Why Do We Need to Study Inks?

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While studying the socio-geographic history of inks, division 4.5 (Analysis of cultural artefacts and assets) of the BAM (Bundesanstalt für Materialforschung und -prüfung) in Berlin together with the Centre for the Study of Manuscript Cultures in Hamburg has developed a protocol for ink analysis. It consists of a primary screening to determine the type of the ink, and a subsequent in-depth analysis using several spectroscopic techniques.

Using this protocol, scientists can assist scholars in addressing a rather broad range of historical questions that cannot be answered unequivocally through scholarly research alone. Among these are investigations on collaboration between scribes and scriptoria, on the usage and annotation of manuscripts and on their path through time and space in general. This research can thus help to reconstruct the circumstances of the production of written heritage as well as their history and transmission.

To facilitate the dialogue between the scholars and the scientists a simple optical tool was developed to allow the scholars to perform preliminary ink analysis required for formulation of the question that in turn can be answered by scientific in-depth investigations. In this paper, ink types and their identification method is accompanied by examples of the recent work conducted on parchment manuscripts in the Austrian National Library.

Key words:
Ink, Manuscripts, Non-destructive analysis, Advanced codicology, XRF.

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INTRODUCTION

Determination of chemical composition of writing materials generates important data for addressing cultural-historical questions that cannot be solved by historical and philological methods alone. In its individual materiality, each manuscript contains evidence of the production processes as well as a wide variety of traces from use, storage, and conservation treatments. The traces that can be measured and ascribed to certain events provide insights into the history of a manuscript.

There are at least two prerequisites for the successful implementation of such measurements. The first and the most important one for the investigation of historical objects is the use of techniques that are non-destructive or require only minimal sampling. In case of sampling, the latter should preferably stay unchanged by analysis and available for further studies. In this respect, "X-ray fluorescence" (XRF) analysis represents one of the best and commonly used methods for obtaining qualitative and semi-quantitative information on a great diversity of materials. In addition to a variety of portable and transportable XRF instruments, a wealth of knowledge and experience has been accumulated in the characterization of historical inks. The second prerequisite is a meaningful question to be answered by scientific analysis. The field of codicology i.e. study of manuscripts as physical objects seems to be the most appropriate field for adoption of a routine determination of the composition of writing materials. In the dialogue between scholars and scientists, a new field of advanced codicology has emerged in the recent years [Hahn et al. 2007; Rabin et al. 2015; Geissbühler et al. 2018; Heiles et al. 2018].

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To further facilitate this dialogue, the BAM division of the analysis of the cultural artefacts has developed a simple optical tool for identification of the ink type since its colour was not well suited for this purpose. It is customary to distinguish between three main types of the historical black writing materials used in manuscript production: soot, plant, and iron-gall inks. Soot ink is a fine dispersion of carbon pigments in a water-soluble binding agent; plant-based ink consists of tree bark (tannin) solution; iron-gall ink, produced by mixing iron (II) sulphate with a tannin extract from gall nuts, presents a boundary case between soot and plant ink – a water soluble preliminary stage (similar to inks from the second group) oxidizes and evolves into a black, insoluble material (similar to the carbon pigments of the first group) when the writing is exposed to air. Each ink class has distinct properties that readily permits their differentiation when used as a solitary type rather than a mixture. Carbon inks do not penetrate the substrate (whether papyrus, parchment or paper) and stay well localized on the surface. In contrast, plant inks and iron-gall inks are absorbed by the substrate, and the degree of their absorption depends to a great extent on the nature of the substrate. A typological differentiation can be also made using the interaction of the inks with the infrared light. IR reflectography has been traditionally used to study soot-based pigments or carbon inks: the colour of soot inks is independent of the illumination wavelength in the range 300-2000 nm; plant inks lose opacity close to 750 nm, whereas iron-gall inks become transparent only at a wavelength > 1000 nm [Mrusek et al. 1995]. Using a conventional usb-microscope with a silicon detector and illumination with visible and near-infrared (940 nm) radiation, it is possible to identify the ink type due to the change of its opacity. When the illumination is changed from the visible to the NIR light carbon ink undergoes no change; plant ink becomes invisible; iron-gall ink changes its opacity but doesn't disappear completely. One has mention, however, that the clear distinction between plant inks and iron-gall inks might be difficult in this spectral range if iron-gall inks contain very little iron and are, in fact very close to the tannin inks in their formulation. In cases of doubt, additional measurements, namely using XRF should be conducted.

XRF ANALYSIS OF IRON-GALL INKS

As a matter of fact, iron-gall inks are best studied by the means of the XRF technique. Natural vitriol, the main component of the historical iron-gall inks, consists of a mixture of metallic sulphates (iron sulphate, copper sulphate, manganese sulphate, and zinc sulphate) with relative weight contributions characteristic of the vitriol source or purification procedure [Krekel 1999]. One uses this very property of the iron-gall inks to compare them and to distinguish among them. Specifically, development of the fingerprint model based on the qualitative and quantitative detection of inorganic components of the iron gall inks leads to their comparison and differentiation [Hahn et al. 2004; Hahn 2010]. Using fingerprints of red and iron-gall inks we were able to tackle a number of specific questions that arose upon the classical codicological analysis. One of such cases, a compilation of different Middle High German texts associated with Arturian tales bound into a single manuscript, *Codex germanicus* 6 is an excellent example for a manuscript with a complex history of production that could be reconstructed using combination of codicological and XRF analysis of the writing materials. Codicological analysis indicated that the sequence of the texts in the bound codex doesn't correspond to the order, in which they were penned. The exact dates of the copying of the large romances *Parzival* and Wigalois, were known from the colophons that accompanied each of the texts. The chronology of the seven out of remaining ten texts could be reconstructed on the basis of classical codicology. Yet three very short texts could be added at any point. Luckily, the codex was written in iron-gall inks and rubricated with red inks, whose composition varied with time. Some two hundred measurements of the inks composition allowed us to establish the chronology of transcription using time dependent variation in the fingerprints of the inks. This chronology confirmed the order of transcription that has been suggested by classical codicology and attributed the time of inscription for the three short texts. Using imaging XRF we were able to document the traces of multiple corrections. These included text cancelling performed in white lead or just crossing out in the red inks as opposed to direct overwriting in black and red inks. We interpret over-painting of initials as a correction of the latter type. It seems that such corrections served to enhance the colour of the inks and were performed during multiple inspections of the manuscript [Rabin et al. 2015; Geissbühler et al. 2018].

XRF ANALYSIS OF CARBON BASED INKS

In the case of carbon based inks, XRF analysis serves to discover the presence of various inorganic contaminants. Carbon inks containing a considerable amount of copper were detected on a small number of the Dead Sea Scrolls [Nir-El and Broshi 1996] and *Tebtunis* papyri [Christiansen 2017] Moreover, carbon inks containing considerable amounts of lead (Pb) were identified on a Herculaneum papyri [Brun et al. 2016]. Since in both cases carbon constitutes the colourant of the inks we consider these varieties to belong typologically to the class of carbon inks. In the last decades, Raman spectroscopy with its distinct spectrum of carbon became very popular in identification of carbon inks. It was even hoped that this method would lead to identification of the organic precursors for the ink. Unfortunately, our preliminary results show that even a clear distinction between charcoal and soot cannot be seen using this type of spectroscopy. Similarly, the claim that Raman spectroscopy can be used as a non-invasive dating tool could not be confirmed [Goler et al. 2016]. XRF is also one of the important methods for identifying the mixed inks, i.e. inks produced by addition of various metals to the soot inks and intentional mixing of iron-gall and soot-based inks, respectively. Mixed inks received no or little attention in the scholarly and material studies because their identification and recognition of their importance belongs to the discoveries of the last years. Their detection, and especially their classification is quite difficult if one is limited to non-invasive methods. Studies of the extant recipe sources indicate that mixed inks played an important role in the medieval Islamicate world. In this respect, the chapter of an excellent study on the written sources of ink recipes, which is dedicated to the inks used by Jews in different epochs and geographical zones is of particular interest to those of us who conduct material analyses and try to correlate the results with written records and existing traditions [Zerdoun Bat-Yehouda 1983]. Comparing the inks proposed by the Jewish philosopher Maimonides, who lived in 12th-century Egypt, with the considerations of Rashi, who lived in 11th-century northern France, we can see that they both advocated use of the inks commonly known and produced in their respective regions. It is Maimonides who proposes to add tannins to the soot inks, but rejects the metallic salt, both of which were practices that were well attested in contemporary Arabic recipes for making ink. In contrast, Rashi was favourable to employing the plant inks in use in contemporary Northern Europe. The last variety is best represented by the thorn or Theophilus' ink whose elaborate recipe is recorded in *De Diversis Artibus* in 12th century AD [Dodwell 1961]. Unfortunately, no systematic study of the historical use of these inks has been ever conducted: its use has been only occasionally reported in different scriptoria. Recently performed studies on the inks used in the Northern Europe in 6-10th centuries CE form a preparation phase of our new project that should explore spreading of the writing materials associated with the insular manuscripts. Here, tannin inks and the appearance of the iron-gall inks build the focus of the research.

When a statistically relevant number of manuscripts with distinct inks from different regions and time periods has been studied the collected data will be assembled into a time-location map that should become a dating and localization tool for the manuscripts of unknown provenance and unsure dating. "Inks of Coptic Manuscripts" [Ghigo et al. 2018], "Ink recipes from the Islamicate world" [Colini 2018], "Writing inks of Hebrew fragments in Genizah collection" [Cohen et al. 2017, Rabin 2017], "Inks of late Antiquity" [Rabin and Krutzsch 2019] belong to the projects that are specifically dedicated to this aim and are being conducted at the "Bundesanstalt für Materialforschung und –prüfung" (BAM) and the "Centre for the Studies of Manuscript Cultures" (CSMC). Similarly, identification of the metallic precursor used for the iron-gall inks seems also to be of great interest. As mentioned above, vitriol is easily recognized due to the presence of sulphur as and metals – companions of iron. In contrast, the recipes known in the Islamic manuscripts report that metallic filings or even nails dissolved in acid were also used as iron precursor to the iron-gall inks [Colini et al. 2018]. The existence of such inks has been confirmed by recent scientific analysis [Ghigo 2018]. We hope do be able to add the results of the systematic studies of tannic and early iron-gall inks of the Northern Europe to the database.

In this paper, preliminary results of the analysis of the manuscripts from the collection of the Austrian National library are shown.

EXPERIMENTAL

Visible spectroscopy was carried out with the aid of the spectral photometer SPM 100 (Gretag Imaging AG company Regensdorf, Switzerland) equipped with a 3 mm sensor and 2W white light bulb. The measurements were conducted with 10 nm wavelength resolution and 0.5 sec illumination time.

One of the XRF spectrometers used in this work, ARTAX (Bruker GmbH) is well known in the field of cultural heritage and belongs to standard equipment in the majority of large museums. It is equipped with focusing lenses and, therefore, has the X-ray beam spot of 70 µm in diameter. It also features a CCD camera for sample positioning and an electrothermally cooled Xflash detector (SDD, area: 30 mm^2) with an energy resolution of <150 eV at 10 kcps. The movable probe is operated by XYZ motors that control positioning for single-spot measurements as well as line and small area scans. Open helium purging in the excitation and detection paths allows for detection of light elements ($Z \geq 13$). All measurements are made using a 30 W low-power Mo tube, operating at 50 kV and 600 µA, and with an acquisition time of 10 - 100 s (live time). The mobile XRF probe moves over the object at a distance of

5 mm and stops for the duration of a single measurement. A line scan consists of several single measurements along a chosen line. With its small beam spot, it is well suited for investigations of fine scripts. For larger areas of interest, a portable XRF spectrometer ELIO (former: XGLab, now: Bruker GmbH) was used. It has a measuring spot of 1 mm in diameter. The instrument is equipped with a 1W low-power rhodium tube and adjustable excitation parameters.

RESULTS

ÖNB Cod. 974: comparison of the red inks

The production of manuscripts in the Cistercian abbey of Heiligenkreuz in Lower Autria (founded 1133) in the 12th century has been thoroughly investigated. On the website www.scriptoria.at examples of hundreds of scribal hands, correctors and rubricators, have already been published in addition to a book dedicated to the early years of the scriptorium [Haidinger and Lackner 2015]. Among the initials in these early books a group of silhouette initials stands out not only in style but also in color. Since the color is also noticeably different from most other Austrian manuscripts of the period information on the composition of the ink is relevant for understanding the workings of the scriptorium. As a point of comparison another initial from a slightly later part of the manuscript with a more standard type of red was also investigated (Fig. 1).

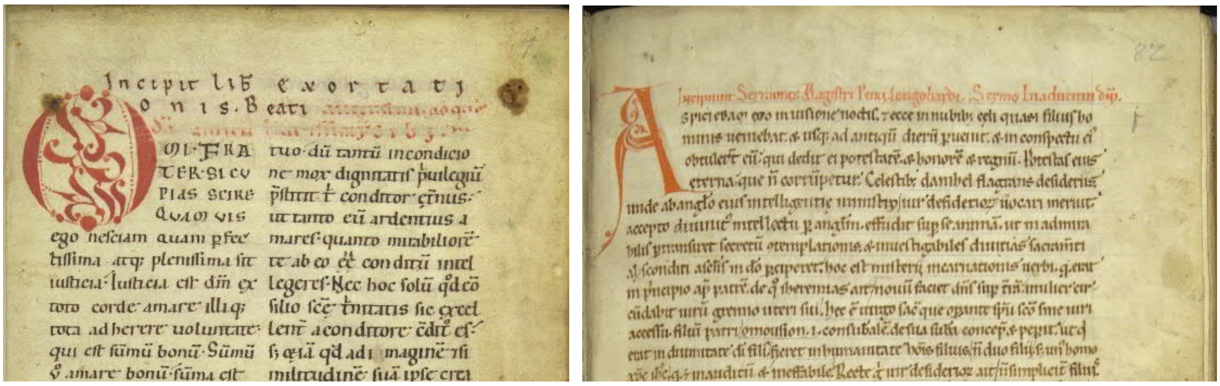

Fig. 1. ÖNB Cod. 974, left: fol. 1r, right: fol. 82r

By means of a spectral photometer, the colour value of a colorant can be quantitatively deter-mined on the basis of its reflective spectrum in the range of visible light (380 nm to 730 nm). With this surface method, the sample to be examined is illuminated with visible light. The sample material interacts with the visible light by absorbing or reflecting it in a specific way, thereby appearing coloured. The reflected light characteristic of a specific pigment is measured with a photometer and recorded in the form of a characteristic reflection curve. This curve represents the correlation between the intensity of the reflected light and its wavelength. Comparison with a databank makes it possible to ascribe the pattern to a certain pigment or dye.

The first derivatives of the reflection curves in Fig. 2 indicate that pure cinnabar (HgS) is responsible for the red colour in fol. 1r (Fig.2, black curve) whereas the colouring on fol. 82r was executed mainly in minium (Pb_3O_4) though a small contribution from cinnabar is clearly seen in the composite curve (Fig.2, red curve). XRF spectra of both colourants confirm the identification made with visible spectroscopy displaying that the pigment from fol. 1r contains basically only mercury whereas the spectrum corresponding to the red from fol. 82r shows lead as a main constituent followed by a small quantity of mercury.

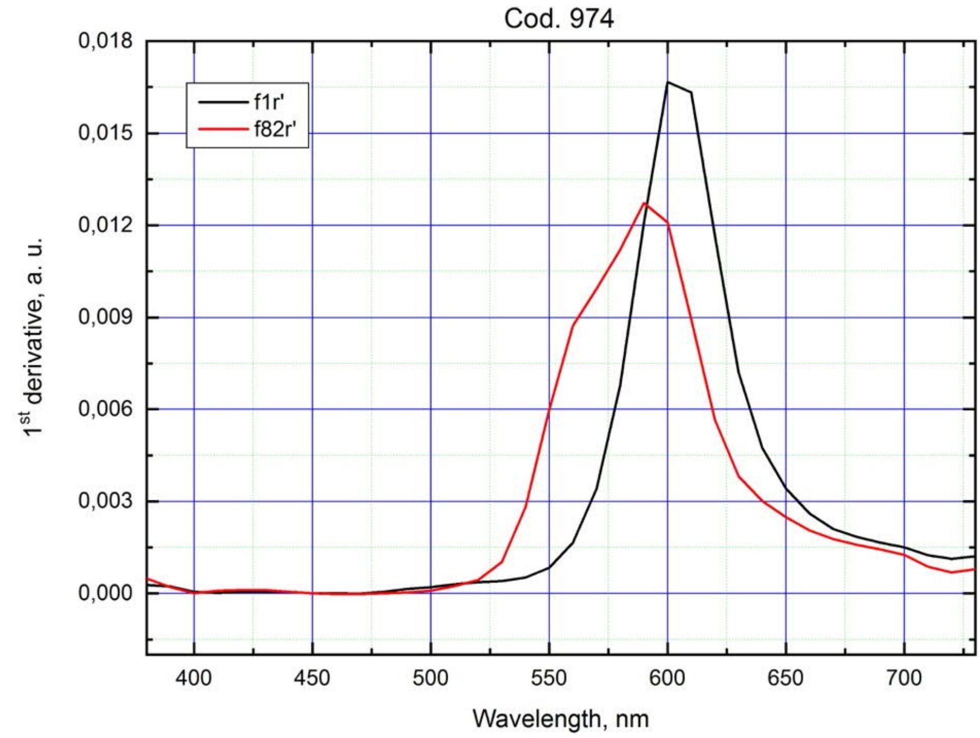

Fig. 2. First derivatives of the reflection curves: black fol. 1r, red: fol. 82r

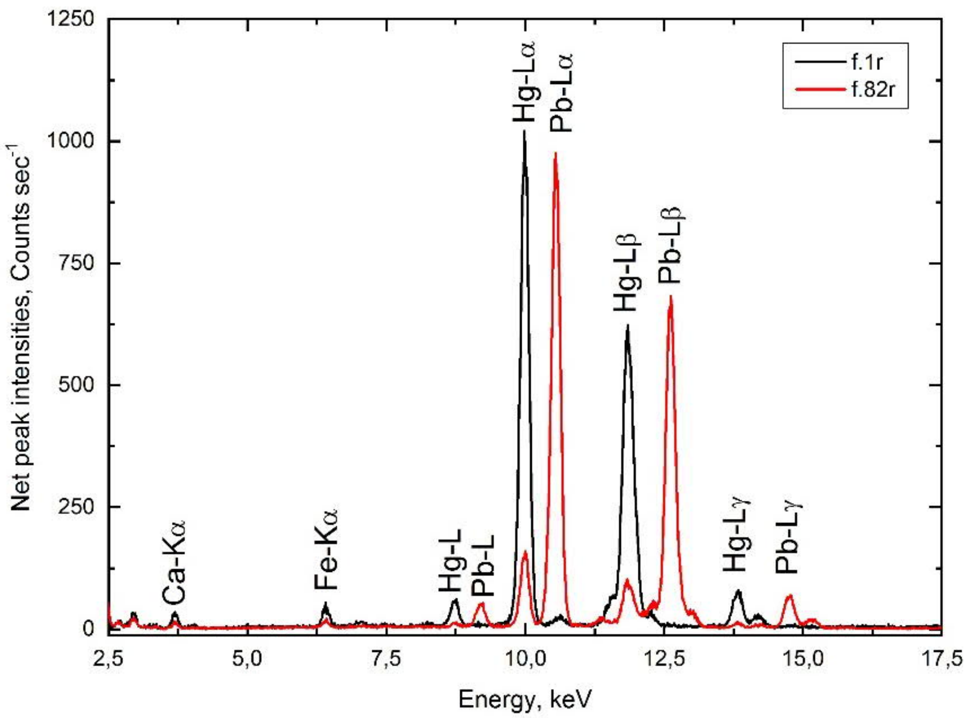

Fig. 3. X-Ray fluorescence spectra of the red pigments from the fol.1r (black) and fol .82r (red)

Analysis with visible spectroscopy and X-Ray fluorescence conducted on the red inks of fol. 1r and 82r of the codex identified two different colourants used: practically pure cinnabar of the first initial in contrast to the minium of the fol. 82r.

ÖNB, Cod. 808 – Comparison of the seemingly different inks on a single page.

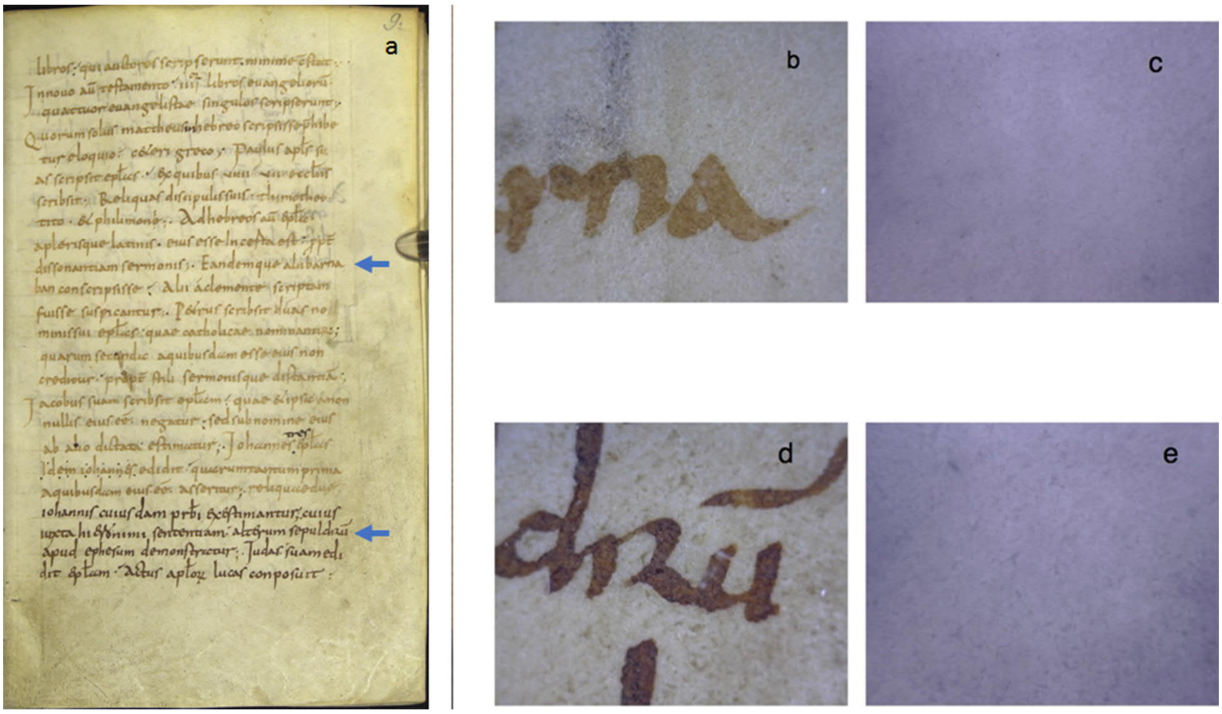

Fig. 4. Cod. 808, fol. 9r with two seemingly different inks. Blue arrow indicates the letters shown in the micrographs on the right, b & d are taken under visible, c & e – under NIR illumination

Working with medieval manuscripts one sometimes encounters strikingly different ink colours within a manuscript or even a single page written by the same scribe. This raises the question whether a different type of ink or a new batch with a slightly different mixture was used, yet again giving hints on how the scriptorium worked. One such sudden change in ink colour can be found in ÖNB Cod. 808 written in Salzburg in the early 9th century and therefore a prime example of Carolingian manuscript production in Austria. Fig.4 demonstrates this ink behaviour of the fol.9r of the manuscript.

Table 1 Elemental composition of the inks (normalized to iron)

Element	Line 22 (±10%)	Line 10 (±10%)
Phosphorus (P)	0.02	0.02
Potassium (K)	0.50	0.27
Calcium (Ca)	0.40	0.40
Manganese (Mn)	0.01	0.01
Iron (Fe)	1.00	1.00

A reflectography test in the Vis / NIR regions demonstrated that the inks from the lines 10 and 22 of the folio 9r disappeared in near infrared light suggesting that the inks are of the tannin type (Fig.4, b/c and d/e). In contrast, XRF analysis of the inks detected elevated intensities of iron indicating that the inks should rather be attributed to the iron-gall type. When one studies and compares various inks in a single manuscript, it is often sufficient to record a characteristic metal pattern of an iron-gall ink and analyze it using a simplified semi-quantitative approach [Rabin et al. 2012]. In this approach, the contributions of the individual metals due to the parchment can be evaluated from the parchment spectrum and subtracted from the measured data of the inked areas. Even in this first-order approximation, the variations due to ink thickness and degradation do not affect the pattern of the ink. To determine a reliable pattern, the micro XRF measurements were conducted in a so-called line-scan mode. In this mode, a minimum of 10 spectra were collected for characterization of the ink and another 10 for characterization of the writing support (parchment in this case). The spectra were evaluated, corrected for the background due to the writing support and normalized to iron as a main element of the iron-gall ink. The resulting pattern described inorganic composition of the ink under investigation (Table 1).

As can be seen from the table 1 the pale ink in the line 10 has almost identical composition to the dark ink of the line 22. Potassium is the only element whose relative amount doesn't stay constant when comparing two inks. Since potassium can be related to the binder used, one can speculate that the ink from the lower part of the page comes from a different batch as compared to the upper part. It is noteworthy that the inks neither contain sulphur nor copper or zinc, the elements that would un-equivocally prove that vitriol was used for the ink production. It is quite possible that the ink used here could be associated with ink preparation known to us from the ink recipe of Theophilus [Dodwell 1961] – tannin ink, to which some metallic iron was added. In such a case, the ink would disappear under the NIR illumination but would contain iron. Presence of the element phosphorus in these inks is most noteworthy – so far it has been detected only in the carbon inks of antiquity.

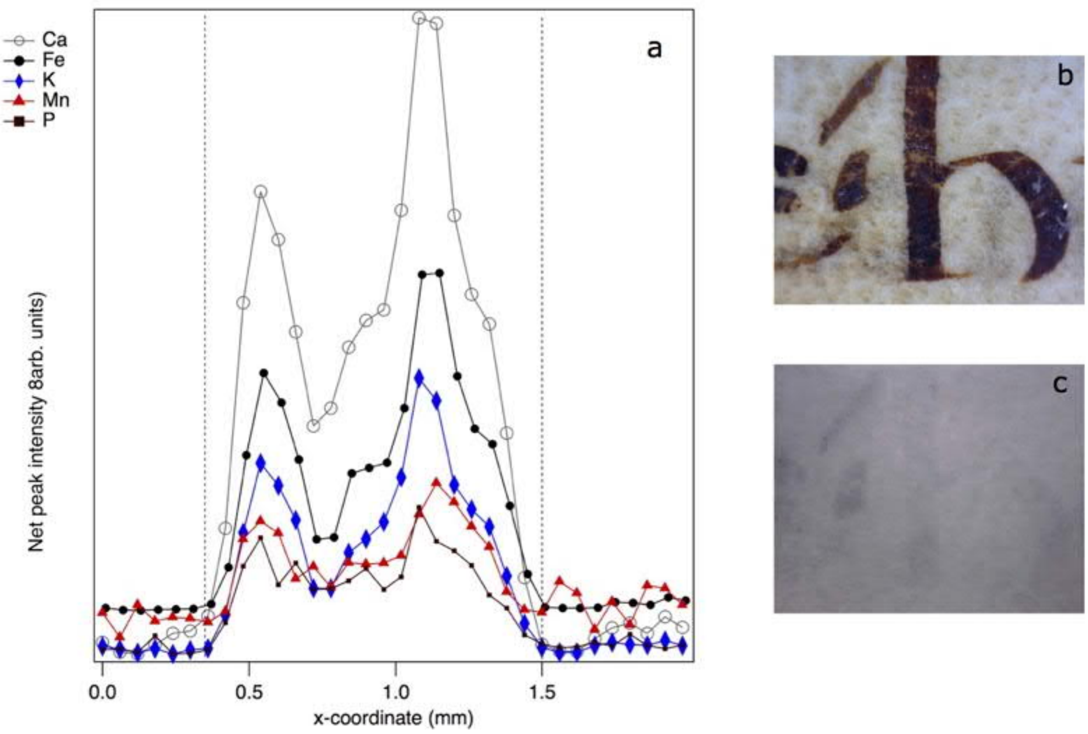

Fig. 5. Intensities of inorganic components of the ink extracted from an XRF-line scan across a letter from fol.3r of (a); micrographs of the letter in visible light (b) and at 940 nm (c). The vertical dotted lines separate the inscribed area from the non-inscribed

ÖNB Cod. Ser. n. 2065

This unusual ink composition has been encountered in this work in another ink, this time in the ink of the codex ÖNB Cod. Ser. n. 2065. The manuscript was written in the late 8th century in yet another famous Carolingian scriptorium in Austria: the Benedictine monastery of Mondsee in Upper Austria. In the 15th century the manuscript was apparently deemed useless and was cut up to be used in new book bindings for the monastic library's ever-expanding collection. What remains today are fragments from over 90 pages that are at the moment being reconstructed on www.fragmentarium.ms.

In Fig. 5a, the authors present the individual intensities of the inorganic components resulting from a line scan across a heterogeneously degraded portion of the ink from fol.3r. Note that the curve form of each contribution follows that of iron, the main component of the ink, the behavior, which is typical for iron-gall ink. The varying thickness and degradation of the ink are reflected by the high variability of the signal for iron and its satellites within the inked area. Note that the elements that constitute the inks are exactly the same elements that were found in the inks of the ÖNB Cod. 808. In contrast to those inks, in this case the ink is still visible at 940 nm (Fig. 5c) unequivocally proving it to be of iron-gall type.

CONCLUSION

This paper shows the successful application of the protocol for ink analysis developed by division “Analysis of Cultural Assets” at the BAM in Berlin in collaboration with the Centre for the Study of Manuscript Cultures in Hamburg. Two-step analysis consisting of XRF and VIS spectroscopy conducted on the red initials of fol. 1r and 82r of the ÖNB Cod. 974 identified two different pigments: practically pure cinnabar of the first initial contrasted with the minium of the fol. 82r. In contrast, seemingly different inks observed on a single page of the ÖNB Cod. 808 demonstrated basically the same composition. The most interesting discovery of this work is identification of an unusual ink composition in the manuscripts without any connection other than geographic proximity and the time period raises a question whether this type of ink could be characteristic for the southern German region and Austrian around 800. Our future research will certainly clarify this question.

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