Case study: **Capuchin prayer book with relics (KU Leuven Maurits Sabbe Library, Leuven)**

Case study image:

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1. Non-invasive analysis

1.1. Methodology

XRF

For the material-technical analyses of a small Capuchin prayer book with relics, X-ray fluorescence (XRF) analyses were performed. Despite a number of limitations and drawbacks, mobile XRF has been successfully used in the past for the identification of pigments in murals, manuscripts, etc.

XRF is an elemental analysis technique that makes it possible to identify the chemical elements present within the irradiated zone, without taking into account the stratigraphy. The chemical elements can thus be present both in the surface layer and / or in the underlying layer(s) (this depends on the nature of the chemical elements and the thickness of the layers).

A second disadvantage of the technique is that the information obtained is “elementary” information and not molecular information. For example, when lead is detected in a paint layer, this may indicate the white pigment lead white [2 $PbCO_3$.Pb $(OH)_2$], or the red lead red (minium) [2PbO.PbO_2], or the yellow massicot [PbO], or a mixture.

Light chemical elements are not detected (positive identification from potassium) and organic pigments / binders cannot be identified.

This technique thus provides a general overview of the chemical elements present in the analysed zone.

The XRF technique is based on the following principle: a primary x-ray (coming from the x-ray source in the equipment) is aimed at the paint layer. Secondary X-rays, characteristic of the chemical elements present, are generated. Due to the high energy of the incident primary x-rays, secondary x-rays are generated not only in the surface layer but also in the underlying layer(s). This obviously complicates the interpretation of measurement results.

All measurements were performed with a µ-XRF instrument ARTAX (Bruker) with a rhodium (Rh) tube with polycapillary optics, which gives the primary x-ray beam a diameter of approximately 70 µm, which allows to analyse very fine details (Figure 1) . The measurement location can be accurately selected using a CCD colour camera (figure 2). All images registered by the XRF-camera (visualisation of the measuring spots) are displayed in this report.

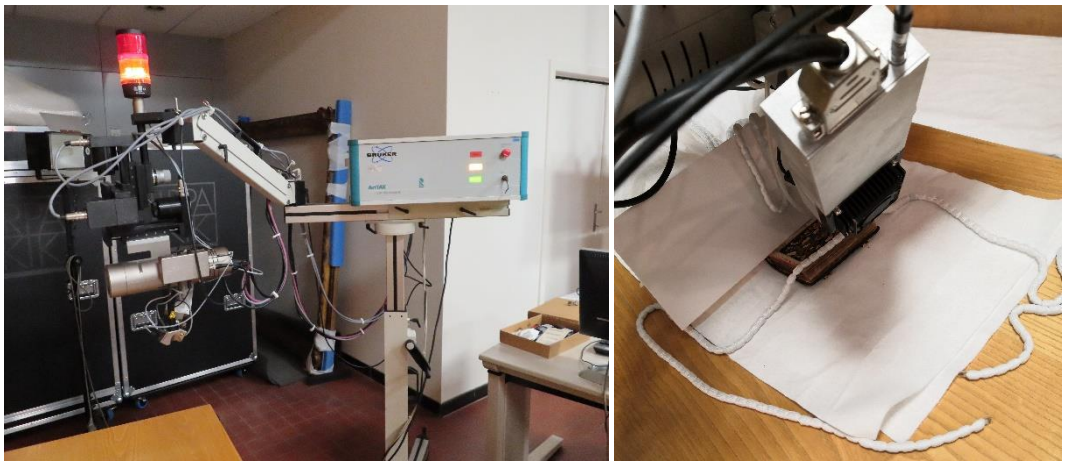
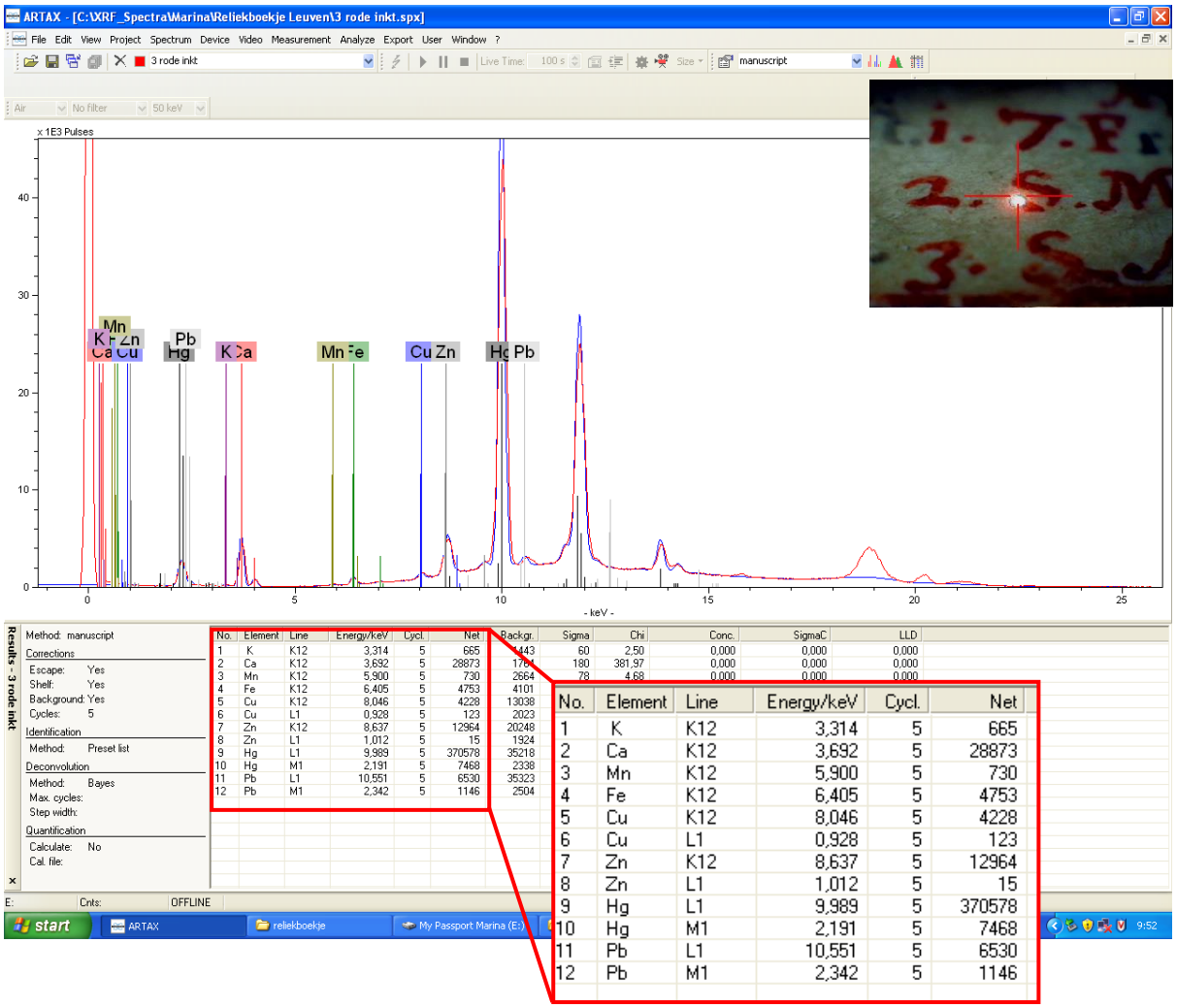


Figure 1: XRF measuring equipment

Figure 2: a small Capuchin prayer book with relics (left) and image taken by the camera of the XRF equipment (detail red ink). The analysed zone (70µm) is in the middle of the cross

A typical analysis result for XRF is presented in Figure 3.

The following experimental conditions were used in all measurements: Rh tube, 50 kV, 500µA, 120 seconds, no filter.



No.	Element	Line	Energy/keV	Cycl.	Net
1	K	K12	3,314	5	665
2	Ca	K12	3,692	5	28873
3	Mn	K12	5,900	5	730
4	Fe	K12	6,405	5	4753
5	Cu	K12	8,046	5	4228
6	Cu	L1	0,928	5	123
7	Zn	K12	8,637	5	12964
8	Zn	L1	1,012	5	15
9	Hg	L1	9,989	5	370578
10	Hg	M1	2,191	5	7468
11	Pb	L1	10,551	5	6530
12	Pb	M1	2,342	5	1146

Figure 3: X-ray spectrum and data processing of the analysed spot presented in figure 2 (red ink)

The interpretation of the spectrum above gives the following result:

The red ink contains the pigment vermilion (mercury sulphide). The detected calcium and zinc (minor amounts) might be present in the paper.

As explained, the interpretation of XRF analyses alone is not always unambiguous: only elementary information is obtained and different pigments can at the same time give rise to secondary X-rays. The interpretation is a combination of the obtained element information and, in case of pigments, also on the observed colour. Sometimes multiple interpretations are possible.

A total of 21 measurement points were selected.

All the selected measurement locations are indicated in the figures 9-12.

Raman

A complementary µRaman (MRS) analysis was performed on some selected places. Unlike XRF, Raman spectroscopy provides molecular information so that the technique can be considered a true 'fingerprint' identification.

The analyses are also carried out non-destructively using a Renishaw Invia Raman spectrometer (figure 4) with 785 nm laser. Interpretation of the resulting spectra is done by comparing with the spectra of an in-house built library of reference spectra of known pigments. A typical analysis result is shown in figure 5.

Figure 4: Raman spectrometer

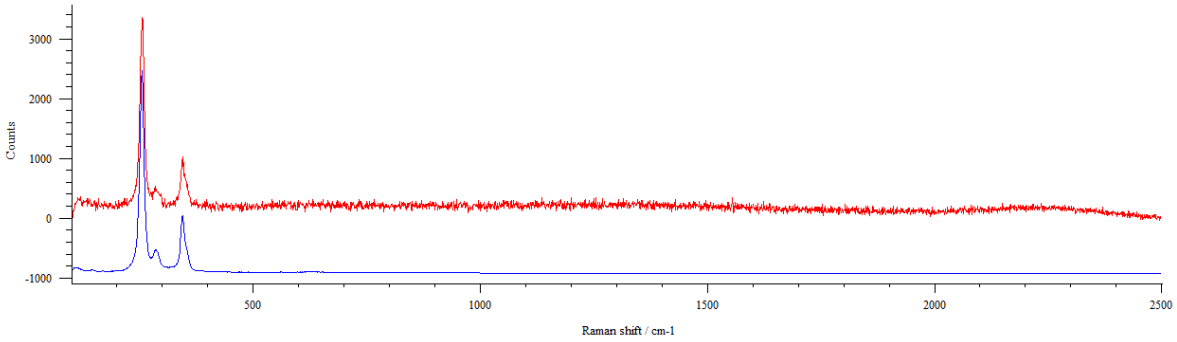


Figure 5: MRS spectrum of a red ink (red spectrum, MRS1). Identification is done by comparing the obtained spectrum with reference spectra from the Raman library (blue spectrum = reference spectrum of vermilion). See table 1

Fibre Optic Reflectance Spectra (FORS)

The reflectance spectra (FORS – Fibre Optic Reflectance Spectra) are registered by the portable spectrometer Gorgias USB4000 (chsopensource, figure 6) with 3648-element Toshiba lineair CCD array, between 350 and 950 nm. The angle of incidence and detection is 45°. Spectra are referenced against a white standard.

The registered spectra are compared to reference spectra of known pigments, both as reflectance spectrum and after log (1/R) transformation. A typical analysis result is shown in figure 7 and 8.

A total of 10 measurement points were selected.

All the selected measurement locations are indicated in figures 9-12.

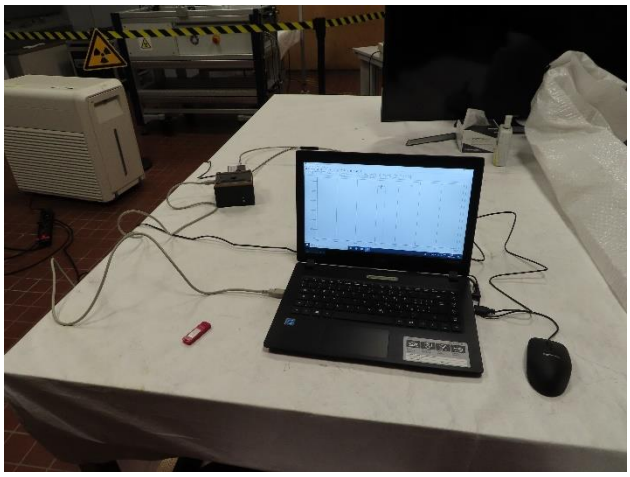


Figure 6: Fibre Optic Reflectance Spectrometer (FORS)

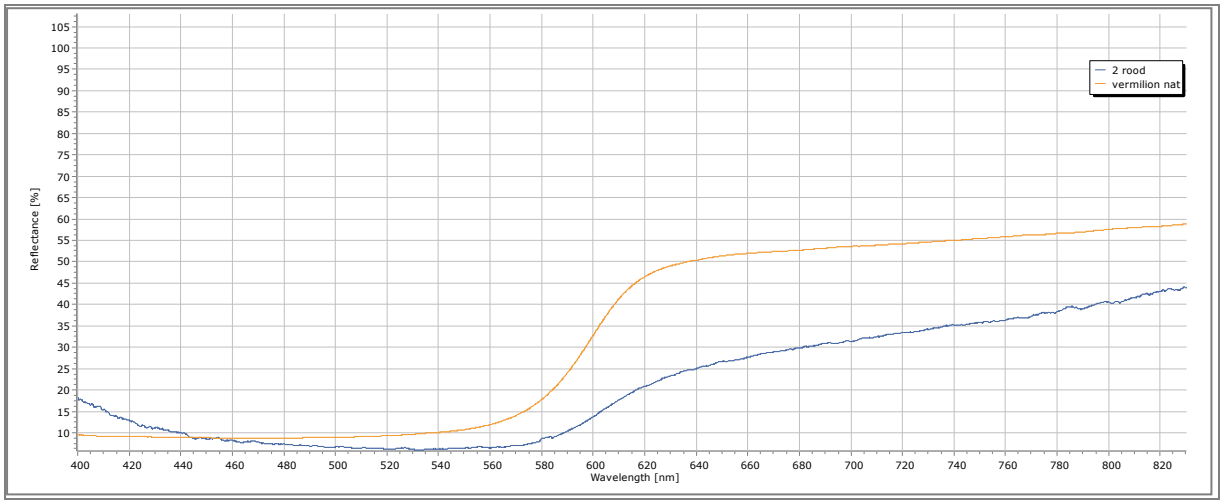


Figure 7: FORS spectrum of a red zone (blue spectrum, FORS2). Identification is done by comparing the obtained spectrum with reference spectra from the FORS library (red spectrum = reference spectrum of vermilion). See table 1

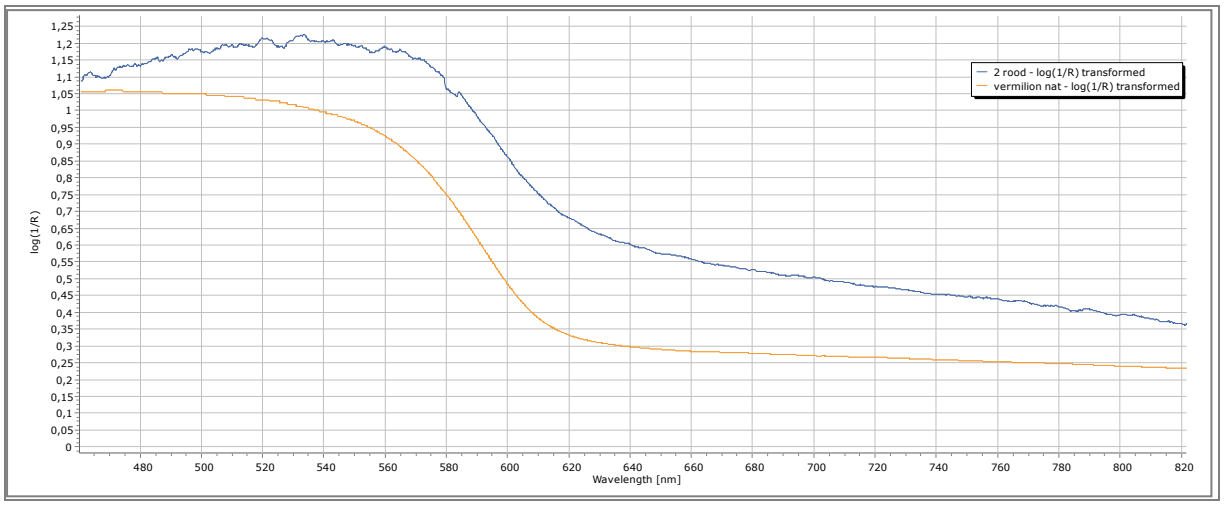


Figure 8: log (1/R) transformation of FORS spectra (cfr figure 7)

1.2. Results

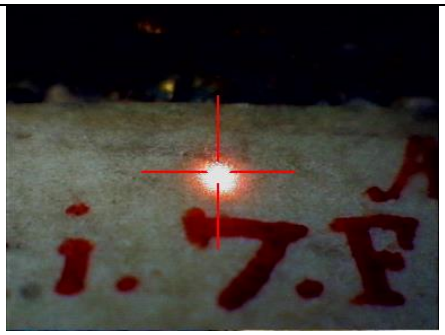
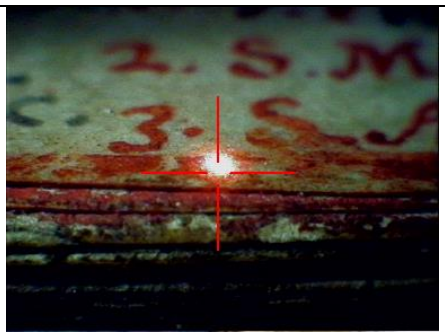
The results are shown below per page.

The results of the analyses are summarized in tables 1-4.

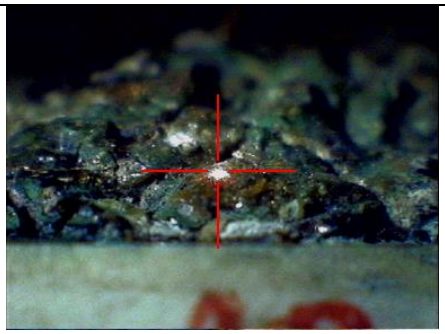
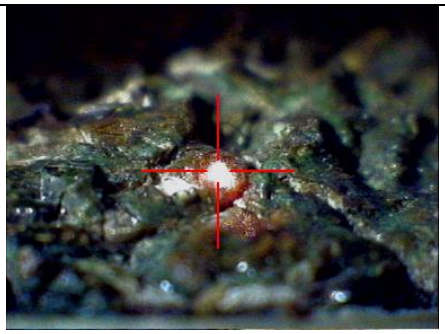
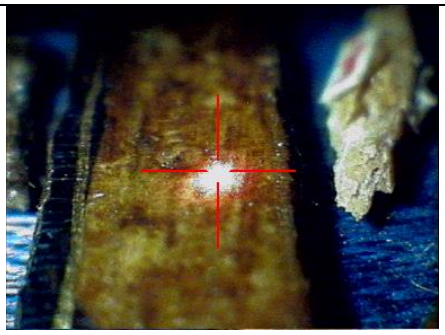
Figure 9: The book with indication of XRF measuring spots (white), MRS measuring spots (light blue) and FORS spots (yellow)

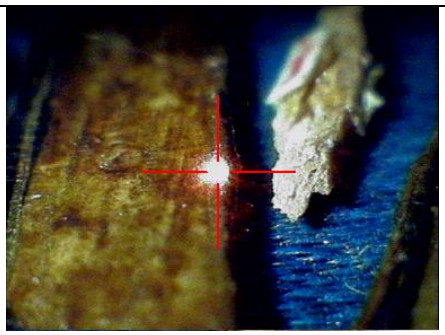
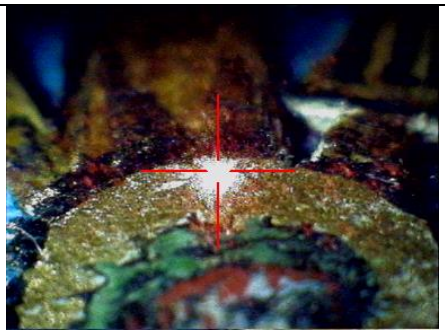
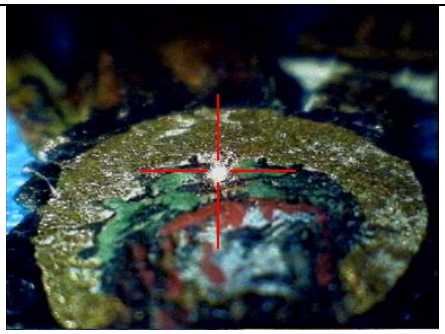
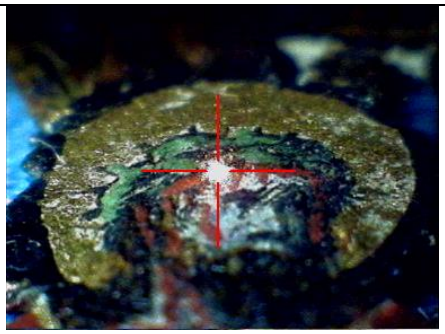
Table 1: description of the analysed zones and results of analyses: potassium (K), calcium (Ca), manganese (Mn), iron (Fe), copper (Cu), zinc (Zn), silver (Ag), gold (Au) mercury (Hg), lead (Pb)

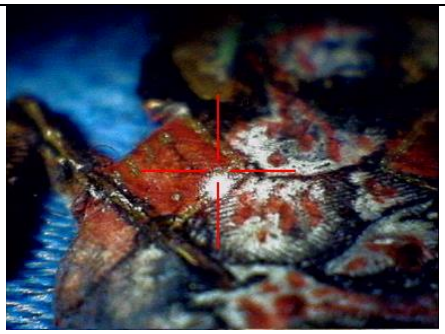
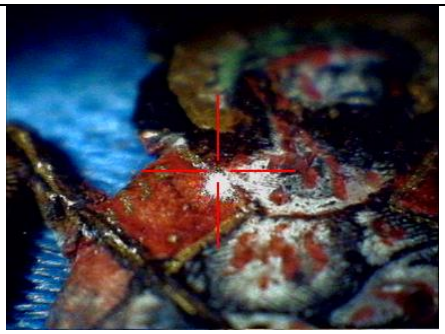
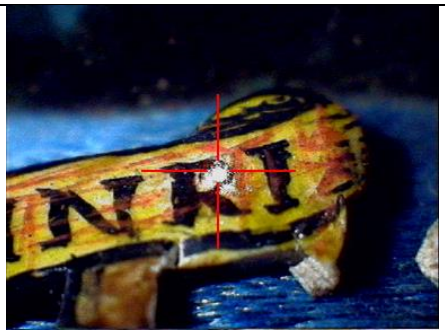
Elements present in low concentrations are in parentheses [1]

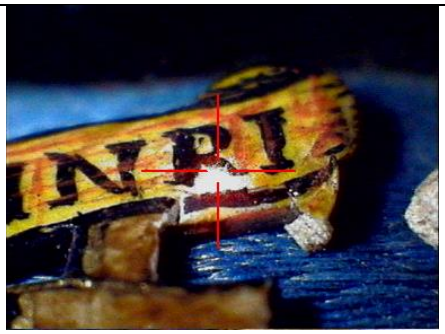
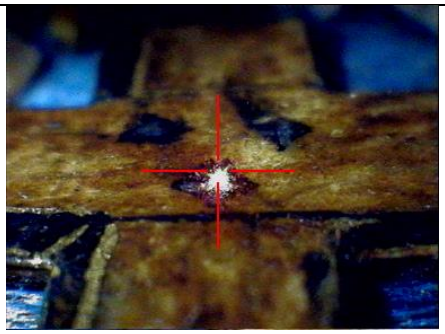
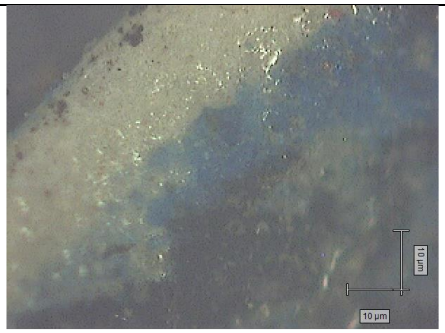
N°	Measuring place (image Artax-camera)	Description	Results of XRF-analyses	Interpretation
XRF1		Red ink	Ca, Hg, Zn, (Cu), (Fe)	vermilion
XRF2		Blanc paper	Ca, Zn, Fe, (Pb), (Cu), (Hg), (Mn)	
XRF3		Red ink	**Hg**, Ca, Zn, (Pb), (Fe), (Cu)	vermilion
MRS1		Red ink		Vermilion
XRF4		Red ink	**Hg**, Ca, Zn, (Pb), (Cu), (Fe), (Au), (Mn)	vermilion

[1] >100.000 in bold, > 10.000 counts in normal letters, >1.000 in parentheses

XRF5		Ink	Ca, Fe, Zn, (Pb), (Cu), (K)	Result not significant different from blanc paper Carbon based ink
XRF6		Ink	Ca, Fe, Zn, (Pb), (Cu), (Au), (K), (Mn)	Idem XRF 5
XRF7		Rock	**Cu**, **Ca**, Au, Fe, (Pb), (K), (Mn)	Copper green Gold (cfr Hirox image in figure 11)
XRF8		Rock	**Cu**, Ca, Au, Fe, (Pb), (K)	Copper green Gold
FORS 6 -7		Rock		No results
XRF9		Cross	Ca, Au, Fe, (Hg), (Pb), (Cu), (K), (Mn)	Result not significant different from blanc paper Organic layer, details in gold

MRS 13		Cross		No result
FORS 4		Cross		No result
XRF10		Cross	Ca, Au, Fe, Pb, (Cu), (Mn), (Hg), (K), (Zn)	Idem XRF 9
XRF11		Nimbus	**Au**, Ca, (Cu), (Hg), (Fe), (Pb), (K), (Mn)	Gold
XRF12		Green thorn wreath	Cu, Au, Ca, Fe, (Pb), (K), (Hg), (Zn), (Mn)	Copper green
MRS 12		Green thorn wreath		Carbon black, lead white and chalk
FORS 5		Green thorn wreath		No result
XRF13		Red face	Hg, Ca, Cu, Pb, Fe, (K), (Au), (Mn), (Zn)	Vermilion, copper green

XRF14		Blood on hand	**Pb**, **Hg**, (Fe), (Ca), (Cu)	Lead white, vermilion
MRS 10		White skin Jezus		No result
MRS 11		Red blood		Vermilion
XRF15		Coat	**Hg**, Ca, Pb, (Fe), (Cu), (K)	vermilion
XRF16		Golden edge on coat	**Hg**, **Au**, Ca, (Cu), (Pb), (Fe), (K)	Gold paint, vermilion
XRF17		Ink inscription	Ca, Fe, (Pb), (Cu), (Hg), (Mn), (K), (Zn)	Result not significant different from blanc paper Carbon based ink

XRF18		Yellow under inscription	Ca, (Fe), (Pb), (Cu), (Hg), (K), (Zn), (Mn)	organic
MRS 2 - 14		Yellow under inscription		No result
FORS 3		Yellow under inscription		Gamboge (see figure 10)
XRF19		Nail on cross	Ca, Fe, (Ag), (Cu), (Pb), (Hg), (Mn), (Zn)	Silver
MRS3		Red bow		vermilion
FORS 2		Red bow		vermilion
MRS4 -5		Blue silk		indigo
FORS 1		Blue silk		Indigo?
MRS 6-7-8		Blue spearhead		Azurite? – carbon black
MRS 9		Black bow		Carbon black

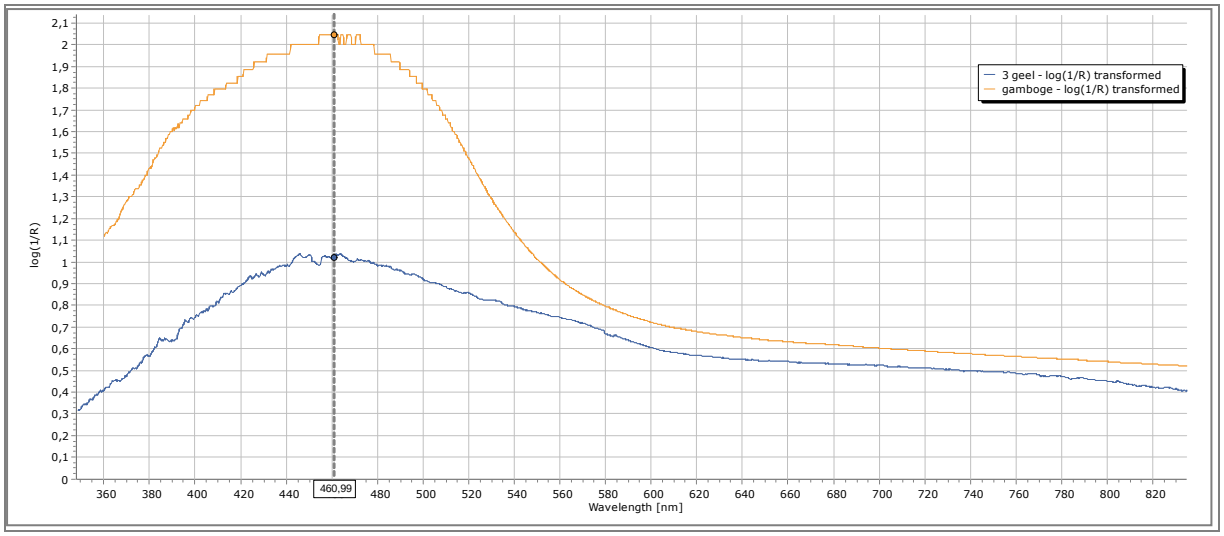


Figure 10: FORS analysis results (FORS measurement area 4 and gamboge reference)

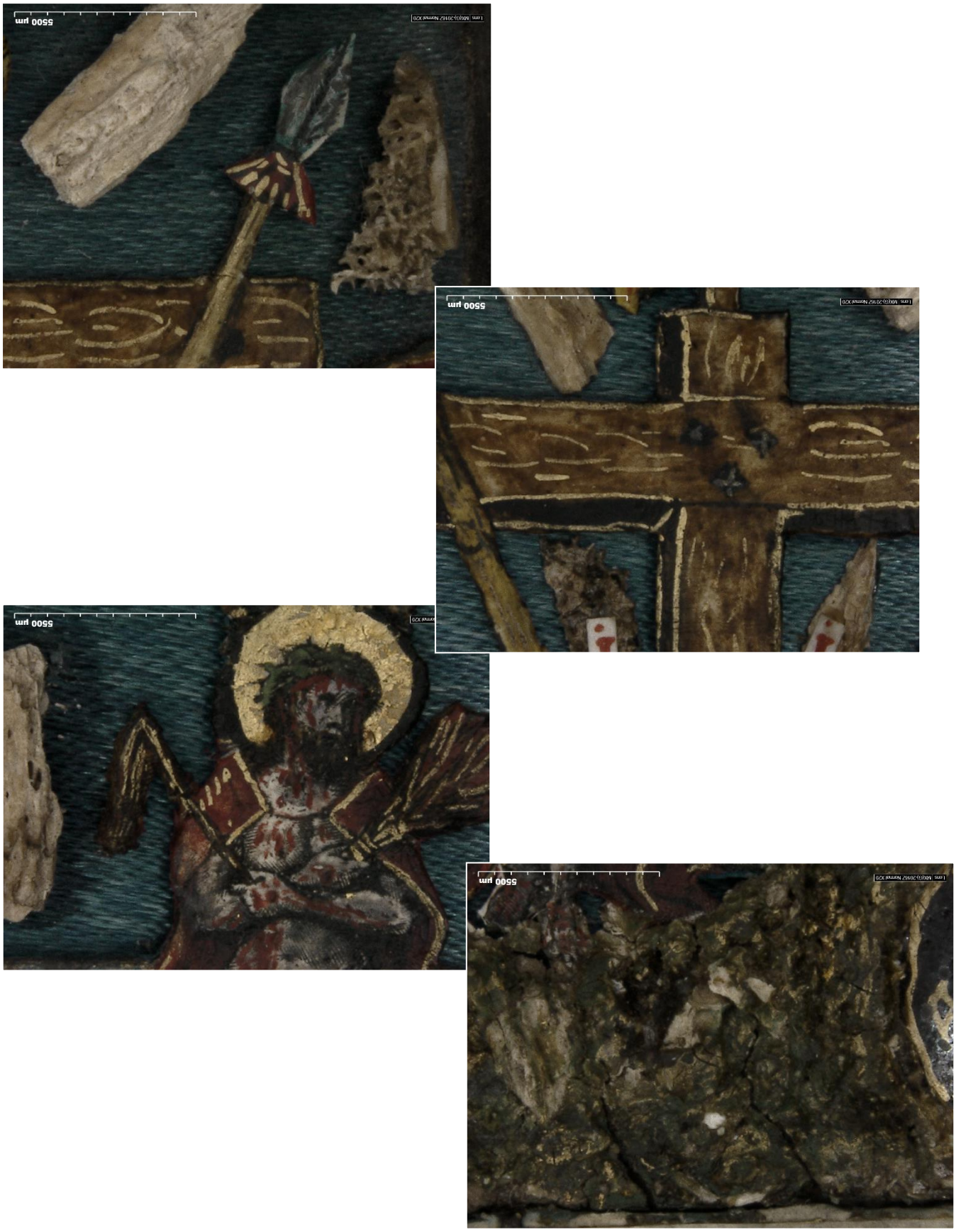


Figure 11: Hirox digital microscope images of some details

Figure 12: The book with indication of MRS measuring spots (light blue) and FORS measuring spots (yellow)

Table 2: description of the analysed zones and results of analyses

N°	Measuring place (image Artax-camera)	Description	Results of XRF-analyses	Interpretation
MRS 15		Blue/green thread (warp/weft)		Indigo
MRS 16		Yellow thread (warp/weft)		Indigo
FORS 8		Green/Yellow textile		*Indigo + ?*
MRS 17		Red thread		No result
FORS 9		Red/yellow textile		Carmine lake? (see figure 13)

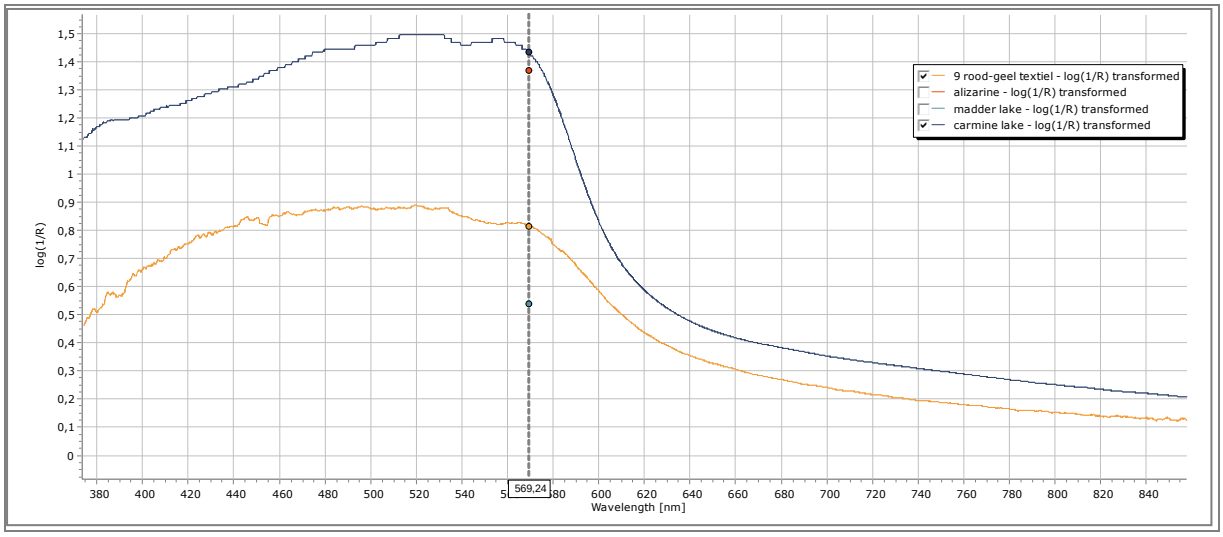


Figure 13: FORS analysis results (FORS measurement area 9 and carmine lake reference)

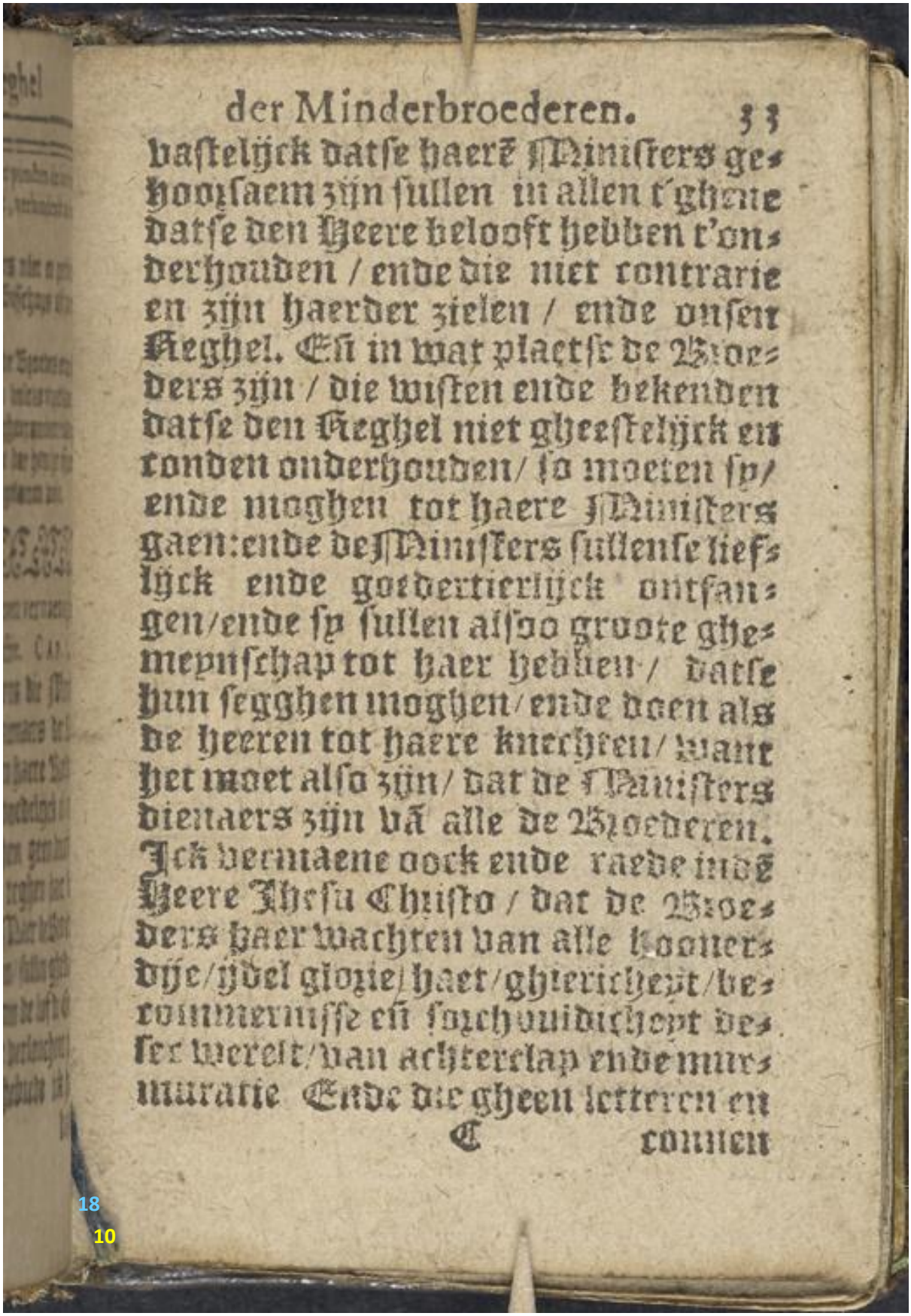


Figure 14: The book with indication of MRS measuring spots (light blue) and FORS measuring spots (yellow)

Table 3: description of the analysed zones and results of analyses

N°	Measuring place (image Artax-camera)	Description	Results of XRF-analyses	Interpretation
MRS 18		Blue sewing thread		Indigo
FORS 10		Blue sewing thread		Indigo (see figure 15)

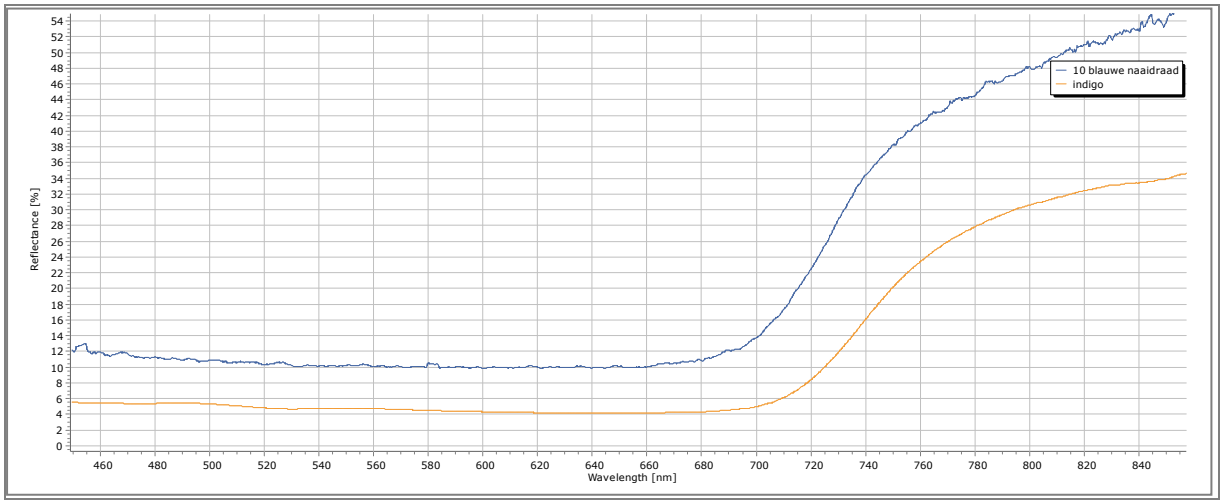
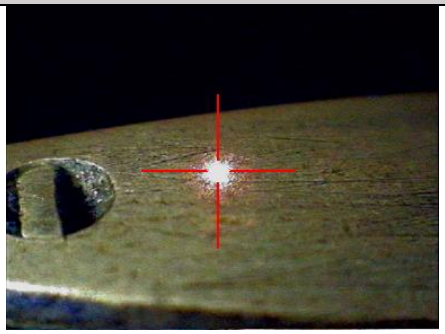
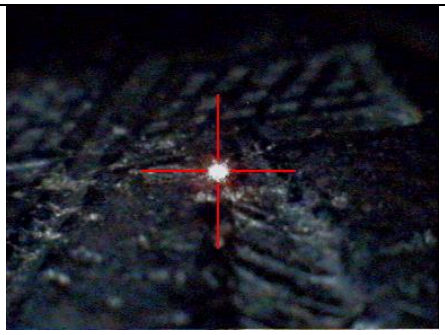


Figure 15: FORS analysis results (FORS measurement area 10 and indigo reference)

Figure 16: The book with indication of XRF measuring spots (white)

Table 4: description of the analysed zones and results of analyses: potassium (K), calcium (Ca), manganese (Mn), iron (Fe), nickel (Ni), copper (Cu), zinc (Zn), tin (Sn), lead (Pb)

Elements present in low concentrations are in parentheses [2]

N°	Measuring place (image Artax-camera)	Description	Results of XRF-analyses	Interpretation
XRF20		Clamp	**Cu**, **Zn**, Pb, Ni, (Fe), (Sn)	Brass
XRF21		Leather	**Fe**, Cu, Zn,, Ca, K, (Pb), (Mn)	Black dying of leather using iron compounds?

[2] >100.000 in bold, > 10.000 counts in normal letters, >1.000 in parentheses

2. Invasive analysis

2.1. Mica

In order to identify the material used to protect the relics in the 'pigeon storage' (see figure 11), a small sample of this material was withdrawn by Lieve Watteeuw.

Digital microscope images of the material inside the book and of the withdrawn sample are shown in figure 17 and 18.

Figure 17: Hirox digital microscope images of the transparent material inside the book

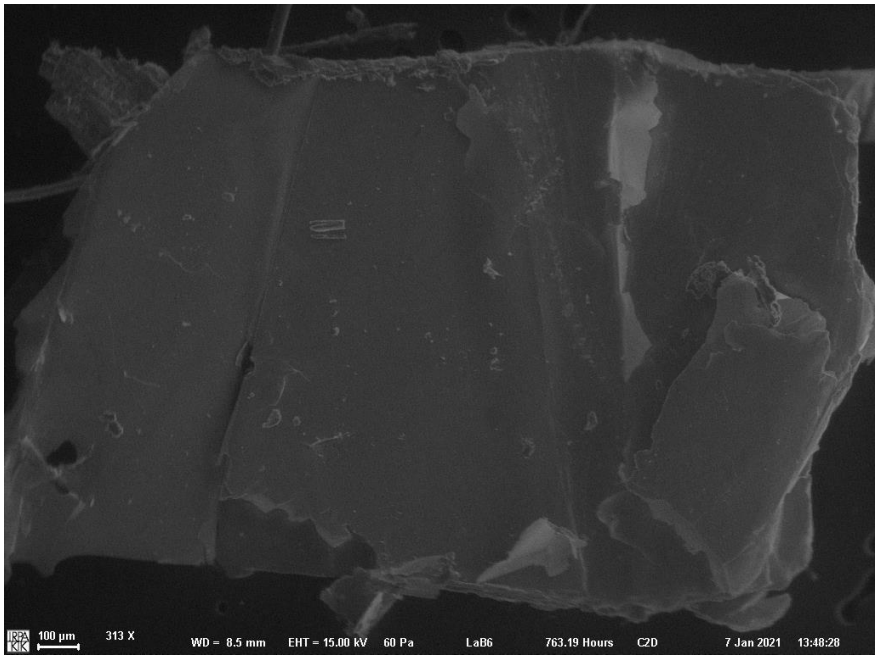
Figure 18: Hirox digital microscope image of the sample of the transparent material

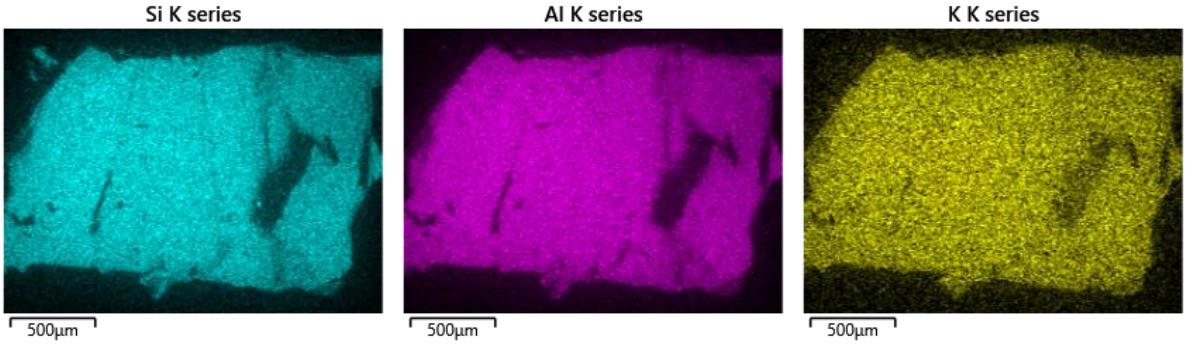
The sample is analysed by scanning electron microscopy – energy dispersive X-ray detection (**SEM-EDX**) and by infrared spectroscopy (**FT-IR**).

For the SEM-EDX analysis, a small fragment is fixed with a carbon-tape on a SEM-sample holder (analysis with a Zeiss EVO LS15 electron microscope and energy dispersive X-ray detector from Oxford instruments).

For the FT-IR analysis, a small fragment (size ≈ needle point) is compressed between two diamond windows of a compression cell (SpectraTech) in order to obtain a transparent thin layer of the sample. The FT-IR spectrum is recorded in transmission mode (64 scans, resolution 4 cm^{-1} , from 4000 to 600 cm^{-1} , Vertex 70 spectrometer coupled to a Hyperion 3000 microscope with MCT detector, Bruker).

The SEM-EDX results are summarized in figure 19.





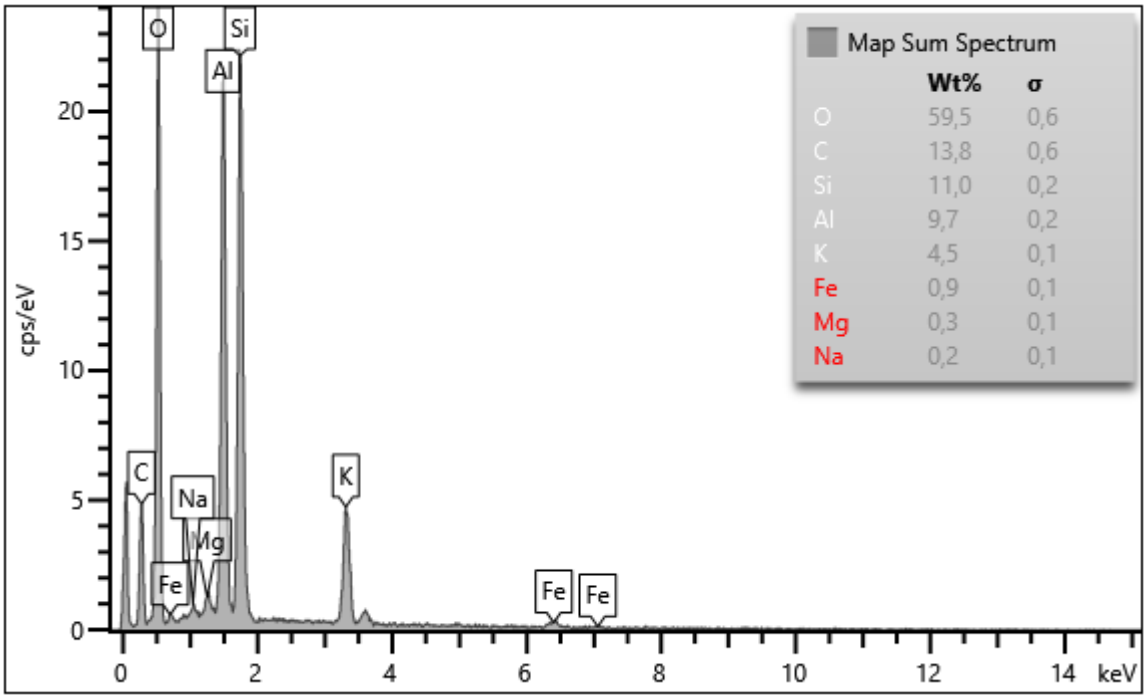


Figure 19: Secondary electron image of the analysed sample fragment (top), SEM-EDX mapping with the distribution of silicon (Si), aluminium (Al) and potassium (K) (middle) and EDX spectrum (carbon (C), oxygen (O), iron (Fe), sodium (Na), magnesium (Mg), silicon (Si), aluminium (Al) and potassium (K) (bottom)

The resulting FT-IR spectrum is shown in figure 20.

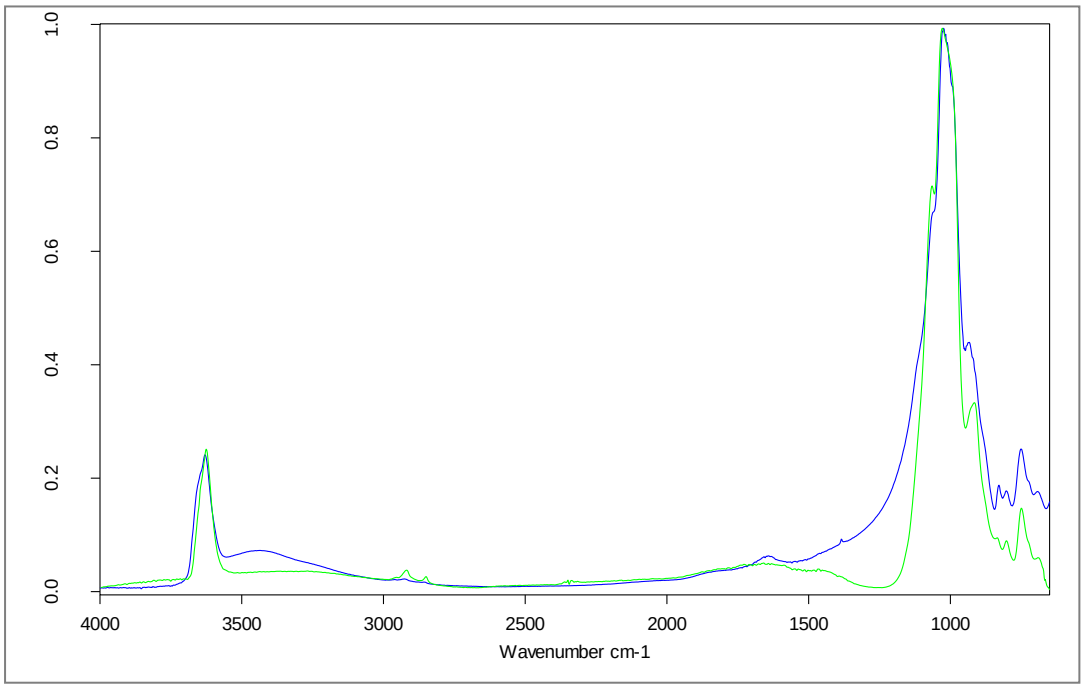


Figure 20: FT-IR spectrum of the analyzed sample (green spectrum) and reference FT-IR spectrum of muscovite (blue spectrum)

Both the SEM-EDX and the FT-IR analysis indicate that the transparent material is 'mica', a mineral composed of potassium aluminum silicates, that cleaves into thin, flexible sheets ("Muscovy glass").

Mica is known as a stable material when exposed to light, moisture and extreme temperatures.

The mechanical stability of the very thin mica sheets is high (its use as 'window'-material in electrical equipment or radiation detectors follows from this property).

In contrast to these properties, the mica film in the Capuchin prayer book does show damage, it is most likely mechanical damage, perhaps caused by frequent manipulation (opening/closing)?

2.2. Textile

Sample Description

By preserving the edges when cutting out the last 128 pages of a Capuchin prayer book, a cavity in the book was created, which was then turned into a miniature reliquary. One side could be described as a pigeonhole storage for approximately sixty relics, protected behind a slightly damaged transparent film, and concealed behind two 'wings' in bright green/red cloth (Figure 21).

Figure 21: Two fabrics and one sewing thread with indication of the sample location

The **green cloth** showed some loose yarns, both in vertical (yellow) and horizontal (green) direction (figure 22). Samples of the yellow (/k01) and green yarn (/k02) were taken for dye analysis, while another sample of the yellow silk (/v01) is used to investigate fibre degradation.

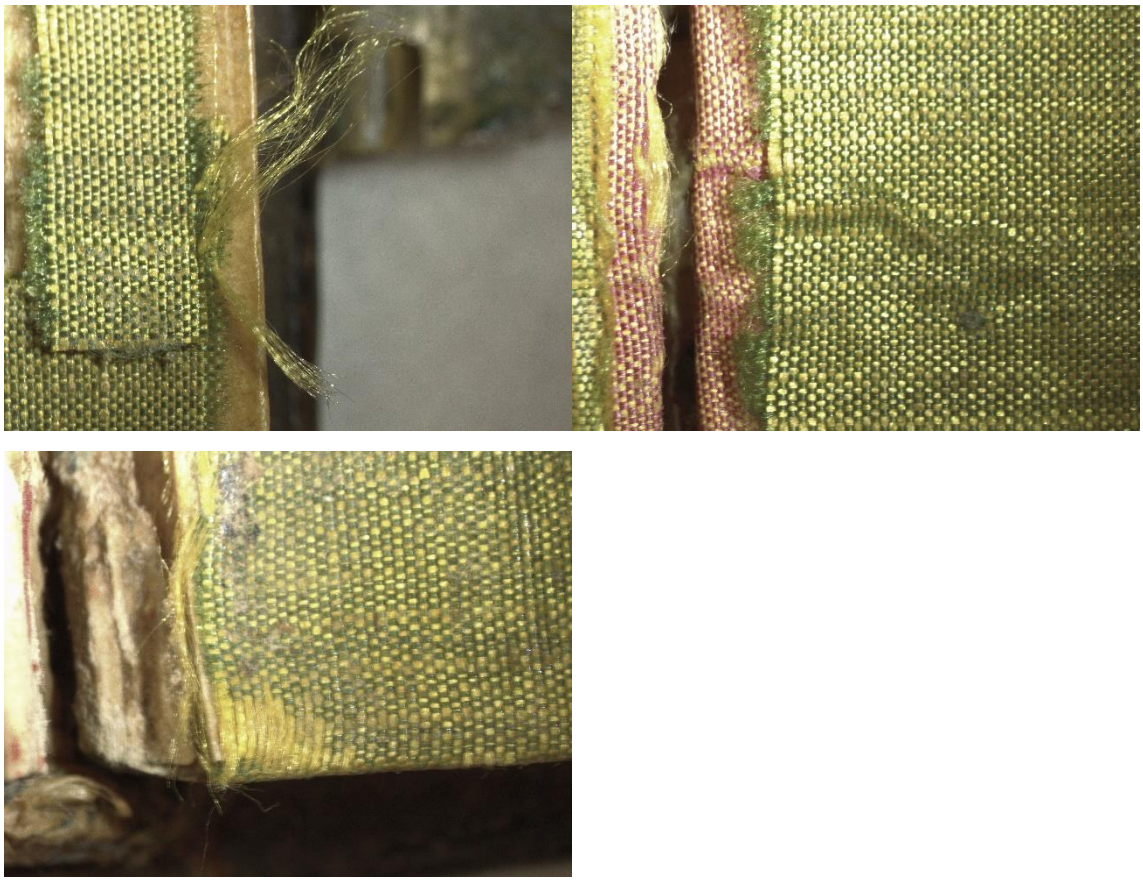


Figure 22: Images (stereomicroscope) of samples for dye identification, yellow (top left) and green (top right); sample for fibre degradation (bottom)

On page 33 of the book, **a blue sewing yarn** was spotted. A sample (/vk03) was taken to identify both dye and fibre.

Figure 23: Image (stereomicroscope) of blue yarn

Information about the samples is shown below (table 5). No images of the samples used for analysis were taken to avoid loss of the already small samples.

Table 5: Overview of the samples with indication of the object, sample description and the KIK sample code and type of analyses

Object	Sample description	KIK/IRPA code	Analysis / Techniques
Green fabric	Yellow silk	14438/k01	Dye identification (HPLC-DAD)
	Yellow silk	14438/v01	Silk degradation (HPLC-FL, FT-IR, MRS)
	Green silk	14438/k02	Dye identification (HPLC-DAD)
Blue sewing yarn	Blue yarn	14438/vk03	Dye identification (HPLC-DAD) Fibre identification (OM)

2.2.1. Methodology

HPLC-DAD

The identification of the organic colorants is performed by High Performance Liquid Chromatography and photo diode array detection system (HPLC-DAD) with Alliance HPLC equipment (Waters, USA). The analyses are interpreted using the Empower software system from Waters. A detailed description of the analytical protocol was published before (Vanden Berghe et al. 2009). The colorants are recovered from the fibres using acidic extraction with hydrochloric acid (HCl)[3]. Hydrochloric acid extraction was preferred to extract the dyes, as to identify a very wide range of organic dyes, either natural or (half) synthetic, by comparison with spectra from the in-house developed textile colorant reference database. Preliminary to the analysis, the samples are examined under binocular in order to avoid any visible surface contamination.

The result of the HPLC-DAD analyses is listed in Table 6. The first column comprises the code of the sample given by KIK-IRPA, followed by the sample colour in the next column. The type of extract analysed and the analysis code are mentioned in the third and fourth columns. The results of the chromatographic analyses are given in the following two columns. The dye composition mentioned in column five is expressed as relative proportions of the dye constituents after calculation of their peak area measured at the wavelength (nm) mentioned in column six.

Fibre

Fibre identification is carried out with optical microscopy under transmitted or polarizing illumination (OM, AxioImager M1, Zeiss). For this, a few fibres are taken from each sample. Prior to the analysis, the sample is examined under reflective light using the digital microscope KH 8700 from Hirox.

The identification of the fibres is carried out on the basis of the fibre morphology in longitudinal view. Vegetable fibres are identified by their diameter and the presence of characteristic properties (1967; Von Bergen and Krauss 1945). Animal fibre are characterized on the basis of their diameter, the presence of a cuticle with specification of the scales, the cortex (pigmented or not) and the presence or absence of a medulla and the type (Petraco and Kubic 2004). If relevant, the medullary index is also determined, based on of which a distinction can be made between hair of animal and human origin (Kshirsagar et al. 2009).

The morphology of the fibres is then compared with that of reference fibres derived from published atlases (1967; Von Bergen and Krauss, 1945), online atlases and / or the internal KIK database (Vanden Berghe).

Identification between the different bast fibres is based on their differences in fibrillar orientation. This test is known as the modified Herzog or red plate test (Petraco & Kubic 2004; Haugan 2013).

[3] Extraction in 250 µL water/methanol/37% HCl (1/1/2, v/v/v) for 10 minutes at 105°C - vacuum evaporation - dissolving the residue in 30/30 µL methanol/water from which 20 µL is injected

2.2.2. Results

Table 6. Result HPLC-DAD analyses. Detected dye composition

KIK code	Colour	Extr.	Analysis n°	Dye composition	λ (nm)
14438/k01	Yellow	HCl	01/210308/05	4 mphb, 32 genistein, 57 luteolin, 4 apigenin, 1 chrysoeriol	255
				93 luteolin, 2 apigenin, 2 chrysoeriol	350
14438/k02	Green	HCl	01/210308/06	2 isatin, 17 genistein, 65 luteolin, 3 apigenin, 10 chrysoeriol, 3 indigotin	255
				83 luteolin, 4 apigenin, 13 chrysoeriol	350
				36 isatin, 64 indigotin	288
14438/k03	Blue	HCl	01/210308/07	26 isatin, 74 ellagic acid	255
				100 isatin	288
				No peaks detected	600

The identification of luteolin, apigenin and genistein is a clear indication of the use of luteolin based yellow dye source. Due to the good preservation of the less stable compound genistein, it is possible to define the exact plant source as dyer's broom, also known as dyer's greenweed (*Genista tinctoria* L.) (Cardon 2007). It is the dye source used in the yellow silk and it is also found in the green silk, where it was combined with a blue dyeing with an indigoid dye source (indigo or woad). The latter can be derived from the detection of indigotin and isatin.

In the blue sewing yarn, ellagic acid was the main compound detected which refers to the presence of ellagitannins, hydrolysable tannins which can derive from a wide range of plant sources (Cardon 2007). In this case, tannins were probably used to obtain a smooth sewing thread. As the sample was very small and not fully dyed (containing white/beige fibres), isatin was the only compound indicating the presence of an indigoid dye source (indigo or woad).

The blue sewing yarn shows the characteristics for bast fibres. The modified Herzog test shows an fibre-orientation in S direction. This confirms that the sewing yarn is made of flax (*Linum usitatissimum* L.) (figure 24).

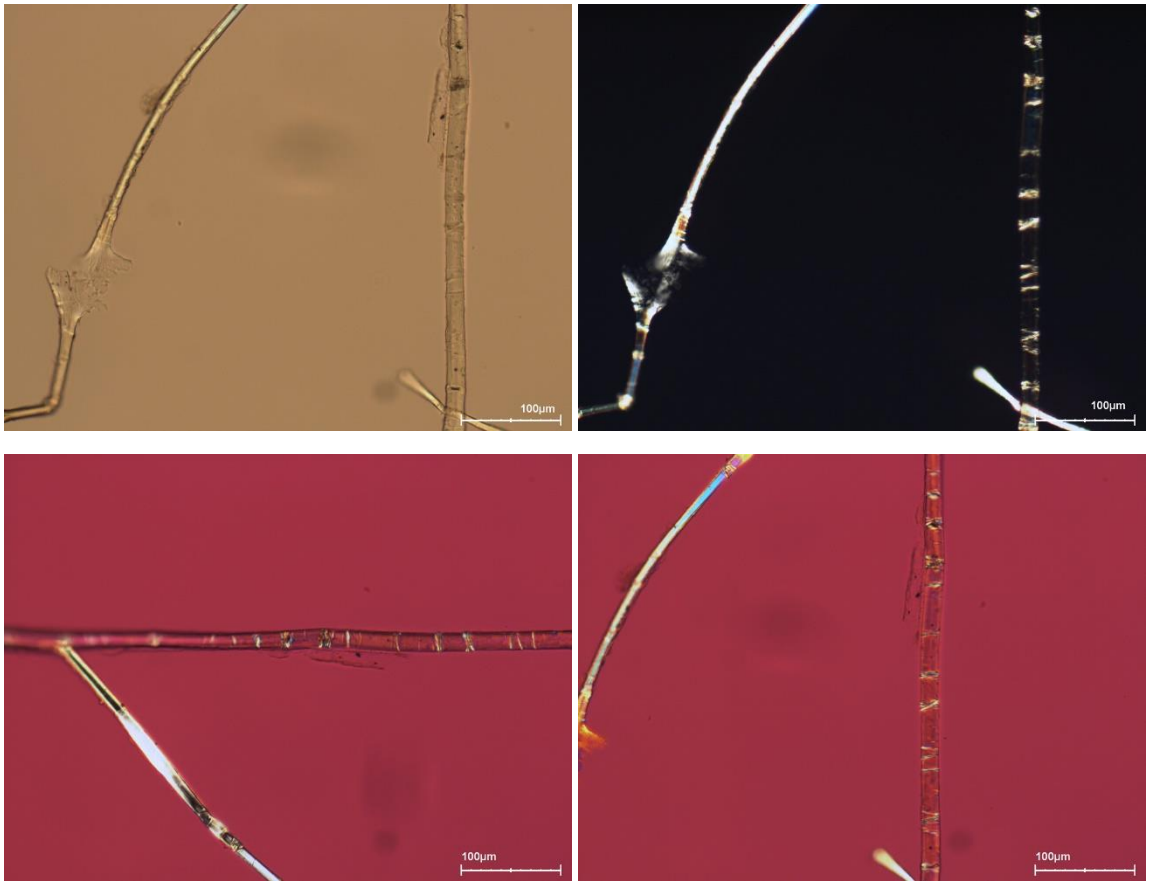


Figure 24: (top) microscopic images of sample 14438/v03 under transmitted light (left) and with the use of the polarisation filter (right) (magnification 200x); (bottom) red plate test images, the sample is blue in horizontal direction (left) and red in vertical direction (right) (magnification 200x)

3. Conclusion

A combination of non-invasive and invasive analyses were performed on the tiny Capuchin prayer book with relics to identify the materials.

On the side where eighteen relics are on display in a scenery of a bloodied Christ seated on an empty tomb before the Cross, vermilion was used to write the red text, for the blood on Christ, his red cloak and also for the red arches around the relics. Carbon black was identified as the black writing ink both on the cross and for the names of the saints. It was also used for the black arches around the relics. For the colour of the tomb, a combination of copper green and gold was used. Copper green was also identified for the green torn wreath, raman analyses showed also the use of carbon black, white lead and chalk. For the incarnation white lead was used. An organic dye was used to color the yellow banner, FORS analysis points in the direction of gamboge. The cross was also painted with an organic dye, details are in gold paint except for the nails that are in silver paint. The red cloak of Christ has also some details in gold paint, his nimbus is also painted gold. Raman analysis of the spearhead points in the direction of azurite and carbon black.

All these objects are mounted on a blue silk, since there was no damage visible only non-invasive measurements were performed. Both FORS and Raman analysis identified indigo.

The other side shows a pigeonhole storage for the approximately sixty other relics, protected behind a slightly damaged transparent film, and concealed behind two 'wings' in bright green/red cloth. After sampling, the transparent film was identified as mica, a mineral composed of potassium aluminum silicates, that cleaves into thin, flexible sheets ("Muscovy glass"). Mica is known as a stable material when exposed to light, moisture and extreme temperatures. The mechanical stability of the very thin mica sheets is high (its use as 'window'-material in electrical equipment or radiation detectors follows from this property). In contrast to these properties, the mica film in the Capuchin prayer book does show damage, it is most likely mechanical damage, perhaps caused by frequent manipulation (opening/closing).

The green cloth showed some loose yarns, both in vertical (yellow) and horizontal (green) direction. Samples of the yellow and green yarn were taken for dye analysis, while another sample of the yellow silk is used to investigate fibre degradation (see other report). Dyer's greenweed (*Genista tinctoria* L.) is the dye source used in the yellow silk and it is also found in the green silk, where it was combined with a blue dyeing with an indigoid dye source (indigo or woad). It was not possible to sample the red silk, but FORS analysis identified the red as carmine lake.

On page 33 of the book, a blue sewing yarn was spotted. A sample was taken to identify both dye and fibre. Ellagic acid was the main compound detected which refers to the presence of ellagitannins, hydrolysable tannins which can derive from a wide range of plant sources. In this case, tannins were probably used to obtain a smooth sewing thread. As the sample was very small and not fully dyed (containing white/beige fibres), isatin was the only compound indicating the presence of an indigoid dye source (indigo or woad). Raman and FORS analysis also identified indigo. Fibre analysis confirms that the sewing yarn is made of flax (*Linum usitatissimum* L.).

To conclude, XRF-analysis on the cover of the book identified a brass clamp and leather blackened by iron.

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