

# PROTOCOL FOR THE CHARACTERISATION OF NON-WATERMARKED HANDMADE PAPER

Amélie Couvrat Desvergnes Paper and book conservator Utrecht the Netherlands

<u>ameliecd@gmail.com</u> www. amelieconservation.com



#### **INTRODUCTION**

The present protocol is mainly adapted to non-watermarked handmade paper, regardless of the geographical region in which it was produced.<sup>1</sup> It consists of a macroscopic analysis including documentation and registration of the paper characteristics, its morphology, surface, colour, fibre distribution, quality and impression of the screen among other fields. it is interesting to combine, if possible, macroscopic examination with microscopic analysis, as the latter can help to resolve questions raised by the former. Microscopic analysis with polarising microscope provides information on pulp and fibre characteristics, identification of plant species, presence of additives, etc. However, this document does not cover the methodology necessary to identify plant species. Although fibres have been identified as part of this project, the method of fibre identification is an area in its own right that has not been addressed as part of this project.<sup>2</sup> In addition, the data collected, combined with information on the paper technology used in a specific context and culture, will provide a better understanding of the characterisation of the finished product. Information can be obtained from historical treatises, travel accounts, technical and trade reports, colonial and international exhibition catalogues, literaturee, anthropological field-works, contemporary academic works, etc.

Today various analytical and non-destructive methods are used to analyse and characterise historical papers. For example, SEM–EDS and XRF were applied for the identification of inorganic elements. FTIR analyses were used to identify the chemical structures of the paper components. SEM–EDS, XFR and FTIR analysis also detect fillers, additives and sizing materials. However, not all professionals have access to these expensive devices, and although they provide a great deal of interesting information, they are no substitute for visual observation at both macroscopic and microscopic levels.

The document is articulated in two parts:

- 1. MACROSCOPIC EXAMINATION
- 1.1. The survey form. It is organised into fields, sub-fields and descriptions and covers general description, surface, pulp distribution, mould impressions and measurements.
- 1.2. Guidelines for using the form: further details are provided to explain certain fields in the form.
- 2. MICROSCOPIC ANALYSIS
- 2.1 The survey form. It is organised into fields, sub-fields and descriptions
- 2.2. Guidelines for the preparation of longitudinal fibre samples with permanent mounting (for long-term storage), including the step-by-step process and materials required.

<sup>1</sup> The present protocol has been developed to examine and analyse the paper used for a collection of drawings from northwest India (known as Pahari) now preserved in the Museum Volkenkunde in Leiden (NL). The paper used was locally handmade using traditional techniques inherited from the Arabs who set up paper mills in Central Asia after the Battle of Talas in 751 AD. In this sense, the paper is not watermarked, although the traces left by the paper mould are visible on the paper sheet. The results of the project have enabled us to characterise this particular paper, which was produced in Sialkot, Pakistan and traded throughout northwest India. For a comprehensive study , see A. Couvrat Desvergnes 2023. <sup>2</sup> For the project, the polarizing microscope ZEISS Axiolab A1 and Olympus B×51 were used.

#### 1. MACROSCOPIC EXAMINATION

**1.1.THE SURVEY FORM** 

The protocol was developed in the form of an Excel spreadsheet with drop-down menus, which remains the most practical format for filling in the information and compiling statistics from the data collected. The development of this form was inspired by other protocols developed by conservators and scientists, to which specific criteria were added or removed according to their relevance, feasibility and usefulness.<sup>3</sup> The present form can be customised according to the requirements of the project and the type of paper being studied. Before starting the survey, it is advisable to have an initial overview in order to define the required fields. During the course of the project, with closer examination, it is likely that new fields will appear, which can still be added after the form has been drawn up.

	Field	Sub-field	Description
MACROSCOPIC EXAMINATION	1. GENERAL DESCRIPTION	1.1 Title (given by the institution)	
		1.2 Item acquisition number	
		1.3 Localisation (if known or geographical area)	
		1.4 Dimensions	1.4.1 Height 1.4.12 width
		1.5 End use of the paper	<ul> <li>1.5.1 drawing or painting paper</li> <li>1.5.2 poor quality, refuse</li> <li>1.5.3 printing paper</li> <li>1.5.4 writing paper</li> <li>1.5.5 blank leaf</li> <li>1.5.6 manuscript folio</li> <li>1.5.7 decorated paper</li> <li>1.5.8 other</li> </ul>
		1.6 Type of paper	<ul><li>1.6.1 Laid western</li><li>1.6.2 Laid eastern</li><li>1.6.3 Laid middle-eastern</li><li>1.6.4 other</li></ul>
		1.7 Colour	<ul><li><b>1.7.1</b> Natural</li><li><b>1.7.2</b> Coloured</li></ul>
		1.8 State of the sheet	<ul><li>1.8.1 fragment</li><li>1.8.2 trimmed</li><li>1.8.3 untrimmed</li></ul>
		1.9 Sound	<b>1.9.1</b> Crispy <b>1.9.2</b> Rustling <b>1.9.3</b> Dull <b>1.9.4</b> no sound
		1.10 Flexibility/resistance	1.10.1 weak 1.10.2 soft

<sup>3</sup> Anna-Grette Rischel, Khartasia Kagi, Bas van Velzen, Geneviève Humbert, IPH International standard for the registration of papers with or without watermarks <u>http://www.paperhistory.org/standard.htm</u>

		1.10.3 stiff
		1.10.4 pliable
	1.11 Manufacture defects	1.11.1 papermaker's tear
		1.11.2 wrinkles
		<b>1.11.3</b> thinning
		1.11.4 fragment of paper from
		another sheet
		1.11.5 splitting
		<b>1.11.6</b> hole
		1.11.7 manufacturing tear
	1.12 Condition	<b>1.12.1</b> very good
		<b>1.12.2</b> good
		<b>1.12.3</b> fair
		<b>1.12.4</b> poor
		1.12.5 very poor

	Field	Sub-field	Description
MACROSCOPIC EXAMINATION	2. SURFACE DESCRIPTION With natural and raking light and with magnification.	2.1 Surface appearance of the recto	2.1.1 Mat 2.1.2 Rough 2.1.3 Irregular 2.1.4 Smooth 2.1.5 Glossy
		2.2 Surface appearance of the verso	2.2.1 Mat 2.2.2 Rough 2.2.3 Irregular 2.2.4 Smooth 2.2.5 Glossy
		2.3 Surface treatment	<ul> <li>2.3.1 No surface treatment</li> <li>2.3.2 application of colour</li> <li>2.3.3 coating</li> <li>2.3.4 primer</li> <li>2.3.5 calendered</li> <li>2.3.6 marks of hammer or other pounding tool</li> <li>2.3.7 marks of smoothing tool</li> </ul>
		2.4 Condition of surface treatment recto	2.4.1 even 2.4.2 non-even
		2.5 Tool recto	<ul> <li>2.5.1 use of a large tool</li> <li>2.5.2 use of a medium tool</li> <li>2.5.3 use of a narrow tool</li> <li>2.5.4 no visible marks</li> </ul>
		2.6 Condition of surface treatment verso	<b>2.6.1</b> even <b>2.6.2</b> non-even none
		2.7 Tool verso	<ul> <li>2.7.1 use of a large tool</li> <li>2.7.2 use of a medium tool</li> <li>2.7.3 use of a narrow tool</li> <li>2.7.4 no visible marks</li> </ul>
		2.8 Drying method	<ul><li>2.8.1 traces of a brush</li><li>2.8.2 traces of the wall</li></ul>

	2.8.3 traces left by a felt
	2.8.4 traces left by a cloth

	Field	Sub-field	Description
MACROSCOPIC EXAMINATION	3. PULP DESCRIPTION On the lightbox	3.1 Pulp repartition	3.1.1 even/clear 3.1.2 moderately even 3.1.3 uneven/cloudy
			3.1.4 Very cloudy
		3.2 Fibre direction	<b>3.2.1</b> dominant
			<b>3.2.2</b> random
		3.3 Fibres appearance	<b>3.3.1</b> presence of long fibres
			<b>3.3.2</b> presence of medium fibres
			<b>3.3.4</b> mixture of short long and
			medium fibres
		3.4 Fibres arrangement	3.4.1 Stand-alone fibres
			<b>3.4.2</b> bundles of unprocessed fibres
			3.4.5 clumps
		3.5 Internal elements	<b>3.5.1</b> uncoloured yarn
			<b>3.5.2</b> coloured yarn
			<b>3.5.3</b> fragment of retree (repulped
			paper cuttings)
			<b>3.5.4</b> mall fibres of different colours
			<b>3.5.5</b> bark or shive
		3.6 Other significant	3.6.1 Black particles
		features	3.6.2 Micro-holes
			3.6.3 Unknown substance

	Field	Sub-field	Description
MACROSCOPIC	4. MOULD	4.1 Type of screen	4.1.1 wire laid lines
EXAMINATION	IMPRINTS		<b>4.1.2</b> bamboo
	On the lightbox		<b>4.1.3</b> reeds
			<b>4.1.4</b> cloth
			4.1.5 presence of rib shadows
			4.1.6 not identified
			<b>4.1.7</b> other
		4.2 Deckle edge	4.2.1 presence of a deckle edge
			<b>4.2.2</b> presence of two deckle edges.
		4.3 Laid lines visibility	4.3.1 clearly visible laid lines
			4.3.2 partially visible laid lines
			4.3.3 no visible laid lines
		4.4 Laid lines repartition	<b>4.4.1</b> even
			<b>4.4.2</b> Random
		4.5 Laid lines feature 1	<b>4.5.1</b> thin
			4.5.2 medium
			4.5.3 thick
		4.6 Laid lines feature 2	4.6.1 Straight
			4.6.1 undulated
		4.7 Chain lines visibility	4.7.1 clearly visible chain lines

		4.7.2 partially visible chain lines
		4.7.3 no visible chain lines
	4.8 Chain lines feature 1 4.8.1 thin	
		4.8.2 medium
		<b>4.8.3</b> thick
	4.9 chain lines feature 2	<b>4.9.1</b> straight
		4.9.2 undulated
	4.10 Chain lines	4.10.1 regular
	arrangement	<b>4.10.2</b> grouped by 2
		<b>4.10.3</b> grouped by 3
		4.10.4 random
		4.10.5 other types of arrangement

	Field	Sub-field	Description
MACROSCOPIC	5. MEASUREMENTS	5.1 Thickness	5.1.1 measurement 1
EXAMINATION	With tools described in		5.1.2 measurement 2
	the next section		5.1.3 measurement 3
			5.1.4 measurement 4
			5.1.5 average thickness
		5.2 Colour	(See next section)
		5.3 Glossiness	5.3.1 measurement 1
			5.3.2 measurement 2
			5.3.3 measurement 3
			5.3.4 measurement 4
			5.3.5 average glossiness
		5.4 Laid lines	5.4.1 number of laid lines/1cm
		number	5.4.2 number of laid lines/2cm
			<b>5.4.3</b> number of laid lines comprised
			in 20 or 30 mm
			5.4.4 Average number of laid lines
		5.5 Chain lines	<b>5.5.1</b> distance between two chain
		number	lines-1
			<b>5.5.2</b> distance between two chain
			lines-2
			<b>5.5.3</b> distance between two chain
			lines-3
			5.5.4 average distance between
			chain lines
		5.6 Number of	[free text]
		visible chain lines	

#### **1.2. GUIDELINES FOR USING THE FORM**

#### 2. Surface description

Besides the use of natural, raking, and UV light, the observation of the surface can be done with the help of two methods:<sup>4</sup>

#### • Thread Counter

An easy-to-use and cheap tool for primary examination.

It is available in all of the conservation suppliers



• **Dinolite**<sup>®</sup> Many models are suitable for paper examination.



#### 2.3 Surface treatment

This field is relevant for papers that have undergone a specific surface treatment. This is the case for Islamic papers, which are generally burnished. Far Eastern papers show a smooth and a rough side resulting from the drying process on hot walls.

#### 3.1 Pulp repartition

This field can tell us a lot about fibre preparation and sheet formation, as well as the skill of the papermaker. Fibre distribution is linked to technological processes in pulp preparation. It also tells us about the quality of the paper and the degree of fibre blending. Uneven fibre distribution in the finished paper is often due to inadequate fibre preparation during the pulping process, but also to the papermaker's ability to distribute the pulp evenly in the mould.<sup>5</sup> The shallow or partial beating of the fibres will interfere with the distribution of the pulp in the finished sheet. The distribution of the pulp is also linked to the size of the sheet: the larger the sheet, the more difficult it is to distribute the

<sup>4</sup> For the project, the portable UV light flashlight Gatzetec WF-501B

https://www.gatzetec.de/Taschenlampen/Gatzetec/UltraFire-WF-501B-CREE-XPL-HI-V3.html <sup>5</sup> Helman-Wazny A., 2016.

fibres evenly. This is why the process has been made easier by adding plant extracts or starch solutions to the pulp to help distribute the fibres evenly and prevent flocculation. The assessment of pulp repartition in a sheet of paper is carried out in transmitted light using a lightbox.

For flat paper works: dimmable LED lightbox<sup>6</sup>



For books: LED light sheet<sup>7</sup>, light-weight, ultrathin (0,5 mm) and flexible.



#### 3.2 Fibre appearance

The presence of fibres of varying lengths indicates the processing method. The presence of long and good-quality fibres may indicate that the paper was produced from native fibres and could have been beaten by hand. Short and fibrillated fibres may suggest that these were recycled or processed with mechanical technology. The presence of bundles of unprocessed fibres indicates that recycled woven or twisted materials were used to produce pulp such as cloth, rags canvas, cordage, sacking, matting etc... <sup>8</sup>

#### 3.3 Internal elements

The presence of various elements that make the paper unclear and irregular is the result of the degree of cleaning of the materials, retting, cooking, beating and washing of the pulp. The presence of small pieces of bark may be the result of careless or partial manual removal as well as a quick retting process. Clumpings are generally loose fibres that have clumped together due to the action of the hammers. Fragments of yarn, coloured or not, suggest that woven products have been recycled in the pulp. Small pieces of recycled retree paper have a distinct, palpable, shape that

<sup>&</sup>lt;sup>6</sup> For the project, I used the Daylight Wafer 2 https://www.vanbeekdesign.nl/p/daylight-wafer-2-lightboxa3/88312/?gclid=CjwKCAjwp6CkBhB\_EiwAlQVyxYDFgM0XBamjQEHf4BRC28i7Hv8MYXTcKk9GiL1h4McDTTnwfwd57xoCsAsQ AvD\_BwE

<sup>&</sup>lt;sup>7</sup> https://www.cxdinternational.com/furniture/lighting-equipment/cxd-light-sheet-a4-eu-eqflse0004

<sup>&</sup>lt;sup>8</sup> Couvrat Desvergnes A. 2023.

protrudes from the surface of the paper, indicating that the paper cuttings have been reused in the pulp.<sup>9</sup>

However, these are just a few examples of how the presence or absence of these elements should be interpreted. Their observation, combined with information on the paper technology used in a specific context and culture, will help to better understand the characterisation of the finished product.

#### 5.1 Thickness

Thickness information is used to determine two main skills: the papermaker's ability to produce sheets of variable thickness and his ability to obtain sheets of regular thickness. The measurements are taken on the four edges with a micrometre. An average of the 4 measurements will be calculated in an additional column with the average formula.

A digital micrometre is easier and more straightforward to use than a manual gauge.



#### 5.2 Colour

The measurement of paper colour is an aspect that is generally not taken into account in paper surveys. The investigators maintain that it is irrelevant because the initial colour of the paper is gradually altered over time. Factors that induce colour alterations are numerous: inherent factors responsible for the natural ageing of the paper or its components (acid hydrolysis) and external factors (pollutants, light, temperature, humidity and fluctuations, etc). While this argument may be valid for archaeological papers or papers from caches such as guenizas that have been damaged by dust or soiling from burial, or for modern wood pulp-based papers that discolour over time, the colour remains nonetheless a relevant criterion that can provide a great deal of information about quality and manufacturing methods, materials used, etc. This is particularly prevalent for papers that were safely kept or for paper folia that are bound inside bindings away from light and other external degradation factors.

Colour is often the result of a specific papermaking technique and depends on the raw materials and their treatment, such as washing, cooking, bleaching and pulping. Colour is often seen as a sign of quality and grading: the whiter the paper, the more expensive and valued it is. However, this feature only makes sense within a group that allows us to compare items of the same construction and composition.<sup>10</sup> This is why it is important to examine other materials outside the initial corpus in order to obtain a global view of paper produced in the same cultural context and used for other types of objects.

<sup>&</sup>lt;sup>9</sup> Velzen 2018, p. 19.

<sup>&</sup>lt;sup>10</sup> Arnaud-Nguyen 2019.

#### **Colour measurement**

The most scientific and precise way is to measure the colour with a **spectrophotometer** which provides **CIE L\*a\*b\*** coordinates which are the norms defined by the International Commission on Illumination. However, if you don't have the budget, the **Munsell colour chart** is a good alternative based on the Munsell classification system. It requires no technology and can be carried out in situ, although measurements must be taken in good natural light to avoid distorting the reading.

- **Subjective visual assessment:** some interviewers indicated that they used their own method by simply characterising the colour of the paper by several different shades (around 5): white, off-white, cream, brown, grey, etc. However, this method remains fairly subjective and depends on the interviewer's own visualisation of the colours and on the ambient light.
- Portable spectrophotometer Konica Minolta

(Minolta CM 700 d and CM-600d have been successfully used in conservation and research project)<sup>11</sup>.

It provides CIE L\*a\*b\* coordinates



### • Munsell Soil Colour Charts<sup>12</sup>

The charts are used in geology, archaeology and forensics to identify the colours of soils. The theory is based on Munsell's classification System. It is an easy, straightforward way to measure colour.





 $<sup>^{11}\,</sup>https://www5.konicaminolta.eu/fr/instruments-de-mesure/produits/couleur/spectrophotometres-portables/cm-700d-cm-600d/introduction.html$ 

<sup>&</sup>lt;sup>12</sup> https://munsell.com/color-products/color-communications-products/environmental-color-communication/munsell-soil-color-charts/

#### 5.2 Glossiness

This field is relevant for papers that have received a specific surface treatment such as burnishing, calendering, etc. An average of the 4 measurements will be calculated in an additional column with the average formula. The glossiness is measured with a glossmeter.

#### **Gloss measurement**

The glossmeter used for the project was purchased from PaintGauges.co.uk (reference GM2).<sup>13</sup> It is a digital glossmeter that optically measures surface reflectivity at multiple angles (20°, 60°, 85°) and can be used to calculate haze.

Its multi-angle measuring capability means it is suitable for measuring the widest variety of surfaces and gloss levels. Its 20° angle is ideal for very high gloss surfaces such as polished metals and its 85° angle is ideal for very low gloss surfaces such as matt paints. This eliminates the need to own multiple meters of different specifications and its capability to take simultaneous readings at each angle speeds the measurement process. If a gloss reading is over 70GU at 60° then the reading at 20° is often selected. If a gloss reading is below 10GU at 60° then the reading at 85° is often selected.



#### 4. Laid lines and Chain lines visibility

Their visibility in a sheet of paper depends on the construction of the papermaking screen and the thickness of the sheet of paper. Chain lines are visible depending on several factors: the material used (horsehair, wire, thread, nylon line, etc.), their condition (over time chain lines become worn and are less likely to leave marks on the sheet of paper) and the way they have been woven around the laid lines (whether they protrude enough over the surface to leave a clear impression on the paper). However, the visibility of these lines can be hampered by the surface treatment of the paper: dyeing, sizing, coating or burnishing often disrupt their observation.

#### 5.4 Laid lines numbers 5.5 Chain lines numbers

The counting of laid and chain lines is an aspect that was initially used for Western watermarked paper. However, it is also accurate for paper with visible impressions left by the paper mould. For instance, it has been used, for example, to characterise Islamic paper. This allows comparisons to be made between different papers and also shows the degree of progress and technology involved in making the mould. Some researchers have highlighted the fact that the number of lines laid means a higher level of achievement and skill in making the mould and, by extension, in making the paper. This is why recording the width, configuration and distribution of the laid and chain lines also provides important data in understanding paper technology.

<sup>13</sup> https://www.paintgauges.co.uk/shop/gm2-gloss-meter/

#### 2. MICROSCOPIC ANALYSIS 2.1 THE SURVEY FORM

Institutions and owners are often reluctant to carry out destructive analyses and therefore to take samples, although the samples are micro-scale; only a few millimetres are required. Recent research projects have demonstrated the use of non-destructive methods to identify fibres, without taking samples, using the high-magnification digital microscope directly on the paper surface.<sup>14</sup> Although this method has certain advantages, there are some reservations about the relevance of the results, as the fibres are interwoven in the substrate, so their characteristics are not fully visible, along with many related features such as crystals, associated cells, fillers, etc. In the author's experience and opinion, sampling and microscopy remain the most straightforward and instructive means of identifying fibres and understanding paper technology by characterizing the pulp as represented in the sample. The analysis should be made with a polarising microscope, which allows us to visualise clearly the fibre features through the polarising lens.<sup>15</sup> Hertzberg and Graff-C staining tests, for example, are commonly used to get a better look at fibre characteristics, but these reagents are no longer commercially available and have to be made by the end users themselves.<sup>16</sup> For this project, no staining tests were carried out, but the fibres were identified on the basis of their physical anatomy and with the help of two fibre atlases (Catling/Grayson; Sisko/Pfäffli). As these atlases refer to untreated textile fibres and not to reused paper fibres, reference samples were also taken from raw and processed materials to broaden the database of comparison materials.

The following table, in the form of an Excel spreadsheet, can be used as a basis for the microscopic study of paper samples. It can be adapted to the means and needs of a specific project.<sup>17</sup>

	Magnification	Field	Sub-field	Description
MICROSCOPY	<b>Overall description</b>	Length of the fibres	Short	
ANALYSIS	of the fibres and the		Medium	
	pulp at 5x		Long	
			Mixture of fibres of	
			various lengths	
		Thickness of the	Thin	
		fibres	Medium	
			Wide	
			Variable	
		Shape of the fibres	Straight	
			Slender	
			Rigid	

<sup>&</sup>lt;sup>14</sup> The method was developed by Japanese scientists Osaka and Sakamoto. See also Emilie Arnaud-Nguyen, 2019. The fibres were analysed in situ by Lucas Llopis (EPHE / CRC) and Léon-Bavi Vilmont (CRC) without sampling, using a Keyence VHX 6000 digital microscope equipped with a VH-Z 1000 R objective. Transmitted and raking light was used. The paper was scanned at x100 magnification to observe the surface appearance. A magnification of between x200 and x500 was then used to observe and identify coarse elements such as threads or clusters of fibres, while a magnification of x1000 was used to identify fibres. <sup>15</sup> Polarized light microscopy involved two polarizers crossed (one above the fiber specimen, the other below the fiber specimen), the image observed has a dark black background. Depending on the molecular order within the specimen, the features of the fiber are seen as brilliant colors of the spectrum (the Michel-Levy chart).

wiki.com/wiki/BPG Spot Tests#Lignin and General Fiber Content

<sup>&</sup>lt;sup>16</sup> The recipes to make these reagents are available on <u>https://www.conservation-</u>

<sup>&</sup>lt;sup>17</sup> For more details on fibre identification and morphology see <u>https://www.conservation-wiki.com/wiki/BPG\_Fiber\_Identification;</u>

https://cameo.mfa.org/wiki/Fiber Morphology Definitions used in the Fiber Reference Image Library and both aforementioned atlases.

	Fibre direction	Angular Flexible Convoluted Twisted Flat Flabby Random Dominant	
		Clumps of short fibres Compact bundles of fibres Unseparated fibres	
	Other substances	Unsubstantial substance Black substance Amorphous substance (cambium cells) Loose wwall cells	
Overall description of the fibres at 10 x	Quality of the fibres	Good quality fibres with visible features Medium-quality fibres with uneven visible features Poor quality fibres with non-visible features Short and very fibrillated fibres	
	Degree of fibrillation	None Light Medium Heavy	
Fibre identification at 20 x and 50 x	Type of fibre	[species]	
	Associated cells of the plant species:	Presence Absence	Parenchyma Vascular vessels Epidermis cells Hairs Other
	Lumen	Not apparent Wide Narrow Even Variable Constricted Interrupted	
	Cell walls	Thick Thin Varying thickness Scimitar like	
	Crossmarkings	None Few Moderate Many	

		Longitudinal	None	
		markings (striations)	Few	
			Moderate	
			Many	
		Dislocations in the	None	
		fibre	Few	
			Moderate	
			Many	
		Dulaina	None	
		Buiging	Few	
			Moderate	
			Many	
			None	
		Bursting	Few	
			Moderate	
			Many	
		Fibre end	Natural end	Tapering
				Pointed
				Rounded
				Branched
		Fibre end	Non-natural end	Cut
				Fibrillated
				fraved
	Other components	Additives: starch	None	
	at 20 x and 50 x	grains	Few	
		Branno	Moderate	
			Many	
		Additives: dispersing	None	[nature]
		agent	Fow	[liature]
		ugent	Moderate	
			Many	
<u> </u>		Additives: mineral	None	[nature]
		filler particles	Fow	[liature]
			Moderate	
			Many	
		Calcium ovalata	Nono	rhamhic
			Fow	clustors
		CI YSLAIS	rew Madarata	
			Marine	clusters in chains
			iviany	
				prismatic

- 2.2. HOW TO PREPARE LONGITUDINAL FIBRE SAMPLE WITH PERMANENT MOUNT (FOR LONG-TERM STORAGE)
  - 2.2.1. **MATERIALS FOR SLIDE PREPARATION** (Most of the materials were purchased from Carl Roth). <sup>18</sup>

Slide superfrost®



Deckaläser

Coverslips





<sup>&</sup>lt;sup>18</sup> https://www.carlroth.com/nl/nl

Microscope slide tablet to keep the slides flat and tidy while the water evaporates



Slide box to store the permanent slides.



Entomologist kit including tweezers, small scissors, and probes to separate the fibre in the paper sample.



#### 2.2.2. MOUNTING MEDIUM

## Nitrile gloves should be used when handling the following products

#### • Canada balsam, 25 ml <sup>19</sup>

It is the oleoresin of the balsam fir tree (Abies balsamea) of boreal North America. The resin, dissolved in essential oils, is a viscous, sticky, colourless or yellowish liquid that turns to a transparent yellowish mass when the essential oils have been allowed to evaporate. Due to its high optical quality and the similarity of its refractive index to that of crown glass (n = 1.55), purified and filtered Canada balsam was traditionally used in optics as an invisible-when-dry glue for glass, such as lens elements. also commonly used for making permanent microscope slides.

The resin very viscous and therefore tedious and time consuming to use, which represents a substantial workload. What's more, it takes at least a week for it to dry, so slide preparation has to be planned well in advance, However, slides made with Canada balsam are renowned for their longevity! It is advisable to buy a small bottle, as the balm dries out in a few weeks in the container, which is not airtight enough. It is also very difficult to clean, even with a large quantity of ethanol.

• Euparal, 100 g. An alternative to Canada balsam<sup>20</sup>. It contains eucalyptol and paraldehyde. It is more fluid than Canada Balsam and therefore much easier to use.











<sup>&</sup>lt;sup>19</sup> https://www.carlroth.com/nl/nl/anhydrous-mounting-media/canada-balsam/p/8016.1

<sup>&</sup>lt;sup>20</sup> https://www.carlroth.com/nl/nl/long-term-mounting-with-euparal/euparal/p/7356.1

• Mounting medium Eukitt<sup>®</sup> from Merck (containing 45% acrylic resin and 55% xylenes) used in microscopy. It is more fluid than Canada Balsam and dry faster (20 minutes). <sup>21</sup>



• For semi-permanent mount: glycerol in dionised water, 50/50.<sup>22</sup> The slide can be used for a set period of time and can be sealed with clear nail varnish.



#### 2.2.3. SLIDE PREPARATION PROCEDURE

- Take a minute sample of fibre and place it on a slide with a drop of deionised water. Label the slide.
   With the help of the pin and separate the fibres. The better the sample preparation, the more visible the fibre characteristics.
- Place a coverslip on top once all the fibers have been well separated. Make sure no air bubbles are trapped between the slide and the coverslip. If it is, press gently with a finger to evacuate the air.

<sup>&</sup>lt;sup>21</sup> https://www.sigmaaldrich.com/NL/en/product/sial/03989

<sup>&</sup>lt;sup>22</sup> https://www.carlroth.com/nl/nl/water-soluble-mounting-media/protein-glycerol/p/p049.1

- 3. Place the slide flat in the tray and allow the water to evaporate for a day or two.
- 4. Open the coverslip and set aside. Place a drop of Canada balsam or other mounting product on the fibers and carefully press the coverslip back into place with your finger.



5. Place the slide on a metal surface and apply a strong neodymium magnet to the top of the coverslip. In this way, the fibres will be compressed in the same plane, making it easier to focus the microscope.







#### SELECTED BIBLIOGRAPHY

#### Paper study and protocol

Arnaud-Nguyen E. 2019. Etude matérielle des manuscrits sur papier en langue tocharienne du fonds Pelliot de la bibliothèque nationale de France, Actes de Colloques Histoire du papier et de la papèterie. Actualités de la recherche.

----2020. Paper Analyses of Tocharian manuscripts of the Pelliot Collection stored in the National Library of France, in *Z Badań nad Książką i Księgozbiorami Historycznymi* [ The Studies into the History of the Book and Book Collections], Vol. 14, no. 3, University of Warsaw.

Beit-Arié, Malachi. 1999. Quantitative typology of Oriental paper patterns. In *Le Papier au Moyen Age: histoire et techniques*. Bibliologia 19. Monique Zerdoun Bat-Yehouda, ed. Turnhout, Brussels: Brepols, pp. 41-54.

Couvrat Desvergnes A. 2023. Sialkoti paper used for the production of Pahari drawings and paintings in northwest India, in *Artists' Paper A Case in Paper History*, edited by Penelope Banou *et al.* Horn/Wien: Verlag Berger.

Helman-Wazny A. 2016. More than meets the eye: Fibre and Paper Analysis of the Chinese Manuscripts from the Silk Roads. STAR: Science & Technology of Archaeological Research, 2:2, 127-140.

---- 2020. Notes on the early history of paper in Central Asia based on material evidence. In *Z Badań nad Książką i Księgozbiorami Historycznymi* [The Studies into the History of the Book and Book Collections] vol. 14, no. 3, University of Warsaw.

Humbert G. 1998. Papiers Non Filigranés Utilisés au Proche-Orient jusqu'en 1450: Essai de Typologie. In Journal Asiatique 286, pp. 1-54.

Irigoin J. 1963. Les Types de Formes Utilises dans l'Orient Méditerranéen (Syrie, Egypte) du XIe au XIVe Siecle. In Papiergeschichte 13 (April 1963), pp. 18-21.

---- 1993. Les Papier Non Filigranes Etat Présent des Recherches et Perspectives d'Avenir. In Ancient and Medieval Book Materials and Techniques. Maniaci M. and Paola Munafo P. eds. Citta del Vaticano: Biblioteca Apostolica Vaticana, 1993. 265-312.

Kropf E. Baker A.C. 2013. A Conservative Tradition? Arab Papers of the 12th–17th Centuries from the Islamic Manuscripts Collection at the University of Michigan. Journal of Islamic Manuscripts, Leiden: Brill. Volume 4: Issue 1

Le Léannec-Bavavéas, M.T. and Humbert G. 1990. Une Méthode de Description du Papier Non Filigrane (Dit "Oriental"). In Gazette du Livre Médiéval 17, pp.24-30.

Rischel A-G. 2001. Through the Microscope Lens: Classification of Oriental Paper Technology and Fibres, Looking at Paper evidence & interpretation, Symposium Proceedings, Toronto 1999, pp.179-188.

---2004. Analysis of Papermaker's Choice of Fibrous Materials and Technology Along the Paper Road. In Rosella Graziaplena (ed.), Paper as a Medium of Cultural Heritage. Archaeology and Conservation (Rome-Verona, August 30th–September 6th 2002, 26th Congress – International Association of Paper Historians), Rome: Instituto centrale per la patologia del libro, 202 – 208 ----2005 Analysis of fibers used for oriental papermaking compared to botanical descriptions, aiming at establishing an identification key Cultures of Silk Road and Modern Science, Vol. 2, Scientific Analysis, Conservation and Digitization of Central Asian Cultural Properties, Osaka 2005, pp. 21-30. ---- 2006. Arab paper - the Oriental link between Asiatic and European paper: a comparative macroscopic and microscopic analysis. In 28<sup>th</sup> International Congress of Paper Historians in Capellades: Papers of the 28th International Congress of Paper Historians, Vol. 16, pp. 137-142. ---- 2009. Permanence and Durability of Paper: A Study Through the Microscope. In *Journal of Paper Conservation: IADA reports* 10, no. 2, pp. 25–30.

----2020. A scientific description of specimens of Asian paper of known origin, in *Z Badań nad Książką i Księgozbiorami Historycznymi* [The Studies into the History of the Book and Book Collections] vol. 14, no. 3, University of Warsaw.

Sakamoto S., Okada Y. (2015), Paper analysis and database of papers of the Pelliot collection, Dunhuang manuscripts, Proceeding in International Conference on Culture and Computing, 203-204 (in Japanese)

Velzen van S.T.J. 2018. The universe between felt and wire: A new look into the typology of Westernmade paper. PhD dissertation, University of Amsterdam.

#### Fibre analysis and identification

Bergfjord, C., & Holst, B. 2010. A procedure for identifying textile bast fibres using microscopy: Flax, nettle/ramie, hemp and jute. *Ultramicroscopy*, 110(9), 1192–1197.

Catling/Grayson. 1982. Identification of vegetable fibres, London, New York : Chapman and Hall.

Sisko/Pfäffli. 1995. Fiber Atlas. Identification of Papermaking Fibers, Berlin : Springer. 1995.

Velzen van STJ. 2018. The universe between felt and wire: A new look into the typology of Western made paper. PhD dissertation, University of Amsterdam.

Wiesner, J. 1887. Mikroskopische Untersuchung der Papiere von El-Faijûm (Microscopic Examination of the Faijûm Papers). *Mittheilungen aus der Sammlung der Papyrus Erzherzog Rainer*, Wien, ---1986. Microscopic Examination of the Faijum Papers (Mikroskopische Untersuchung der Papiere von El-Faijùm). Transl. Gudrun Aurand. Edited, with an introduction by Jack C. Thompson. Portland: the Caber Press.

#### Scientific characterisation of paper

Espejo T. et al. 2010. Microscopic and spectroscopic techniques for the study of paper supports and textile used in the binding of hispano-arabic manuscripts from Al-Andalus: A transition model in the 15th century. Journal of Cultural Heritage 11, pp.50–58.

Mahgoub H, John R. Gilchrist JR, Fearn T, Strlič M. 2017. Analytical robustness of quantitative NIR chemical imaging for Islamic paper characterization. Proc. SPIE 10331, Optics for Arts, Architecture, and Archaeology VI.

Mahgoub H, Bardon T, Lichtblau D, Fearn T, Strlic M. 2016. Material properties of Islamic paper. Heritage science, 4:34.

Mahgoub H, Chen H, Gilchrist JR, Fern T, Strlic M. 2017. Quantitative chemical near-infrared hyperspectral imaging of Islamic paper. In: Bridgland J, editor. ICOM-CC 18th triennial conference preprints, Copenhagen, 4–8 September 2017, Art. 1606. Paris: International Council of Museums.

Manso, Marta and Maria Luisa Carvalho. 2009. Application of Spectroscopic Techniques for the Study of Paper Documents: A Survey. Spectrochimica Acta. Part B, Atomic Spectroscopy 64, pp. 482–490.