REMOVING DYE BLEED FROM A SAMPLER: NEW METHODS FOR AN OLD PROBLEM

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ABSTRACT – One common problem that textile conservators confront is dye bleed on historic textiles. This paper describes the successful treatment of an important sampler, dating from 1832, in the collection of the Philadelphia Museum of Art. An earlier wet cleaning had caused extensive bleeding from the green and red silk embroidery threads onto the un-dyed wool ground, making the sampler unsuitable for exhibition.

A series of tests with various cleaning solutions and solvents failed to reduce the dye bleed, and bleaching methods were deemed too risky. A chelating solution of ethylenediaminetetraacetic acid (EDTA), brought to pH 8.0 with triethanolamine (TEA), was found to considerably reduce the dye bleed with no discernable damage to the wool fibers. This solution was delivered using an agarose poultice to control exposure and maximize contact time. The embroidery threads were protected from the cleaning solution with layers of cyclododecane (CDD) applied with a modified *kistka* tool. This cleaning system produced a dramatic visual improvement, significantly reducing the dye bleed on the wool ground while protecting the silk embroidery, and allowed the sampler to be exhibited.

RESUMEN – Uno de los problemas más comunes con los que se enfrentan los conservadores es la tintura corrida en las telas históricas. Este documento describe el tratamiento exitoso de un importante muestrario, que data del año 1832, de la colección del Museo de Arte de Filadelfia. El muestrario había sido limpiado en húmedo, lo que hizo que los hilos del bordado de seda verdes y rojos destiñeran sobre la base tejida no teñida, y fuera imposible exhibirlo.

Una serie de pruebas con diferentes soluciones limpiadoras y solventes no lograron reducir el desteñido, y los métodos de blanqueamiento eran demasiado riesgosos. Se encontró que una solución quelante de ácido etilendiaminotetraacético (EDTA), con un pH llevado a 8.0 con trietanolamina (TEA), redujo considerablemente el desteñido sin ningún daño visible en las fibras del tejido. Esta solución se colocó usando un emplasto de agarosa para controlar la exposición y maximizar el tiempo de contacto. Los hilos del bordado se protegieron de la solución limpiadora con capas de ciclododecano (CDD) aplicado con una herramienta *kistka* modificada. Este sistema de limpieza produjo una drástica mejora visual, reduciendo significativamente el desteñido sobre la base tejida y protegiendo el bordado de seda. De este modo, se pudo volver a exhibir el muestrario.

1. INTRODUCTION

The subject of this paper is a Scottish sampler in the collection of the Philadelphia Museum of Art (PMA), made in 1832 by Susanna Gillies Smith (Figure 1). The sampler entered the PMA's collection in 1969 as part of the Whitman Sampler collection, which comprises 392 American and European samplers dating from the mid-17th through the mid-20th centuries. The sampler is made with a plain-woven, un-dyed wool ground and multicolored silk floss embroidery [1] depicting a detailed pastoral scene, along with a central verse, family names, and traditional alphabets. In addition to being quite beautifully executed, this object has a well-established provenance and history. The subject of the embroidered scene, Stobcross House, is thoroughly documented: historic photographic images and written documentation regarding the site and residing families have been found in the Glasgow Digital Library (2012) (Figure 2).

Figure 1: The sampler by Susanna Gillies Smith, overall front, before treatment.

Figure 2: Photographic image of Stobcross House, date unknown, from the Glasgow Digital Library, based at the University of Strathclyde.

With regard to its condition, the problem with this particular sampler was extensive dye bleed from the dark green and red embroidery threads that was so disfiguring that many of the details of the fine embroidery were largely obscured by colored halos on the once neutral ground. The sampler was not considered suitable for display in this state. Reducing the dye bleed presented several challenges: the proteinaceous fibers in both the wool ground and the silk embroidery precluded treatment options such as bleaching, and the finely executed embroidery would be difficult to protect from the chosen cleaning method for the ground fabric. The successful method would require removal of the dye bleed from the ground without damaging the wool fibers, while at the same time preventing further dye bleed from the embroidery.

2. HISTORICAL CONTEXT AND PHYSICAL CONSTRUCTION

The main subject of the sampler is Stobcross House, built in the 18th century within what is now the city of Glasgow, Scotland. In 1745 businessman John Orr bought Stobcross and the associated lands and made several additions to the house. After passing through the Watson family, the house was bought in 1783 by John Phillips, the grandfather of Susanna Gillies Smith. Susanna worked on the sampler in this house in 1832; the date and names of her extended family are included in the design. Many of these extended family members lived at the house with her. The house remained in the Phillips family until 1844, when the family sold the house and the associated land. The house was demolished in the 1870s (Glasgow Digital Library 2012).

In addition to Stobcross House, the sampler design features a central verse: “Make much of Precious time / while in your power / Be careful well to Husband / Every hour / The time will come when you / will sore lament / The useful moments that you / have misspent.” The name “Stob-Cross” is also embroidered just below the verse, and there are several alphabets and numbers separated by decorative borders as well as family names and initials. The finely worked embroidery stitches have many small flourishes and details, and include cross, satin, eyelet, stem, 4-sided, fishbone, back, double-running, and split stitches.

The sampler is in generally fair to good structural condition, with some embroidery loss - mostly in the longer floats where there is significant abrasion and loss. This is particularly noticeable with the missing floats of many flower petals in the border as well as the larger green patches of the pastoral scene. There are also significant losses in several of the letters in the largest and most elaborate alphabet. The wool ground is generally intact and stable with the exception of three small losses near the borders.

3. ANALYSIS OF DYES

Samples of the red and green threads were analyzed using high performance liquid chromatography with photodiode array detection (HPLC-PDA) (Shibayama 2012). Carminic acid was the major component detected in an extract from the red thread, suggesting the use of cochineal. In an extract from the green thread, luteolin, apigenin and several other flavonoid components were detected with a composition that matched well with a reference sample of weld [2]. The green color was achieved by the use of weld in combination with indigo carmine, which was also detected by HPLC-PDA, and its identity supported by Fourier transform infrared microspectroscopy (MFTIR) analysis (Figure 3) [3].

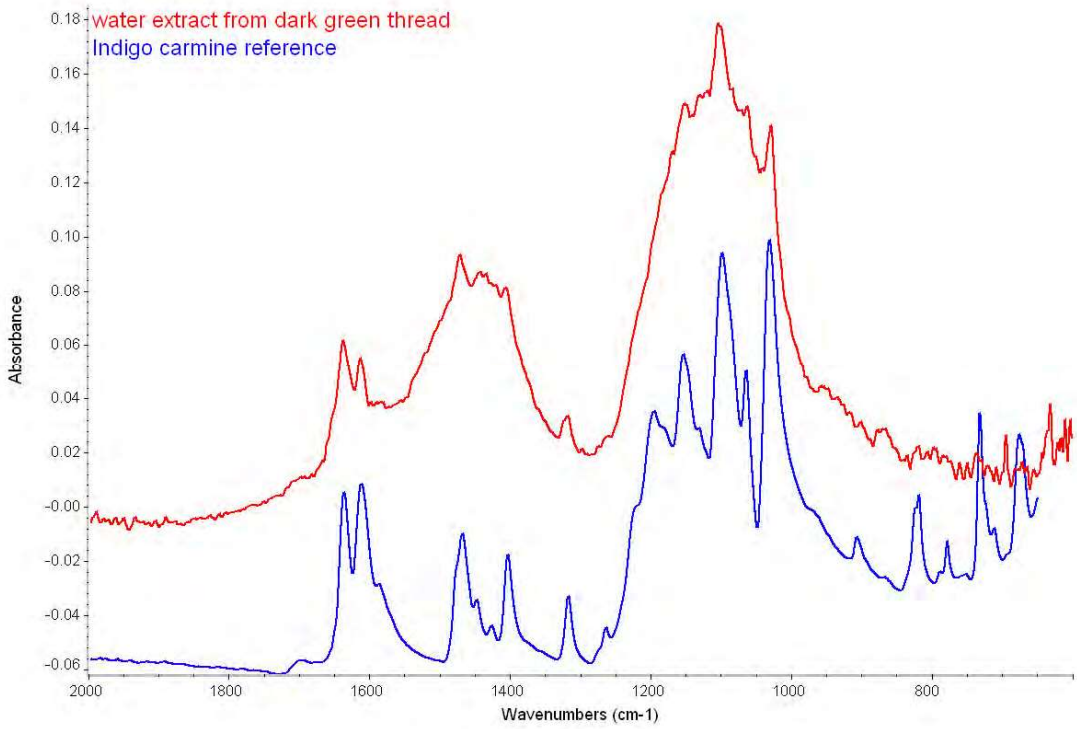


Figure 3: Detail of infrared spectrum of extract from dark green thread (top), showing close correspondence of a series of peaks with those in a reference spectrum for indigo carmine (bottom).

Indigo carmine, also called Saxon blue or indigo extract, is a direct acid dye made by treating powdered indigo with concentrated sulphuric acid. The dyestuff was introduced in England in 1748 and became quite popular due to ease of use over indigo, requiring no mordant although sometimes used with alum or cream of tartar to improve the fastness (Ponting 1980, Hofenk de Graaff 2004). Indigo carmine has low light-fastness and wash-fastness properties (de Keijzer et al 2012) unlike the related indigo vat dye, explaining the extensive bleed of the blue colorant in the sampler.

Cochineal is a dyestuff derived from the dried bodies of female beetles of *Dactylopius coccus* Costa (Hofenk de Graaff 2004), and can produce a range of colors from purple to bright red, depending on the mordant and pH of the dyeing solution. The dye was brought to Europe after the discovery of the Americas and commonly used to attain a bright red hue on wool and silk until newer synthetic aniline dyes, capable of producing a similarly vibrant red hue, were introduced around 1870. Although generally considered a washfast dye, Hofenk de Graaff says about cochineal that "Fastness… to washing is poor." (2004). Additional studies have found that cochineal dye is potentially sensitive to washing under certain conditions of temperature, pH, and surfactant choice (Duff et al 1977). The exact history of this sampler is unknown; however, a combination of problematic processing when the silk floss was originally dyed and/or particular conditions during the sampler's wet cleaning treatment could have provided an optimal situation for release of the red dye.

4. TESTING

The green silk embroidery threads still bled in some areas from contact with moisture, although water did not release the dye on the wool ground. Initial testing proceeded with surfactants and solutions commonly used in conservation, including a solution of 0.5% (w/v) Orvus WA paste in reverse osmosis (RO) water, and several solvents including acetone, Stoddard solvent, denatured ethyl alcohol, and isopropyl alcohol. None of these cleaning agents had any noticeable effect in reducing the dye bleed.

Further treatment testing was informed by the lectures and workshops of Richard Wolbers, Associate Professor at the University of Delaware Department of Art Conservation, as well as consultations with several fellow textile conservators. Testing proceeded with aqueous solutions using several parameters: pH, conductivity, and chelators. Surfactants were not tested further due to their lack of efficacy in initial testing. The following reagents were chosen for the test solutions: triethanolamine (TEA) and ammonium hydroxide were used to raise the pH of a solution, and sodium citrate and disodium ethylenediaminetetraacetic acid (EDTA) were tested as chelators or sequestering agents.

Small samples of the green embroidery thread were removed from the back of the sampler and placed onto glass slides. Each test solution was dropped onto a thread sample. After about 10 minutes, the samples were placed between clean blotters and allowed to dry. Two of the samples, those treated with EDTA and TEA, showed the most significant transfer of dye to the blotters after the testing.

Chelating agents are compounds that have two or more functional groups that can bond with metal atoms to form complexes. The resulting chelate-metal complexes behave quite differently from the individual chelator or metal ions in solution. The pH of a solution can have a significant effect on the sequestering capabilities of chelators as this will affect the ionization of the functional groups that are required to bind the metal ions and form the resulting complex (Adler and Eaton 1995). EDTA is a fairly strong chelating agent for most di- and trivalent metal ions (Timar-Balazsy and Eastop 1998), including calcium, copper, and iron. An aqueous solution of EDTA tends to be slightly acidic, with a pH of about 4.5 for a 0.5% solution in RO water. Under alkaline conditions, the hydroxyl groups will ionize, facilitating the formation of metal complexes. A higher pH also helps the salt to fully dissolve. The EDTA molecule can effectively wrap around many metal ions, taking advantage of all six possible coordination sites to bind the metal ion (Wolbers 2000). In this particular case, the efficacy of a chelator such as EDTA in reducing the dye bleed is likely due to complexes formed with possible mordants based on metals such as aluminum, tin, or iron.

TEA is a strong base and buffers to a range of 6.9-8.3. The proteinaceous materials of the sampler dictated that a pH of 9 was considered the upper limit for safely treating the object without damaging the fibers. Because of the individual qualities of these two agents, a solution combining the two seemed a good starting point for further testing on the dye bleed.

4.1 TREATMENT PARAMETERS AND MATERIAL

Several considerations were important for determining a delivery system for the cleaning solution: the system would ideally allow for prolonged contact with the affected areas, so that the solution would have maximum efficacy; wetting and spreading of the solution, however, would need to be minimized. A barrier would also be necessary to prevent the solution from coming into contact with the silk

embroidery and causing further dye bleed or color shift. Because the sampler had been wet cleaned within its more recent history, as indicated by the extensive dye bleed, tideline formation did not appear to be an issue. This is because any build-up of water-soluble degradation products was likely reduced with this most recent cleaning.

Initially, the delivery system for the cleaning solution was a methyl cellulose poultice made to about the consistency of soft putty, based on the 2009 TSG tip presented by textile conservator Maya Naunton (Figure 4, Figure 5). Although this method was successful, there were several limitations, most importantly the extremely long contact time required for successful dye bleed reduction in the wool ground, as well as the difficulty of fully clearing the poultice from the ground fabric. A more successful delivery system was agarose gel, which achieved the desired dye bleed reduction with application to the sampler for a much shorter period of time. In addition, the rigid gel structure of agarose meant that there was less likelihood of poultice residues being left on the fibers. Materials and methods used for the treatment are described below.

Figure 4 (left): Detail of proper right border area before initial testing with cleaning solution in a methyl cellulose poultice. Figure 5 (right): Detail of proper right border area after initial testing with cleaning solution in a methyl cellulose poultice.

Barrier

Embroidery threads were protected with an application of cyclododecane (CDD), a waxy but volatile compound commonly used in conservation applications as a consolidant or binder, which sublimes directly from a solid to a gas phase at atmospheric pressure. CDD is a non-polar cyclic hydrocarbon ($C_{12}H_{24}$) with a melting point of 58-61˚C and is soluble in non-polar solvents (Scharff and Nielsen 2000, Larochette 2004).

Kistka application

The application of CDD was complicated by the intricate embroidery on the sampler. High levels of precision and accuracy were needed to coat the silk threads without covering the ground fabric. A *kistka* was used to apply the CDD. This hand-held tool was developed to decorate Easter eggs with wax resist designs. While a variety of tip sizes are available for kistkas, even the extra-fine tip had too large of an aperture for the delicate embroidery. For this project, a modification was made based on the schematic in the paper by Brückle et al (1999): a hypodermic needle with beveled tip was inserted into the aperture

of a medium kistka tip. To create a large reservoir for CDD, the kistka tip was fitted with a small metal funnel manufactured for decorating cakes with icing (Figure 6).

Figure 6: The modified kistka tool used for cyclododecane application.

Agarose

Agarose is a natural polysaccharide that has many scientific uses, especially in DNA electrophoresis. It is derived from several species of red marine algae that are processed to extract agar, also known as agar-agar. Agar is composed of the polysaccharides agarose and agaropectin. In gel form, the porous lattice structure of agarose facilitates a slow diffusion of liquids via capillary action (Araki 1956).

Agarose is well-suited for use as a poultice material for textile conservation. It can support a variety of solutions including enzymes. To make the gel, dry agarose powder is added to the aqueous solution, brought to a boil, and then poured into a container to cool. It is nonionic and will accommodate a pH range of 4.5-10 (Warda et al 2007). The gel is clear, which allows for continuous observation during treatment.

There are two properties of agarose that can be easily altered to suit particular applications: gel density and thickness. A gel with a higher percentage of agarose will have smaller pores and thus slower diffusion than a gel with a low percentage of agarose. A 4% gel was useful for initial tests, because the impact on the object is slower and more easily controlled. For the treatment of the sampler, a 1% gel was used to speed the diffusion process. The thickness of the gel is related to its flexibility and drying time. For the sampler treatment, the gel was cast to ~0.5cm thick. The gel was supple and could be draped over portions of embroidery (coated with CDD) and still maintain contact with the ground fabric (Figure 7).

Figure 7: Gel poultice made with the cleaning solution shown in place on the sampler.

5. TREATMENT

5.1 PROCESS

Agarose gel recipe:
1% w/v EDTA in RO water
1% w/v agarose in solution
TEA – sufficient to bring solution pH to ~8.0

1. Add EDTA to RO water and mix thoroughly
2. Bring solution pH to ~8.0 by adding TEA dropwise
3. Add agarose and heat until boiling – solution will become clear
4. Pour into container and allow to cool (a shallow tray or petri dish is well suited for this). After the gel has cooled, it should be covered to prevent drying.

5.2 APPLICATION

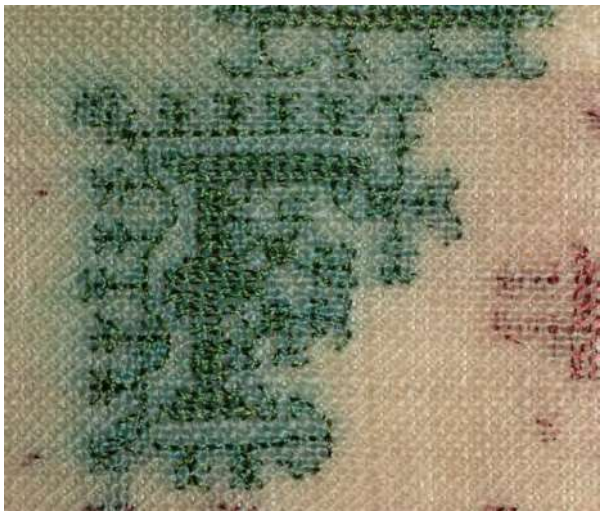
1. Select a small area to treat – up to a few square inches. For your safety, use an elephant trunk, or other source of ventilation, while applying CDD. On the front side of the sampler, cover all embroidery threads with 2 coats of CDD. Create a perimeter of 2 coats of CDD around the area to be treated. Turn sampler to reverse side and repeat application of CDD (with a kistka tip, the application motions are more patting and sweeping; with the needle tip, the application motions are more like tattooing).
2. Position sampler face down on suction disk so that rinsing can begin as soon as gel is removed.
3. Cut a piece of gel to fit within the CDD perimeter. If the gel was cast in a clear container (such as a petri dish), the container can be placed on the sampler so that the desired shape of gel can be accurately cut. Alternately, transfer the gel to a piece of Mylar placed over the treatment area, and cut the gel to the needed shape. Remove the piece of gel with tweezers and place on the sampler. Use the back end of the tweezers to gently press the gel into place. It may be helpful to

use the back end of the tweezers to gently break up the gel to allow it to have greater contact with the textile. Leave the gel in place for about an hour. Watch the gel to make sure that none of the solution moves beyond the CDD perimeter. If this happens: remove the gel, rinse area with RO water and dry using low suction, reinforce CDD perimeter, and replace gel.

4. After the dye has moved from the sampler to the gel, remove the gel with tweezers. Use a piece of blotter to remove more dye bleed before turning on the suction. Turn the suction disk on to a low setting and rinse and dry the treated area with RO water (~30-50mL). Continue to use blotter paper along with suction. NOTE: this rinse is to flush the EDTA/TEA solution from the sampler.
5. Check the CDD perimeter and coating on the back embroidery threads. Add more CDD if needed. Turn sampler to be face up on the suction disk. Check the CDD perimeter and coating on the front embroidery threads. Add more CDD if needed.
6. Cut a new piece of gel to fit within the CDD perimeter. Place this new gel on the front side of the sampler and leave in place for about 15 minutes. Watch the gel to make sure that none of the solution moves beyond the CDD perimeter.
7. After the dye has moved from the sampler to the gel, remove the gel with tweezers. Use a piece of blotter to remove more dye bleed before turning on the suction. Turn the suction disk on to a low setting and rinse and dry the treated area with RO water (~30-50mL). Continue to use blotter paper along with suction. Dry the area completely on the suction disk.
8. If possible, leave the sampler uncovered near a low fan or elephant trunk to speed the sublimation of the CDD. To expedite the sublimation, the piece can be left on a suction table on a very low setting.

6. DISCUSSION

The treatment protocol using EDTA and TEA as a cleaning solution in an agarose gel poultice effectively removed the dye bleed on the sampler ground (Figure 8, Figure 9). After treatment, many of the sampler designs and motifs are much more readable and there is a dramatic improvement in the appearance of many of the small embellishments and flourishes within the design (Figure 10). This treatment system is one that can be applied to many other situations, and the materials are considered non-hazardous and can be used without elaborate fume extraction systems. Agarose gel can be used with a variety of aqueous cleaning solutions and is therefore adaptable to many different cleaning situations.



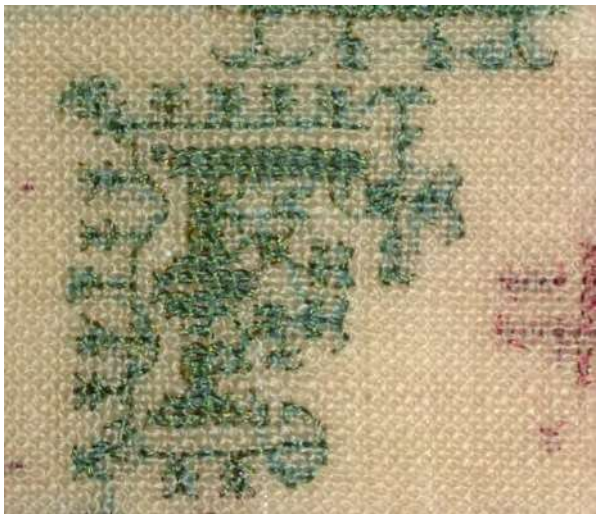


Figure 8 (left): Detail of the letter “F” before cleaning with the gel poultice. Figure 9 (right): Detail of the letter “F” after cleaning with the gel poultice.

Figure 10: The sampler by Susanna Gillies Smith, overall front, after treatment.

NOTES

1. The wool and silk were identified by visual examination and polarized light microscopy.

2. For HPLC-PDA analysis, carried out at the Metropolitan Museum of Art, the red thread was extracted in a 6:4 (v/v) mixture of 1N aqueous hydrochloric acid: methanol. The green thread was extracted in a 6:4 (v/v) mixture of 0.001M aqueous EDTA: methanol. The analyses were performed using a Waters Corporation HPLC system (1525µ binary HPLC pump, 2996 PDA detector, 1500 series column heater, in-line degasser) equipped with a Rheodyne 7725i manual injector with 20 µl loop and Waters Xterra RP_{18} reverse-phase column. The mobile phase was a gradient system of formic acid (0.88% in deionized water) and methanol, with linear gradients from 90% to 12% formic acid (v/v). Components were identified on the basis of chromatographic retention times and UV-visible spectra, in comparison to data from standards and dyed fabric references. Full analytical details are on file in the Scientific Research and Analysis Laboratory of the PMA.

3. For MFTIR analysis, carried out at the PMA, the dried residue of an aqueous extract from the green thread was mounted on a Spectra-Tech diamond window. The IR data were collected in transmission mode between 4000 and 600 cm^{-1} at 4 cm^{-1} resolution and 200 scans per spectrum using a Thermo Nicolet Continuum microscope with MCT-A detector, attached to a Nexus 670 spectrometer bench, and processed using Happ-Genzel apodization. The sample spectra exhibited a series of distinctive, sharp

bands in the 1700-600 cm^{-1} region that matched closely with those in a reference spectrum for indigo carmine.

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SOURCES OF MATERIALS

Electric Kistka with interchangeable tips
Polish Art Center
www.polartcenter.com

Hypodermic needle, size 27 G ½
CVS Pharmacy
www.cvs.com

Icing tip, size 5 round
Fante's Kitchen Shop
http://www.fantes.com/

Agarose, molecular biology grade
Benchmark Scientific
http://benchmarkscientific.com/

Cyclododecane
Kremer Pigment
http://kremerpigments.com/shopus/index.php?lang=ENG

disodium EDTA, Ethylenediaminetetraacetic acid disodium salt dehydrate
Alfa Aesar
http://www.alfa.com/en/go160w.pgm?srchtyp=coa

TEA, Triethanolamine-99%
Conservation Support Systems
http://www.conservationsupportsystems.com/product/all-products

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