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► **To cite this version:**

Marie Messenger, Véronique Rouchon. Damaged Blue Papers: Optimising Consolidation while Preserving Original Colour. *Journal of Paper Conservation*, 2013, 14 (2). hal-01439838

HAL Id: hal-01439838

<https://hal.science/hal-01439838>

Submitted on 18 Jan 2017

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Damaged Blue Papers

Optimising Consolidation while Preserving Original Colour*

This article presents the case of blue paper in a sample book that once belonged to Victor Poterlet, a French wallpaper designer operating in Paris in the 19th century. Prussian blue is the main colourant used to dye the paper. The condition of the paper is very poor, probably due to the presence of free iron ions. However the paper could not be replaced or chemically treated because it supports 954 samples, which could not be dismantled due to time considerations. Moreover the paper is indubitably of high aesthetic value and an inherent part of the original. This study was carried out in order to optimise the reinforcement of the blue paper through local treatments, such as resizing and/or lining. Tests

were also undertaken to evaluate improvements in mechanical properties induced by these treatments. In addition it was found necessary to estimate any colour changes that could result from the application of an adhesive to a deep blue paper. Several adhesives were evaluated: Tylose, Klucel, gelatine, starch, Funori, Lascaux 498HV and Lascaux 360HV. The best results were obtained with Tylose. Finally, the book was consolidated with linings that were applied by reactivating the adhesive. This method offered a satisfactorily compromise between the best reinforcement and the least colour change.

Blue-Coloured Papers

The manufacture of Western blue papers emerged in Italy in the middle of the 15th century (Brückle 1993, Brückle 2003, Charbey 1989). This type of paper was initially the result of blue rag residues remaining in the pulp because the light-bleaching methods used in that period were ineffective for removing indigo (Piette 1863). Blue rags could additionally be separated from other rags and used specifically for making blue-coloured paper. Initially an economical response to rag wastage, blue-coloured paper [1] rapidly gained a distinct aesthetic value and was used in various ways.

Blue-coloured paper was commonly used for drawings for its aesthetic value but more rarely for printed books and engravings. The example of Pierre-Jean Mariette (1694-1774), a French collector who systematically mounted his collection in blue frames, illustrates the fascination these appealing papers could hold. Yet the occurrence of blue paper is not restricted to fine arts. This paper was also appropriate for the manufacture of functional objects such as temporary bookbindings. In addition, they were used largely as wrapping material to protect goods during transport. In the late 18th century, blue-coloured papers were commonly made by adding colour. The development of trade in the 19th century greatly boosted the production of blue-coloured paper, which became a real industry. At that time, blue-coloured recycled paper was developed to overcome the shortage of raw material, and, among other considerations, blue was used to mask the grey appearance of the sheet. Thus in the 19th century the blue colour was often associated, rightly or wrongly, with the low quality of recycled paper.

Blue-coloured paper was achieved with pigments and/or dyes that were usually added directly to the pulp. Until the end of the 18th century, natural substances were mostly used (indigo, woad, logwood, etc.). Innovations in colouring materials began at the end of the 18th century with the introduction of synthetic pigments, such as Prussian blue. In the 19th century, many new pigments and dyes were discovered and transferred within a few decades to the industrialization of the paper making process.

The compounds producing the blue colour of paper in the

latter half of the 19th century are impossible to identify without the help of analytical tools. Several pigments/dyes could have been used: Prussian blue, Guimet ultramarine, smalt, indigo, woad, logwood, cochineal, litmus, aniline dyes, etc. Moreover, these compounds were mixed to obtain the desired hue. As there were several ways to produce the same colour (Piette 1863), choices were made depending on availability, cost, colourant strength, easy of blending with the pulp, etc. The final use of the paper was sometimes an additional determinant aspect in the choice of blue: for instance, when blue-coloured paper was designed for wrapping sugar blocks, there was a choice of two colours: violet-blue for export sales and deep blue for national sales (Figuier 1873: 296).

Blue-coloured paper is quite numerous in archival collections. In sample book collections for example, several types of blue-coloured paper can be found. Support of the samples was one consideration, but it was generally chosen for aesthetic reasons because the blue colour nicely highlighted the samples (Fig 1). These sample books also show a large variety of blues, from purple-blue to light blue, which were obtained by mass tinting or surface coating.



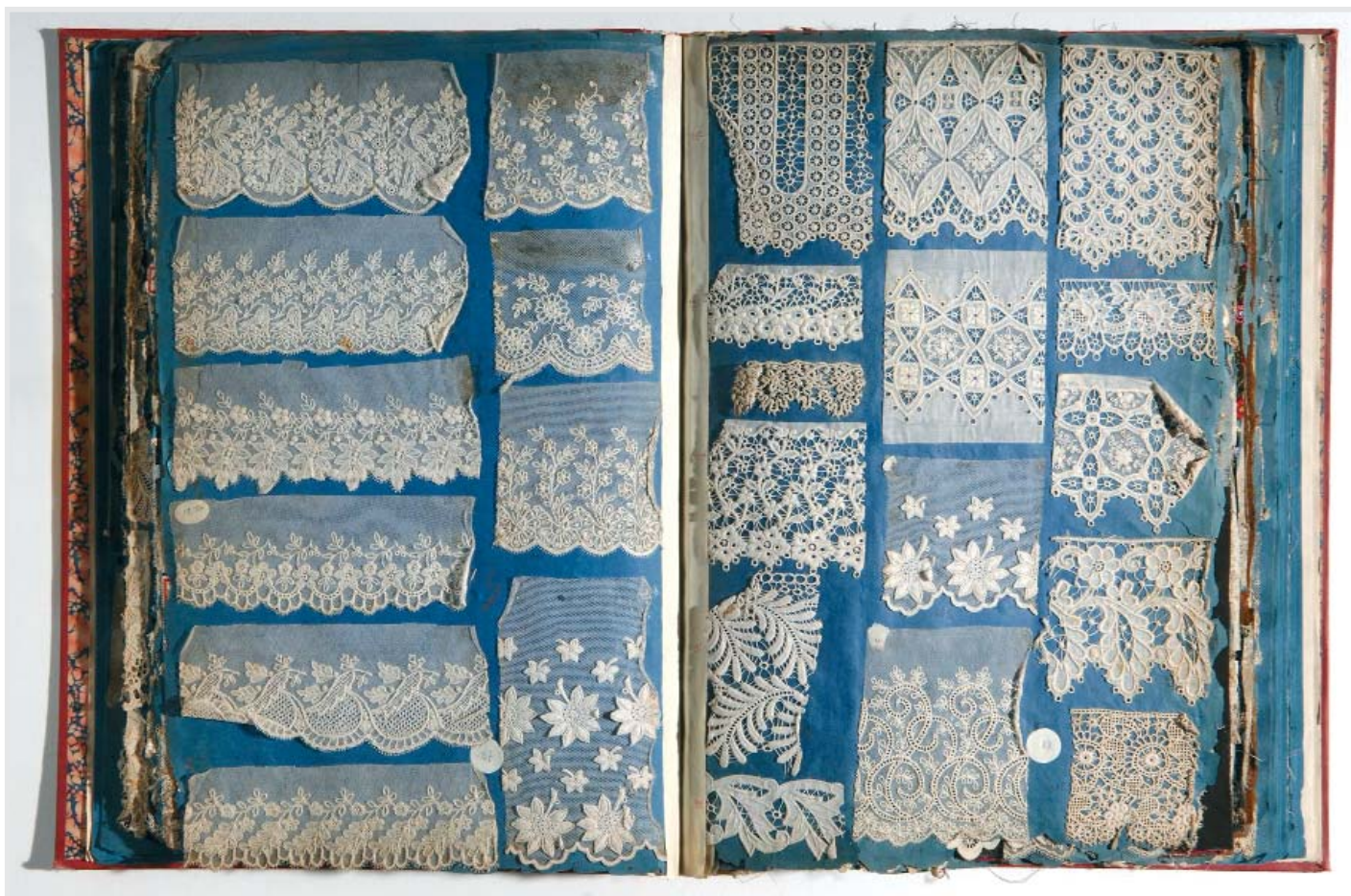
1 Example of a blue-coloured paper used to highlight textile samples (Victor Poterlet sample book). © INP/G. Vanneste.

The Case of the Victor Poterlet Sample Book

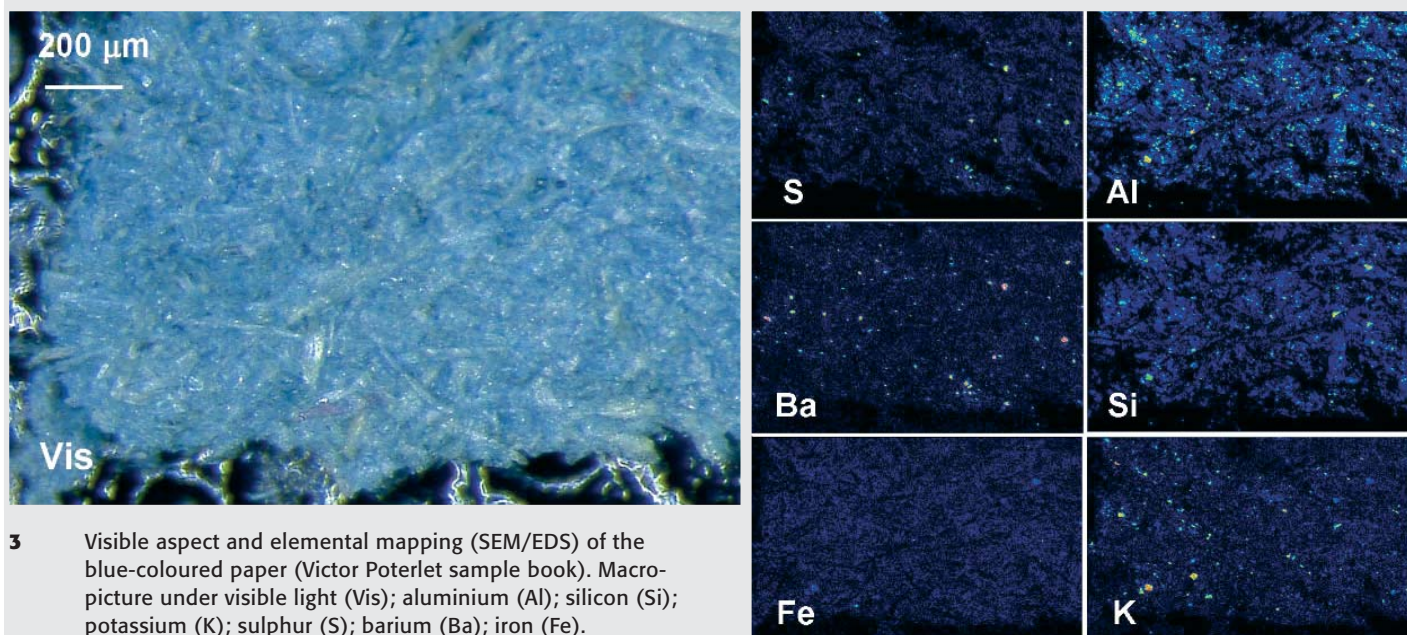
The following case study is based on the conservation treatment carried out on sample book nr. 1162spl from the Forney Library, Paris. It was the property of Victor Poterlet (1811-1889), a famous French wallpaper designer and was sold to the Forney Library by his widow on 26th July 1889. The sample book dates

from the second half of the 19th century and is unique in the Victor Poterlet collection. There is no evidence to suggest how it was used. It might have been for design inspiration but no direct correlation could be established between Victor Poterlet's designs and the collected samples.

The Victor Poterlet sample book comprises 56 dark blue wove-paper pages (51 x 36 cm) supporting 954 samples, which



2 Overall view of Victor Poterlet sample book before conservation treatment. © INP/G. Vanneste.



3 Visible aspect and elemental mapping (SEM/EDS) of the blue-coloured paper (Victor Poterlet sample book). Macro-picture under visible light (Vis); aluminium (Al); silicon (Si); potassium (K); sulphur (S); barium (Ba); iron (Fe).

Tab 1 Blue paper characterisation (GC/MS = Gas Chromatography/Mass Spectrometry; SEM/EDS = Scanning Electron Microscopy coupled with Energy Dispersive Spectrometry).

	Tests/Methods	Results/Interpretation
Weight, thickness	Micrometer, balance	0.2 mm, ~140 g·m ⁻²
Affinity toward liquid water	Time necessary for a 0.1 µL drop of water to penetrate into the sheet. Average of 10 measurements performed in an air-conditioned room (23°C, 50% RH)	1.5 ± 0.5 minute
pH	Cold aqueous extracts (TAPPI T509: 2011) Average of 3 measurements	3.8 ± 0.5
Fibres	Herzberg and Lofton-Meritt reagents	Cotton, linen, alpha, coloured wool fibres, straw and a few wood fibres
Sizing	GC/MS	Natural resins (sandarac and rosin)
Blue pigment/dye	SEM/EDS	Presence of iron throughout
	Fourier Transform Infrared spectrometry	Presence of Prussian blue
Free iron II	Bathophenanthroline test paper (iron II test: Conservation by Design) on the edge and in the middle of the sheets	Highly positive on the margins (damaged areas); slightly positive in the middle of the sheets
Other inorganic products	SEM/EDS	Aluminum and potassium silicate (filler) Barium sulphate
Other organic products	GC/MS	Gum Arabic Proteins

are mostly machine or hand-made embroideries, laces and cut papers (Fig 2). The sheets are collected in a tab binding definitely dating from the 1960s even though all other materials are original. This binding gave rise to several conservation issues and the solutions found have been described elsewhere (; Messager and Aubry 2013; Messager et al. 2012). This article focuses specifically on the paper itself and the research done to improve its condition.

Characterization of the Paper

Several tests were carried out to further characterize the paper (Tab 1). No blue coating layer, blue fibres, or blue pigment particles could be seen under the microscope (Fig 3). Therefore, the pulp must have been coloured with a blue dye or a pigment with small particle size. It has a mixed composition, which is typical of recycled pulps: wool, wood fibres, straw, and a significant amount of Esparto fibres (alpha), a fibre introduced in the 1850s for the manufacture of low quality papers. Gas Chromatography/Mass Spectrometry (GC/MS) measurements (CPG 3800, SM Saturn 2000: Varian; electronic impact 70 eV, ion trap dispersive system 200°C) showed that the paper is sized with rosin. All these characteristics are consistent with the paper being made in the second half of the 19th century.

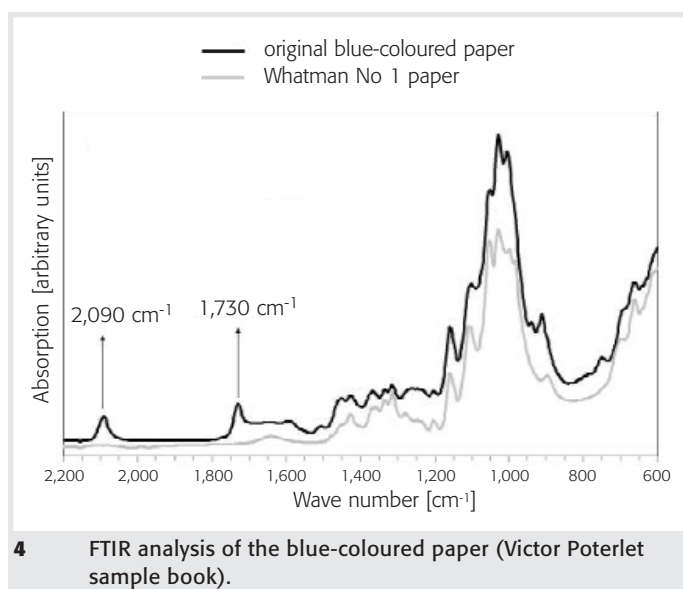
The presence of gum arabic and proteins was also indicated by GC/MS, but these two products are probably not from the paper itself. They are more from the glues used to attach the samples.

The presence of Prussian blue (PB) in the paper colour was proved by Fourier Transform Infrared spectrometry (Diamond ATR macro system: Thermo Scientific). A significant infrared absorption was observed at 2,090 cm⁻¹ (Fig 4), which corresponds to the CN stretching mode of cyanide groups. This feature is characteristic of the presence of PB (Kuckova et al. 2012; Pitarch et al. 2012; Christensen et al. 1990). Two kinds of PB have been

proposed in the literature: Fe^{III}₄[Fe^{II}(CN)₆]₃, which is called 'insoluble PB', and KFe^{III}[Fe^{II}(CN)₆], which is called 'soluble PB'. It has been suggested that these terms refer to ease of peptisation rather than solubility as both kinds can form colloidal dispersions (Liu et al. 2010).

Elemental mappings were performed with Scanning Electron Microscopy (JSM 5410 LV: Jeol) coupled to Energy Dispersive X-Ray Spectrometry (SI[Li] Links PentaFET: Oxford Instruments). They show a quite even distribution of iron (Fig 3), suggesting that Prussian blue is present as sub-micrometric particles. A large quantity of small particles rich in aluminium, potassium and silicon were also detected, indicating that clay was used as a filler.

Many particles rich in sulphur and barium were detected by SEM/EDS (Fig 3) and attributed to barium sulphate, which could be used as a filler for PB pigments. Indeed in the late 19th cen-



4 FTIR analysis of the blue-coloured paper (Victor Poterlet sample book).

tury, PB, which has a high colour strength, was commonly diluted by the addition of fillers (Perego 2005; Halphen 1895). This practice gave rise to several names for PB based pigments that were sold to paper manufacturers: in France for instance, PB mixed with barium sulphate only was called 'bleu Charron'; PB mixed with barium sulphate and kaolin was called 'bleu mineral', etc. (Coffignier 1908). Thus the detected clay was possibly used to fill out the PB.

The presence of a PB-based pigment here was proven, but further investigations would appear necessary to assess the exact composition of this pigment. Moreover, the additional use of a dye cannot be excluded. Some authors (Piette 1863) mention that Prussian blue was often mixed with indigo, logwood or Brazil wood, which may be the case here.

A Paper in Poor Condition

The paper, originally a deep blue colour, was in poor condition. The margins were locally discoloured toward a greenish hue. The paper was torn and often fragmented (Fig 5) near the margins but also in the middle of the sheet. Thus numerous small parts were missing. These damages are attributable to handling, the weight and the three-dimensionality of the textile samples, as well as the paper itself, which initially looked attractive but is of poor quality for long-term preservation.

PB is a pigment rich in iron, which may release free iron into paper when it deteriorates. The ability of PB to participate in Fenton reactions has already been pointed out (Liu et al. 2010). Spot tests with bathophenanthroline-impregnated papers (iron-gall ink test paper: Conservation by design) confirmed the substantial presence of free iron in the paper. Additionally, some pH measurements were made using cold aqueous extracts (TAPPI T509 om-11, 2011) on paper samples of a few micrograms. This showed that the blue-coloured paper is significantly acidic, with a pH value of 3.8 ± 0.5 . Finally, FTIR measurements (Fig 4) showed a strong absorption at $1,730 \text{ cm}^{-1}$ characteristic of the C=O stretching mode, thus indicating that the blue-coloured paper is highly oxidised.

The presence of iron in the paper sheets, together with an acidic environment, is quite similar to the cases of iron-gall ink damaged paper that becomes brown and brittle with ageing.



5 Damaged paper edges of the Victor Poterlet sample book (detail). © INP/G. Vanneste.

This damage is attributed to the combination of two degradation phenomena: acid hydrolysis caused by the low pH of the ink and oxidative depolymerisation caused by the presence of free iron. The question of whether oxidation or acidic hydrolysis is the dominant damaging mechanism is quite open and currently under debate (Rouchon et al. 2011; Kolar et al. 2006). Nevertheless, the fact remains that the degradation of the Victor Poterlet sample book paper is quite similar to that of an iron-gall ink manuscript on which the ink has migrated deep into the paper.

Aim of the Treatment

Replacing the blue paper with new was considered to be inappropriate as the book would have lost part of its original material and historical value. It was therefore decided to retain the original paper even though it was acidic and badly damaged. Initially, chemical treatments were investigated for stabilizing the paper, but all pages would then have to be treated and taking apart the 954 samples was too time-consuming. This option rapidly appeared unrealistic.

The focus changed to a less ambitious objective, i.e., finding an optimal local consolidation treatment. This was a challenging aim because

- > the unity of the paper pages and the book character should be maintained,
- > applications of adhesive to dark coloured paper often produces undesirable stains, and
- > the consolidation should be robust as the book would remain available for consultation.

Several adhesives were applied to artificially damaged paper and then mechanically tested to evaluate the resulting reinforcement. The colour changes caused by these adhesives were visually evaluated before and after artificial ageing on small fragments of the original blue paper. The final objective was to select adhesives that offered the strongest reinforcement while causing the smallest colour change.

Experimental

Selection of Adhesives

Tylose, Klucel, gelatine, and starch, all commonly used by French paper conservators, were first selected (Tab 2). Tylose, Klucel and gelatine were prepared at a concentration of 2% w/v, which is quite usual for sizing purposes in paper conservation. Starch was prepared in a traditional way and its concentration was worked out by eye.

Two additional adhesives, Funori and MMA/BMA, were also chosen. In Japan, Funori is traditionally used as a sizing agent (Swider and Smith 2005) in painting conservation for re-adhering and facing matte painting layers. It was suggested for use on western artworks already in the 80s (Evans 1984). It is known to cause no colour changes or additional gloss on the treated surface (Geiger and Michel 2005). More recently, its adhesive properties have been tested by paper conservators (Pataki 2009) for making remoistenable tissue, but results were inconclusive.

MMA/BMA is a mixture of two adhesives, Lascaux 498HV and Lascaux 360HV, both designed by the Swiss company Lascaux

Tab 2 List of tested adhesives.

Type	Adhesives and comments	Names	Conc. (w/v)	Solvents	Suppliers
Cellulosic ethers	Tylose [®] MH 300P (méthylhydroxyethylcellulose)	Tylose	2%	De-ionised water	CTS
	Klucel [®] G (hydroxypropylcellulose)	Klucel	2%	Ethanol 96%	
	Mixture of Tylose [®] MH300 P and Klucel [®] G (1:1)	Tylose/Klucel	2%	De-ionised water / ethanol 96%	
Proteins	Gelatine	Gelatine	2%	De-ionised water	GMW Gabi Kleindorfer
Polysaccharides	Wheat starch Zin Shofu [®] , Japanese origin	Starch	~4.5%	Prepared in water (1:4 in volume) and diluted in water	Atlantis France
	Funori, produced from the red seaweed <i>Gloiopeltis furcat</i> (Japanese origin)	Funori	1%	De-ionised water	Kremer Pigmente GmbH & Co
Aqueous dispersion of acrylic polymers	Mixture of Lascaux 498HV and Lascaux 360HV with a ratio of 2:1 v/v Both adhesives are made with methylmethacrylate (MMA) and butylmethacrylate (BMA). Butylester (BU) is added as a thickener.	MMA/BMA	40%	De-ionised water	Lascaux Colours & Restauro

Colours and Restauro for conservation purposes. They are made of methylmethacrylate and butylmethacrylate and include butylester as thickener. This mixture aims to combine the elasticity properties of Lascaux 498HV and the flexibility properties of Lascaux 360HV. It has been used in the conservation field (Pretzel 1997) and the adhesive ratio chosen for this study (2:1 v/v) has been already satisfactorily tested for the consolidation of the textile of a fan (David 2007).

Preliminary testing was carried out to help estimate the desired degree of paper reinforcement in order to formulate suitable concentrations. Funori substantially reinforced the laboratory samples but becomes difficult to prepare above 2% w/v because of its high viscosity. Therefore a concentration of 1% w/v seemed appropriate for these tests (Tab 2).

MMA/BMA effectively reinforced the paper but could not be used over 40% v/v because it makes the paper sticky. Therefore a concentration of 40% v/v seemed appropriate (Tab 2).

Assessment of Paper Reinforcement

> *Preparation of Laboratory Samples:* It was initially intended to work on historical blue-coloured papers, but those available presented such a huge variation in their mechanical properties (standard deviation: 25% to 45%), that it seemed impossible to undertake a viable comparative study using this material. Thus small sheets (10 x 12 cm) of Whatman No 1 paper (Fisher Scientific) were chosen. As this paper is unsized, it absorbs the adhesives quite evenly, which results in a smaller variation in mechanical measurements.

The original blue paper is brittle, which is not the case of virgin Whatman paper. This paper was made brittle by artificial ageing after being impregnated with a diluted iron gall ink solution [2]. Ageing was carried out in a climatic chamber (Vötsch V0020: Servathin) monitored at 65% RH and 70°C until the paper lost 30% of its initial zero-span tensile strength (TAPPI T 231: 2007). After this treatment, the paper was easy to split and breaks

like the original paper and thus was found suitable for the mechanical tests.

Re-sizing was performed as follows: damaged papers were placed on a non woven polyester fabric (Reemay[®], 17 g·m⁻²: Stouls) on a blotting paper and the adhesive was applied with a soft, 5 cm wide, synthetic brush, first vertically then horizontally, on the recto and verso of each sample.

Japanese paper (RK1, 8 g·m⁻²: Atlantis) was used for the lining. It was coloured blue with diluted acrylic paint (Heavy Body: Liquitex) in a direct brush application with the paper placed on a silicone-coated polyester film (Stouls). Later the papers were pre-coated with the adhesives in a similar method to that used for the resizing treatments, but on one side only. Note that thinner Japanese papers, although available, were not considered here because they were not strong enough to adequately reinforce the original paper. Thicker Japanese papers were also found unsuitable because they would have significantly increased the thickness of the book, which was undesirable.

The laboratory papers were lined on the recto by reactivating the adhesive by applying the appropriate solvent (water or ethanol) with a brush through the Japanese paper lining. The whole was gently brushed, then sandwiched between two pieces of non-woven polyester fabric (Reemay[®], 17 g·m⁻²: Stouls) and two blotting papers, then left under a gentle pressure of 90 kg·m⁻².

All samples were weighed before and after application in order to estimate the quantities of blue paint and adhesive applied. These estimates were quite similar across the samples; those deviating from the average value by more than 20% were removed from the tests.

Three sets of samples were made for the tests: type S samples were sized only; type L samples were lined only; type S+L samples were sized and lined. The type S series was duplicated into two sets called S and S' in order to evaluate the reproducibility of the preparation of samples and of the mechanical testing.

Artificial ageing was this time performed in closed vessels

(hybridisation tube: Fisher Scientific), which were left in an oven for two months. A relatively low temperature of 50°C was chosen in order to (i) create ageing conditions that are close to natural ones and (ii) avoid the denaturation of the gelatine. Small monitors placed in the vessels (hygrobutton: ProgesPlus) recorded temperature and humidity during the process.

This ageing procedure was inspired by previous work (ASTM D6819-02 : 2007; Sawoszczuk et al. 2008), and includes the following variant: samples were preconditioned for 2 days at 80% RH instead of 50% RH by the use of small enclosures containing potassium bromide (KBr) saturated solutions (ISO 483: 1999). This pre-conditioning maintained a relative humidity of 70% during ageing.

- > **Mechanical testing:** The altered stiffness caused by the applied adhesives was perceptible by touch. Initially these alterations were evaluated with a zero-span tensile tester, but this technique was insufficiently sensitive to show significant differences between samples. It was therefore opted for double-fold test measurements (ISO 5626: 1993), using a frequency of 175 folds per minute and a 0.5 kg weight. All tests were performed on 1.5 x 12 cm strips of dried papers, pre-conditioned for at least four hours at 23°C and 50% RH. For each sample, 12 measurements were performed and the average taken. The standard deviations of the 12 measurements underwent an additional calculation in order to estimate experimental error.

Visual Assessment of Colour Changes

Small fragments (approx. 2 cm²) of the original blue paper were found in the page folds. These were sacrificed for the evaluation of colour changes as they were detached from the margins and could not be replaced in the initial position.

The fragments were gently cleaned with a vinyl eraser (Staedler: Stouls) then adhesives were applied on half of the fragment surface with a soft, 2 cm wide, synthetic brush on the recto and verso. Samples were then allowed to dry naturally at room temperature.

The samples were artificially aged for two months in closed vessels as described above. The visual assessment of colour changes was performed by the use of macro-photographs that were taken before and after artificial ageing in similar conditions (same apparatus, same capture parameters) under natural light (3200 K) and UV-light (312 and 365 nm)

Results

Evaluation of Reinforcement by Sizing Treatments

Fig 6 collates the mechanical test performed on the two sets of type S samples. These two sets were prepared independently from the beginning and the paper aged in separate climate chambers before being sized. A minor but systematic difference in temperature between the climate chambers explains why the S' set appears less damaged than the S set. However, comparison of the different adhesives shows similarities in both the S and S' sets, which demonstrates the reproducibility of the approach.

Sizing with Tylose, gelatine or MMA/BMA significantly rein-

forces the paper as can be seen by the increase in the double fold number by a factor of 2 to 3. Klucel, Funori and starch appear much less effective. Finally, combining Tylose and Klucel (Tylose/Klucel) did not significantly improve the reinforcement in comparison to Klucel alone.

Modification of Paper Permeability after Sizing

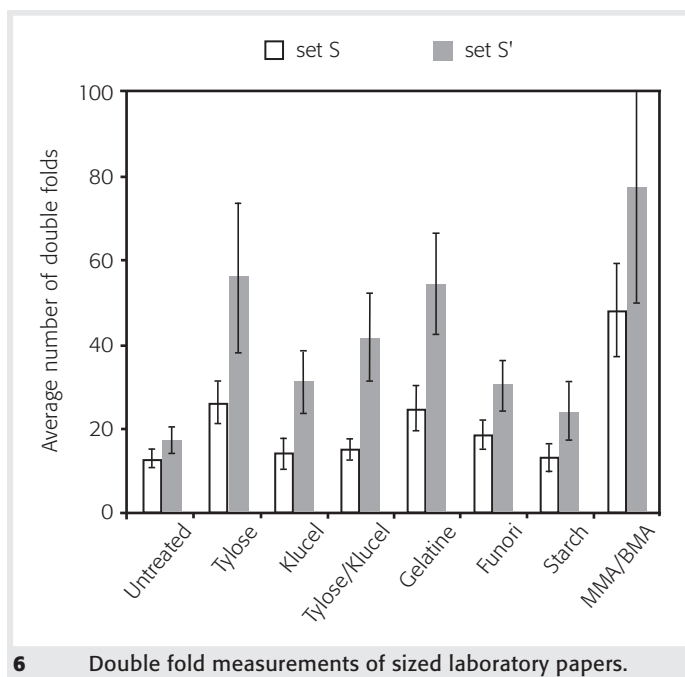
In this test, the re-sizing treatment was chiefly investigated for consolidation purposes. However, in papermaking, sizing is usually undertaken to make the paper less permeable to water, and thus more suitable for water-based inks. To investigate this aspect, some absorption tests were made by measuring the time necessary for a 0.1 µL drop of de-ionised water to penetrate the paper. An average of ten measurements was taken.

Whatman paper is highly absorbent, even after being impregnated with ink and artificially aged. Thus the unsized laboratory sample instantaneously absorbed the 0.1 µL drop of water. The application of Tylose, Tylose/Klucel and starch did not alter this property. The use of Klucel and Funori had only a slight impact by increasing the absorption time from zero to a few seconds. Gelatine however significantly increased the absorption time up to 34 ± 7 s before ageing, but this time decreased with ageing (6 ± 1 s after two months of ageing). Finally MMA/MBA appeared to be the most effective adhesive to make the paper more impermeable to water, as the absorption time reached the average value of 130 ± 30 s, and slightly increased with ageing (190 ± 30 s after two months).

Evaluation of the Reinforcement Attained by Lining Treatments

Mechanical tests were impossible to perform with the Funori samples. Due to its poor adhesion, the lining paper peeled off during the to-and-fro motion of the machine. Hence no data was produced for samples lined with Funori.

On most of the adhesives, it was observed (Fig 7) that the



6 Double fold measurements of sized laboratory papers.

Tab 3 Peeling tests. These tests aim to quickly evaluate the reversibility of the lining (- difficult to remove; + easy to remove; ++ extremely easy to remove [poor adhesion]; – impossible to remove without damaging the original paper).

Adhesive	Dry peeling				Solvent	Peeling with the use of a solvent			
	Before ageing		After ageing			Before ageing		After ageing	
	Type L	Type S+L	Type L	Type S+L		Type L	Type S+L	Type L	Type S+L
Tylose	-	-	++	+	Water	+	+	++	++
Klucel	++	+	++	+	Ethanol	++	++	++	++
Tylose/Klucel	-	-	+	+	Water	+	+	++	++
Gelatine	-	+	-	+	Water	+	+	++	++
Starch	-	-	+	-	Water	+	+	++	++
MMA/BMA	--	--	--	--	Ethanol	-	-	+	-
Funori	++	++	++	++	Water	++	++	++	++

reinforcement was much better after lining than after re-sizing: lined papers (type L and S+L) attained a largely superior average number of double folds (up to ten times more) than that of re-sized papers (type S). This was expected and is largely attributable to the strength and flexibility of the Japanese paper lining. With most adhesives (Klucel, Tylose, gelatine), no significant difference could be observed between lined-only papers (type L) and re-sized and lined papers (type S+L). It was nevertheless observed that lining was radically easier on the samples that were re-sized (type S+L) than on the samples that were not (type L). This is probably due to the fact that re-sized papers are already coated with a thin layer of adhesive, which is reactivated during lining.

Starch samples behaved differently: the average number of double folds of type S+L samples is significantly below that of type L samples. This could be attributed to the adhesive itself: starch is known to lose its flexibility when too concentrated, which may be the case with the type S+L samples.

Results obtained with MMA/BMA were unexpected as type S and L samples show similar average numbers of double folds. Here the presence of a lining paper had no significant impact on the paper reinforcement, probably because it is mainly due to

the adhesive itself. The average number of double folds was much higher for type S+L samples than for type S or L samples: thus the more adhesive, the more flexible the paper becomes.

Similar tests were performed on artificially aged papers (not shown). The same tendencies were observed, yet with lower values of double fold numbers. This shows that the previously indicated properties endure over time.

Evaluation of the Reversibility of the Lining

A final but crucial aspect is the reversibility of the lining. Empirical peeling tests were carried out by hand on dry samples and also by using a solvent applied with a brush beforehand (Tab 3). In these tests, linings with MMA/BMA appear poorly reversible, which rules out its use for direct application on original objects. All other linings showed good reversibility, at least with the use of a solvent.

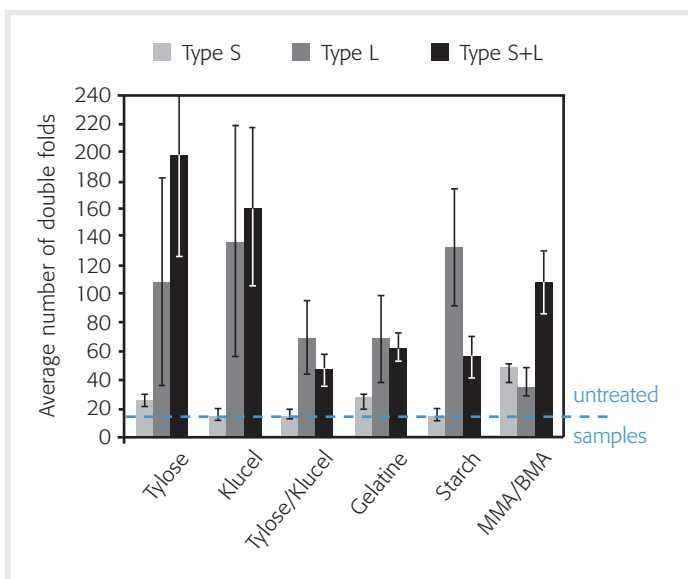
In the case of gelatine, a strong adhesive, the dry removal of the lining was easier when the paper was sized, probably because sizing makes the surface of the paper more even. In this case, the application of a sizing before lining is advised. This is also the case for Klucel, but for opposite reasons: Klucel is a weak adhesive and its application as a size before lining improves the adherence of the lining paper, which can be considered to be positive.

The question of an adhesive's reversibility is often related to its ageing. A lining may be removable immediately after its application, but the evolution of the adhesive over time may significantly change this property. Peeling tests were therefore replicated after artificial ageing (Tab 3), which showed that the reversibility of the linings increased over time for all tested adhesives.

Visual Assessment of Colour Changes on the Original Blue Paper

The examination of fragments under UV light was not very conclusive: the paper was differentially fluorescent and it was nearly impossible to distinguish between the mark of the adhesive and the paper itself. Colour changes were thus chiefly evaluated under daylight by the naked eye, and by the examination of macro-photographs taken before and after ageing (Tab 4).

On all fragments, the contrast between the untouched and the re-sized parts were much less perceptible after ageing than before,



7 Double fold measurements of sized laboratory papers.

Tab 4 Assessment of colour changes due to sizing, before and after artificial ageing (- no significant change; + subtle change; ++ significant change; +++ obvious change).

Adhesive or solvent	Day-light (3200 K)	
	Before ageing	After ageing
Water	+	-
Ethanol	+	-
Tylose	+	-
Klucel	+	-
Gelatine	+++	+
Starch	++	-
Funori	+	-
MMA/BMA	+	-

mostly because the paper had become greener and darker. Before ageing, a more or less obvious darkening of the paper was observed with all adhesives and also with the application of water or alcohol, meaning that the darkening is not only due to the adhesive, but also to the solvent itself.

Gelatine was the adhesive that caused the greatest colour change by far (Fig 8) and the mark of the adhesive remained perceptible even after artificial ageing. Slight but significant colour changes were also observed with starch. Finally, the least colour changes were obtained with Tylose, Klucel, Funori and MMA/BMA.

Application to the Victor Poterlet Sample Book

The Choice of Adhesive

This investigation confirmed that the choice of adhesive is crucial to the possible results of the sizing and lining treatments. In the case of the Victor Poterlet sample book, an adhesive was sought that would optimise paper reinforcement and flexibility while remaining removable and not altering the blue colour of the original paper.

Many of the adhesives could not meet all these objectives: MMA/BMA was irreversible; gelatine and starch induced the most significant colour changes; Funori and, to a lesser extent, Klucel had poor adhesion, which favours the reversibility of the lin-

ing but rules out their use on an object that will remain available for consultation.

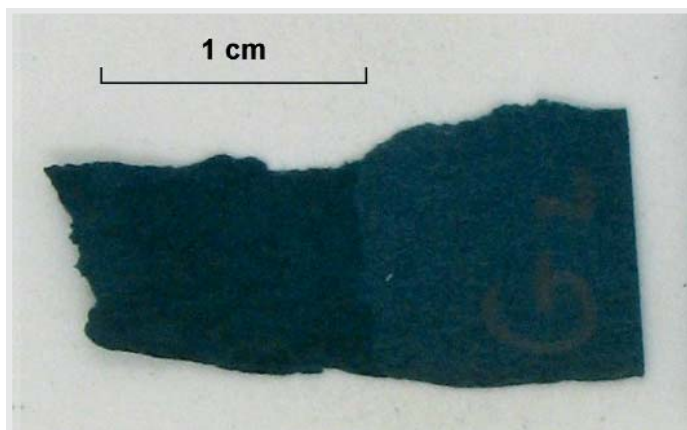
Finally, among all tested adhesives, Tylose appeared to be the most suitable: it remains adequately reversible, induces little colour change, and effectively reinforces the paper when used either for re-sizing or lining.

Description of the Conservation Treatment

The sample book's binding was taken apart and the consolidation of each page carried out. Lacuna outlines were first sketched on a tracing paper. For insets, Sekishu Shi paper (Stouls) was chosen for its opacity and its thickness that is about half that of the original paper. Two pieces of Sekishu Shi paper were cut out following the outer shape of the lacuna using a needle. These were glued together with diluted wheat starch over their entire surface, but leaving approx. 1 mm at the edge of the lacuna free. The original paper was then sandwiched between these two edges (Fig 9).

This investigation highlighted the positive mechanical impact of re-sizing. Nevertheless, this treatment was not carried out on the Poterlet sample book: it would have been necessary to re-size most of the page surface which could not be done with a standard application because of the high number of textile samples. Therefore it was considered that the risk of halos could not be ignored and that a more cautious approach would be to carry out local linings to consolidate the paper.

Consolidation was carried out with the Japanese paper RK1 (8 g·m⁻²: Atlantis) that was stained blue with diluted acrylic paint (Heavy Body: Liquitex), pre-coated with Tylose then cut out in the desired shape (Fig 9). Linings were applied by reactivating the adhesive following a previous study (Van Velzen 2011): the lining paper was placed between two humidified 100% cellulose sponges. The weight of a glass plate placed on top ensured an even humidification. After one minute, the paper could be manipulated and carefully placed on the area to be lined. A gentle pressure applied with a Teflon folder during the drying produced a good adherence.



8 Colour change caused by gelatine application on the original blue-coloured paper of the Victor Poterlet sample book. Only the left part of the sample was sized. © M. Messenger.



9 Detail of a page during treatment. The inset paper is already positioned and the lining paper (blue paper below) is cut out according to the shape of the textile sample. © M. Messenger.



10 Detail of a sheet before and after mending. © M. Messenger.

The pre-coated paper did not expand during reactivation due to the acrylic paint. Hence it was possible to cut out the paper exactly to the required shape and to work with large pieces of lining paper, thus avoiding joints.

This method was effective, rapid and achieved a satisfactory recovery of the original appearance of the paper (Fig 10). No halos or colour changes were perceptible. The drying was quicker than in the case of a direct reactivation with a brush. The original blue paper did not distort, being less humid. Furthermore, there was no need to put the original paper under weights, which was appreciated because the textile samples would have made this operation rather tricky.

Conclusion

This study provided the opportunity to explore different techniques for the consolidation of blue-coloured papers. The properties of various adhesives were compared in order to identify those that would ensure a satisfactory compromise between consolidation and preservation of the original blue colour. Some new adhesives, such as MMA/BMA (scantly documented in the field of paper conservation) were tested, but gave deceptive results. The best results were obtained with Tylose and Klucel. The results of this study were applied to a sample book of the

Forney Library, comprising badly damaged, deep blue paper and a large number of textile samples. As it was not realistic to temporarily remove all these samples, or to undertake chemical treatments on the paper pages, work was focused on formulating an optimal consolidation treatment and a strategy that would be both effective and easy to manage. Finally, the treatment of the 56 sheets took 60 hours and the reinforcement treatment 20 hours, representing an average of 37 minutes per sheet.

Acknowledgements

We warmly thank Thierry Aubry, conservation adviser at the Bibliothèque nationale de France, Paris and Véronique Deangeli Cayol, responsible for the wallpaper and textile collection of the Bibliothèque Forney, Paris, for their advice and for their help in conservation choice.

Endnotes

- * This study is part of a diploma work carried out in 2011 at the Institut National du Patrimoine, Saint Denis, Paris. Some of the analyses mentioned in this article were performed in the laboratory of this institute.
- [1] Blue paper sometimes refers to a large-scale manufacture process that began in the Netherlands in the 16th century and was then spread largely over Europe. This consisted of blending a

small quantity of blue pigments in paper pulps in order to whiten the paper. Although called 'blue paper', this type of paper appears white and is thus not the focus of this article, which deals with paper with a definite blue appearance. To distinguish between the two types, the latter paper is henceforth referred to as 'blue-coloured papers'.

- [2] Iron-gall ink was prepared using the following ingredients and concentrations: gallic acid monohydrate (27645: Sigma Aldrich) 0.6 g·L⁻¹; iron II sulphate heptahydrate (215422: Sigma Aldrich), 2.66 g·L⁻¹; Gum Arabic (G9752: Sigma Aldrich), 6 g·L⁻¹. Two days later, Whatman paper sheets were immersed for ten minutes in the inks using a ratio of 1mL of ink per 2 cm² of paper. After immersion, the excess solution was mopped away in a similar way to that described in the Cobb test (TAPPI T441: 2009).

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Suppliers

- Atlantis France, 35, rue du Ballon, 93 160 Noisy le grand, France, Tel +33-148-155151, www.atlantis-France.com (Japanese paper Paper Nao RK1, 100% Kozo; starch, Zin Shofu®).
- Conservation by design, Timecare Works, 5 Singer Way, Woburn Road Industrial Estate, Kempston, Bedford, MK42 7AW, United Kingdom, Tel +44-123-4846300, www.conservation-by-design (iron gall ink test paper, SUIGIT3000).
- CTS France S.A.R.L., 26, Passage Thiéré, 75 011 Paris, France, Tel +33-143-556044, www.ctseurope.com (Klucl G; Tylose MH300P).
- Fisher Scientific S.A.S., Boulevard Sébastien Brant, Parc d'innovation, BP 50 111, F-67 403 Illkirch cedex, France, Tel +33-

388-671414, www.fishersci.com (Hybridisation tube, 11541325; Whatman n°1, 11541325).

Kremer Pigmente GmbH & Co, Hauptstr. 41-47, 88317 Aichstetten, Germany, Tel +49-756-591120, www.kremer-pigmente.de (Funori).

GMW Gabi Kleindorfer GMW, Aster Str. 9, 84186 Vilsheim, Germany, Tel +49-870-61094, www.gmw-gabikleindorfer.de (Gelatine type B, 40 321).

Jeol Europe SAS, Allée de Giverny, 78, 290 Croissy sur Seine, France, Tel +33-130-153737, www.jeol.fr (Scanning Electron Microscope JSM 5410 LV).

Lascaux Colours & Restauro, Zürichstr. 42, CH-8306 Wangen-Brüttisellen, Switzerland, Tel +41-448-074141, www.lascaux.ch (Lascaux Acrylic Adhesive 360HV, Lascaux Acrylic Adhesive 498HV).

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Oxford Instruments, 77 ZA de Monvoisin, 91400 Gometz la Ville, France, Tel +33-169-852524, www.oxford-instruments.com (Energy Dispersive X-ray Spectrometer, Si[Li] Links PentaFET).

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Servathin S.A, B.P. 54015, 78570 Chanteloup les Vignes, France, Tel: +33 1 340 11100, www.servathin.com (climate chamber, Vötsch V0020).

Sigma Aldrich Chemie Sarl, L'Isle d'Abbeu Chesnes, 38297 Saint Quentin Fallavier, France, Tel +33-800-211408, www.sigmaldrich.com (Gallic acid monohydrate, 27645; iron II sulphate heptahydrate, 215422; gum Arabic, G9752).

Stouls, 9-11, rue de l'Orme St-Germain, 91 165 Champlan Cedex, France, Tel +33-169-101070, www.stouls.fr (Reemay®, RI17; silicone coated polyester film 12 µm thick, POS12RL; Sekishu Shi paper, 50% Thailand kozo, 50% Chemical Bleached Pulp, 31 g·m⁻²; blotting paper; Mars plastic Staedler eraser).

Thermo Fisher Scientific, 16 Avenue du Québec, SILIC 765, 91 963 Courtaboeuf Cedex, France, Tel +33-160-924800, www.thermo.fr (Nicolet 6700 spectrophotometer, Diamond ATR macro system Smart endurance).

Varian SA, Avenue des Tropiques, 91940 Les Ulis, France, Tel +33-169-863838, www.varianinc.com (GC/MS, CPG 3800, SM Saturn 2000).

German Title and Abstract

Beschädigte blaue Papiere: Optimierung der Konsolidierung unter Wahrung der ursprünglichen Farbe

Dieser Artikel befasst sich mit der Behandlung des blauen Papiers eines Musterbuches aus dem Besitz von Victor Poterlet, einem französischen Tapeten-Designer, der im 19. Jahrhundert in Paris arbeitete. Hauptbestandteil des Papierfarbstoffes ist Preußisch Blau. Der Zustand des Papiers war sehr schlecht, wahrscheinlich wegen der Anwesenheit freier Eisenionen. Das Papier konnte weder ersetzt, noch chemisch behandelt werden, weil eine Demontage der darauf befestigten 954 Musterproben zeitlich unmöglich realisierbar war. Darüber hinaus ist das Papier zweifellos von hohem ästhetischem Wert und inherenter Bestandteil des Originalen. Die Untersuchung zur Optimierung der Festigung des blauen Papiers durch lokale Behandlungen, wie Nachleimen und/oder eine Kaschierung mit Japanpapier, wird beschrieben. Es wurden Tests durchgeführt, um auftretende Veränderungen der mechanischen Eigenschaften durch diese Behandlungen zu bewerten. Darüber hinaus wurde es als notwendig erachtet, Farbveränderungen die durch die Anwendung eines Klebstoffes auf einem tiefblauen Papier entstehen könnten, einzuschätzen. Letztendlich wurde das Buch mit einer Methode behandelt, die einen zufriedenstellenden Kompromiss zwischen der besten Verstärkung und den geringsten Farbveränderungen zeigte.

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