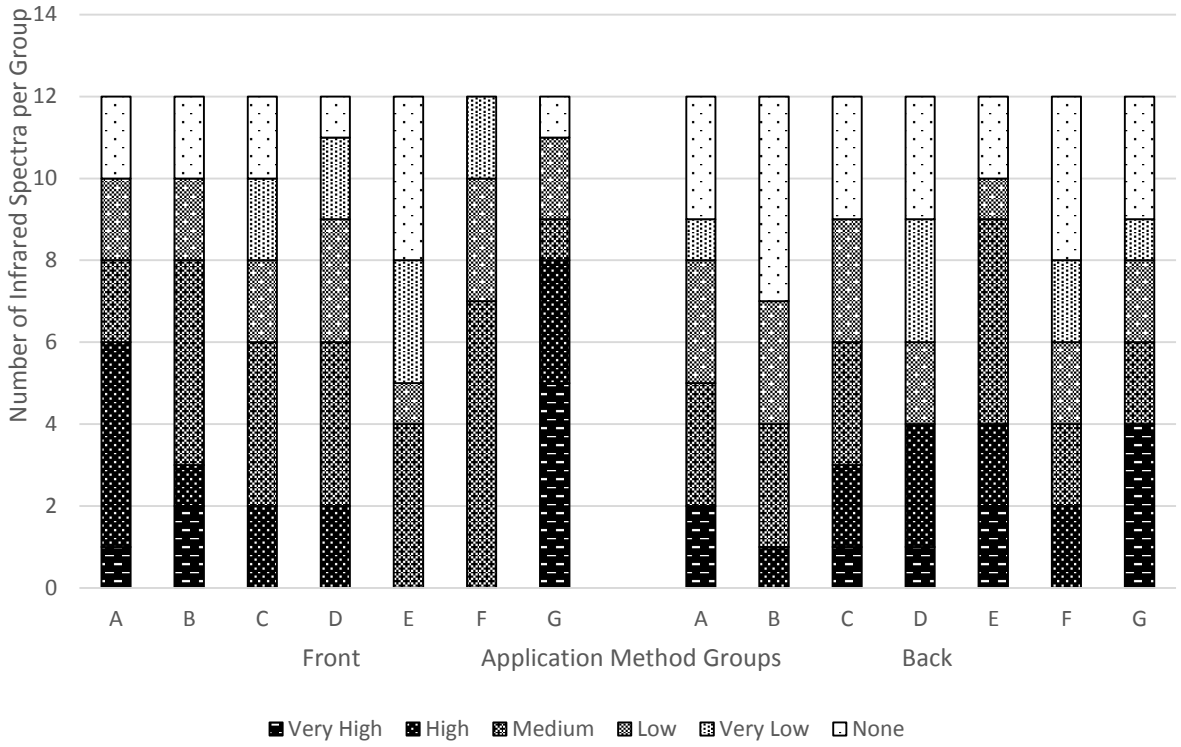
[19] Smith, 26-28.

	Table 9					
Group	**Level of Cyclododecane Present on Sample**					
	Very High	High	Medium	Low	Very Low	None
A Front	1	5	2	2	0	2
B Front	2	1	5	2	0	2
C Front	0	2	4	2	2	2
D Front	0	2	4	3	2	1
E Front	0	0	4	1	3	4
F Front	0	0	7	3	2	0
G Front	5	3	1	2	0	1
A Back	2	0	3	3	1	3
B Back	0	1	3	3	0	5
C Back	1	2	3	3	0	3
D Back	1	3	0	2	3	3
E Back	2	2	5	1	0	2
F Back	0	2	2	2	2	4
G Back	4	0	2	2	1	3

Looking at the infrared spectra of the samples from both the ATR-FTIR and the DRIFTS analysis, it can be seen that the cyclododecane layers on the front of the samples generally performed better than those on the back. A similar overall difference between front and back was also noticed during the visual analysis, and was likely caused by one side possibly being exposed to the open environment more than the other although the samples were turned often to promote even sublimation of both sides. Because the front and the back of each sample were being analyzed separately since they had been treated separately during the application method, this discrepancy did not affect the analysis of the infrared spectra.

Unlike the previous analytical methods, “good” and “bad” results were not readily apparent. For example, it might initially appear that a sample group that showed a lot of “very high” levels of cyclododecane would be sublimating very slowly, which would indicate good performance. However, many sample groups had a large amount of “very high” levels quickly followed by “very low” or even “none” levels, which would indicate that the cyclododecane was sublimating very quickly and unevenly and that it was not very well embedded within the fibers. Instead samples with similar high amounts of “medium”, “high”, and “low” levels, with only a few “very high”, “very low”, and “none” levels, would indicate the best results. This is because the combined “medium”, “high”, and “low” levels indicate that the cyclododecane is sublimating slowly and evenly while a significant amount still remains within the fibers. The results of Table 8 have been graphed below in Figure 20 for easier evaluation and comparison.

Fig. 20: Levels of Cyclododecane Present on Samples Per Application Method, Front and Back of Samples



Evaluating the spectra taken from the front of the samples, and following the parameters for "good" and "bad" results established above, it can be seen that group B performed very well with a combined majority of "high" and "medium", low amounts of "low" and "none", and even some "very high". Groups C and D also performed well with half of their spectra showing "high" and "medium" levels of cyclododecane and the other half showing "low", "very low", and a few "none" levels, indicating a slow and even rate of sublimation. Spectra from groups A and G showed many "very high" and "high" levels, and then degraded quickly into mainly "low" and "none", indicating that the cyclododecane began sublimating recently but did so rapidly and so was not being contained by being embedded within the fibers. Group F performed moderately with levels being "medium" or lower, and the E group spectra were very poor with a majority of "very low" and "none".

Looking at the spectra taken from the back of the samples, group E performed very well with a majority of "high" and "medium" levels, with a few "very high", "low", and "none". Groups C and B were very similar to, but not as good as, the spectra from the front of group C. The D group samples performed moderately, and groups A and G performed poorly with results similar to those from the front of group G.

Results of the evaluations of spectra from both the samples' front and back could then be combined. This shows that, overall, the groups B and C consistently performed well whereas other groups performed well on one side and poorly on the other side. Group G was consistently poor in performance as both sides had "very high" levels of cyclododecane that was not embedded into the fibers and so rapidly sublimated to "low" or "none" levels.

Careful visual examination of the infrared spectra provided ample information for the purposes of this experiment. However, if needed it would also have been possible to complete a highly rigorous

statistical analysis of the ATR-FTIR and DRIFTS data by using a type of multivariate data analysis called Principal Components Analysis, a technique that enhances the visibility of trends in a dataset and that has been used previously to evaluate spectroscopic data.[20]

Combining the Results

The B and C groups showed the absolute best performance during the dye bleed testing. Groups G and D had the best overall results for mass retention in the gravimetric analysis while C and F tied for second and B and E tied for third best. The B and C groups also showed the absolute best performance for infrared spectroscopy. In the visual analysis of the cyclododecane layers during sublimation, A and G performed the best on the front of the samples while G also performed the best on the back. However, A and G performed the worst in the visual analysis of the samples' dye bleed after sublimation. Group A also performed the worst in the gravimetric analysis and A and G showed the worst results during the infrared spectroscopy. In the visual analysis groups C, E, and F performed the worst on the front, while B performed the worst on the back.

Overall, groups B and C performed well and showed consistent results in all tests except for the visual analysis of the sublimation of the cyclododecane layers where they both performed poorly. This means that B and C not only protected the sample against dye bleeding, but it sublimated steadily and slowly, indicating that the cyclododecane was well embedded within the fibers. Both B and C application methods involved applying the cyclododecane to one side of the sample while it was room temperature, while applying cyclododecane to the opposite side while it was heated. However, it is not known why these two consistently performed better than the other methods that involved heating the sample. Inversely, the two methods A and G, where the substrate was not heated in any way during the application of cyclododecane, generally performed the worst except in the visual analysis and, for G samples, the gravimetric analysis. This indicates that a tenacious layer of cyclododecane was formed on the surface of these samples, particularly in group G, but it did not protect the sample from dye bleeding whatsoever and then sublimated rapidly. This is further supported by the fact that during the visual analysis 9 of 16 anomalous "surface" layers appeared on G samples and another 5 appeared on A samples. These were thin cyclododecane layers that sat on the uppermost surface of the substrate but are not embedded within the fibers whatsoever. It is possible that the cyclododecane layers on the A and G samples were never particularly embedded within the fibers and that air gaps could have existed or appeared easily between the substrate and the cyclododecane. This would account for how easily the dye bled on these samples and why sublimation occurred so suddenly despite such an apparently high amount of cyclododecane showing during the visual and spectroscopic analysis.

The overall results of both the brush and the *kistka* were also compared. During the dye bleed testing, the *kistka*-created samples conistently performed more poorly than those made with a brush except for the B and F groups where two tools performed exactly the same. The poor performance of the *kistka* samples may have been caused by the fact that there was more cyclododecane initially applied to the brush samples than to the *kistka* samples. However, in the visual analysis of the dye bleeding after the cyclododecane had sublimated the *kistka* samples performed similarly to the brush samples except for those from brush groups A and G, which performed the absolute poorest. Also, the *kistka* samples, particularly

[20] Teodor, E.s., E.d. Teodor, M. Virgolici, M.m. Manea, G. Truică, and S.c. Liţescu, "Non-destructive Analysis of Amber Artefacts from the Prehistoric Cioclovina Hoard (Romania)," *Journal of Archaeological Science*: 2386-396.

those with cotton substrates, consistently performed better than the brush samples at retaining mass during the gravimetric analysis. And although both tools created a similar number of best-performing samples during the visual analysis of the sublimating cyclododecane layers, there were 25% more worst-result samples made with a brush than with a *kistka*. Overall in these tests, the *kistka* samples performed better than the brush samples except where the difference in the initially applied amounts of cyclododecane may have had an influence. If it had been possible to apply equal amounts to both *kistka* and brush samples, the *kistka* samples may have performed even better.

In the spectroscopy analysis, the *kistka* and brush samples showed similar numbers of "very high" and "high" levels of cyclododecane present. The *kistka* samples had significantly fewer "medium" and "very low" levels, though, with the majority being "low" and "none" levels. This indicated that the *kistka* samples sublimated more quickly than the brush samples, which was unexpected since this would suggest that the cyclododecane was not as embedded in the fibers as it was on the brush samples. However, during the creation of the *kistka* samples, it was noted that the melted cyclododecane often easily saturated the substrate, ensuring that the cyclododecane was embedded within the fibers. Instead, the discrepancy that appeared in the spectroscopy results may have been caused by the fact that much more cyclododecane had been initially applied to the brush samples than to the *kistka* samples. The presence of this difference in spectroscopy results could not be excluded due to the small number of samples tested, however it should not have interefered with the interpretation of the results since all of the evaluated groups included an equal number of *kistka* samples and so were all influenced the same by their presence.

Chapter Five: Conclusions

Conclusion

Dye bleeding is a common problem in textile conservation that can negatively affect the treatment of historic objects. Cyclododecane is sometimes used to create a hydrophobic barrier over such water-sensitive dyes in order to protect them during wet cleaning. Building on previous research, seven different methods for creating these cyclododecane barriers were devised and tested for their effectiveness against dye bleed. This study not only confirmed the results of a previous study, but provided a larger set of results for evaluation and comparison. More importantly, several analytical methods were used to develop explanations for why some methods were more effective than others. The results of the study were used to create instructions for the application of melted cyclododecane to textiles that textile conservators can use during their treatments.

The seven methods devised involved variations of the temperature of the fabric to which cyclododecane was applied, as well as variations in the temperature of the surrounding environment. Each method was executed using two tools common for cyclododecane application: a paintbrush and a wax-melting pen called a *kistka.* Each method was also used to treat both cotton and silk samples as these two fibers are commonly applied with cyclododecane in real-world application. The methods were first tested for their efficacy as a hydrophobic barrier via a dye bleed test where samples were submerged in water for 10 minutes. Any active dye bleeding that occurred during the test was noted, as well as any signs of dye bleeding noticed on the samples after they had dried. Gravimetric, visual, and infrared spectroscopic analyses were performed in order to determine the amounts of cyclododecane present on the samples throughout the experiment. This allowed correlations to be drawn between the behavior of the cyclododecane during sublimation and the application method with which each sample had been created.

Samples from the application methods group B and group C had the best consistent results overall, except in the case of the visual analysis. B and C were methods that required cyclododecane to be applied to one side of the fabric while it was warm and to the other side while it was room temperature, which corroborates the findings of previous research. During the experiment it was noted that the cyclododecane on these samples sublimated slowly and steadily. This indicates that B and C protected against dye bleeding because the cyclododecane was well embedded within the fibers. Inversely, groups A and G generally performed the worst except in the visual analysis and, for G samples, the gravimetric analysis. This indicates that a tenacious layer of cyclododecane was formed on the surface of these samples, particularly in group G, but it did not protect the sample from dye bleeding whatsoever because the cyclododecane sat only on the surface instead of being embedded within the fibers. The paintbrush and *kistka* were also compared and it was discovered that, generally, the *kistka* samples performed better than the brush samples, likely because it allowed the cyclododecane to become better embedded within the fibers.

Although a reasonably large number of variable were included within this study, there are several other aspects that unfortunately could not be explored at the time. If the material could have been sourced, a dense balanced plain-weave silk with an equivalent cotton would have been the most preferable substrate materials as the difference in substrates in this study may have altered the results somewhat. There was also an unavoidable difference in the amounts of cyclododecane that was applied by a brush and by a *kistka.* If this could be rectified it could alter the results, possibly further in favor of the *kistka.* It would also be highly beneficial to test the methods on actual historic objects in order to provide more accurate expectations during real-world applications. Creating microscope cross-sections of the various samples

could allow visual analysis of to what extent cyclododecane embedded the fibers. Analyzing the infrared spectra with the multivariate data analysis method called Principal Component Analysis would be mathematically rigorous but it would emphasize patterns between the application methods and the amount of cyclododecane present on the samples. It would also provide quantitative data comparing the infrared spectra, as this study only generated qualitative data about the spectra.

Although cyclododecane is often used in the field of textile conservation, its use is often based on the treatments of objects that are vastly different from textiles. Because of this, it is likely that the cyclododecane is sometimes used ineffectively, which could possibly cause damage to the historic objects that are being treated. This study not only expanded on and confirmed the results of previous research, it provided qualitative and quantitative evidence that was used to explain those results and inform the use of cyclododecane within textile conservation. At the end of the study, the results and their causes were extrapolated in order to create instructions for applying cyclododecane to textiles which conservators can use for the optimum treatment of their objects.

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Appendix A: Recommended Method of Application

A Recommended Method for Applying Cyclododecane to Textiles to Protect Against Dye Bleed during Aqueous Treatments

Please note that this method has only been tested on light/medium weight textiles without any type of dimensional decoration such as embroidery or a dimensional weave. The method has also not been tested on historic objects at this time.

1. Choose a tool to apply the cyclododecane to the object. An electric wax-melting pen called a *kistka* provides the best results and is suitable for smaller areas that need protection during treatment. A paintbrush provides good results and is more practical when applying cyclododecane to larger areas. The *kistka* is easy to use since solid pieces of cyclododecane can be loaded into the tool's reservoir where it is melted. However, this reservoir is small. Even with the use of a specialized *kistka* reservoir extender only small amounts of cyclododecane can be loaded at a time. Using "sticks" of cyclododecane make loading the reservoir easier and can be made by filling plastic pipettes with melted cyclododecane, letting it solidify, and then cutting it free. If using a brush a double-boiler, particularly an electric one, is very useful to ensure that they cyclododecane remains molten. Cyclododecane easily solidifies and can become encrusted on the brush, hampering application. However, letting the brush sit in the molten cyclododecane within a double-boiler melts the problematic encrustation and clears the brush. Both tools deposit a relatively large amount of cyclododecane when initially touched to the object. This can be beneficial for adding extra protection to particularly susceptible areas.

2. Evaluate the object and determine which is the "back", or the side that has the least amount of the water-sensitive area exposed. Apply a layer of cyclododecane to back while the object is at room temperature. The cyclododecane is applied to this area first since this creates a thicker cyclododecane layer on the opposite side later.

3. After the back layer has dried, heat the opposite side (the "front") with a heated spatula set to 60-65° C. 20 seconds of heating is enough for light-weight textiles. However, 60 seconds may be more appropriate for thicker substrates. Immediately apply cyclododecane to the heated front as was done in step 2.

4. After the initial layers of cyclododecane have dried, extra cyclododecane can be applied to areas of the layers that seem particularly thin. Adding whole second coats to both sides may also be beneficial, particularly if using a *kistka* as it creates thinner layers than a brush. However, this has not been tested.

5. Progress with treating the object. Clean any tools or supplies coated with cyclododecane by physically scraping off any solid cyclododecane possible. Then let the tools sit under ventilation or in a warmed environment in order to promote the sublimation of any remaining traces of cyclododecane.

6. After treatment, the cyclododecane will sublimate from the object at room temperature. However, even thin layers can take a week to sublimate this way. If safe for the object, sublimation can be sped up by placing the object under ventilation or in a warmed environment.

Appendix B: Health and Safety

University of Glasgow

RISK ASSESSMENT FORM

School:	Section:	Location:	Reference Form Number:	Related COSHH Form Number:
Culture and Creative Arts	Centre For Textile Conservation and Technical Art History	Room number(s) 308, 309A, 310, 315	R_____	C_____

Description of activity:
Cyclododecane and Indigo Carmine were applied to fabric substrates in order to test cyclododecane's efficacy as a hydrophobic barrier.
The cyclododecane will be heated to 60-90 degrees Celsius using heated metal tools and an electric double-boiler.
The cyclododecane will then be allowed to sublimate at room temperature in an open environment until it is no longer present.
The cyclododecane will be tested using aqueous submersion, stereomicroscopic analysis, gravimetric analysis, and ATR-FTIR and DRIFTS analysis.

Persons at risk:	Is operator training/supervision required? If yes, please specify:
Students, Staff	Yes, training is required to operate the ATR-FTIR and DRIFTS analytical machines.

Hazards/ Risks	Current controls	Are these adequate?	What action is required if not adequately controlled?
Chemical -Cyclododecane -Indigo Carmine	PPE, keeping within closed and labelled container, knowing proper disposal methods	**No**	**Read and Complete appropriate COSHH forms**
Spillages	Keep lid tightly on containers that can be closed. Keep all containers away from counter edges.	**Yes**	
Sharps, Broken Glass -Cuts	Keep all breakable containers away from counter edges. Use care when handling. Dispose of broken glass in appropriate disposal container.	**Yes**	
Hot Surfaces -Burns	Keep all heated instruments away from counter edges. Use extreme care when handling and heat-proof PPE when necessary. Know placement of first aid kit.	**Yes**	
Electrical Equipment -Trip Hazard -Fire Hazard	Keep all cords out of the way of foot traffic. Ensure that the electronics have been inspected recently. Turn the equipment off, unplug it, and turn off the electrical socket when not in use.	**Yes**	

Completed by: Gennifer Majors, student	**Date:** 30, 5, 2015
Approved by: Dr Anita Quye, Lecturer in Conservation Science	**Date:** 18/8/2015

University of Glasgow

COSHH Assessment Form

School: Culture and Creative Arts
Section: Centre for Textile Conservation and Technical Art History
Project Title: Dissertation **Date:** 30-5-2015

File ref: C_____
Related Assessment Form: R_____

Room Number(s): 308, 309A, 310, 315
Building: Robertson Building, University of Glasgow

Persons involved:
Student, Staff

Description of procedure:
Cyclododecane and Indigo Carmine were applied to fabric substrates in order to test cyclododecane's efficacy as a hydrophobic barrier.
The cyclododecane will be heated to 60-90 degrees Celsius using heated metal tools and an electric double-boiler.
The cyclododecane will then be allowed to sublimate at room temperature in an open environment until it is no longer present.
The cyclododecane will be tested using aqueous submersion, stereomicroscopic analysis, gravimetric analysis, and ATR-FTIR and DRIFTS analysis.

Substance used	Quantities used	Frequency of use	Hazards identified	Exposure route
Cyclododecane	200g	3 hours every weekday for 6 weeks	None	Inhalation, Skin, Eye, Ingestion

Could a less hazardous substance (or form of the substance) be used instead? **NO**
Justify not using it: N/A

What measures have you taken to control risk?

Engineering controls: Adequate ventilation used in case of dust buildup.

Personal Protective Equipment: Gloves, safety glasses, heat-resistant gloves, long-sleeves, shoes that cover the toes.

Management measures: Store in a tightly-sealed container in a cool, well-ventilated area. Do not store with food or drink.

Checks on control measures: N/A	**Health surveillance required?** No	**Training required:** None

Emergency procedures:
Skin: wash with soap and water; if symptoms persist, consult a doctor.
Inhalation: move to fresh air; if symptoms persist, consult a doctor.
Eye: rinse with water for 15 minutes; if symptoms persist, consult a doctor.
Ingestion: rinse mouth, drink water; if symptoms persist, consult a doctor.

Waste disposal:
Treat as toxic waste. Do not discharge into drains or surface/ground water. Dispose into suitable waste container.

Name and position of assessor: Gennifer Majors, Student **Signature**:

Name of supervisor (student work only): Dr Anita Quye **Signature**:

Name of Head of School or nominee: **Signature**:

University of Glasgow

COSHH Assessment Form

School: Culture and Creative Arts
Section: Centre for Textile Conservation and Technical Art History
Project Title: Dissertation **Date:** 30-5-2015

File ref: C_____
Related Assessment Form: R_____

Room Number(s): 308, 309A, 310, 315
Building: Robertson Building, University of Glasgow

Persons involved:
Student, Staff

Description of procedure:
Cyclododecane and Indigo Carmine were applied to fabric substrates in order to test cyclododecane's efficacy as a hydrophobic barrier.
The cyclododecane will be heated to 60-90 degrees Celsius using heated metal tools and an electric double-boiler.
The cyclododecane will then be allowed to sublimate at room temperature in an open environment until it is no longer present.
The cyclododecane will be tested using aqueous submersion, stereomicroscopic analysis, gravimetric analysis, and ATR-FTIR and DRIFTS analysis.

Substance used	Quantities used	Frequency of use	Hazards identified	Exposure route
Indigo Carmine	10g	2 days	Eye and skin irritant	Inhalation, Skin, Eye, Ingestion

Could a less hazardous substance (or form of the substance) be used instead? **NO**
Justify not using it: N/A

What measures have you taken to control risk?

Engineering controls: Adequate ventilation used to keep airborne particles low if user operations generate dust, fume, or mist.

Personal Protective Equipment: Gloves, lab coat, dust mask.

Management measures: Store in a tightly-closed, light-resistant container in a cool, well-ventilated area.

Checks on control measures: N/A	**Health surveillance required?** No	**Training required:** None

Emergency procedures:
Skin: wash with soap and water; if symptoms persist, consult a doctor.
Inhalation: move to fresh air; if symptoms persist, consult a doctor.
Eye: rinse with water for 15 minutes; if symptoms persist, consult a doctor.
Ingestion: rinse mouth, drink water; if symptoms persist, consult a doctor.

Waste disposal:
Place solid waste into waste disposal container, clean area with water.

Name and position of assessor: Gennifer Majors, Student **Signature**:

Name of supervisor (student work only): Dr Anita Quye **Signature**:

Name of Head of School or nominee: **Signature**:

Appendix C: Supplies and Tools

Cyclododecane
500g
Purchased August 2011
87100 Cyclododecan
CAS-Nr. 294-62-2
EINECS-Nr. 206-033-9
Kremer Pigmente
GmbH & Co KG
Hauptstr. 41-47
D-88317 Aichstetten
Phone: +49-7565-91120
Fax: +49-7565-1606

Indigo Carmine
CASRegistryNumber1: 860-22-0
MFCD Number: MFCD00005723
Grade: Certified Biological Stain
Dye Content: 80% minimum
Fisher Scientific
300 Industry Drive
Pittsburgh, PA 15275
Phone: 1-800-766-7000
Fax: 1-800-926-1166

Electric *Kistka* Wax-Melting Pen
Electric Kistka Multi Tip Interchangeable with 3 tips. 220 Volt (Europe, Australia, Asia)
#1 Fine, #2 Medium and #3 Heavy tips
WaxArtSupply
WaxArtSupply.com
Phone: (443)492-9278
Email: orders@waxartsupply.com

Fourier Transform Infrared Spectroscopy Analytical Machine
PerkinElmer®
Spectrum One FT-IR Spectrometer
940 Winter St.
Waltham , Massachusetts 02451
US Phone: 800-762-4000
US Fax: +1 203-944-4904
US Email: CustomerCareUS@perkinelmer.com
UK Phone: 0800-89.60.46
UK Fax: 0800-89.17.14
UK Email: cc.uk@perkinelmer.com

Attenuated Total Reflectance Sampling Accessory
PerkinElmer®
Spectrum One ATR top plate for the Spectrum One FT-IR Spectrometer
940 Winter St.
Waltham , Massachusetts 02451
US Phone: 800-762-4000
US Fax: +1 203-944-4904
US Email: CustomerCareUS@perkinelmer.com
UK Phone: 0800-89.60.46
UK Fax: 0800-89.17.14
UK Email: cc.uk@perkinelmer.com

Software used for ATR-FTIR analysis
PerkinElmer®
Spectrum version 5.0.1
Used on Default Settings for Absorption Spectra
940 Winter St.
Waltham , Massachusetts 02451
US Phone: 800-762-4000
US Fax: +1 203-944-4904
US Email: CustomerCareUS@perkinelmer.com
UK Phone: 0800-89.60.46
UK Fax: 0800-89.17.14
UK Email: cc.uk@perkinelmer.com

Diffuse Reflectance Infrared Fourier Transform Spectroscopy Analytical Machine
Agilent Technologies
4100 ExoScan Series FTIR handheld tool
5301 Stevens Creek Blvd.
Santa Clara, CA 95051
contact_us@agilent.com
Phone: +1 877-424-4536
Phone: +1 408-345-8886

Diffuse Sampling Accessory
Agilent Technologies
Diffuse sampling accessory for the 4100 ExoScan Series FTIR handheld tool
5301 Stevens Creek Blvd.
Santa Clara, CA 95051
contact_us@agilent.com
Phone: +1 877-424-4536
Phone: +1 408-345-8886

Software use for DRIFTS analysis
MicroLab FTIR software
Used on Default Settings for Absorption Spectra
5301 Stevens Creek Blvd.
Santa Clara, CA 95051
contact_us@agilent.com
Phone: +1 877-424-4536
Phone: +1 408-345-8886

Appendix D: Resulting Data and Graphs

Appendix D.1: Results of the Dye Bleeding Analysis

The tables below show the analysis of any active bleeding noticed during the dye bleed test. The key immediately below explains the notations used as well as the levels of dye bleeding that occurred.

Amount of Dye Bleed		
"H"	high levels of dye bleed	Dye bled profusely onto the sample substrate and/or into the water when the sample was submerged.
"L"	low levels of dye bleed	Dye bled somewhat onto the sample substrate and/or into the water when the sample was submerged.
"N"	no bleed	Dye never bled during the time when the sample was submerged.
Characteristic of Dye Bleed		
"I"	immediate bleed	Dye began to bleed immediately when the sample was submerged.
"W"	water-only bleed	Dye bled only into the water, not onto the sample substrate, when the sample was submerged.

Sample label	**Dye bleed types**	**Sample label**	**Dye bleed types**	**Sample label**	**Dye bleed types**	**Sample label**	**Dye bleed types**
cA1b	I, L	sA1b	N	cA1k	L	sA1k	N
cA2b	I, H	sA2b	N	cA2k	L	sA2k	L
cA3b	N	sA3b	N	cA3k	H, W	sA3k	L
cA4b	I, H, W	sA4b	N	cA4k	L	sA4k	N
cB1b	L	sB1b	N	cB1k	L, W	sB1k	N
cB2b	L	sB2b	N	cB2k	L, W	sB2k	N
cB3b	L	sB3b	N	cB3k	L, W	sB3k	N
cB4b	L	sB4b	N	cB4k	L, W	sB4k	N
cC1b	I, L	sC1b	N	cC1k	I, H, W	sC1k	N
cC2b	I, H	sC2b	N	cC2k	L, W	sC2k	N
cC3b	L	sC3b	N	cC3k	I, H, W	sC3k	N
cC4b	I, L, W	sC4b	N	cC4k	L, W	sC4k	N
cD1b	I, H	sD1b	N	cD1k	L, W	sD1k	L
cD2b	H	sD2b	N	cD2k	L, W	sD2k	N
cD3b	L	sD3b	N	cD3k	I, L, W	sD3k	N
cD4b	I, H, W	sD4b	N	cD4k	L, W	sD4k	N
cE1b	I, H, W	sE1b	L, W	cE1k	I, H, W	sE1k	I, L, W
cE2b	I, H, W	sE2b	I, L, W	cE2k	I, H, W	sE2k	L, W
cE3b	I, L, W	sE3b	N	cE3k	I, H, W	sE3k	I, L, W
cE4b	I, L, W	sE4b	L, W	cE4k	I, H, W	sE4k	L, W
cF1b	I, H, W	sF1b	L, W	cF1k	I, L	sF1k	L
cF2b	I, L, W	sF2b	L, W	cF2k	I, H	sF2k	I, L
cF3b	I, H, W	sF3b	N	cF3k	I, L	sF3k	N
cF4b	I, L, W	sF4b	N	cF4k	H	sF4k	N
cG1b	L	sG1b	N	cG1k	I, L	sG1k	I, L
cG2b	L	sG2b	N	cG2k	L	sG2k	N
cG3b	I, L	sG3b	N	cG3k	I, L	sG3k	N
cG4b	I, L	sG4b	N	cG4k	I, H, W	sG4k	N

Appendix D.2: Gravimetric Measurements of the Samples During Sublimation

The tables below show the total masses (cyclododecane and sample substrate) of the samples during the course of their sublimation. All masses were measured in grams. In order to complete the calculations for the chapter "Analyzing the Data" the masses of the substrates themselves, given below, were subtracted from the total masses. These substrate masses were calculated from control pieces of substrates which were not added until 6/19/2015. The substrate masses before this date are the calculated average of the measured substrate masses and the original mass of the substrate.

Substrate Masses	Original Mass	6/12/2015 12:00	6/15/2015 13:30	6/17/2015 12:30	6/17/2015 16:20	6/18/2015 13:30	6/19/2015 11:00	7/6/2015 11:00	7/6/2015 16:30	7/7/2015 14:30	7/7/2015 16:45	7/8/2015 10:00	7/9/2015 15:30	7/10/2015 11:30	7/13/2015 12:00	7/14/2015 16:00	7/15/2015 16:00
Cotton	1.2	1.205	1.205	1.205	1.205	1.205	1.2	1.217	1.202	1.209	1.209	1.205	1.193	1.212	1.211	1.198	1.198
silk	0.75	0.753	0.753	0.753	0.753	0.753	0.75	0.757	0.752	0.755	0.755	0.754	0.749	0.758	0.755	0.75	0.751

	6/12/2015 12:00	6/15/2015 13:30	6/17/2015 12:30	6/17/2015 16:20	6/18/2015 13:30	6/19/2015 11:00	7/6/2015 11:00	7/6/2015 16:30	7/7/2015 14:30	7/7/2015 16:45	7/8/2015 10:00	7/9/2015 15:30	7/10/2015 11:30	7/13/2015 12:00	7/14/2015 16:00	7/15/2015 16:00
cA1b	1.887	1.778	1.689	1.669	1.586	1.516	1.504	1.466	1.382	1.367	1.303	1.218	1.22	1.211	1.197	1.2
cA2b	1.978	1.871	1.763	1.745	1.657	1.584	1.549	1.51	1.443	1.425	1.369	1.265	1.25	1.239	1.225	1.227
cA3b	2.257	2.143	2.045	2.025	1.93	1.849	1.829	1.787	1.698	1.68	1.609	1.465	1.421	1.22	1.202	1.204
cA4b	2.133	2.008	1.911	1.892	1.805	1.727	1.706	1.684	1.588	1.571	1.485	1.366	1.312	1.21	1.193	1.196
sA1b	1.315	1.157	1.113	1.125	1.015	0.971	0.956	0.927	0.857	0.844	0.799	0.761	0.767	0.768	0.759	0.76
sA2b	1.254	1.105	1.061	1.051	0.967	0.911	0.901	0.878	0.816	0.806	0.779	0.754	0.764	0.764	0.755	0.756
sA3b	1.312	1.209	1.118	1.045	0.976	0.961	0.938	0.902	0.841	0.83	0.812	0.761	0.767	0.767	0.759	0.758
sA4b	1.291	1.178	1.078	1.06	0.984	0.916	0.887	0.861	0.81	0.799	0.772	0.745	0.756	0.765	0.736	0.747
cA1k	1.552	1.514	1.479	1.469	1.435	1.407	1.406	1.391	1.362	1.355	1.327	1.267	1.264	1.224	1.208	1.21
cA2k	1.57	1.519	1.482	1.473	1.436	1.405	1.406	1.384	1.353	1.346	1.323	1.259	1.263	1.221	1.206	1.204
cA3k	1.549	1.503	1.475	1.467	1.439	1.415	1.411	1.399	1.372	1.366	1.343	1.285	1.28	1.218	1.195	1.194
cA4k	1.582	1.533	1.492	1.482	1.444	1.408	1.403	1.384	1.352	1.341	1.321	1.255	1.258	1.216	1.199	1.2
sA1k	0.944	0.893	0.866	0.859	0.833	0.814	0.814	0.795	0.788	0.785	0.778	0.76	0.765	0.762	0.755	0.753
sA2k	0.888	0.836	0.806	0.8	0.781	0.774	0.774	0.766	0.79	0.769	0.767	0.757	0.767	0.767	0.76	0.759
sA3k	0.901	0.868	0.847	0.841	0.822	0.807	0.809	0.796	0.788	0.79	0.779	0.758	0.767	0.767	0.759	0.758

sA4k	0.873	0.84	0.817	0.812	0.794	0.78	0.781	0.773	0.764	0.762	0.756	0.745	0.755	0.756	0.746	0.745

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cB1b	2.007	1.897	1.795	1.77	1.672	1.592	1.553	1.515	1.437	1.422	1.355	1.253	1.235	1.214	1.2	1.2
cB2b	2.118	1.085	1.878	1.852	1.761	1.689	1.668	1.642	1.558	1.491	1.458	1.337	1.314	1.228	1.207	1.208
cB3b	1.783	1.693	1.605	1.582	1.497	1.434	1.422	1.391	1.33	1.318	1.267	1.222	1.237	1.238	1.224	1.225
cB4b	1.754	1.653	1.563	1.544	1.452	1.387	1.379	1.35	1.294	1.279	1.231	1.192	1.209	1.21	1.195	1.196
sB1b	1.136	1.042	0.941	0.924	0.856	0.81	0.797	0.786	0.774	0.772	0.769	0.758	0.77	0.769	0.762	0.759
sB2b	1.199	1.999	0.983	0.96	0.883	0.829	0.765	0.759	0.763	0.765	0.766	0.755	0.764	0.764	0.757	0.757
sB3b	1.181	1.077	0.979	0.939	0.884	0.832	0.825	0.806	0.783	0.778	0.768	0.755	0.765	0.766	0.758	0.756
sB4b	1.122	1.028	0.941	0.923	0.854	0.808	0.805	0.786	0.769	0.767	0.761	0.753	0.763	0.763	0.754	0.756
cB1k	1.582	1.542	1.509	1.496	1.462	1.438	1.44	1.422	1.394	1.391	1.368	1.312	1.309	1.249	1.233	1.233
cB2k	1.553	1.515	1.477	1.466	1.43	1.403	1.407	1.389	1.362	1.357	1.332	1.275	1.265	1.213	1.196	1.197
cB3k	1.543	1.528	1.492	1.482	1.45	1.426	1.42	1.404	1.382	1.376	1.353	1.298	1.291	1.199	1.196	1.194
cB4k	1.572	1.537	1.503	1.493	1.456	1.426	1.424	1.41	1.384	1.377	1.351	1.351	1.285	1.226	1.208	1.209
sB1k	0.918	0.896	0.872	0.866	0.832	0.826	0.828	0.815	0.801	0.798	0.785	0.762	0.768	0.767	0.757	0.759
sB2k	0.978	0.949	0.923	0.916	0.888	0.87	0.873	0.855	0.838	0.831	0.813	0.776	0.777	0.766	0.758	0.758
sB3k	0.931	0.909	0.887	0.879	0.853	0.835	0.827	0.872	0.804	0.802	0.788	0.761	0.768	0.763	0.757	0.755
sB4k	0.916	0.896	0.871	0.864	0.84	0.825	0.828	0.817	0.801	0.798	0.787	0.762	0.769	0.767	0.759	0.758
cC1b	1.724	1.633	1.542	1.517	1.452	1.409	1.396	1.37	1.315	1.304	1.266	1.203	1.219	1.218	1.204	1.205
cC2b	2.049	1.939	1.827	1.8	1.724	1.673	1.639	1.608	1.543	1.53	1.482	1.382	1.365	1.228	1.197	1.199
cC3b	2.184	2.097	2.019	1.993	1.916	1.857	1.842	1.817	1.734	1.718	1.666	1.513	1.479	1.2	1.247	1.228
cC4b	1.994	1.912	1.827	1.799	1.733	1.688	1.677	1.648	1.574	1.56	1.492	1.38	1.353	1.249	1.226	1.227
sC1b	1.1	0.897	0.951	0.939	0.879	0.848	0.844	0.827	0.793	0.788	0.778	0.757	0.766	0.766	0.757	0.757
sC2b	1.214	1.141	1.053	1.043	0.967	0.929	0.901	0.879	0.833	0.827	0.801	0.766	0.769	0.763	0.754	0.755
sC3b	1.15	1.059	0.993	0.971	0.912	0.871	0.866	0.85	0.801	0.792	0.771	0.751	0.76	0.76	0.752	0.752
sC4b	1.347	1.275	1.188	1.174	0.106	1.056	1.047	1.019	0.968	0.929	0.913	0.825	0.807	0.764	0.757	0.757
cC1k	1.571	1.531	1.486	1.472	1.439	1.417	1.419	1.386	1.355	1.348	1.325	1.262	1.265	1.215	1.197	1.199
cC2k	1.577	1.546	1.505	1.493	1.467	1.439	1.435	1.417	1.391	1.385	1.363	1.294	1.296	1.238	1.221	1.222
cC3k	1.565	1.531	1.481	1.483	1.456	1.435	1.431	1.418	1.39	1.382	1.362	1.301	1.299	1.239	1.213	1.211
cC4k	1.538	1.505	1.474	1.462	1.432	1.414	1.415	1.394	1.373	1.366	1.344	1.282	1.28	1.23	1.209	1.206
sC1k	0.965	0.943	0.911	0.901	0.882	0.874	0.873	0.862	0.844	0.841	0.827	0.803	0.797	0.769	0.758	0.754
sC2k	0.931	0.796	0.878	0.805	0.821	0.84	0.834	0.828	0.815	0.812	0.803	0.778	0.787	0.765	0.756	0.758
sC3k	0.936	0.916	0.888	0.88	0.857	0.847	0.845	0.836	0.821	0.818	0.803	0.768	0.777	0.766	0.758	0.759
sC4k	0.911	0.889	0.865	0.859	0.838	0.827	0.827	0.816	0.805	0.801	0.793	0.769	0.773	0.762	0.757	0.753

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cD1b	1.796	1.711	1.613	1.599	1.522	1.463	1.453	1.434	1.365	1.35	1.308	1.241	1.238	1.204	1.19	1.19
cD2b	1.736	1.66	1.573	1.557	1.492	1.441	1.434	1.413	1.362	1.351	1.311	1.251	1.254	1.252	1.239	1.24
cD3b	1.877	1.775	1.666	1.648	1.574	1.512	1.487	1.464	1.398	1.388	1.335	1.241	1.23	1.208	1.194	1.195
cD4b	2.072	1.983	1.887	1.869	1.789	1.726	1.715	1.693	1.633	1.622	1.567	1.449	1.414	1.264	1.23	1.227
sD1b	1.234	1.137	0.999	1.018	0.939	0.882	0.88	0.86	0.817	0.811	0.784	0.756	0.761	0.761	0.754	0.754
sD2b	1.499	1.406	1.305	1.285	1.208	1.151	1.144	1.126	1.056	1.044	0.987	0.899	0.874	0.771	0.754	0.753
sD3b	1.414	1.307	1.198	1.178	1.102	1.047	1.014	0.995	0.943	0.933	0.872	0.832	0.818	0.766	0.75	0.751
sD4b	1.353	1.26	1.17	1.152	1.084	1.037	1.025	1.006	0.957	0.946	0.898	0.825	0.797	0.759	0.753	0.752
cD1k	1.602	1.562	1.522	1.514	1.481	1.457	1.451	1.436	1.411	1.407	1.384	1.323	1.319	1.241	1.212	1.208
cD2k	1.586	1.543	1.495	1.486	1.449	1.426	1.416	1.402	1.377	1.372	1.344	1.288	1.285	1.219	1.197	1.195
cD3k	1.584	1.548	1.504	1.496	1.462	1.437	1.435	1.421	1.397	1.39	1.363	1.305	1.301	1.232	1.207	1.206
cD4k	1.523	1.487	1.441	1.433	1.401	1.379	1.374	1.363	1.335	1.331	1.306	1.252	1.251	1.207	1.186	1.196
sD1k	0.91	0.893	0.872	0.81	0.829	0.8	0.835	0.825	0.814	0.81	0.798	0.778	0.783	0.765	0.751	0.757
sD2k	0.933	0.919	0.895	0.89	0.872	0.856	0.861	0.851	0.841	0.838	0.826	0.796	0.796	0.766	0.741	0.756
sD3k	0.959	0.937	0.911	0.906	0.883	0.871	0.862	0.854	0.841	0.84	0.827	0.803	0.806	0.781	0.767	0.761
sD4k	0.944	0.909	0.891	0.887	0.86	0.852	0.848	0.84	0.83	0.827	0.813	0.786	0.788	0.766	0.752	0.751
cE1b	1.961	1.872	1.757	1.727	1.622	1.526	1.503	1.465	1.396	1.384	1.334	1.245	1.252	1.25	1.236	1.237
cE2b	2.056	1.961	1.85	1.822	1.715	1.623	1.576	1.544	1.461	1.447	1.383	1.266	1.247	1.219	1.205	1.205
cE3b	2	1.905	1.781	1.75	1.632	1.537	1.517	1.487	1.405	1.391	1.329	1.238	1.239	1.239	1.223	1.225
cE4b	2.112	2.004	1.855	1.831	1.716	1.624	1.604	1.572	1.473	1.457	1.391	1.284	1.263	1.237	1.223	1.226
sE1b	1.159	0.985	0.967	0.943	0.867	0.822	0.824	0.808	0.778	0.773	0.765	0.753	0.764	0.764	0.755	0.755
sE2b	1.17	1.066	0.964	0.943	0.869	0.818	0.802	0.789	0.775	0.771	0.764	0.754	0.764	0.765	0.756	0.757
sE3b	1.106	1.011	0.917	0.9	0.807	0.782	0.775	0.765	0.761	0.761	0.759	0.748	0.758	0.76	0.75	0.749
sE4b	1.135	1.039	0.942	0.922	0.843	0.79	0.781	0.77	0.757	0.755	0.755	0.746	0.756	0.756	0.747	0.749
cE1k	1.573	1.546	1.509	1.498	1.467	1.438	1.435	1.418	1.4	1.395	1.375	1.322	1.326	1.253	1.23	1.226
cE2k	1.528	1.492	1.457	1.445	1.411	1.384	1.377	1.364	1.342	1.335	1.316	1.266	1.268	1.216	1.195	1.19
cE3k	1.591	1.556	1.515	1.501	1.459	1.428	1.414	1.401	1.372	1.367	1.346	1.29	1.292	1.235	1.213	1.209
cE4k	1.57	1.546	1.506	1.492	4.453	1.425	1.421	1.404	1.372	1.366	1.340	1.28	1.28	1.226	1.205	1.207
sE1k	0.964	0.943	0.917	0.899	0.847	0.872	0.871	0.861	0.848	0.844	0.830	0.799	0.802	0.763	0.75	0.748
sE2k	0.926	0.907	0.862	0.873	0.837	0.837	0.823	0.831	0.82	0.817	0.807	0.781	0.784	0.756	0.754	0.755
sE3k	0.943	0.918	0.889	0.88	0.853	0.836	0.826	0.818	0.803	0.801	0.790	0.764	0.772	0.76	0.751	0.752
sE4k	0.918	0.868	0.877	0.873	0.845	0.837	0.838	0.83	0.822	0.82	0.810	0.788	0.794	0.773	0.76	0.76

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cF1b	2.125	2.053	1.952	1.932	1.841	1.762	1.706	1.706	1.605	1.587	1.536	1.406	1.388	1.253	1.241	1.241
cF2b	2.075	2.002	1.902	1.882	1.793	1.736	1.717	1.677	1.577	1.561	1.499	1.394	1.354	1.23	1.21	1.211
cF3b	1.789	1.707	1.619	1.598	1.513	1.433	1.392	1.37	1.306	1.298	1.258	1.206	1.224	1.222	1.206	1.209
cF4b	1.903	1.83	1.725	1.706	1.62	1.572	1.555	1.524	1.451	1.438	1.377	1.285	1.27	1.242	1.227	1.228
sF1b	1.243	1.143	1.044	1.013	0.941	0.876	0.864	0.842	0.798	0.79	0.775	0.756	0.767	0.765	0.759	0.757
sF2b	1.159	1.078	0.996	0.973	0.903	0.843	0.833	0.811	0.777	0.775	0.767	0.756	0.76	0.766	0.759	0.761
sF3b	1.195	1.123	1.004	1.009	0.937	0.887	0.854	0.831	0.799	0.793	0.775	0.754	0.765	0.763	0.757	0.756
sF4b	1.257	1.184	1.077	1.08	0.976	0.929	0.947	0.927	0.883	0.874	0.845	0.793	0.788	0.764	0.756	0.756
cF1k	1.549	1.53	1.497	1.485	1.455	1.427	1.424	1.407	1.384	1.378	1.354	1.296	1.3	1.229	1.205	1.205
cF2k	1.567	1.542	1.5	1.491	1.448	1.413	1.405	1.39	1.36	1.356	1.328	1.272	1.272	1.224	1.205	1.208
cF3k	1.571	1.531	1.514	1.504	1.467	1.435	1.426	1.409	1.382	1.376	1.344	1.283	1.289	1.222	1.207	1.207
cF4k	1.569	1.551	1.515	1.505	1.467	1.439	1.435	1.415	1.392	1.381	1.359	1.294	1.299	1.227	1.206	1.206
sF1k	0.938	0.86	0.909	0.86	0.881	0.836	0.865	0.851	0.837	0.832	0.821	0.789	0.793	0.77	0.762	0.762
sF2k	0.923	0.912	0.888	0.882	0.862	0.845	0.838	0.829	0.818	0.813	0.801	0.775	0.781	0.766	0.758	0.757
sF3k	0.926	0.914	0.892	0.887	0.802	0.848	0.847	0.835	0.824	0.821	0.807	0.791	0.788	0.768	0.768	0.757
sF4k	0.921	0.871	0.876	0.876	0.859	0.837	0.838	0.828	0.813	0.811	0.795	0.77	0.774	0.761	0.755	0.755
cG1b	1.958	1.896	1.811	1.795	1.721	1.656	1.644	1.603	1.522	1.504	1.436	1.323	1.479	1.215	1.2	1.201
cG2b	2.202	2.147	2.056	2.036	1.96	1.89	1.867	1.821	1.733	1.712	1.63	1.5	1.452	1.252	1.212	1.212
cG3b	2.214	2.15	2.064	2.044	1.967	1.896	1.884	1.845	1.761	1.744	1.666	1.547	1.506	1.313	1.259	1.243
cG4b	1.915	1.851	1.764	1.744	1.663	1.594	1.561	1.523	1.451	1.433	1.368	1.268	1.248	1.223	1.211	1.213
sG1b	1.406	1.347	1.265	1.25	1.183	1.122	1.095	1.061	1	0.985	0.93	0.846	0.853	0.761	0.752	0.754
sG2b	1.409	1.307	1.25	1.242	1.17	1.113	1.106	1.082	1.012	0.998	0.95	0.88	0.849	0.763	0.77	0.754
sG3b	1.99	1.931	1.838	1.818	1.73	1.647	1.635	1.604	1.505	1.489	1.424	1.294	1.252	1.001	0.907	0.858
sG4b	1.839	1.769	1.678	1.659	1.581	1.51	1.49	1.47	1.378	1.365	1.309	1.193	1.159	0.924	0.854	0.82
cG1k	1.598	1.572	1.524	1.515	1.48	1.447	1.446	1.425	1.388	1.378	1.346	1.281	1.235	1.246	1.232	1.236
cG2k	1.623	1.596	1.563	1.554	1.518	1.491	1.491	1.473	1.44	1.433	1.401	1.34	1.339	1.245	1.215	1.212
cG3k	1.572	1.548	1.516	1.506	1.473	1.448	1.439	1.42	1.389	1.383	1.348	1.289	1.289	1.24	1.224	1.226
cG4k	1.541	1.435	1.484	1.476	1.442	1.417	1.419	1.4	1.374	1.365	1.34	1.285	1.287	1.221	1.2	1.2
sG1k	0.942	0.928	0.896	0.897	0.879	0.831	0.865	0.853	0.838	0.833	0.816	0.786	0.789	0.761	0.752	0.754
sG2k	0.935	0.921	0.821	0.833	0.874	0.857	0.854	0.846	0.832	0.828	0.815	0.79	0.783	0.764	0.748	0.751
sG3k	0.908	0.893	0.872	0.866	0.846	0.828	0.829	0.82	0.802	0.798	0.785	0.762	0.768	0.761	0.754	0.755
sG4k	0.909	0.894	0.873	0.867	0.848	0.833	0.83	0.818	0.804	0.799	0.784	0.758	0.761	0.758	0.749	0.752

Appendix D.3: Visual Analysis of the Cyclododecane Layers During Sublimation

The tables below show state of the cyclododecane layers present on a sample throughout sublimation, as based on visual analysis. The samples in the tables are listed from "best performance" to "worst performance" and are grouped by substrate and application tool. The key below explains the symbols used in the tables and the visible characteristics of the cyclododecane as it sublimated.

Categories of Visible Sublimation Characteristics		
○	"Complete"	Complete layer of cyclododecane.
Δ	"Partial"	Partial layer of cyclododecane.
◊	"Embedded"	Cyclododecane embedded within the fibers but not laying on the substrate surface.
∩	"Surface"	Cyclododecane layered on the substrate surface but not embedded within the fibers.
□	"None"	No visible cyclododecane remaining.

Sample Front	6/12/2015 12:00	6/15/2015 13:30	6/17/2015 16:20	6/18/2015 13:30	6/19/2015 11:00	7/7/2015 14:30	7/8/2015 10:00	7/9/2015 15:30	7/10/2015 11:30	7/13/2015 12:00	7/14/2015 16:00	7/15/2015 16:00
sG4b	○	○	○	○	○	○	Δ	Δ	Δ	◊	□	□
sD4b	○	○	○	○	○	Δ	Δ	Δ	Δ	□	□	□
sG1b	○	○	○	○	○	Δ	Δ	Δ	◊	□	□	□
sD3b	○	○	○	○	Δ	Δ	Δ	Δ	Δ	□	□	□
sG3b	○	Δ	Δ	Δ	Δ	Δ	Δ	Δ	Δ	◊	□	□
sG2b	○	Δ	Δ	Δ	Δ	Δ	Δ	Δ	◊	□	□	□
sC4b	○	Δ	Δ	Δ	Δ	Δ	Δ	◊	◊	□	□	□
sD2b	○	Δ	Δ	Δ	Δ	Δ	Δ	◊	◊	□	□	□
sA1b	○	Δ	Δ	Δ	Δ	Δ	□	□	□	□	□	□
sA4b	○	Δ	Δ	Δ	Δ	Δ	□	□	□	□	□	□
sA2b	○	Δ	Δ	Δ	Δ	◊	□	□	□	□	□	□
sB2b	○	Δ	Δ	Δ	Δ	◊	□	□	□	□	□	□
sF1b	○	Δ	Δ	Δ	Δ	□	□	□	□	□	□	□
sF2b	○	Δ	Δ	Δ	Δ	□	□	□	□	□	□	□
sF3b	○	Δ	Δ	Δ	Δ	□	□	□	□	□	□	□
sB1b	○	◊	◊	◊	◊	◊	□	□	□	□	□	□
sB3b	○	◊	◊	◊	◊	◊	□	□	□	□	□	□
sC1b	○	□	□	□	□	□	□	□	□	□	□	□
sC3b	○	□	□	□	□	□	□	□	□	□	□	□
sE1b	○	□	□	□	□	□	□	□	□	□	□	□
sE2b	○	□	□	□	□	□	□	□	□	□	□	□
sE3b	○	□	□	□	□	□	□	□	□	□	□	□
sE4b	○	□	□	□	□	□	□	□	□	□	□	□

Sample Front	6/12/2015 12:00	6/15/2015 13:30	6/17/2015 16:20	6/18/2015 13:30	6/19/2015 11:00	7/7/2015 14:30	7/8/2015 10:00	7/9/2015 15:30	7/10/2015 11:30	7/13/2015 12:00	7/14/2015 16:00	7/15/2015 16:00
cA3b	○	○	○	○	○	◊	◊	◊	◊	⊃	□	□
cB2b	○	○	○	○	Δ	◊	◊	◊	◊	◊	□	□
cA2b	○	○	○	○	Δ	◊	◊	⊃	⊃	□	□	□
cB3b	○	○	○	○	Δ	◊	◊	□	□	□	□	□
cB4b	○	○	○	○	◊	◊	◊	□	□	□	□	□
cD2b	○	○	○	○	◊	◊	◊	□	□	□	□	□
cG2b	○	Δ	Δ	Δ	Δ	Δ	Δ	Δ	⊃	⊃	□	□
cG1b	○	Δ	Δ	Δ	Δ	Δ	Δ	⊃	⊃	□	□	□
cG4b	○	Δ	Δ	Δ	Δ	Δ	Δ	⊃	⊃	□	□	□
cA4b	○	Δ	Δ	Δ	Δ	Δ	◊	⊃	⊃	□	□	□
cF2b	○	Δ	Δ	Δ	Δ	◊	◊	◊	◊	◊	□	□
cA1b	○	Δ	Δ	Δ	Δ	◊	◊	◊	□	□	□	□
cE3b	○	Δ	Δ	Δ	Δ	◊	◊	◊	□	□	□	□
cE4b	○	Δ	Δ	Δ	Δ	◊	□	□	□	□	□	□
cC3b	○	Δ	Δ	Δ	◊	◊	◊	◊	◊	◊	□	□
cC4b	○	Δ	Δ	Δ	◊	◊	◊	◊	◊	□	□	□
cD1b	○	Δ	Δ	Δ	◊	◊	◊	◊	◊	□	□	□
cD4b	○	◊	◊	◊	◊	◊	◊	◊	◊	◊	□	□
cF1b	○	◊	◊	◊	◊	◊	◊	◊	◊	□	□	□
cC1b	○	◊	◊	◊	◊	◊	◊	□	□	□	□	□
cC2b	○	◊	◊	◊	◊	◊	◊	□	□	□	□	□
cD3b	○	◊	◊	◊	◊	◊	□	□	□	□	□	□
cE2b	○	◊	◊	◊	◊	◊	□	□	□	□	□	□
cF4b	○	◊	◊	◊	◊	◊	□	□	□	□	□	□

Sample Front	6/12/2015 12:00	6/15/2015 13:30	6/17/2015 16:20	6/18/2015 13:30	6/19/2015 11:00	7/7/2015 14:30	7/8/2015 10:00	7/9/2015 15:30	7/10/2015 11:30	7/13/2015 12:00	7/14/2015 16:00	7/15/2015 16:00
sG2k	○	○	○	○	○	Δ	Δ	Δ	Δ	Δ	□	□
sG1k	○	○	○	○	○	Δ	Δ	Δ	Δ	□	□	□
sG4k	○	○	○	○	○	Δ	Δ	Δ	◊	□	□	□
sD4k	○	○	○	○	Δ	Δ	Δ	Δ	Δ	Δ	◊	□
sD2k	○	○	○	○	Δ	Δ	Δ	Δ	Δ	□	□	□
sA1k	○	Δ	Δ	Δ	Δ	Δ	Δ	Δ	Δ	□	□	□
sB1k	○	Δ	Δ	Δ	Δ	Δ	Δ	Δ	Δ	□	□	□
sB2k	○	Δ	Δ	Δ	Δ	Δ	Δ	Δ	Δ	□	□	□
sB4k	○	Δ	Δ	Δ	Δ	Δ	Δ	Δ	Δ	□	□	□
sC1k	○	Δ	Δ	Δ	Δ	Δ	Δ	Δ	Δ	□	□	□
sC3k	○	Δ	Δ	Δ	Δ	Δ	Δ	Δ	Δ	□	□	□
sD3k	○	Δ	Δ	Δ	Δ	Δ	Δ	Δ	Δ	□	□	□
sE2k	○	Δ	Δ	Δ	Δ	Δ	Δ	Δ	Δ	□	□	□
sE3k	○	Δ	Δ	Δ	Δ	Δ	Δ	Δ	Δ	□	□	□
sF3k	○	Δ	Δ	Δ	Δ	Δ	Δ	Δ	Δ	□	□	□
sA3k	○	Δ	Δ	Δ	Δ	Δ	Δ	Δ	□	□	□	□
sB3k	○	Δ	Δ	Δ	Δ	Δ	Δ	Δ	□	□	□	□
sF2k	○	Δ	Δ	Δ	Δ	Δ	Δ	Δ	□	□	□	□
sA4k	○	Δ	Δ	Δ	Δ	Δ	Δ	□	□	□	□	□
sC4k	○	Δ	Δ	Δ	Δ	Δ	Δ	□	□	□	□	□
sC2k	○	Δ	Δ	Δ	Δ	Δ	□	□	□	□	□	□
sE4k	○	Δ	Δ	Δ	Δ	Δ	□	□	□	□	□	□
sF1k	○	Δ	Δ	Δ	Δ	Δ	□	□	□	□	□	□

Sample Front	6/12/2015 12:00	6/15/2015 13:30	6/17/2015 16:20	6/18/2015 13:30	6/19/2015 11:00	7/7/2015 14:30	7/8/2015 10:00	7/9/2015 15:30	7/10/2015 11:30	7/13/2015 12:00	7/14/2015 16:00	7/15/2015 16:00
cB1k	o	Δ	Δ	Δ	Δ	Δ	Δ	Δ	Δ	Δ	□	□
cC4k	o	Δ	Δ	Δ	Δ	Δ	Δ	Δ	Δ	Δ	□	□
cB4k	o	Δ	Δ	Δ	Δ	Δ	Δ	Δ	Δ	◊	□	□
cD3k	o	Δ	Δ	Δ	Δ	Δ	Δ	Δ	Δ	◊	□	□
cD4k	o	Δ	Δ	Δ	Δ	Δ	Δ	Δ	Δ	◊	□	□
cA1k	o	Δ	Δ	Δ	Δ	Δ	Δ	Δ	Δ	□	□	□
cA2k	o	Δ	Δ	Δ	Δ	Δ	Δ	Δ	Δ	□	□	□
cA3k	o	Δ	Δ	Δ	Δ	Δ	Δ	Δ	Δ	□	□	□
cA4k	o	Δ	Δ	Δ	Δ	Δ	Δ	Δ	Δ	□	□	□
cB2k	o	Δ	Δ	Δ	Δ	Δ	Δ	Δ	Δ	□	□	□
cB3k	o	Δ	Δ	Δ	Δ	Δ	Δ	Δ	Δ	□	□	□
cC3k	o	Δ	Δ	Δ	Δ	Δ	Δ	◊	◊	◊	□	□
cD2k	o	Δ	Δ	Δ	Δ	Δ	Δ	◊	◊	◊	□	□
cE1k	o	Δ	Δ	Δ	Δ	Δ	Δ	◊	◊	◊	□	□
cE4k	o	Δ	Δ	Δ	Δ	Δ	Δ	◊	◊	◊	□	□
cF1k	o	Δ	Δ	Δ	Δ	Δ	Δ	◊	◊	◊	□	□
cF4k	o	Δ	Δ	Δ	Δ	Δ	Δ	◊	◊	◊	□	□
cC1k	o	Δ	Δ	Δ	Δ	Δ	Δ	◊	◊	□	□	□
cC2k	o	Δ	Δ	Δ	Δ	Δ	Δ	◊	◊	□	□	□
cD1k	o	Δ	Δ	Δ	Δ	Δ	Δ	◊	◊	□	□	□
cE2k	o	Δ	Δ	Δ	Δ	Δ	Δ	◊	◊	□	□	□
cE3k	o	Δ	Δ	Δ	Δ	Δ	Δ	◊	◊	□	□	□
cG2k	o	Δ	Δ	Δ	Δ	Δ	Δ	◊	◊	□	□	□
cG3k	o	Δ	Δ	Δ	Δ	Δ	Δ	◊	◊	□	□	□
cG4k	o	Δ	Δ	Δ	Δ	Δ	Δ	◊	◊	□	□	□
cF2k	o	Δ	Δ	Δ	Δ	Δ	◊	◊	◊	◊	□	□
cF3k	o	Δ	Δ	Δ	Δ	Δ	◊	◊	◊	□	□	□
cG1k	o	Δ	Δ	Δ	Δ	Δ	◊	◊	◊	□	□	□

Sample Back	6/12/2015 12:00	6/15/2015 13:30	6/17/2015 16:20	6/18/2015 13:30	6/19/2015 11:00	7/7/2015 14:30	7/8/2015 10:00	7/9/2015 15:30	7/10/2015 11:30	7/13/2015 12:00	7/14/2015 16:00	7/15/2015 16:00
cF2b	o	o	o	o	o	o	o	Δ	Δ	□	□	□
cC3b	o	o	o	o	o	Δ	Δ	Δ	Δ	Δ	Δ	Δ
cC4b	o	o	o	o	o	Δ	Δ	Δ	Δ	Δ	□	□
cF1b	o	o	o	o	o	Δ	Δ	Δ	Δ	□	□	□
cE4b	o	o	o	o	o	Δ	Δ	◊	◊	□	□	□
cG1b	o	o	o	o	o	◊	◊	◊	◊	◊	□	□
cF4b	o	o	o	o	o	◊	◊	◊	◊	□	□	□
cG2b	o	o	o	o	o	◊	◊	◊	◊	□	□	□
cA3b	o	o	o	o	Δ	Δ	Δ	Δ	Δ	□	□	□
cD1b	o	o	o	o	Δ	Δ	Δ	Δ	Δ	□	□	□
cE2b	o	o	o	o	Δ	Δ	◊	◊	◊	□	□	□
cA4b	o	o	o	o	Δ	◊	◊	◊	◊	□	□	□
cD3b	o	o	o	o	Δ	◊	◊	◊	◊	□	□	□
cA2b	o	o	o	o	Δ	◊	◊	◊	□	□	□	□
cE3b	o	o	o	o	◊	◊	◊	□	□	□	□	□
cC2b	o	o	o	Δ	Δ	Δ	Δ	Δ	Δ	⊃	⊃	□
cG4b	o	o	o	Δ	Δ	Δ	◊	◊	◊	□	□	□
cB1b	o	o	o	Δ	Δ	□	□	□	□	□	□	□
cB4b	o	o	o	Δ	◊	◊	□	□	□	□	□	□
cD2b	o	o	Δ	Δ	Δ	◊	◊	◊	◊	□	□	□
cB3b	o	o	Δ	◊	◊	◊	□	□	□	□	□	□

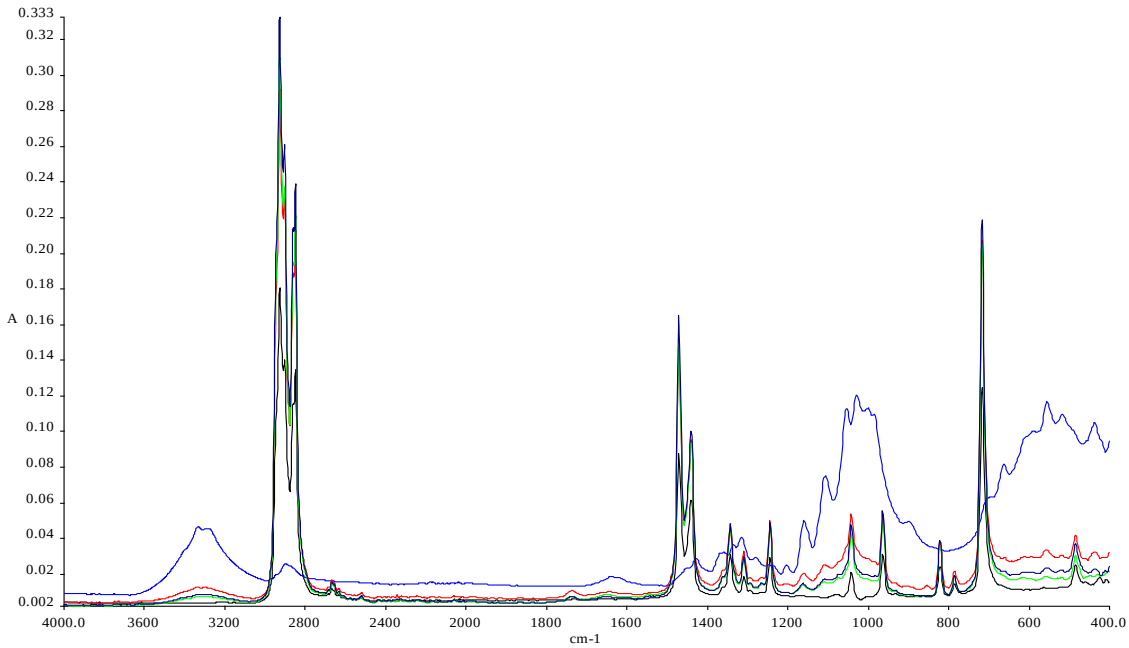
Sample Back	6/12/2015 12:00	6/15/2015 13:30	6/17/2015 16:20	6/18/2015 13:30	6/19/2015 11:00	7/7/2015 14:30	7/8/2015 10:00	7/9/2015 15:30	7/10/2015 11:30	7/13/2015 12:00	7/14/2015 16:00	7/15/2015 16:00
sG1b	○	○	○	○	○	○	○	Δ	Δ	□	□	□
sG2b	○	○	○	○	○	○	○	Δ	Δ	□	□	□
sG4b	○	○	○	○	○	○	○	Δ	Δ	Δ	Δ	Δ
sC2b	○	○	○	○	Δ	Δ	Δ	□	□	□	□	□
sA3b	○	○	○	○	Δ	◊	◊	□	□	□	□	□
sC1b	○	○	○	○	Δ	◊	□	□	□	□	□	□
sA1b	○	○	○	○	◊	◊	◊	□	□	□	□	□
sD4b	○	○	○	Δ	Δ	Δ	Δ	◊	◊	◊	□	□
sD3b	○	○	○	Δ	Δ	Δ	Δ	◊	□	□	□	□
sC3b	○	○	○	Δ	Δ	Δ	□	□	□	□	□	□
sD1b	○	○	○	Δ	Δ	◊	◊	□	□	□	□	□
sE2b	○	○	○	Δ	Δ	◊	□	□	□	□	□	□
sF1b	○	○	○	Δ	Δ	◊	□	□	□	□	□	□
sF2b	○	○	○	Δ	Δ	◊	□	□	□	□	□	□
sB2b	○	○	○	Δ	◊	◊	◊	◊	□	□	□	□
sA2b	○	○	○	Δ	◊	◊	◊	□	□	□	□	□
sB4b	○	○	○	◊	◊	◊	◊	□	□	□	□	□
sB1b	○	○	○	□	□	□	□	□	□	□	□	□
sE3b	○	○	○	□	□	□	□	□	□	□	□	□
sE4b	○	○	○	□	□	□	□	□	□	□	□	□
sF3b	○	○	Δ	Δ	Δ	◊	□	□	□	□	□	□
sE1b	○	○	Δ	◊	◊	◊	□	□	□	□	□	□
sB3b	○	○	Δ	□	□	□	□	□	□	□	□	□

Sample Back	6/12/2015 12:00	6/15/2015 13:30	6/17/2015 16:20	6/18/2015 13:30	6/19/2015 11:00	7/7/2015 14:30	7/8/2015 10:00	7/9/2015 15:30	7/10/2015 11:30	7/13/2015 12:00	7/14/2015 16:00	7/15/2015 16:00
cE1k	o	o	o	o	o	Δ	Δ	Δ	Δ	Δ	Δ	Δ
cE2k	o	o	o	o	o	Δ	Δ	Δ	Δ	Δ	Δ	Δ
cE4k	o	o	o	o	o	Δ	Δ	Δ	Δ	Δ	□	□
cG2k	o	o	o	o	Δ	Δ	Δ	Δ	Δ	Δ	Δ	Δ
cF2k	o	o	o	o	Δ	Δ	Δ	Δ	Δ	Δ	□	□
cF3k	o	o	o	o	Δ	Δ	Δ	Δ	Δ	Δ	□	□
cF4k	o	o	o	o	Δ	Δ	Δ	Δ	Δ	Δ	□	□
cG4k	o	o	o	Δ	Δ	Δ	Δ	Δ	Δ	Δ	Δ	□
cB4k	o	o	o	Δ	Δ	Δ	Δ	Δ	Δ	Δ	□	□
cD3k	o	o	o	Δ	Δ	Δ	Δ	Δ	Δ	Δ	□	□
cG3k	o	o	o	Δ	Δ	Δ	Δ	Δ	Δ	Δ	□	□
cG1k	o	o	o	Δ	Δ	Δ	Δ	Δ	Δ	□	□	□
cA3k	o	o	Δ	Δ	Δ	Δ	Δ	Δ	Δ	Δ	Δ	Δ
cB3k	o	o	Δ	Δ	Δ	Δ	Δ	Δ	Δ	Δ	Δ	Δ
cC3k	o	o	Δ	Δ	Δ	Δ	Δ	Δ	Δ	Δ	Δ	Δ
cC4k	o	o	Δ	Δ	Δ	Δ	Δ	Δ	Δ	Δ	Δ	Δ
cD2k	o	o	Δ	Δ	Δ	Δ	Δ	Δ	Δ	Δ	Δ	□
cA2k	o	o	Δ	Δ	Δ	Δ	Δ	Δ	Δ	Δ	□	□
cA4k	o	o	Δ	Δ	Δ	Δ	Δ	Δ	Δ	Δ	□	□
cB2k	o	o	Δ	Δ	Δ	Δ	Δ	Δ	Δ	Δ	□	□
cC1k	o	o	Δ	Δ	Δ	Δ	Δ	Δ	Δ	Δ	□	□
cC2k	o	o	Δ	Δ	Δ	Δ	Δ	Δ	Δ	Δ	□	□
cB1k	o	o	Δ	Δ	Δ	Δ	Δ	Δ	Δ	◊	□	□
cA1k	o	o	Δ	Δ	Δ	Δ	Δ	Δ	Δ	□	□	□
cD4k	o	o	Δ	Δ	Δ	Δ	Δ	Δ	Δ	□	□	□

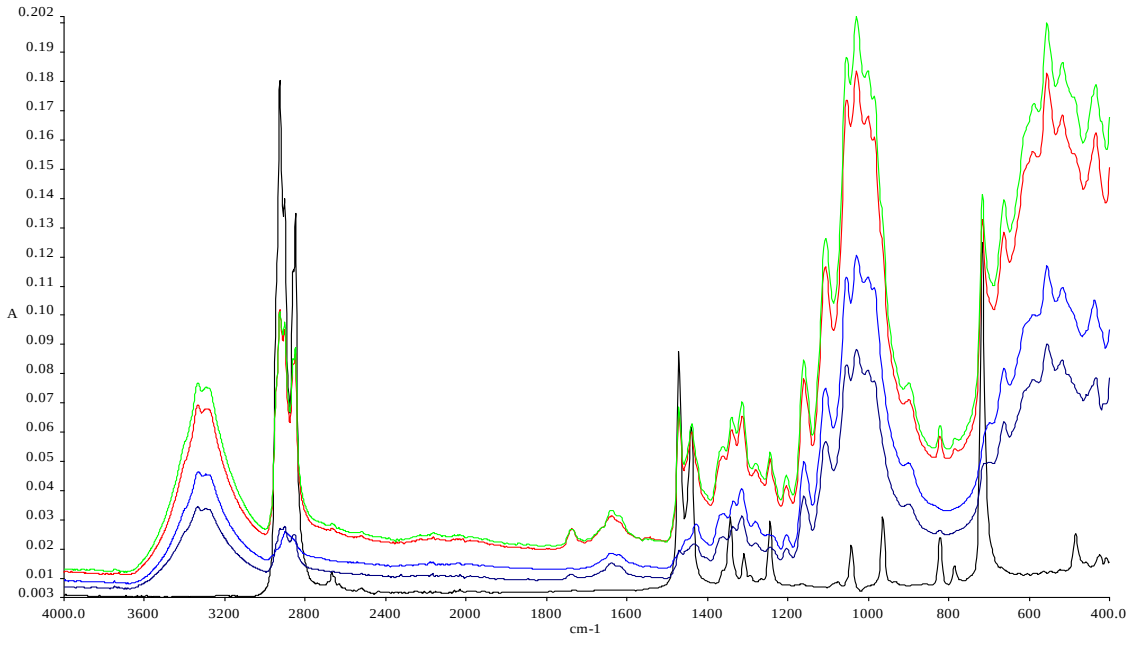
Sample Back	6/12/2015 12:00	6/15/2015 13:30	6/17/2015 16:20	6/18/2015 13:30	6/19/2015 11:00	7/7/2015 14:30	7/8/2015 10:00	7/9/2015 15:30	7/10/2015 11:30	7/13/2015 12:00	7/14/2015 16:00	7/15/2015 16:00
sE1k	o	o	o	o	o	Δ	Δ	Δ	Δ	Δ	Δ	□
sD2k	o	o	o	o	o	Δ	Δ	Δ	Δ	Δ	□	□
sE2k	o	o	o	o	o	Δ	Δ	Δ	Δ	□	□	□
sD3k	o	o	o	o	Δ	Δ	Δ	Δ	Δ	Δ	Δ	□
sG1k	o	o	o	o	Δ	Δ	Δ	Δ	Δ	Δ	□	□
sD1k	o	o	o	o	Δ	Δ	Δ	Δ	Δ	□	□	□
sG2k	o	o	o	o	Δ	Δ	Δ	Δ	Δ	□	□	□
sE4k	o	o	o	Δ	Δ	Δ	Δ	Δ	Δ	Δ	Δ	□
sF1k	o	o	o	Δ	Δ	Δ	Δ	Δ	Δ	Δ	□	□
sF3k	o	o	o	Δ	Δ	Δ	Δ	Δ	Δ	Δ	□	□
sB4k	o	o	o	Δ	Δ	Δ	Δ	Δ	Δ	□	□	□
sC2k	o	o	o	Δ	Δ	Δ	Δ	Δ	Δ	□	□	□
sE3k	o	o	o	Δ	Δ	Δ	Δ	Δ	Δ	□	□	□
sF2k	o	o	o	Δ	Δ	Δ	Δ	Δ	Δ	□	□	□
sC1k	o	o	Δ	Δ	Δ	Δ	Δ	Δ	Δ	Δ	□	□
sA3k	o	o	Δ	Δ	Δ	Δ	Δ	Δ	Δ	□	□	□
sB3k	o	o	Δ	Δ	Δ	Δ	Δ	Δ	Δ	□	□	□
sC4k	o	o	Δ	Δ	Δ	Δ	Δ	Δ	Δ	□	□	□
sG3k	o	o	Δ	Δ	Δ	Δ	Δ	Δ	Δ	□	□	□
sG4k	o	o	Δ	Δ	Δ	Δ	Δ	Δ	◊	□	□	□
sA1k	o	o	Δ	Δ	Δ	Δ	Δ	Δ	□	□	□	□
sB1k	o	o	Δ	Δ	Δ	Δ	Δ	□	□	□	□	□
sA2k	o	o	Δ	Δ	□	□	□	□	□	□	□	□
sA4k	o	o	Δ	Δ	□	□	□	□	□	□	□	□

Appendix D.4: Infrared Spectra of the Analyzed Samples

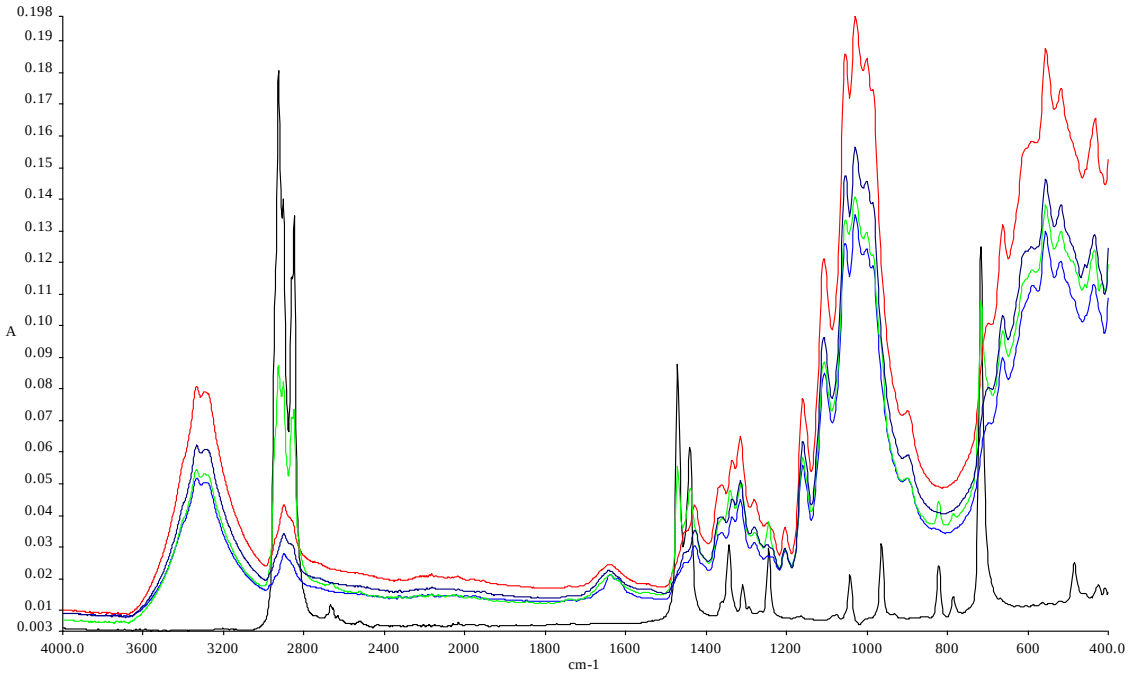
Approximately 400 infrared spectra comparison overlays similar to the following were created during the experiment. These include the absorption spectra from both ATR-FTIR and DRIFTS analysis, as well as derivatives of the spectra used to better analyze the results. Due to practicality issues, not all of these spectra could be included here. However, digital images of the spectra will be kept with this study at the Centre for Textile Conservation. The following are examples of each type of sample tested, and show the differences between spectra from ATR-FTIR and DRIFTS for the same sample.



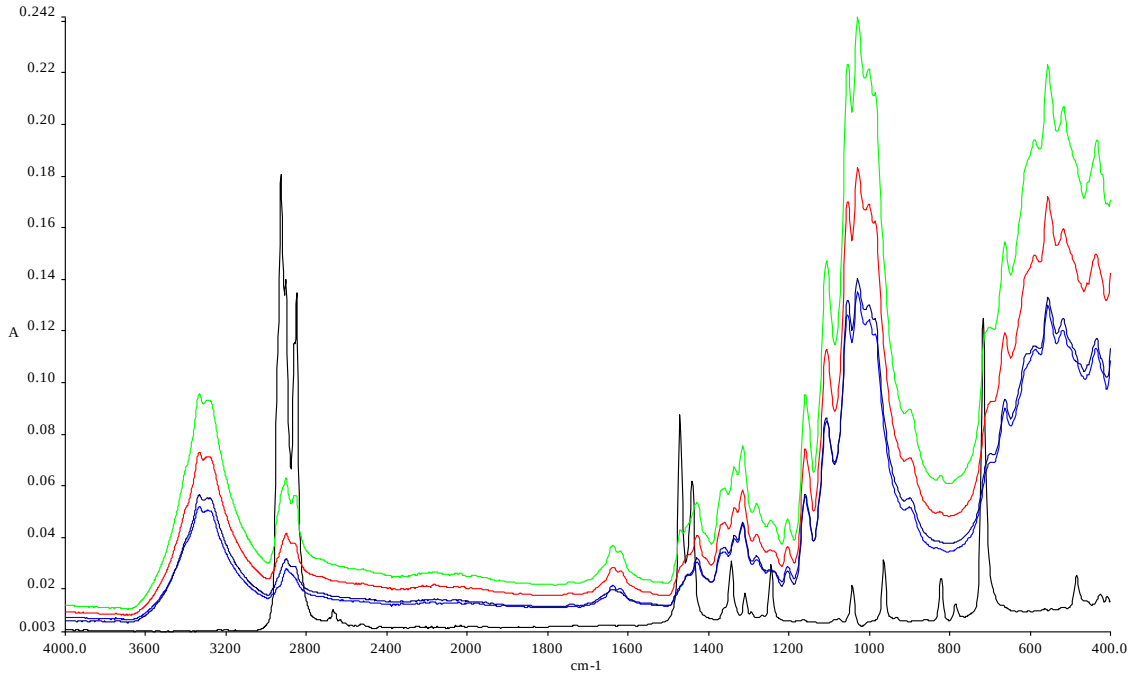
CDD (black) *cotton (blue)* *cA2bF1 (navy)* *cA2bF2 (green)* *cA2bF3 (red)*



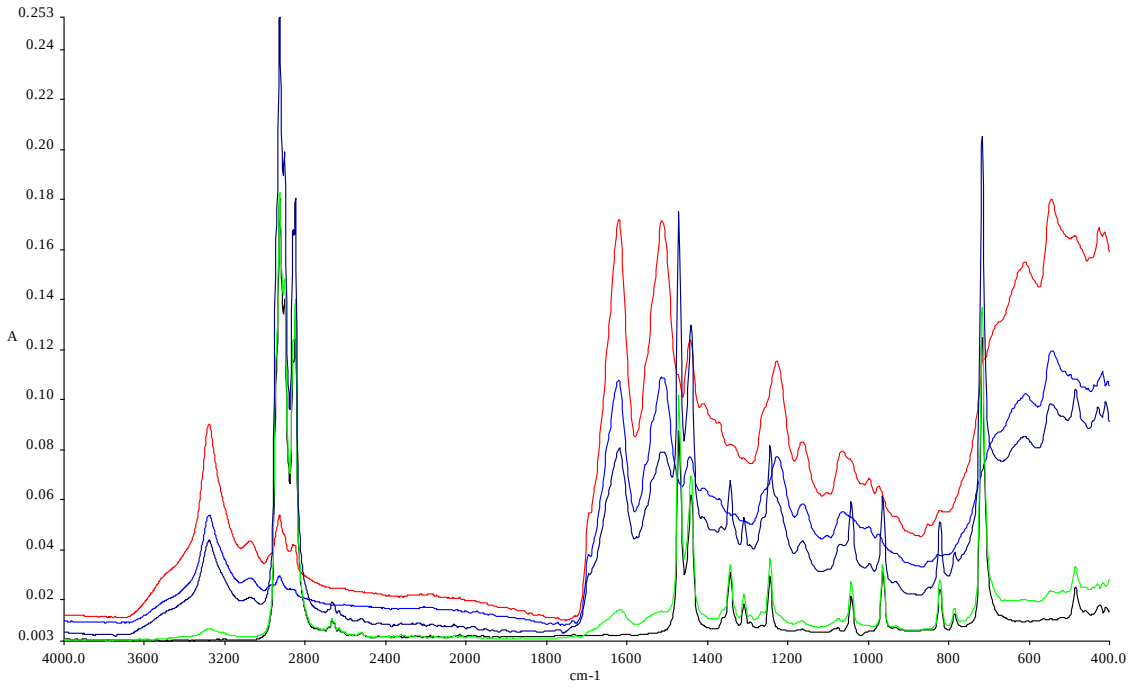
CDD (black) *cotton (blue)* *cA2bB3 (red)* *cA2bB2 (green)* *cA2bB1 (navy)*



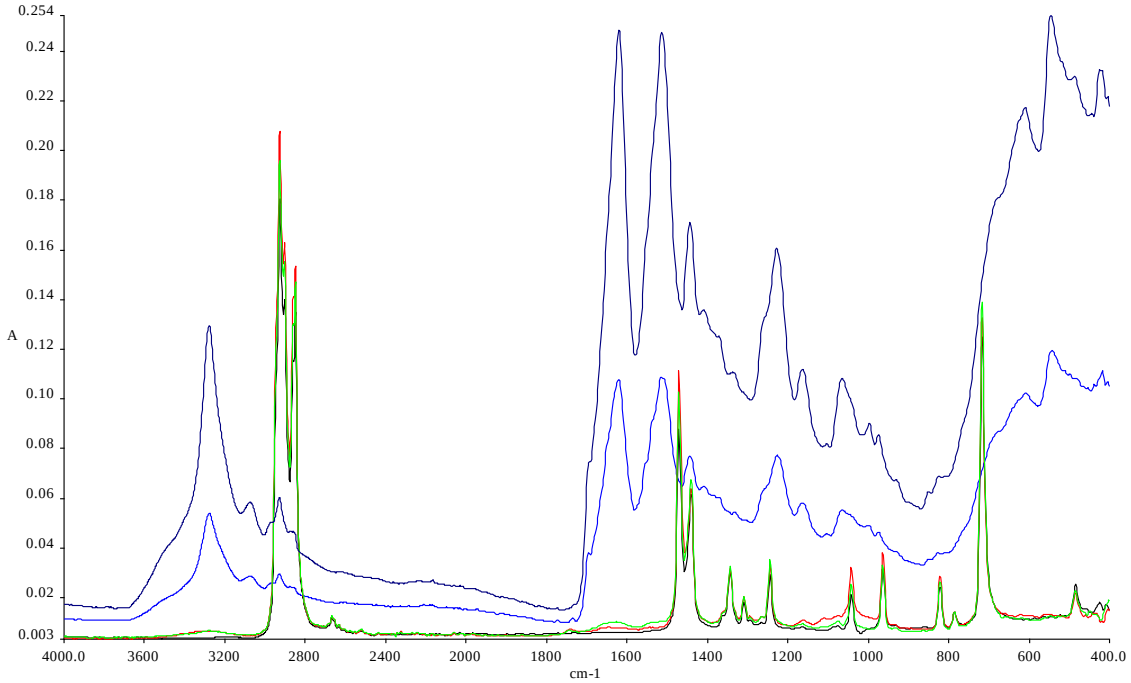
CDD (black) *cotton (blue)* *cA4kF1 (navy)* *cA4kF2 (green)* *cA4kF3 (red)*



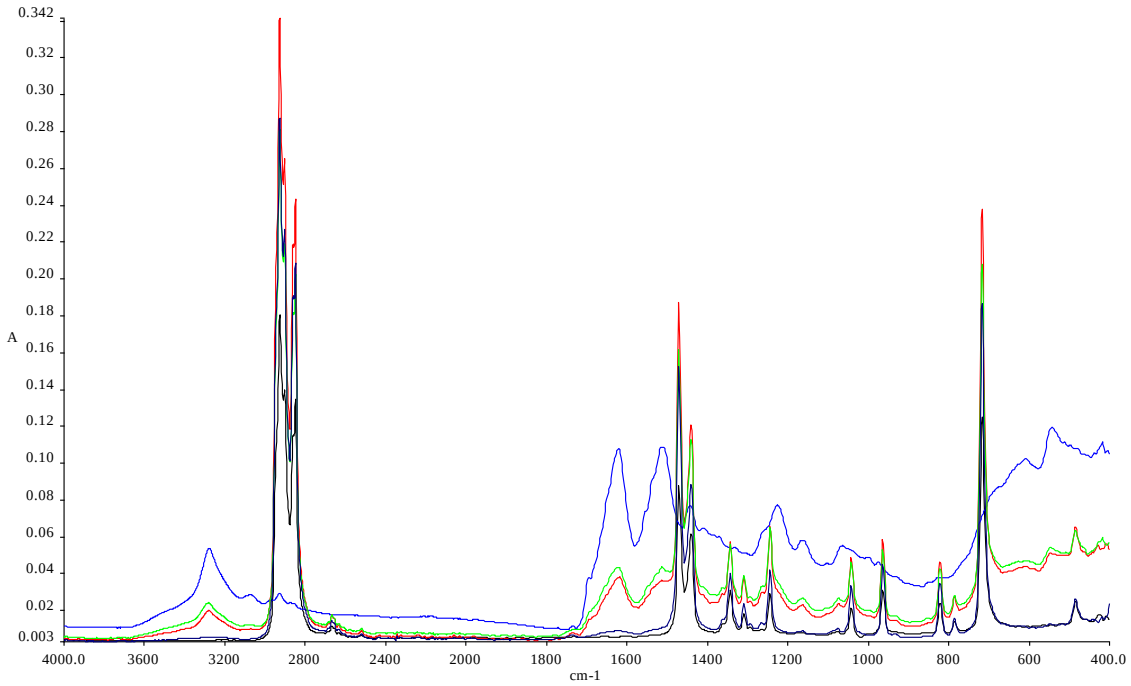
CDD (black) *cotton (blue)* *cA4kB3 (navy)* *cA4kB1 (green)*



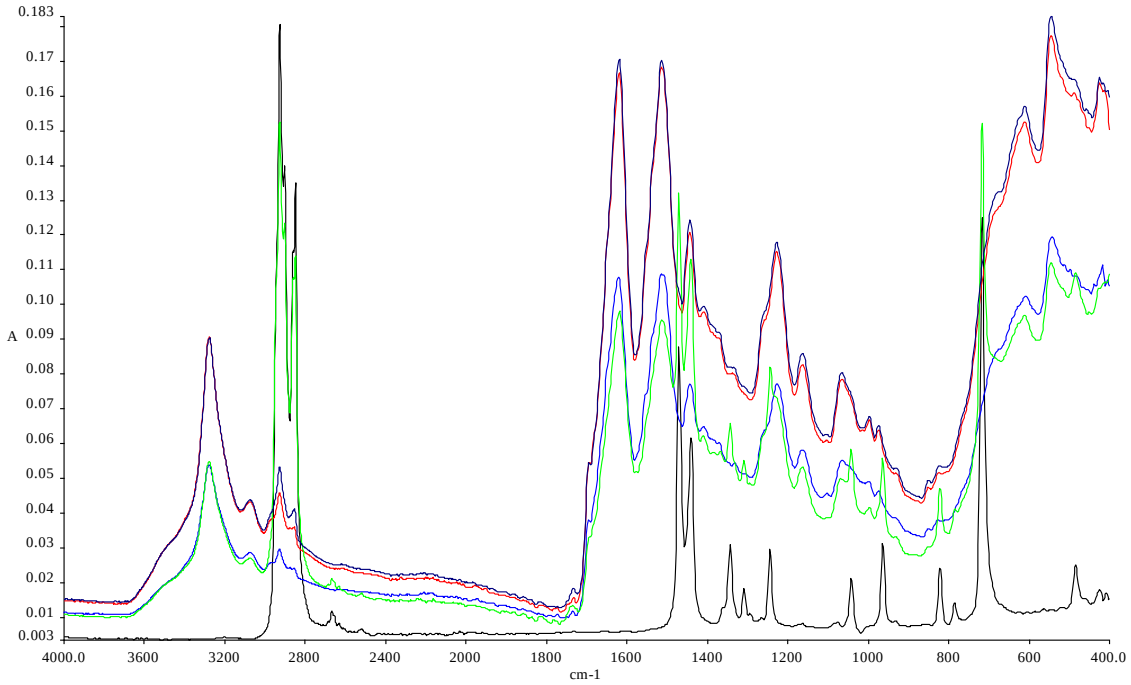
CDD (black) *silk (blue)* *sA3kF1 (navy)* *sA3kF2 (green)* *sA3kF3 (red)*



CDD (black) *silk (blue)* *sA3kB1 (navy)* *sA3kB2 (green)* *sA3kB3 (red)*



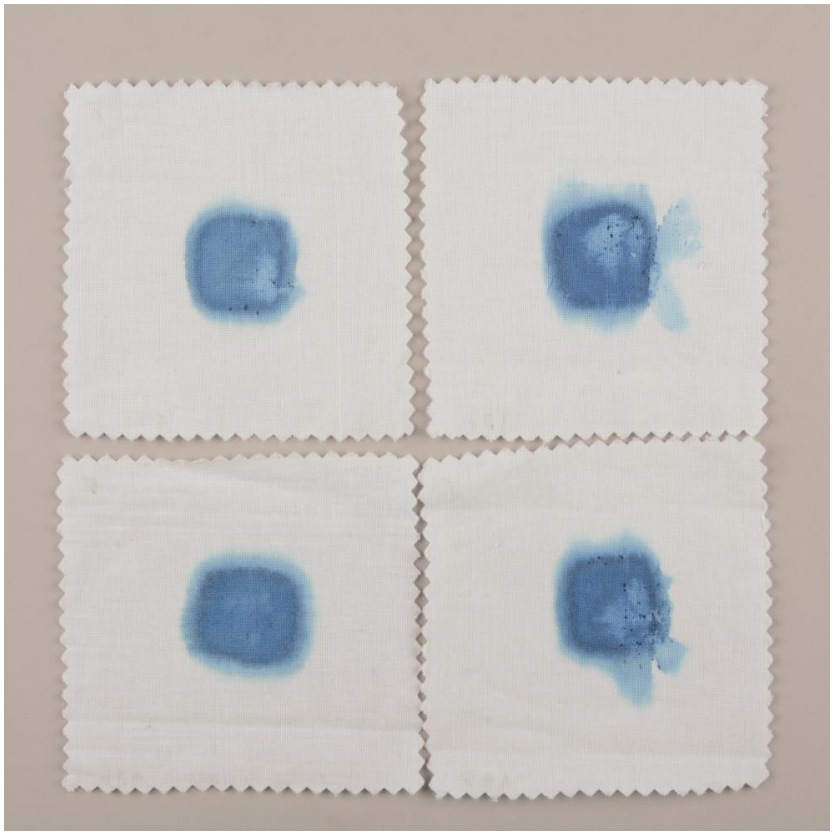
CDD (black) *silk (blue)* *sA4bF1 (navy* *sA4bF2 (green)* *sA4bF3 (red)*



CDD (black) *silk (blue)* *sA4bB1 (navy)* *sA4bB2 (green)* *sA4bB3 (red)*

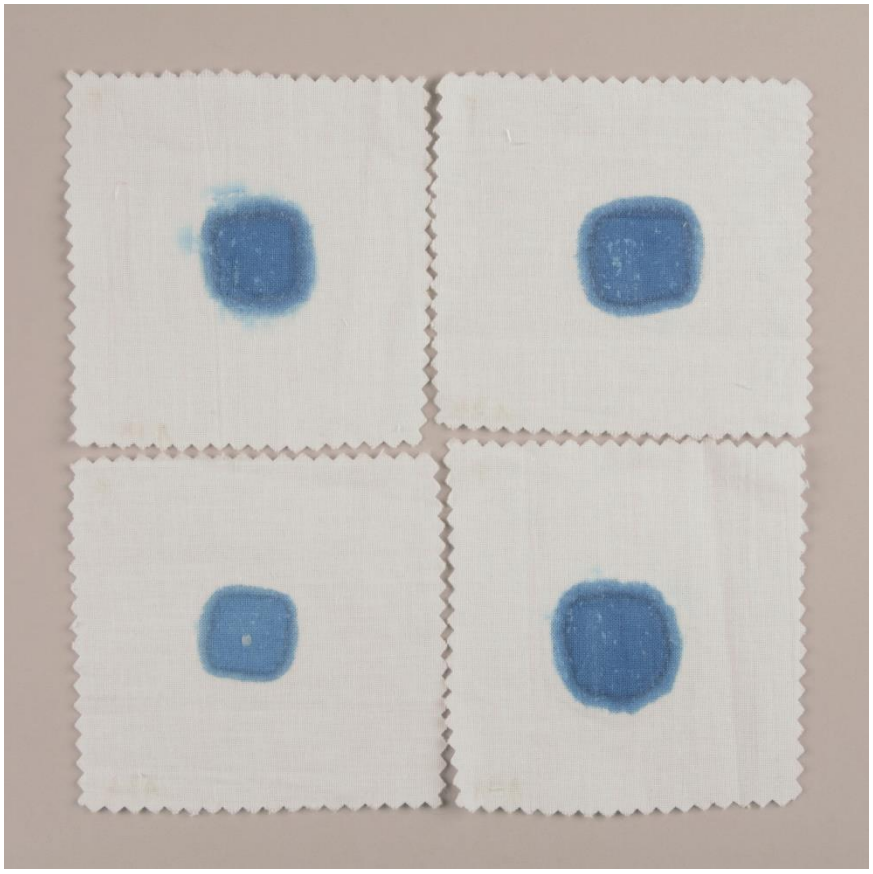
Appendix E: Image Results from Dye Bleed Testing

Appendix E: Image Results from Dye Bleed Testing

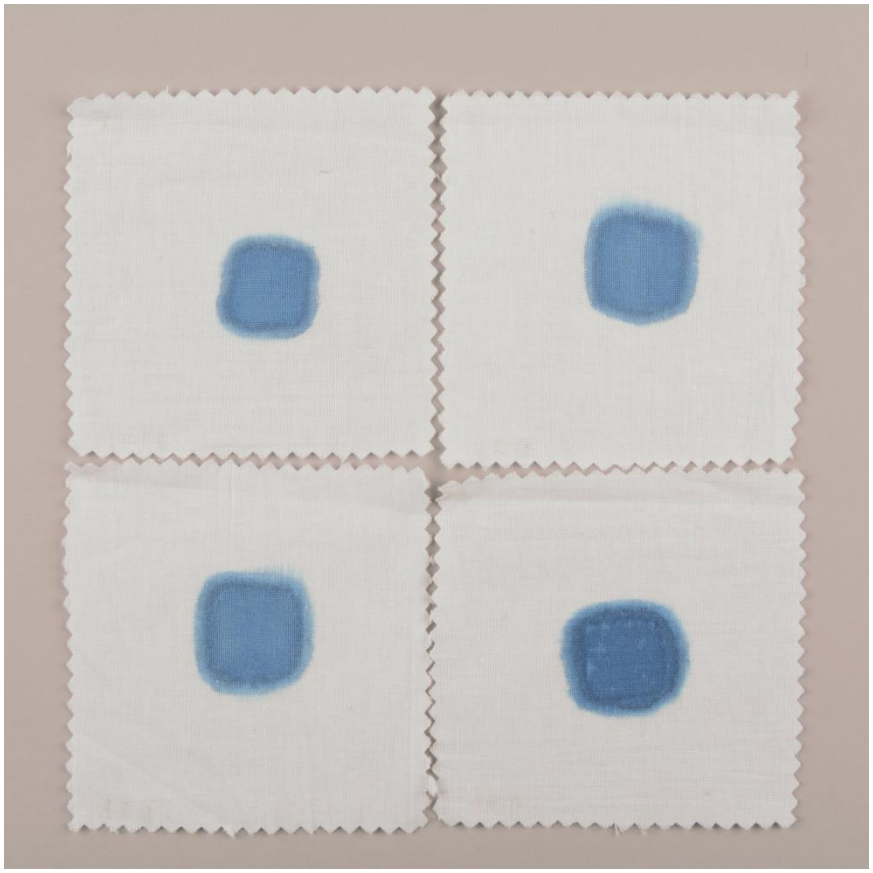


The images in this appendix are of dye bleeding visible on the samples after the dye bleed test. Due to practicality issues, not all of the images could be presented here. Instead, the samples that were of the most interest and use to the experiment are shown here. Such samples were all cotton since there was little variation in any of the silk samples. Two representative examples of silk samples have been included as well.


F
A 1K
F
A 2K
F
A3K
F
A4K

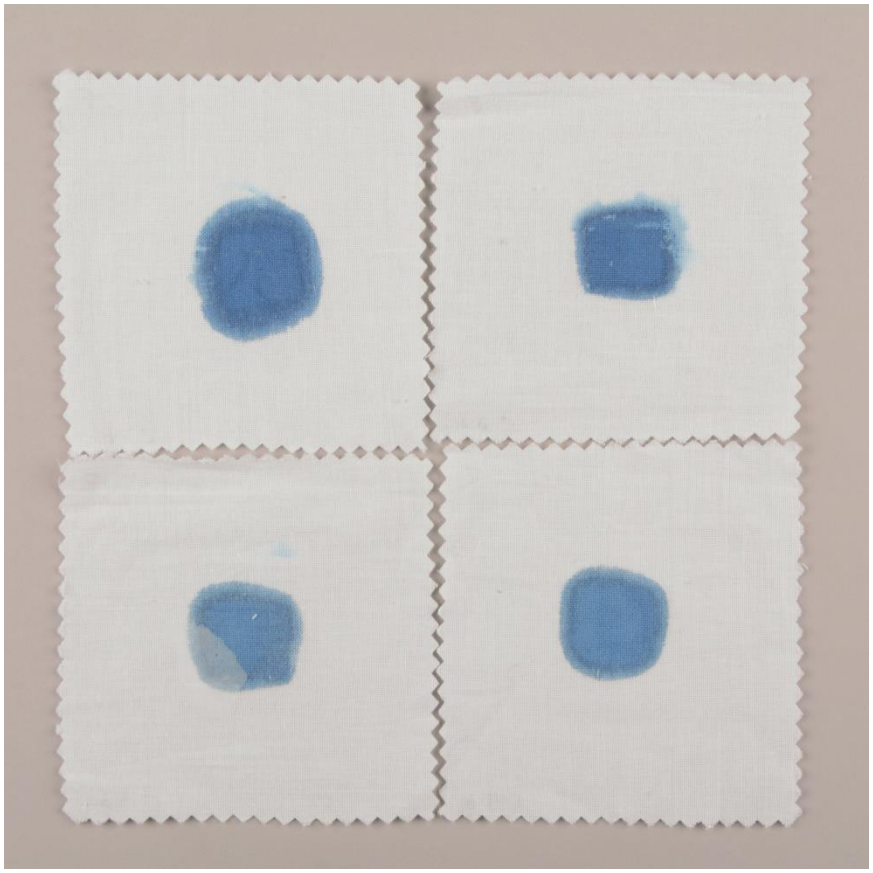



F
B1b
F
B2b
F
B3b
F
B4b

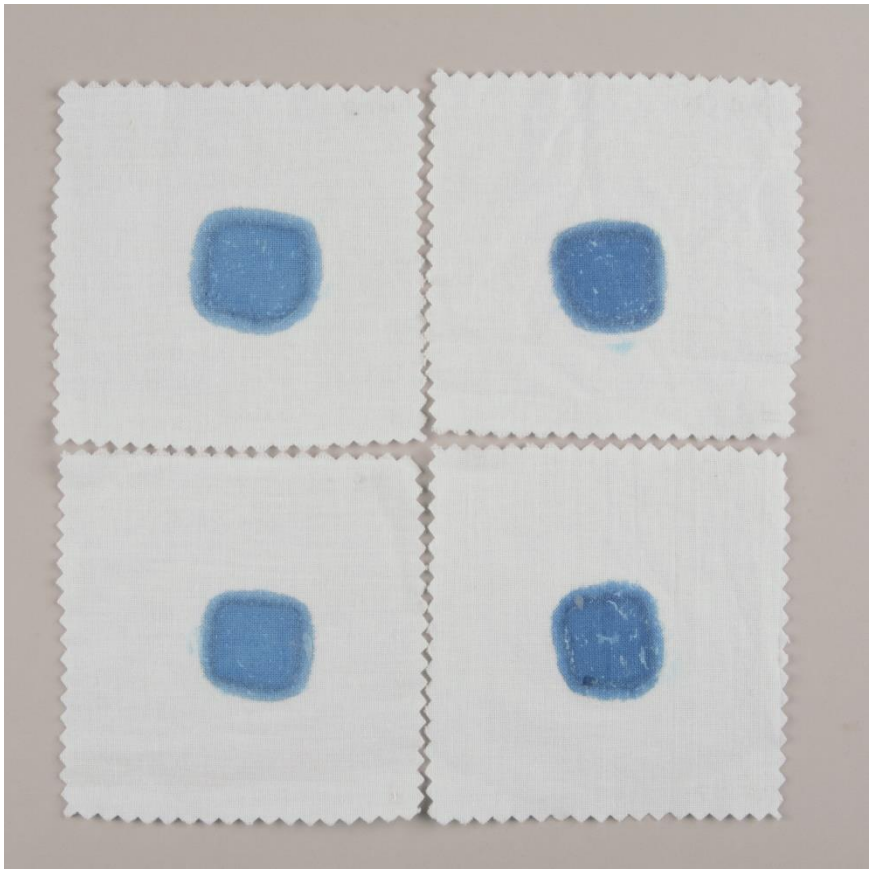



F
F
B14
B24
F
F
B34
B44

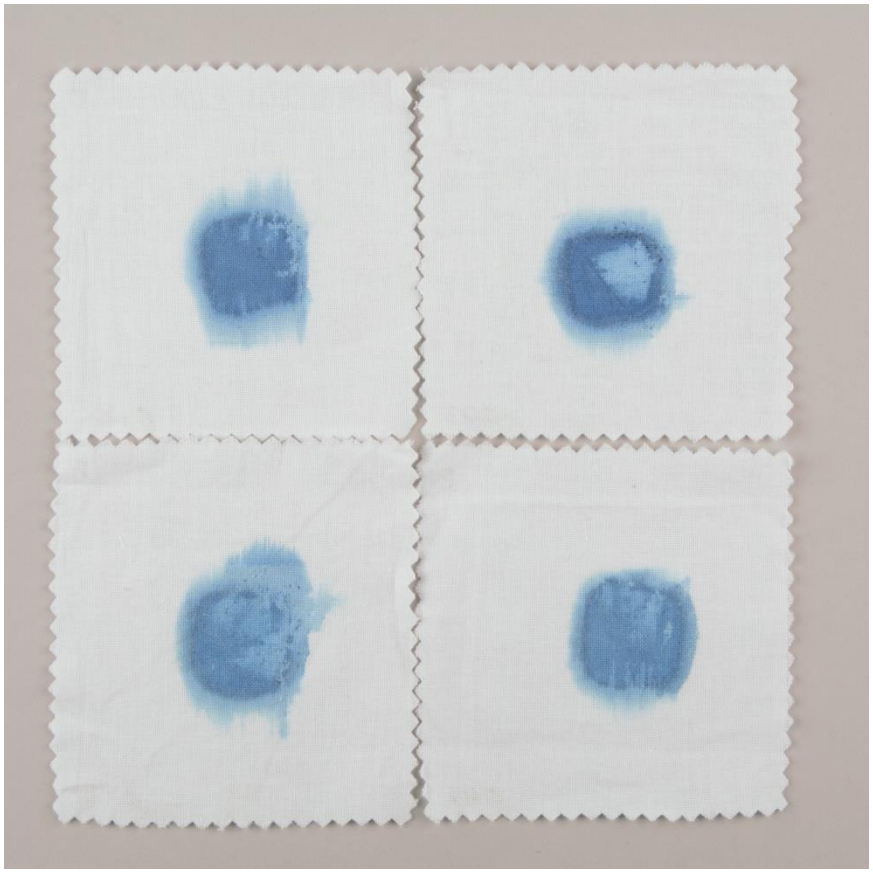

F
C1b
F
C2b
F
C3b
F
C4b



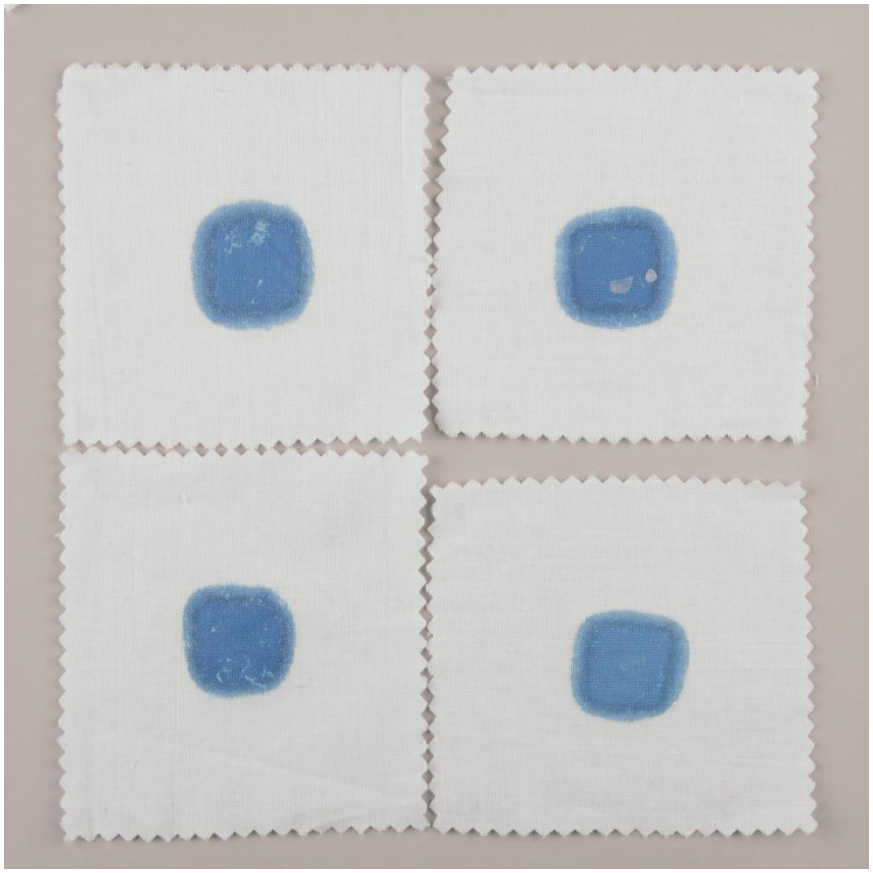

F
C14
F
C24
F
C34
F
C44



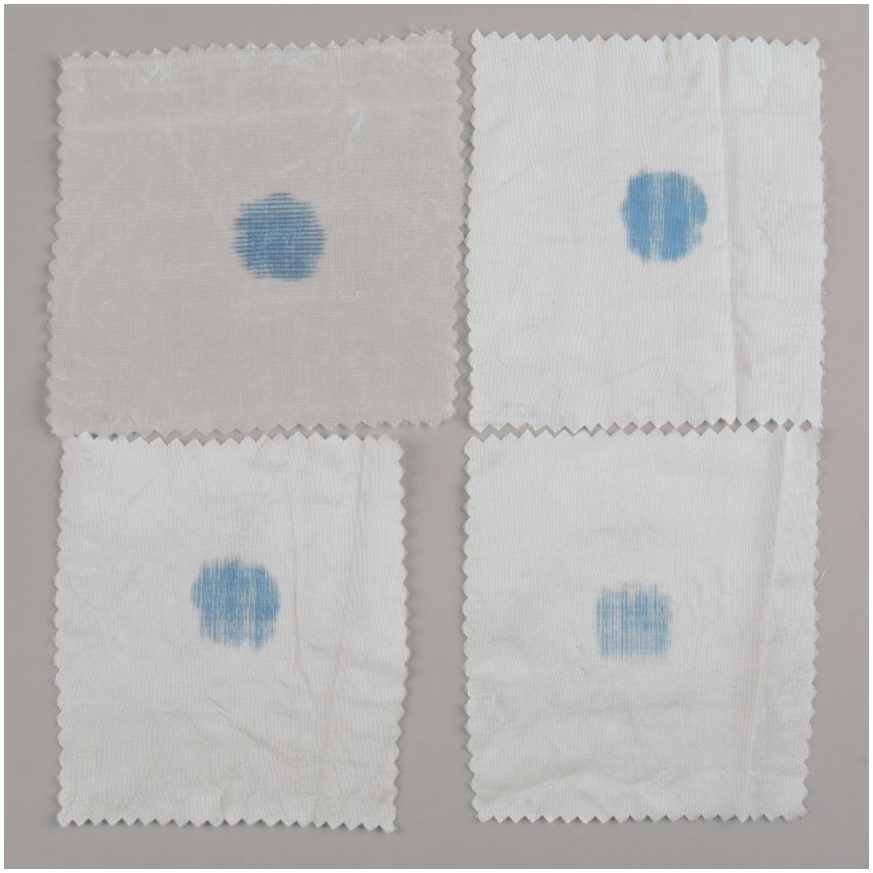

F
G1b
F
G2b
F
G3b
F
G4b

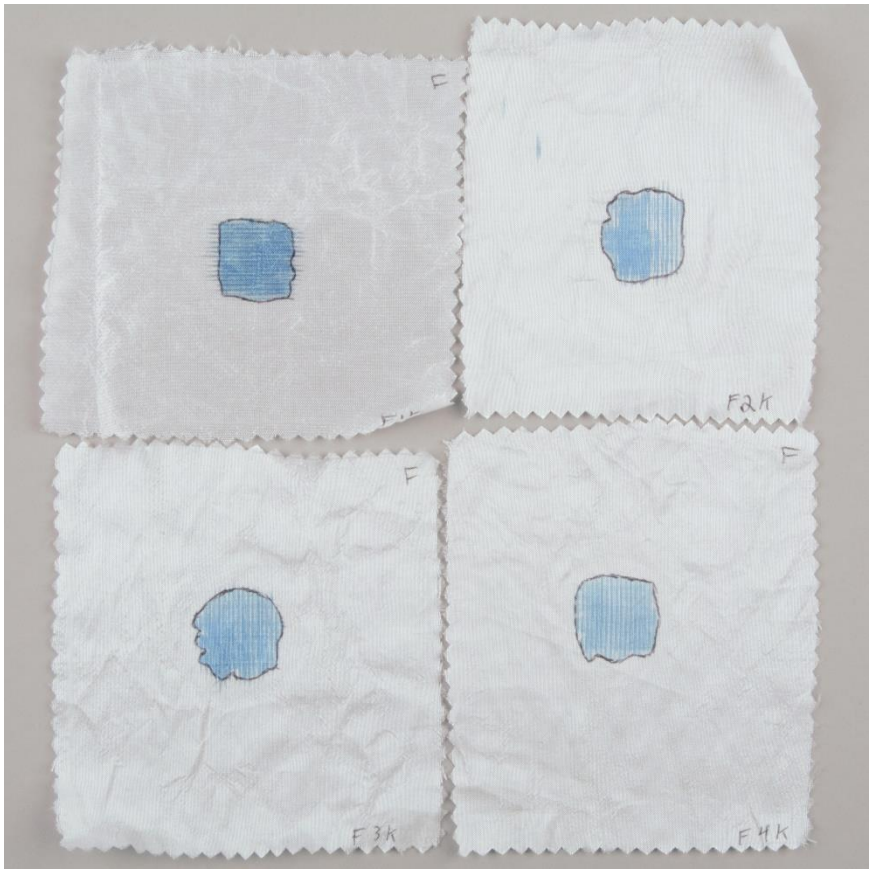


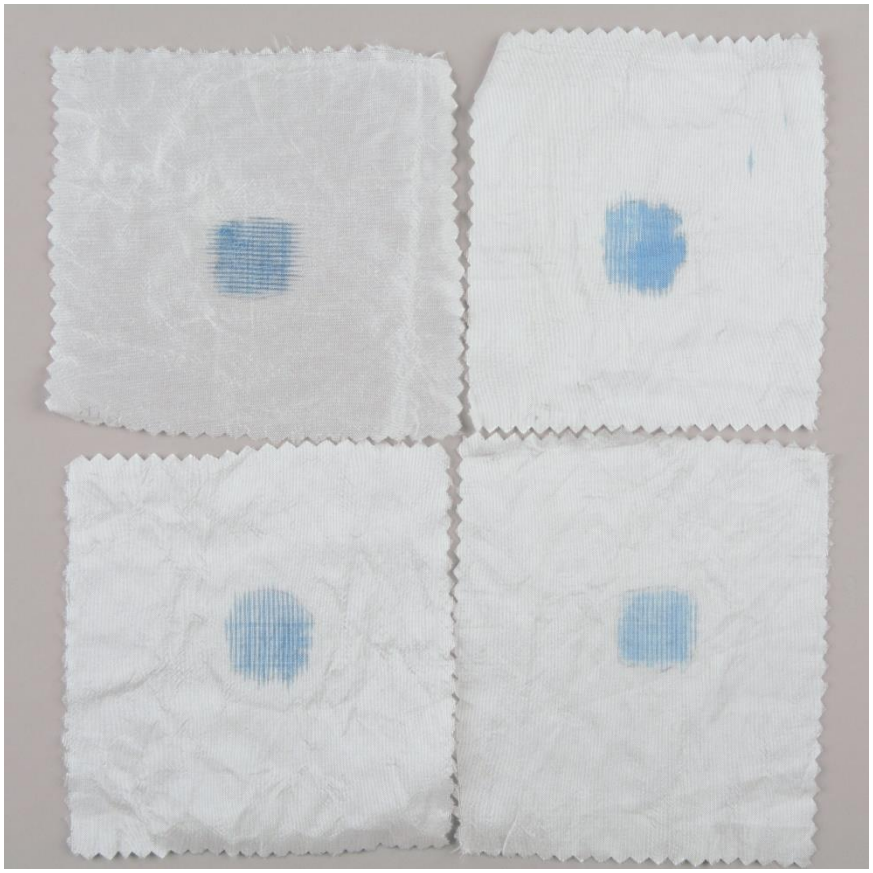

F
F
S1K
S2K
F
F
S3K
S4K




F
A1K
F
A2K
F
A3K
F
A4K




F
F2K
F
F
F3K
F4K



Appendix F: The Samples

(Included only in the original document)