
Article:
Calà, Elisa, Agostino, Angelo, Fenoglio, Gaia et al. (5 more authors) (2019) The Messale Rosselli:Scientific investigation on an outstanding 14th century illuminated manuscript from Avignon. Journal of Archaeological Science: Reports. pp. 721-730. ISSN: 2352-409X

https://doi.org/10.1016/j.jasrep.2018.12.001

Manuscript Details

Manuscript number	JASREP_2018_289_R1
Title	The Messale Rosselli: scientific investigation on an outstanding 14th century illuminated manuscript from Avignon
Short title	Scientific investigation on the Messale Rosselli
Article type	Research Paper

Abstract

The manuscript D.I.21 kept at Biblioteca Nazionale Universitaria in Torino, better known as Messale Rosselli, is one of the richest fully illustrated missals surviving from the mid-14th century. It was produced in Avignon for the Aragonese Cardinal Nicolas Rossell (1314-1362) but after the patron's death, it passed from hand to hand until it reached its final destination in Torino. The Messale Rosselli has recently been the object of a thorough interdisciplinary study, involving full characterisation of the colourants with non-invasive techniques (FORS, fluorimetry, XRF spectrometry, optical microscopy, IR photography). The full set of colourants was identified, highlighting the systematic use of precious pigments such as lapis lazuli, cinnabar and gold, a feature reinforcing the symbolic value of the manuscript; in addition, less valuable but interesting dyes such as brazilwood and folium were also identified, used either pure or in a mixture with pigments in order to obtain a wide range of hues. The palettes used by the various artists have been evaluated according to the availability of raw materials in the geographic area around Avignon, finding that most of the colourants could be at easy disposal of the artists. Information has also been obtained concerning the preparation of the parchment. The systematic measurement of the width of folios allowed hypothesising the number of the animals slaughtered to produce parchment, and the way of using skins. XRF analysis on the folios suggested that different preparations were used. Finally, ZooMS, a non-invasive technique able to provide information on the animal species from which parchment was produced, evidenced that calf and goat, but not sheep, were used to produce the parchment of the Messale Rosselli.

Keywords	Manuscripts; Avignon; FORS; XRF; non-invasive; eZooMS; folium.

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Suggested reviewers	Marcello Picollo, pietro baraldi, Paola Ricciardi, Abigail Quandt, Cheryl Porter

Submission Files Included in this PDF

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Data will be made available on request

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to the attention of Editorial Board of Journal of Archaeological Science: Reports

Alessandria, 10/23/2018

Object: *Submission of revised paper to Journal of Archaeological Science: Reports*

Dear Sir,

The paper "The *Messale Rosselli*: scientific investigation on an outstanding 14th century illuminated manuscript from Avignon" by Elisa Calà, Angelo Agostino, Gaia Fenoglio, Valerio Capra, Franca Porticelli, Francesca Manzari, Sarah Fiddyment and Maurizio Aceto has been revised following strictly the indication of the two referees. All issues have been addressed; in the main text, corrections have been highlighted in yellow. We believe that the present version is suitable for publication.

Looking forward to hear news from you

Best wishes Prof. Maurizio Aceto

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Comments from the editors and reviewers:

- **Reviewer 1**

This paper presents the results of the non-destructive diagnostic campaign, without any sampling, carried out on the Messale Rosselli. The paper presents itself as a case study aimed at the characterization of palette and decoration technique of the whole masterpiece.

Moreover, the study has its peculiarity in the fact that a new technique as the EZooMS one has been applied to provide information on the animal species used for the parchment production. The work is well written; the division into paragraphs is very clear and the contents are explained.

However, for the palette characterization important information are missing (see below).

The English is correct.

Some modifications need to be performed before publication. Namely:

Q1. The acronym of the Electrostatic Zooarchaeology by Mass Spectrometry must always be written in the same way (ZooMS in the abstract; zooms in the keywords; eZooMS in the introduction…).

A1. We changed to eZooMS for all instances.

Q2. In the paragraph 3a there is a relevant discussion about the depth of the foils, but in the text no information about how this measurement has been performed. Please add all the information about it.

A2. The information on the measurement of parchment thickness has been added.

For the palette:

Q3. Blue: how you individuate the woad? Please specify

A3. The identification of woad instead of indigo is only based on practical considerations, not on diagnostic evidences. Due to the well-established trade of woad in the region of Toulouse, it is reasonable thinking that the artists used the local pigment and not a pigment – such as indigo – coming from Far Eastern Asia. At any rate we added the phrase "even in the lack of diagnostic evidences".

Q4. Gold: what means "assiso"? Please add in the text. How you deduce the Armenian bole presence? How you distinguish yellow bole from the Armenian one?

A4. "Assiso" is the Italian term for the material used as basis for gold lead involving chalk; I think the same term can be used in English. We added this definition in the text. The distinction of yellow bole from Armenian one by means of FORS is very simple: the first shows the spectrum of yellow ochre, while the second shows the spectrum of red ochre. Of course, they can be identified - and distinguished – only if small gaps are present in the gold leaf.

Q5. Green. You mention the use of indigo, but in the table 1 woad has been reported. Why?

A5. See answer A3.

Q6. Pink: in the text you mention cinnabar, but not in the Table 1, why?

A6. The use of cinnabar in incarnate tones was erroneously cited, we cancelled it from Pink.

Q7. Violet: please add the FORS spectra of folium and ultramarine as reference in the fig. 5. Please add the spectrum of parchment in the fig. 6.

A7. Figg. 5 and 6 have been changed following the indications of the referee; captions have been changed accordingly.

Q8. Inks: from the data shown in fig.7, you deduce that Fe/Cu ratio is 20:1: please add the equation of the interpolated straight line and add the units on the axes.

A8. The equation of the interpolated straight line and the units on the axes have been added to Fig. 7.

Q9. The different palettes: for the pigments mentioned in table 1, please provide the techniques used to determine their presence and which is the element, or emission band or… peculiar for each pigment identification (e.g. EDXRF detects the Hg in the red pigment and you can deduce that there is cinnabar…).

A9. This information was added for every colourant identified.

Q10 - Details from FT-IR photography: are you sure that the technique used is FT-IR photography?

A10. It is, indeed, IR photography, we changed the term.

Q11 - Reading the bibliography it is easy to understand the identity of the authors because 7 references out of 17 are from the same research group; please, delete / replace some references.

A11. This comment is not clear to us, please specify.

The paper can be published on JASREP after a major revision

- **Reviewer 2**

The paper presents the results of an interdisciplinary study performed on a 14th Century illuminated manuscript, which was deeply examined with a multi-analytical approach with the occasion of the restoration. A notable amount of data was acquired thus leading to new findings about the case-study considered. Some of these results supported some of the scholars' hypothesis about the manuscript. Since an increased knowledge has been gained with this research on the examined case-study, the work deserves attention and is worthy to be published. However, in my opinion, a main flaw of the manuscript is a weakness of rationale and organization of contents. Indeed, being the scientific approach adopted in this research well-established and based on consolidated analytical techniques, the novelty of the work mainly lies in the findings about the specific case-study examined, which is reported as an outstanding example of the 14th Century illuminated manuscript production from the Avignon area.

Q1. However, if this is the case, the Introduction should better focus this aspect, and a discussion of the results of under the broader perspective of the interdisciplinary approach should be introduced in the Results section.

A1. A brief discussion on this argument has been added in the Introduction section, and the discussion of results has been improved.

Q2. I would recommend adding in the Introduction a paragraph illustrating the specific aim of the investigations, and clearly stating the open questions raised by scholars about this manuscript, illustrating the wider historical and artistic context, etc.

A2. A short paragraph has been added in the Introduction, specifying the main aims of the scientific investigation.

Q3. In addition, since the use of non-invasive analytical techniques applied to the study of illuminated manuscripts has greatly grown in recent years, authors should dedicate a brief paragraph to this topic, with a short state of the art of other relevant studies published in the field.

A3. A brief paragraph has been added with references to the works lately published.

That said as general comment, I also found several specific issues which should be addressed to reach the standards required for publication, as reported in the detailed list below.

In conclusion I would recommend to resubmit the paper after major revisions.

Detailed list of comments and recommended changes.

1) Introduction: see comments above.

Q4. 2) P.4 l. 97, "many of the questions…". Please add further details about these questions, at least by mentioning those addressed by the scientific analysis

A4. The sentence has been improved.

3) Materials and methods.

Q5. P. 4 l. 106 and following. Please indicate the set-up adopted for the FORS measurements. Include a description of the probe head used. Rather than (or in addition to) the detector spectral range, the overall operational range of the device should be indicated (combination of lamps + detector).

A5. This information has been added in the experimental section.

Q6. P. 5 l. 150. Please include the number of samples extracted per each folium, their location and their dimension /weight.

A6. The information on samples for eZooMS analysis has been added to the text.

4) Results

Q7. P. 6, l. 205. A bibliographic reference or the source of Figure 2 has to be included, here or in the caption of figure 2.

A7. A reference has been included in the caption of Figure 2.

Q8. P. 8 from l. 227 to l. 229. Authors concede that data and samples they acquired are not sufficient to draw general conclusions about the constituting materials of all the folios. However, this motivation is not sufficient to justify a lack of conclusions in a scientific paper. If data are not enough to answer the question tackled, the data-set could be enriched. Alternatively I would suggest to omit this paragraph and present the data as preliminary result, or publish more complete results in a future publication.

A8. The knowledge on the animal source of parchment is only in its infancy at present; therefore we reputed that the result provided by eZooMS could be nevertheless of interest for scholars, despite being partial. At any rate we recognize that the data-set could be enriched in a future publication.

Q9. P. 8 l. 241. “several” is not informative for a scientific paper. The number of points acquired should be indicated P. 8 l. 241. Some” see above.

A9. The total number of XRF measurements on parchment has been added to the text.

Q10. P. 12. Fig 5 x-axis and y-axis scales are not optimised for readability. Minor ticks are uselessly too dense.

A10. We think that the readability of the figure is good as it is now.

Q11. P.15 l. 428. “The order is the following”. The reasons and observations which lead to hypothesize this sequence should be reported.

A11. The rationale on the sequence has been added to the text.

Q12. P. 16 l. 447, and l. 453 “FT-IR Photography”. This is strongly incorrect! IR Reflectography is the right name for the technique used. FT-IR is a different working principle, not used in this context.

A12. The referee is definitely right, it was a big mistake of ours.

- A precious 14th century manuscript has been analysed with different complementary techniques
- Measurements yielded information on all colourants used by ancient artists
- Different hands at work in the decoration were identified, confirming the hypothesis of scholars
- Many colourants were available in the area surrounding Avignon

The *Messale Rosselli*: scientific investigation on an outstanding 14th century illuminated manuscript from Avignon

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Abstract

The manuscript D.I.21 kept at Biblioteca Nazionale Universitaria in Torino, better known as *Messale Rosselli*, is one of the richest fully illustrated missals surviving from the mid-14th century. It was produced in Avignon for the Aragonese Cardinal Nicolas Rossell (1314-1362) but after the patron's death, it passed from hand to hand until it reached its final destination in Torino.

The *Messale Rosselli* has recently been the object of a thorough interdisciplinary study, involving full characterisation of the colourants with non-invasive techniques (FORS, fluorimetry, XRF spectrometry, optical microscopy, IR photography). The full set of colourants was identified, highlighting the systematic use of precious pigments such as lapis lazuli, cinnabar and gold, a feature reinforcing the symbolic value of the manuscript; in addition, less valuable but interesting dyes such as brazilwood and folium were also identified, used either pure or in a mixture with pigments in order to obtain a wide range of hues. The palettes used by the various artists have been evaluated according to the availability of raw materials in the geographic area around Avignon, finding that most of the colourants could be at easy disposal of the artists.

Information has also been obtained concerning the preparation of the parchment. The systematic measurement of the width of folios allowed hypothesising the number of the animals slaughtered to produce parchment, and the way of using skins. XRF analysis on the folios suggested that different preparations were used. Finally, eZooMS, a non-invasive technique able to provide information on the animal species from which parchment was produced, evidenced that calf and goat, but not sheep, were used to produce the parchment of the *Messale Rosselli*.

Keywords (3-7)

Manuscripts; Avignon; FORS; XRF; non-invasive; eZooMS; folium.

1) Introduction

a) The history of Messale Rosselli

The *Messale Rosselli* or *Rossell Missal* (*Messale* thereafter) is one of the richest fully illustrated Missals surviving from the mid-14th century (Ragusa, 1975). It was produced most probably in Avignon for the Aragonese Cardinal Nicolas Rossell (1314-1362), as indicated both by the *colophon* at the end of the manuscript (f. 423v) where the scribe Alamannus, with a riddle, indicates that the book was completed in 1361, and by a beautifully decorated note (f. Iv), at the beginning of the book (Manzari, 2006). This note was drawn in coloured inks by a gifted pen-flourisher, recognisable throughout the *Messale* and in other manuscripts produced in Avignon, identified with the illuminator Bernard de Toulouse (Manzari, 2014).

The vast illustrative programme, comprising hundreds of historiated initials at the beginning of the Masses throughout the liturgical year and fully illuminated borders highlighting the most important festivities, was painted in *tempera* by a different workshop. This was led by an artist whose style combined a basically Southern French culture – especially recognisable in the type of foliage used in the decorated letters and borders – with Catalan components, visible for example in the expressionistic elements in the full-page Crucifixion. The first quire of the body of the *Messale* (ff. 19r-26v) was illuminated by a different artist, possibly later and in an equally elegant style (Manzari, 2006).

The *Messale* stayed in the Avignon curia after Cardinal Rossell's death, which occurred in Spain in March 1362, then passing into the hands of Cardinal Guillaume de Bragose. The arms of Cardinal Bragose have been added on ff. 286r and 287v, showing that the *Messale* must have been acquired by the prelate between Rossell's death in 1362 and his own in 1367. As the other arms, present on the *incipit* page (f. 19r), are surmounted by a crozier topped with a cross, a third owner can be identified with the Archbishop Pierre II de Cros, who must have bought the manuscript between 1370, when he was named Archbishop of Bourges, and 1383, when he became a Cardinal (Manzari, 2006). After that, it was reported in a Franciscan monastery in Pinerolo (northern Italy) in 17th century and eventually in the Savoy ducal library in Torino, from where it reached the Regia Biblioteca Universitaria (now Biblioteca Nazionale Universitaria) after the foundation of this library by Duke Vittorio Amedeo II di Savoia.

The arms of the original owner, Cardinal Rossell, have been overpainted in many borders, usually with decorative elements and by the same workshop responsible for the illustrative programme, perhaps in preparation for its sale on the market, after the patron's sudden death. Cardinal Rossell's arms survive, however, in certain cases, such as an initial with St. John the Evangelist (f. 34r), or in some of the illuminated borders, where they were frequently accompanied by the arms of the Crown of Aragon (Manzari, 2006).

b) The interdisciplinary study

It was opinion of the authors of this study that some open questions raised by scholars about the manuscript could be addressed by means of analytical investigations, with particular concern to the identification of the different hands at work in it and to the chemical nature and geographic provenance of the colourants used. Therefore, taking the opportunity given by its restoration, the *Messale* has recently been the object of a thorough interdisciplinary study, involving full characterisation of colourants, inks and parchment with non-invasive

techniques. The multi-techniques approach applied to the study of illuminated manuscripts has greatly grown in recent years. Elemental, molecular and imaging techniques are combined in order to yield as most complete information as possible concerning the materials and the techniques used by ancient artists. Considering only the last two years, good examples are the studies by Cucci et al. (2018), Fruhmann et al. (2018), Legrand et al. (2018), Mounier & Daniel (2017) and de Viguerie et al. (2018). In this study, a combination of molecular (UV-visible diffuse reflectance spectrophotometry with optic fibres and Spectrofluorimetry), elemental (X-Ray Fluorescence spectrometry) and visual (optical microscopy) techniques yielded a vast amount of information; in addition, the application of mass spectrometry using the eZooMS (electrostatic Zooarchaeology by Mass Spectrometry) method provided information on the preparation of parchment.

The combination of the cited techniques allowed addressing many of the questions raised by scholars, in particular those concerning the story of the *Messale* and its changes of ownership , its manufacture and its geographic provenance in relation with the raw matters used, its symbolic value and more generally its role inside the production of precious books in Avignon.

2) Materials and Methods

a) UV-visible diffuse reflectance spectrophotometry with optic fibres (FORS)

FORS analysis was performed with an Avantes (Apeldoorn, The Netherlands) AvaSpec-ULS2048XL-USB2 model spectrophotometer and an AvaLight-HAL-S-IND tungsten halogen light source; detector and light source were connected with fibre optic cables to an FCR-7UV200-2-1,5x100 probe. In this configuration, both the incident and detecting angles were 45° from the surface normal, in order not to include specular reflectance. The spectral range of the detector was 200-1160 nm; the overall operational range of the device (combination of lamp + detector) was 375-1100 nm. Depending on the features of the monochromator (slit width 50 µm, grating of UA type with 300 lines/mm) and of the detector (2048 pixels), the best spectra resolution was 2,4 nm calculated as FWHM. Diffuse reflectance spectra of the samples were referenced against the WS-2 reference tile provided by Avantes and guaranteed to be reflective at 98% or more in the spectral range investigated. The investigated area on the sample had a 1 mm diameter. The probe was inserted into an aluminum block, in order to exclude external light and to hold firmly the probe in place. During analysis, the block is laid on the sheet; therefore the side in contact with the manuscript was covered in Tyvek®, a soft tissue. In all measurements the distance between probe and sample was kept constant to 2 mm. To visualise the investigated area on the sample, the probe contained a USB endoscope inserted as well in the block. The instrumental parameters were as follows: 10 ms integration time, 100 scans for a total acquisition time of 1.0 s for each spectrum. The whole system was managed by means of AvaSoft v. 8 dedicated software, running under Windows 7™.

b) X-Ray Fluorescence spectrometry (XRF)

XRF measurements were performed with an EDXRF Thermo (Waltham, USA) NITON spectrometer XL3T-900 GOLDD model, equipped with an Ag tube (max. 50 kV, 100 µA, 2 W), a large area SDD detector, energy resolution of about 136 eV at 5.9 keV. Analysed spot had an average diameter of 3 mm and was focused by a CCD camera, with a working distance of 2 mm. Total time of analysis was 240s. The instrument is held in position with a moving

stage allowing micrometric shifts, in order to reach the desired probe-to-sample distance; the stage is laid on a tripod. The obtained spectra have been processed with the commercial software WinAxil, derived by the academic software QXAS from IAEA.

c) Spectrofluorimetry

An Ocean Optics (Dunedin, Florida, USA) Jaz model spectrophotometer was employed to record molecular fluorescence spectra. The instrument is equipped with a 365 nm Jaz-LED internal light source; a QF600-8-VIS/NIR fibre fluorescence probe is used to drive excitation light on the sample and to recover the emitted light. The spectrophotometer works in the range 191-886 nm; according to the features of the monochromator (200 µm slit width) and detector (2048 elements), the spectral resolution available is 7.6 nm calculated as FWHM. The investigated area on the sample is 1 mm in diameter. In all measurements the distance between probe and sample was kept constant to 12 mm, corresponding to the focal length of the probe. To visualise the investigated area on the sample, the probe contained a USB endoscope. Instrumental parameters were as follows: 2 s integration time, 3 scans for a total acquisition time of 6 s for every spectrum. The system is managed with SpectraSuite™ software under Windows 7™.

d) Optical Microscopy

A USB Dino-Lite (New Taipei City, Taiwan) AM4113T-FV2W model microscope was used to acquire digital images at 50x and 200x magnification ratios. The instrument is equipped with 375 nm and visible LED lights and a digital camera with 1.3 Megapixel resolution.

*e) electrostatic Zooarchaeology by Mass Spectrometry (*eZooMS*)*

Seventeen folios of the *Messale* were sampled using the dry non-invasive eraser-based sampling technique of Fiddyment *et al.* (2015) for protein analysis. One eraser sample per folio was taken from the border of the page over an area of approximately 2 cm^2. Samples were analysed using the eZooMS methodology following the protocol of Fiddyment *et al.* (2015). Briefly, samples were incubated in 75 µL of 0.05 M NH_4HCO_3 (AmBic) buffer (pH 8) with 1 µL of trypsin (0.4 µg/µL) at 37 °C for 4 h. Samples were desalted and concentrated using Pierce™ (Thermo Fisher Scientific, Waltham, USA) C18 resin, following the manufacturer's instructions. Peptides were eluted in a final volume of 50 µL of 50% acetonitrile/0.1% trifluoroacetic acid (vol/vol). Samples were spotted in triplicate and analysed using a calibrated Bruker Daltonics (Bremen, Germany) Ultraflex III NLD1 model MALDI-TOF-MS instrument in reflector mode. Spectral analysis was performed using the open-source cross-platform software mMass (www.mmass.org) (Strohalm et al., 2010).

f) IR photography

IR pictures were taken with a Canon (Tokyo, Japan) EOS 20D camera equipped with an infrared filter blocking light below 750 nm. Lighting was obtained by means of 2 Profoto (Sundbyberg, Sweden) flashes free from UV components.

g) Thickness measurements

Thickness measurements on parchment were taken with a Vogel (Leno, Italy) Käfer model analogue thickness gauge with reading 0.01.

3) Results

a) The structure of Messale Rosselli

The *Messale* is composed of 425 leaves with size 383x278 mm, divided into 57 quires: 51 quaternions, 3 ternions, 1 binions, 1 bifolium, 1 single leaf for a total of 216 bifolia. It is divided into two main sections: (1) a Calendar with the most important liturgical feasts, containing illustrations of the labours of the months and signs of the zodiac (ff. Ir-VIIv); (2) the main part containing the texts necessary for the performance of the mass (ff. 1r-425v). The true beginning of the main part is actually the quire at ff. 19r-26v, containing the liturgies for First Sunday of Advent; this quire was apparently decorated by an artist different from the others, particularly accomplished and possibly later.

The parchment is very smooth and was apparently prepared according to the transalpine style (Maniaci, 1996) which, after a fine and accurate working, rendered the hair and the flesh sides indistinguishable.

In order to evaluate the building sequence of the manuscript, thickness measurements were taken into 5 points of every leaf: 4 at the corners and 1 in the centre of the leaf. The 4 corner points gave average values comprised between 0.1644 and 0.1695 mm, while the central points yielded an average value of 0.1709 mm, slightly but systematically higher than the corner points: this means that the two central points of a bifolium (Fig. 1) corresponded to the back part of a beast, and suggests that one skin, after trimming, provided one bifolium; therefore the building of the *Messale* required not less than 216 beasts to be slaughtered.

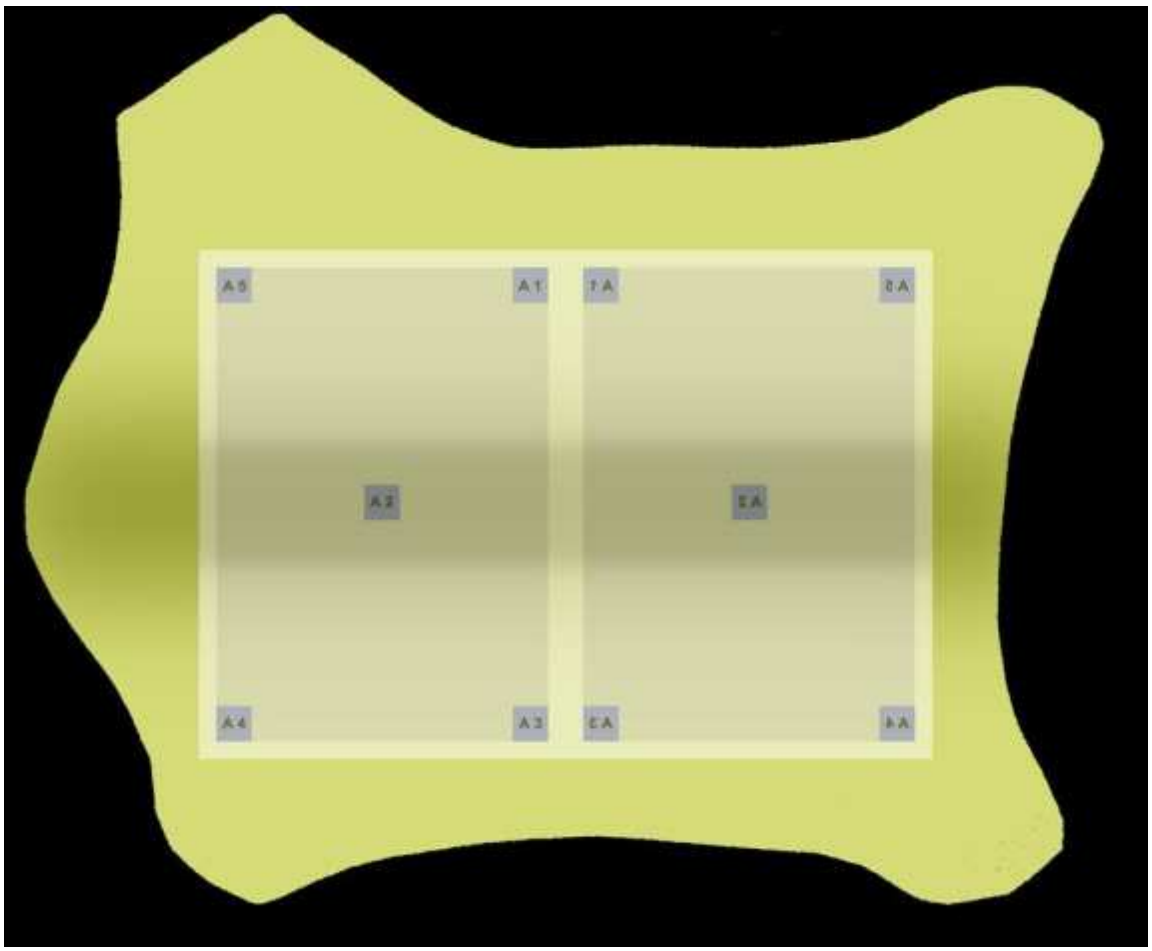

Fig. 1 - Reconstruction of the making of a bifolium from a skin

A different situation was found in the bifolium containing ff. 206-207, located at the very centre of the manuscript: in this case the thickness ranged between 0.22 mm and 0.35 mm. This must be regarded, however, as a particular bifolium as it contains the most important miniatures of the whole manuscripts, most probably realised by the main artist.

b) Analysis of the parchment with identification of the animal source

In the study on the *Messale* the previously developed non-invasive eZooMS technique was used to analyse a small collection of samples (n = 17). From this analysis we have identified 7 folios to be made from calf parchment (41%) and 10 folios to be made from goat parchment (59%). The presence of both goat and calf, although intriguing, fits well into our geographic distribution as the *Messale* is thought to be produced in Avignon, a location with influences from both France (predominance of calf parchment) and Italy (predominance of goat parchment) (Fiddyment et al., 2015). When comparing specifically to contemporary documents (14th century) we can see that these trends are still observable (Fig. 2).

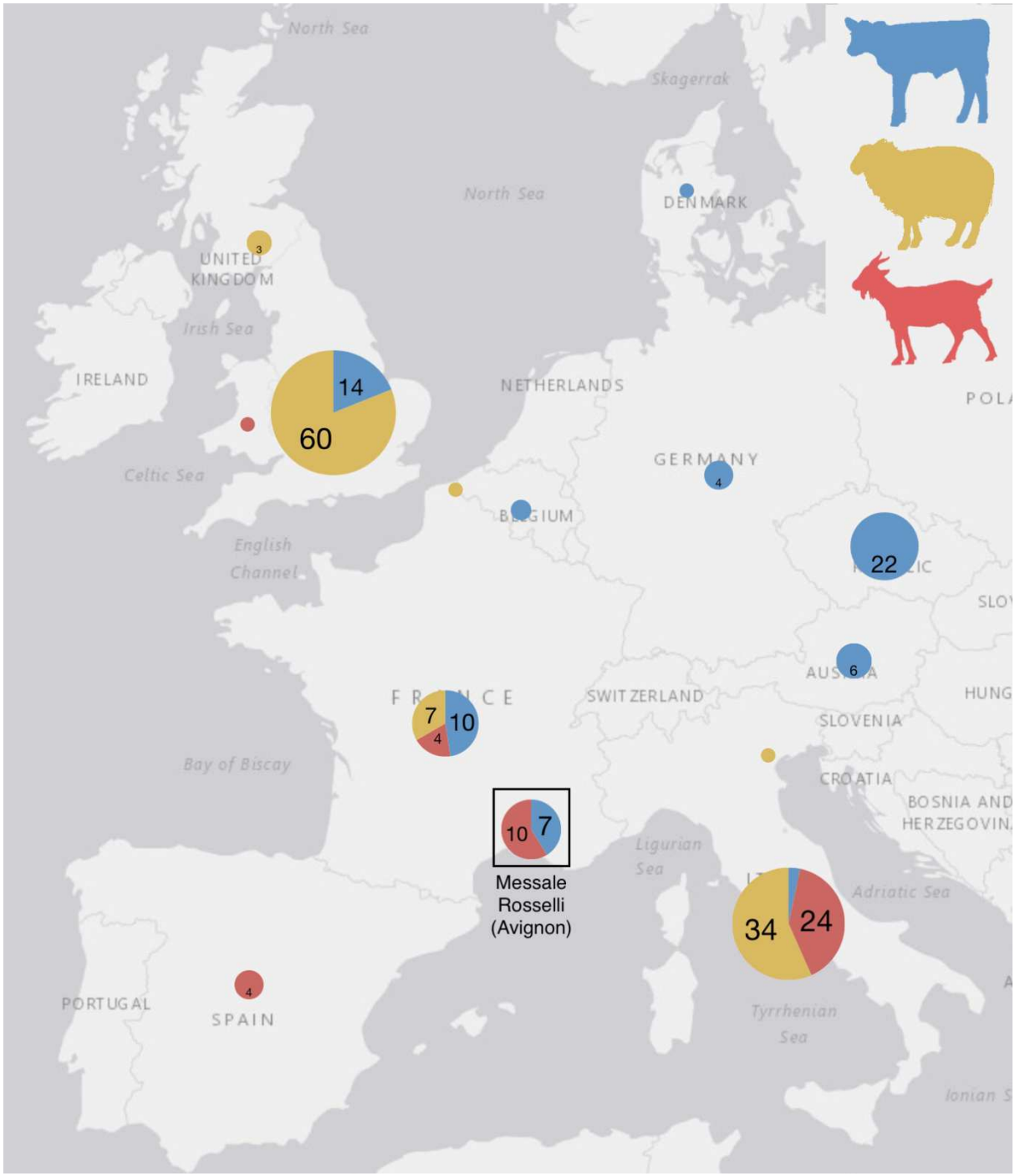

Fig. 2 - Geographic distribution of 14th century European parchment samples including 17 folios sampled from the *Messale Rosselli.* Map is based on data published in Fiddyment et al. (2015).

The absence of sheep is not unexpected due to the nature of the document, a missal, which is considered to be a more prestigious and personal object, and a manuscript usually made with the most precious material as it was to be used on the altar, together with the chalice and paten, and would therefore be made from finer quality materials. We know from accounts at Beaulieu Abbey (Gullick, 1991) that even the best prepared sheep parchment was cheaper

(and presumably considered inferior) to even the worst prepared calfskin parchment, highlighting the prestige attached to the latter. It is interesting to note that the distribution of the animal species is mixed through the document (Fig. 3), with the first folios sampled (1 and 7) being made from goat, followed by a section predominantly made from calf (with the exception of bifolium 206-207 which is also goat) and finally the second half of the manuscript seems to be exclusively goat once more.

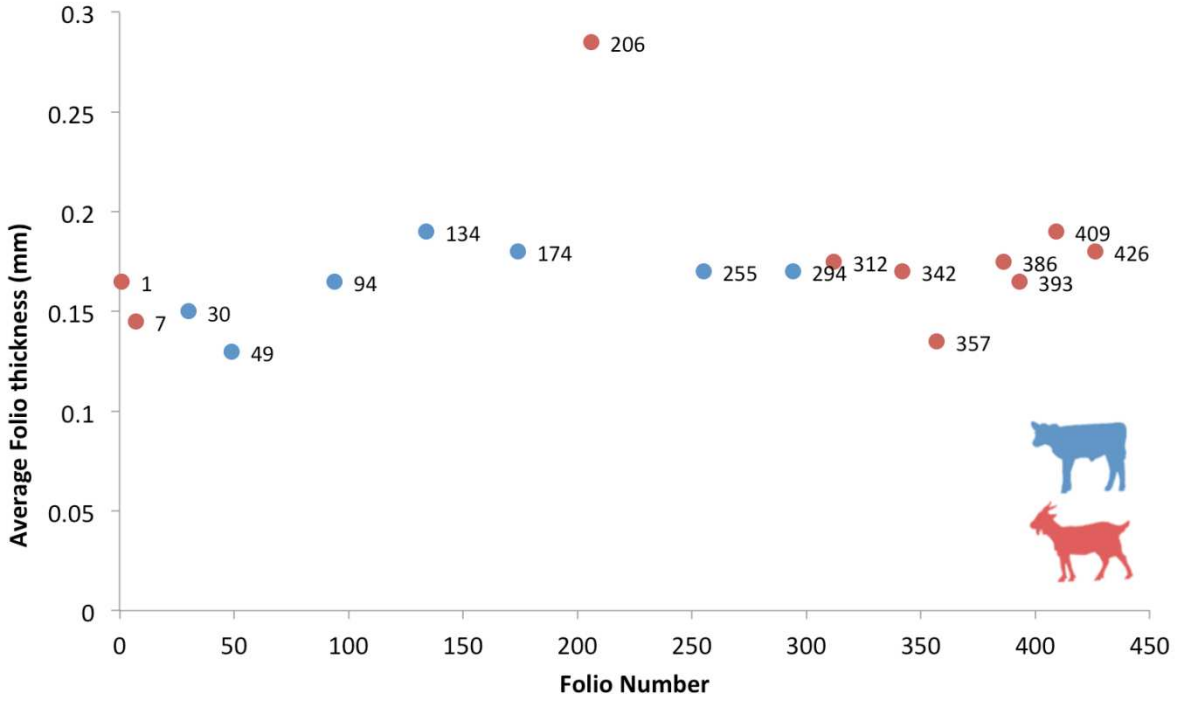

Fig. 3 - Species identification and average thickness of 17 folios sampled from the *Messale Rosselli*

However, as we have not sampled all the folios in the *Messale*, we cannot say whether this is definitely the case or if there is a more structured pattern of alternation that we cannot currently observe with this limited number of samples; the data-set will be enriched in a future publication. Regarding the thickness of the parchment, there doesn't seem to be a notable difference between the calf and goat parchment with the exception of bifolium 206-207 which is significantly thicker than any other folio sampled, as stated before. Although it is identified as goat this is not reason enough to warrant this thickness as the other folios identified as goat are much thinner and present similar thicknesses to the calfskin folios. This might indicate a problem with manufacturing where the skin was not shaved enough or could possibly indicate a problem with the skin itself. However, given that this is also the folio which contains the most important miniatures, it is likely that it may have been a deliberate choice to use a thicker parchment to provide a more stable substrate on which to paint.

c) Elemental analysis of the parchment

40 XRF measurements were taken on the parchment folios in order to obtain information on the preparation of parchment itself. The presence of specific elements, in fact, could suggest that particular compounds or treatments had been applied for rendering the surface smooth and ready for writing/painting. The results of this survey highlighted that the parchment

makers did not use one single treatment but rather different kinds of treatments. Apart from Ca which is ubiquitous and is due to the traditional use of lime, significant amounts of Pb suggested the use of *lead white* – $(PbCO_3)_2 \cdot Pb(OH)_2$ - generally employed as white pigment but in this case used to compact collagen fibres; the combined presence of Si, Al and K, instead, suggested the use of clay materials, possibly to remove the residual greasiness of parchment. The different treatments, however, are not related to the different animal source (cow vs. goat). It is apparent, then, that the makers of the *Messale* purchased different sets of parchment folios, maybe from different *pergamenarii*, e.g. parchment makers.

d) The miniatures

The restoration of the *Messale* was a very good opportunity for carrying out non-invasive measurements, due to the fact that it was completely unbound into quires and therefore all interesting features (miniatures, inks, pen-work decoration, etc.) were much more available for probing. The decorative apparatus of the *Messale* is breath-taking for its richness: it is composed of 2 full-page miniatures (at ff. 206v and 207r), 15 historiated frames, 285 historiated initials and 144 initials decorated with pen-work. In the following, the colourants identified with non-invasive measurements will be discussed, trying to highlight the differences among the different hands who worked at the decoration.

i) Black

Carbon-based pigments were used for black hues all through the *Messale*. It was not possible to define the exact type of pigment; only *bone black* can be excluded, according to the lack of phosphorus evidenced by XRF analysis.

ii) Blue

One of the most striking features of the *Messale* is the wide use of *ultramarine blue*, the precious pigment made from lapis lazuli stone, identified according to the absorption band at 600 nm in FORS spectrum. According to Delamare (2013) in the late Middle Ages the price of lapis lazuli on the Paris market was equivalent to its weight in gold. There was only one known source, the mines of Badakshan (modern north-western Afghanistan), from which the stone after a long trip, through the harbours of the near-eastern Asia coast, reached Venice and eventually Europe.

Another blue pigment used on the *Messale*, identified according to the absorption band at ca. 660 nm in FORS spectrum, was *indigo*, the organic colourant extracted from *Indigofera tinctoria*, a plant native to south-eastern Asia, or from *Isatis tinctoria*, a plant native to Asia but widely cultivated in Europe; in this case the colourant is termed *woad*. In the *Messale* it is obviously pointing to the use of woad rather than indigo (even in the lack of diagnostic evidences), considering that the cultivation of *Isatis tinctoria* represented in Middle Ages the richness of the area called *Pays de cocagne*, the triangle between Toulouse, Albi and Carcassonne, very close to Avignon. Woad was used in the *Messale* mostly in mixtures: with yellow ochre for obtaining greens, with brazilwood for obtaining violet-purplish backgrounds.

iii) Brown

All brown hues were obtained with *red ochre*, identified according to the absorption band at ca. 850 nm and the inflection point at ca. 580 nm in FORS spectrum. The use of iron oxide pigments is absolutely common in painting; nevertheless it is worth noting the availability of high quality ochres at Roussillon (Vaucluse department), less than 50 Km from Avignon.

iv) Gold

To assess the value of the *Messale*, it would be sufficient to evaluate the huge number of features decorated with gold, which account to almost 700. Gold was identified according to the inflection point at ca. 510 nm in FORS spectrum and characterised with XRF analysis. Some features are made with gold shell, such as clothes of important characters and the beautiful interweaving on the background of several historiated initials. Most of gold features are made in leaf and particularly relevant is the fact that gilding was carried out using different preparations. Rather than to the action of various artists, this variety of preparation could be due to the desire of the main artist to obtain different final hues of gold or to adapt the preparation to the morphology of the parchment. The combination of XRF, yielding information on the elements present below gold leaf, and FORS analysis carried out on leaf losses, was suitable in elucidating this aspect. All the preparations were of the *assiso* type, that is involving the use of a small layer of chalk. The following preparations could be identified:

- *assiso* with gypsum: the presence of gypsum below gold is suggested by XRF analysis which evidences Ca, S and Sr; the last element has been recently suggested (Franceschi & Locardi, 2013) as a marker for natural gypsum;
- *assiso* with gypsum and Armenian bole;
- *assiso* with gypsum, lead white and Armenian bole;
- *assiso* with gypsum, Armenian bole and cinnabar;
- *assiso* with yellow bole, cinnabar and lead white;

v) Green

Three different green paints were identified, which characterised three different hands working on the *Messale*. In the Calendar part, the painter used a mixture of ultramarine blue and yellow ochre; this last can be hypothesised according to the detection of iron by means of XRF. In the main part, instead, the artists used indigo/woad (in FORS spectrum the absorption band at ca. 660 nm is well detectable) mixed with yellow ochre. Finally, only in the central bifolium (ff. 206v and 207r) which is assigned to the main artist, all greens are made with *verdigris*, a synthetic pigment largely used in miniature painting; verdigris was identified according to the absorption band at ca. 720 nm and the detection of copper by means of XRF. Rather than indicating three different hands, this variety could mean that the main artist wanted to give different symbolic value to the different features depicted.

vi) Grey

Grey parts were mostly painted with a mixture of lead white, suggested by the detection of Pb by means of XRF, and a carbon pigment. Some features, however, were painted with silver, identified by XRF. In some cases, such as for the helmets of soldiers at f. 206v (Fig. 4), small Ag leaves were probably used, perhaps applied on a yellow bole, lead white and cinnabar according to the underlying presence of yellow ochre (identified according to the absorption band at ca. 900 nm in FORS spectrum), Pb, Hg and S as detected by means of XRF.

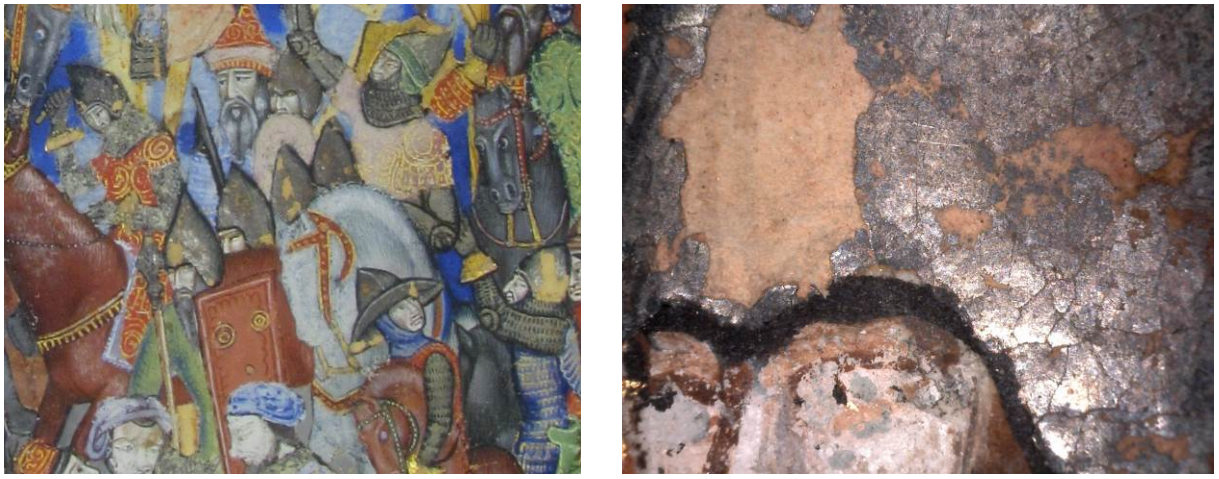

Fig. 4 - Exploded view of the miniature at f. 206v (left) and 200x image of a grey helmet (right)

In other cases Ag was used powdered, similarly to shell gold. A particular feature is the shield with grey bands at f. 19r which shows an unusually high amount of Pb: most probably the shield is a later addition painted by another artist.

vii) Orange

In the Calendar, *red lead* or *minium* – Pb_3O_4 – a very common and cheap synthetic pigment, identified according to the inflection point at ca. 560 nm in FORS spectrum, was used for orange hues. In the main part, red lead was used either pure and in mixture of red ochre.

viii) Pink

For rendering pink hues, i.e. for incarnates, the artists used only red pigments such as *red lead* diluted with lead white (this last suggested by the detection of Pb by means of XRF); no dyes or lakes were identified on such instances.

ix) Purple

For a wide range of hues, from dark red to purple and violet, the painters used *brazilwood*, the dye extracted from different tree species native of South-eastern Asia such as *Caesalpinia sappan*. Brazilwood was identified according to the absorption band at ca. 560 nm in FORS spectrum. This dye was traded in Europe from Asia since at least 12th century (Roger et al., 2003), so its use was all but usual in late Middle Ages. The dye was used for several decorative features, but mostly initials and backgrounds of inhabited initials. In the Calendar, pure brazilwood was used, while in the main part the painters used also brazilwood mixed with cinnabar (suggested by the detection of Hg by means of XRF) for dark red tones and brazilwood mixed with indigo, according to two absorption bands at ca. 560 and 660 nm in FORS spectrum, for violet tones.

x) Red

In the Calendar, the only red pigment was *cinnabar*, identified according to the inflection point at ca. 600 nm in FORS spectrum and confirmed by the detection of Hg by means of XRF. In the main part, instead, the various artists used cinnabar but also red lead, red ochres and cinnabar mixed (or adulterated!) with red lead; the mixture cinnabar/red lead was

identified according to two inflection points at 560 and 600 nm in FORS spectrum and confirmed by the detection of Hg and Pb by means of XRF.

xi) Violet

One remarkable feature of the *Messale* is the extensive use of delicate pen-work decorating small red and blue initials. While the pen-flourishes decorating blue initials were made with cinnabar, the pen-work decorating red initials was made with *folium*, the dye extracted from *Chrozophora tinctoria* plant; folium was identified according to its typical absorption bands at 550 and 580 nm in FORS spectrum. Though the composition of this dye is largely unknown (Aceto et al., 2015), its use in miniature painting was documented in many instances (Aceto et al., 2017a; Aceto et al., 2017b) and above all in 14th and 15th centuries manuscripts of Italian and French production. The wide use of folium on the *Messale* is not surprising considering that Avignon is only 60 Km far from Grand-Gallargues (present day Gallargues-le-Montueux), a small hamlet in the Gard department which was in Middle Ages the European centre of cultivation of *Chrozophora tinctoria* and of production of the dye.

In many instances, folium was added with ultramarine blue as evidenced by FORS analysis (Fig. 5) in which the spectral features of ultramarine blue (absorption band at 600 nm) overlap to those of folium (absorption bands at 550/580 nm); XRF analysis confirmed the presence of ultramarine blue according to a higher amount of Si, S and K. The mixture folium/ultramarine blue was perhaps done by the artists in order to obtain a more intense blue hue.

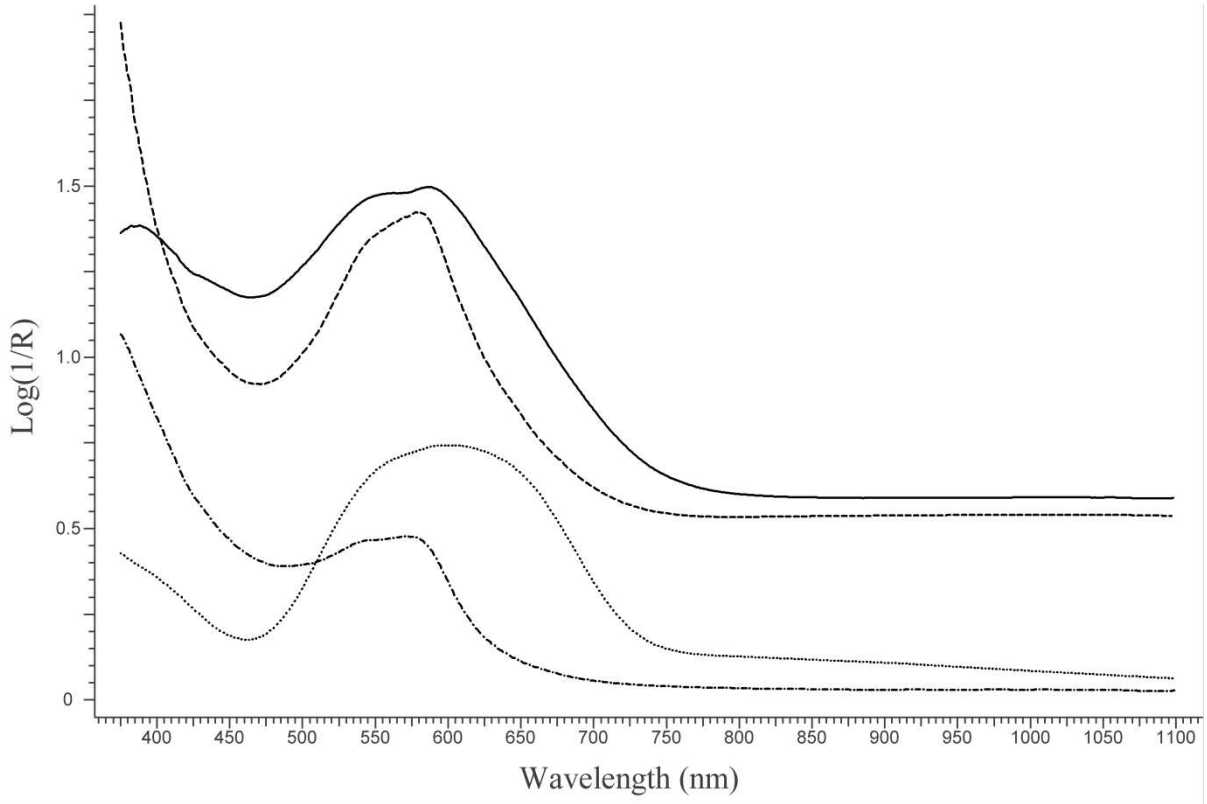

Fig. 5 - FORS spectra in Log(1/R) coordinates of a violet pen-flourished decoration filigree (dashed line) and an intensely blue pen-flourished decoration (solid line) compared with a standard paint of folium (dashed-dotted line) and a standard paint of ultramarine blue (dotted line)

Under the stylistic point of view, at least two different pen-flourishers can be recognised in the making of red and violet pen-work: one more gifted artist, who can be identified as the illuminator Bernard de Toulouse (Manzari, 2014) and one more conventional craftsman. Indeed, the analysis of violet pen-work – which in France is specifically restricted to manuscripts produced in the South (Manzari 2006) – by means of spectrofluorimetry (Fig. 6) evidenced two slightly different preparations corresponding to the two different styles of drawing, according to spectral features at 605/625 nm (Aceto et al., 2015) in the pen-work attributed to Bernard de Toulouse and at 605/625/665 nm in the pen-work attributed to a second hand. Apparently, the second illuminator used a recipe which contained a compound with a further fluorophore in addition to folium.

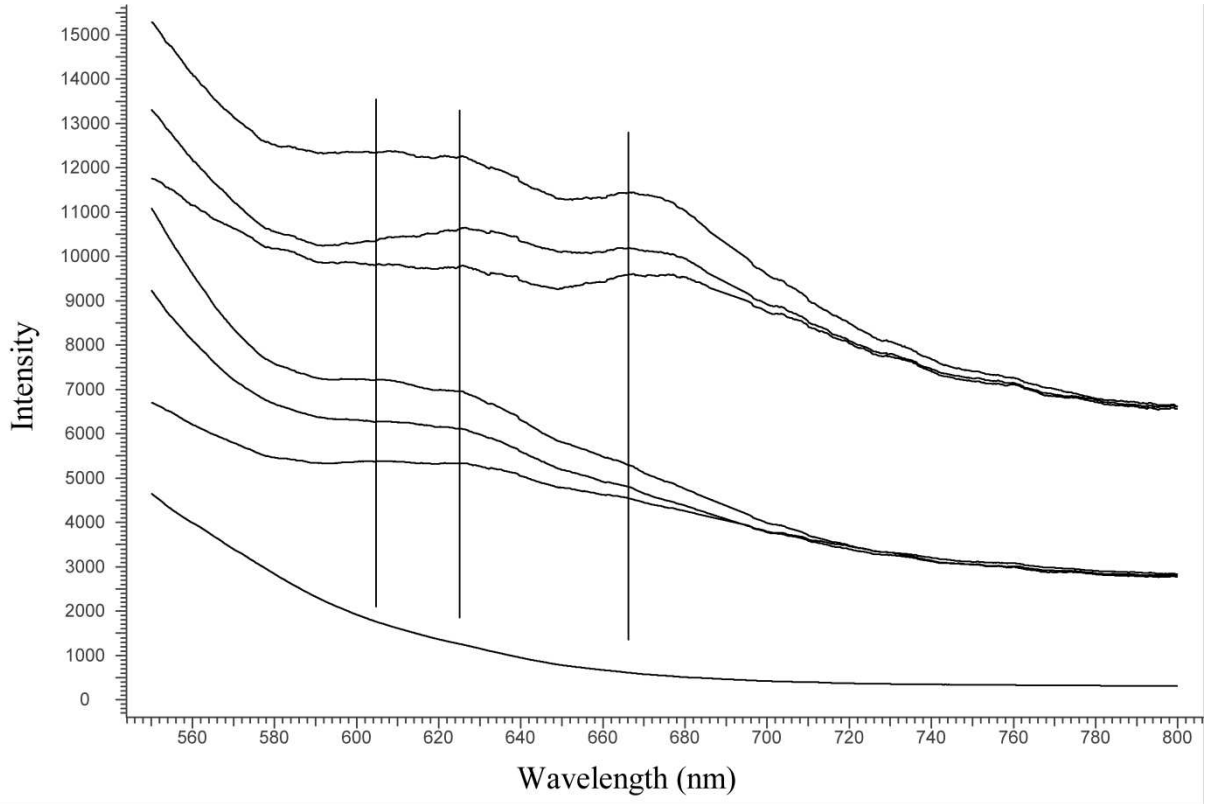

Fig. 6 - Fluorimetry spectra of violet pen-work made by Bernard de Toulouse (center lines) and by another illuminator (top lines); the spectrum of parchment (bottom line) is given for reference

xii) White

The only white pigment used throughout the manuscript was *lead white*, suggested by the detection of Pb by means of XRF.

xiii) Yellow

The yellow hue is rare, probably due to the wide use of gold. The main use of yellow seems to be as preparatory ground for gold and silver leaf, as described before; in this case, as in the rare cases of miniature details, the pigment used was *yellow ochre*, identified according to the absorption band at ca. 900 nm in FORS spectrum and confirmed by the detection of Fe by means of XRF.

xiv) Inks

The coloured inks were made with the same pigments used for miniatures, that is cinnabar for red inks and ultramarine blue for blue inks. For the main text and musical notations, iron gall inks (IGI) were used, as identified by means of FORS analysis (Aceto & Calà, 2017c). The composition of IGI, as it results from XRF analysis, seems to be homogeneous all through the manuscript, as if it had been written by a single scribe. In addition, there is no apparent difference between the ink used for text and that used for musical notations: both show a Fe/Cu ratio of ca. 20:1 (Fig. 7) with a small amount of Zn. The absolute amounts of Fe and Cu in musical notations are higher only because the spots analysed with XRF (3 mm) were in those cases completely filled with ink, while the text lines were thinner (1-1.5 mm).

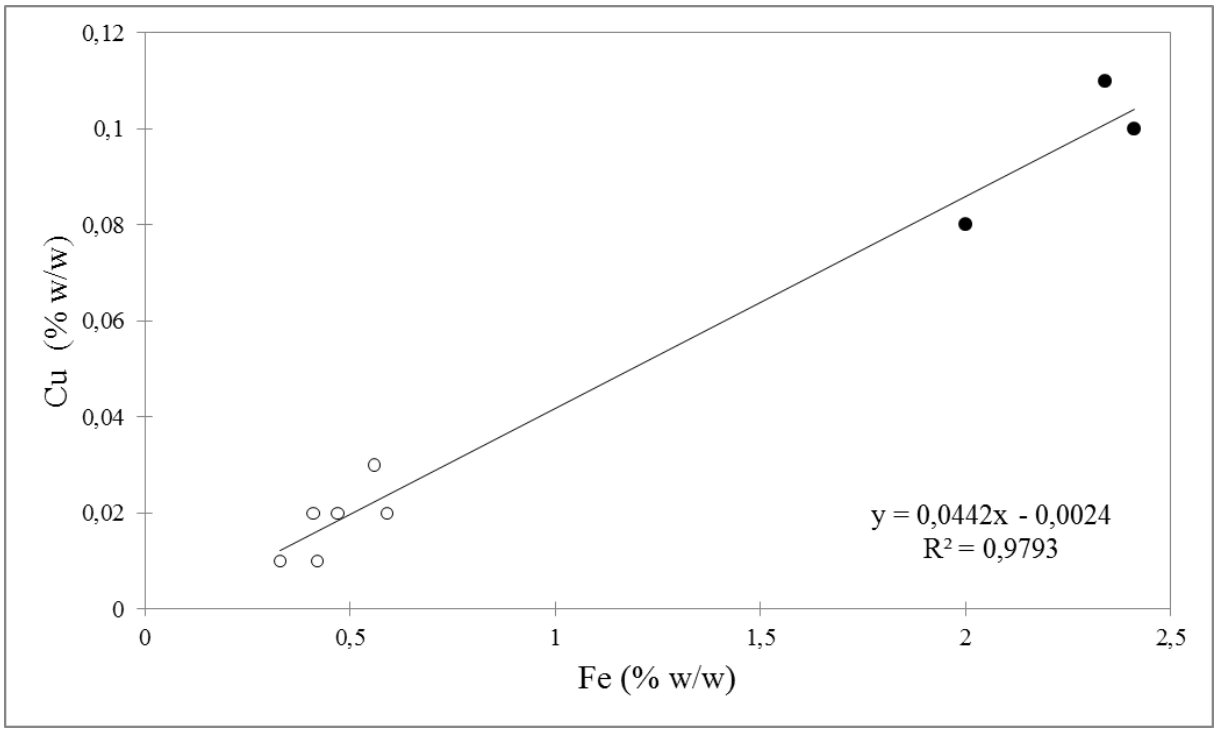

Fig. 7 - Fe vs Cu plot as determined by XRF analysis on text (white circles) and on musical notations (black circles)

The overall condition of inks is very good all through the *Messale*, so it seems like the scribes used a well-balanced recipe for IGI.

xv) The different palettes

According to the results of non-invasive measurements, it is possible to define the palettes used in the different sections in which different artists were at work. The sections seem to be at least four: (1) the Calendar; (2) the main part; (3) the starting quire of the main part at ff. 19r-26v; (4) the bifolium 206v-207r. The main colourants used are listed in Table 1.

Colours	Calendar	Main	ff. 19r-26v	ff. 206v-207r
black	carbon	carbon	carbon	carbon
blue	ultramarine blue	ultramarine blue, woad	ultramarine blue, woad	ultramarine blue, woad
brown	red ochre	red ochre	red ochre	red ochre

gold	shell gold, gold leaf	shell gold, gold leaf	shell gold, gold leaf	shell gold, gold leaf
green	ultramarine blue/yellow ochre	woad/yellow ochre	woad/yellow ochre	verdigris
grey	shell silver	shell silver	shell silver	silver leaf
orange	red lead	red lead/red ochre	red lead/red ochre	red lead/red ochre
pink	red lead/lead white	red lead/lead white	red lead/lead white	red lead/lead white
purple	brazilwood	brazilwood	brazilwood	brazilwood
red	cinnabar	cinnabar, cinnabar/red lead, brazilwood/cinnabar	cinnabar, cinnabar/red lead, brazilwood/cinnabar	cinnabar, cinnabar/red lead, brazilwood/cinnabar
violet	folium, folium/ultramarine blue	folium, folium/ultramarine blue, brazilwood/indigo	folium, folium/ultramarine blue, brazilwood/indigo	folium, folium/ultramarine blue, brazilwood/indigo
white	lead white	lead white	lead white	lead white
yellow	yellow ochre	yellow ochre	yellow ochre	yellow ochre

Table 1 - Palettes used in the different parts of the *Messale*

e) Sequence of manufacture of the Messale

The results of the different analyses, including micro images taken on particular features, complemented the technical-artistic knowledge on miniature painting and allowed hypothesising a sequence of operations in the manufacture of the manuscript, from the point of view of the materials involved. The order, outlined after discussion involving different competences, is the following:

1) First at all, the ruling, that is the lines used to delimit the written surface, are traced; in the Calendar part ruling was traced either with cinnabar (red) and with folium (violet), while in the main part they it was traced with IGI;

2) Then text and musical notations were written by the scribes with IGI;

3) Then comes the decoration of the text with red and blue initials, ornamented by, respectively, violet and red pen-flourished decoration: this sequence can be appreciated in Fig. 8, where the red pen-work apparently overlaps the blue initial previously laid.

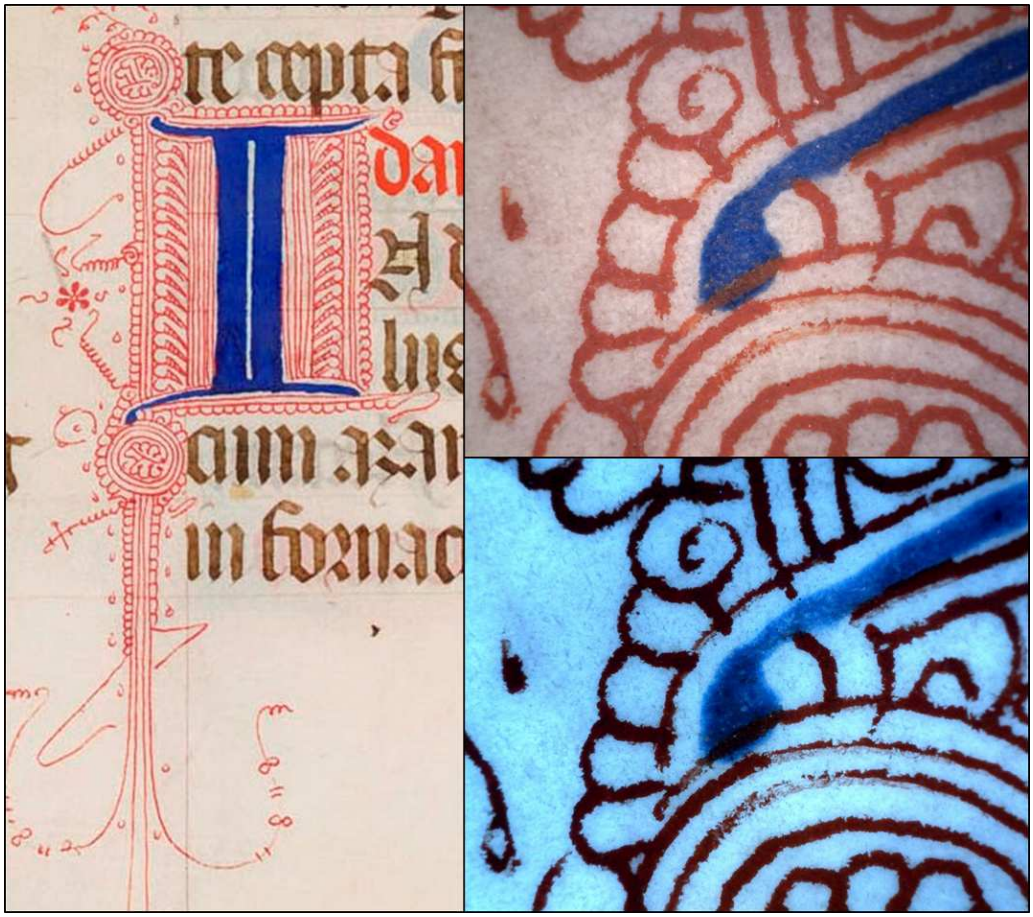

Fig. 8 - Decorated blue initial with red pen-work at f. 66v (left), 50x image (top right) and 50x UV image (bottom right)

4) gold leaves are laid and contours are defined.
5) Miniatures are then painted in the fields specifically left by scribes; usually the leading artist carried out the preparatory drawings, while less-experienced artists painted the first layers and the main artist finished hands and faces (Alexander, 1992);
6) In the Calendar part, the zodiac signs were the last features painted.

f) Details from IR photography

In the Introduction paragraph, the various changes in ownership of the *Messale* have been described. The arms of Cardinal Rossell, the original owner, still appear in few instances: the five roses, the red and yellow *Barras de Aragón* (the coat of arms of the Crown of Aragon) and the red Cardinal's hat. Many others, though, were hidden by the artists who changed some decorative features in order to adapt the manuscript to the needs of the new owners. By means of IR photography it was possible to confirm this sequence. Some examples can be appreciated in Fig. 9. At f. 287v the three roses of Cardinal Guillaume de Bragose, owner from 1361 to 1367, were overpainted on the five roses of Cardinal Rossell, adding two animals supporting the shield. At f. 252r the *Barras de Aragón* appear below a generic decorative feature when the image is seen under IR light: in this case, after the patron's sudden death, the artists overpainted his arms perhaps in preparation for the sale of the *Messale* to a new, but still unknown, purchaser.

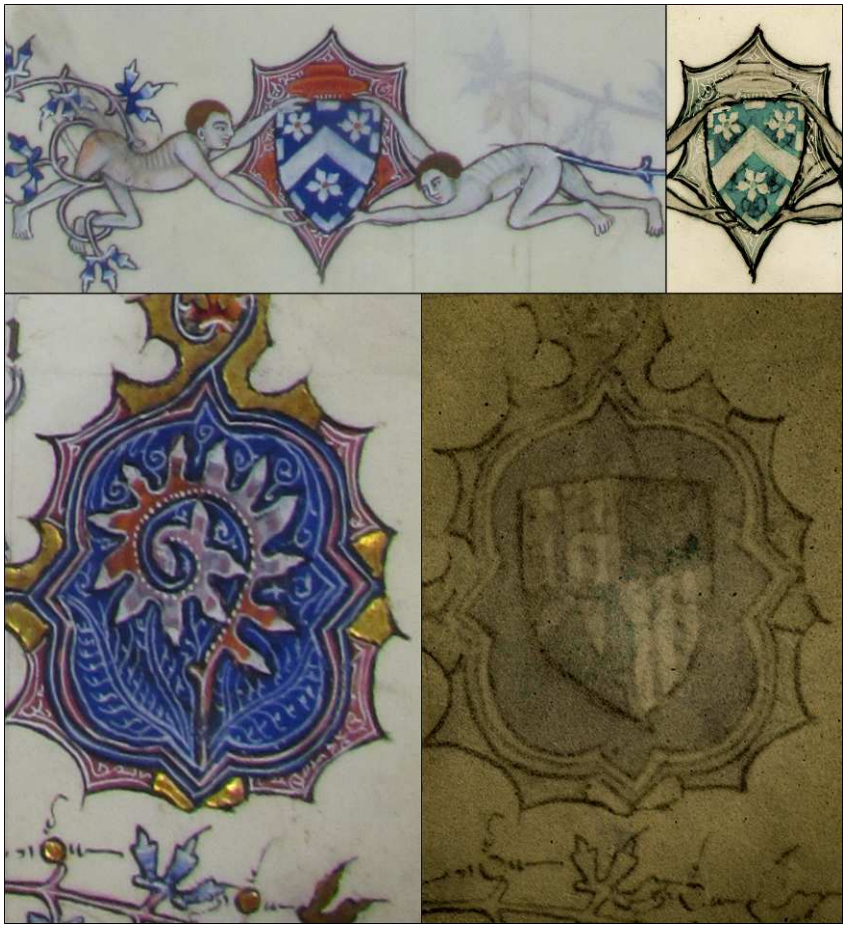

Fig. 9 - The three roses of Cardinal Guillaume de Bragose (top left) over the five roses of Cardinal Rossell (top right) at f. 287v; a decorative feature at f. 252r (bottom left) and the same photographed under IR light (bottom right)

4) Conclusions

The combined use of non-invasive techniques, and the opportunity of applying them with ease due to the restoration of the artwork, offered the perfect situation to obtain a very considerable amount of information on the history of the *Messale Rosselli*, its manufacture and the raw materials used. The different artists who contributed in its decoration employed the most precious pigments known at the age: lapis lazuli, cinnabar, gold, silver. These and other colourants were used both pure and in mixture, in order to obtain a wide range of hues. Chemical analysis provided the characterisation of the palettes used by the different artists, reinforcing the hypotheses raised by scholars. In addition, the overall palettes have been evaluated according to the availability of raw materials in the geographic area around Avignon, finding that most of the colourants could be at easy disposal of the artists.

Information has also been obtained concerning the preparation of the parchment and the volume itself. The systematic measurement of the width of folios allowed hypothesising the number of the animals slaughtered to produce parchment, and the way of using skins. XRF analysis on the folios suggested that different preparations were used. Finally, eZooMS analysis evidenced that calf and goat, but not sheep, were used to produce the parchment of the *Messale Rosselli*.

By means of IR photography, it was also possible to obtain information on the sequence of owners of the manuscript, identifying the materials used in the coats of arms of each ones.

5) Acknowledgements

The authors would like to thank Dr. Paolo Giagheddu (Dipartimento di Studi Storici, Università degli Studi di Torino) for taking IR pictures at the miniatures of the *Messale*; Dr. Guglielmo Bartoletti, head of Biblioteca Nazionale Universitaria in Torino, for allowing thorough inspection of the manuscript; Sig. Massimo Saccon (Studio Vellum) for helpful suggestions. Authors are also grateful to Rotary Club Torino Castello for sponsoring the restauration of the manuscript.

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