Improving Iron Gall Ink Palimpsest X-ray Fluorescence Element Mapping Analysis

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In the recent years multispectral imaging and X-ray Fluorescence element mapping have established themselves as a well working two step approach, when dealing with faded, erased or overwritten text in iron gall ink. During the last years a lot was done to improve the mere technical side of the techniques. Especially in the case of XRF mapping experiments the acquisition speed needed to be increased to allow mapping in the high resolution demanded by the challenges at hand. High resolution in moderate times can now be achieved, but with the price of proportionally increased amount of data that needs to be processed ideally in parallel to the measurement. The classical approach in XRF spectroscopy of elemental fitting of the spectral data does not even come close to fulfill any on the fly processing time constraints and will consume too much processing time in the foreseeable future. In this paper we will discuss the applicability of modern statistical data processing methods to XRF mapping of iron gall ink palimpsests. We will present a comparison of a new approach based on principle and independent component analysis (PCA and ICA) and the standard element mapping method. We will show that a reduction in processing time of almost two orders of magnitude can easily be achieved.

Key words:
Archaeometry, Statistical analysis, PCA, XRF, Iron Gall Ink.

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INTRODUCTION

Parchment has been a well-established writing support in the last two thousand years. As a sturdy material parchment allowed to be reusing after prior writing had been scraped off or chemically removed. Reused parchment is called palimpsest and can contain any ink type used at the time of its creation, as e.g. iron gall, plant – or soot-ink. A significant portion of known palimpsests contain at least one erased layer of iron gall ink, while a new text in iron gall ink has been written on top. Iron gall ink can be removed mechanically or chemically. Mechanical removal of preexisting writing is very time consuming but usually leaves no detectable residuals of the removed text, only scratch marks from the scraping. Chemical removal of iron gall ink writing is much less time consuming and very effective for the naked eye, hence ink was mainly removed by chemical means with a sponge preferentially soaked in lemon juice. The lemon juice destroys the black colored iron gallic acid complex and removes some of the ink, but leaves residuals of the metallic contribution of the ink at the position of the erased text. After centuries the erased writing often reappears faintly in yellowish-brown characters due to continuous slow forming of iron oxide from the residual iron of the erased ink. These ghostly traces of slightly brown text often identify a page as a palimpsest. Sometimes iron gall ink just fades due to the unfavorable storage conditions, while again the metallic contributions of the ink stay put at the position of the former text.

The recovering of the overwritten texts from palimpsests went through several stages of technology [Albrecht 2015; Rabin et al. 2015 and references within], of which the earlier were strongly, the latter slightly or non-invasive. During the beginning often chemicals were used that worked for a short time and sometimes allowed to have photographs taken from the under-text, but often left the document in a much more degraded state. This approach

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was mostly replaced when artificial “ultraviolet” (UV) light became available. Reading texts under constant illuminations with UV light caused less damage to a manuscript than the chemical treatment, but still affected the state of the object by increased aging due to the UV radiation. Additionally the newer technology caused damage to the readers’ eyes and often delivered still inadequate results. UV light causes parchment to fluoresce, while this fluorescence is suppressed in regions where the parchment had been in contact with gallic acid, as in places where iron gall ink had been. Developments during the last 20 years in the field allow now non-destructive investigations without any harm to object and reader. A nice set of tools is available for scholars and scientists to digitally recreate the lost texts. Especially when dealing with hidden (faded, covered or erased) iron gall ink the following processing protocol has proven most effective: Objects with suspected hidden writing in Iron Gall Ink will first be measured with “multispectral imaging” (MSI) [Easton et al. 2010] where a special photographic camera with an optional set of filters is used together with with LED light panels. In most cases this first step is already sufficient to recover the lost text completely. In the other cases or for very delicate regions that remain illegible, the document will be tested for the presence of iron gall ink residues using an “X-ray fluorescence” (XRF) spectrometer. If that test is positive the ink to be recovered possesses metallic components and X-ray based scanning techniques can be applied. XRF mapping [Bergmann 2011; Glaser and Deckers 2015] often allows recovering more or other parts of the lost text. MSI digitally reconstructs text by statistical analysis of the local dispersive surface response function of the investigated object in the “ultraviolet” (UV), “visible” (VIS) and “infrared” (IR) range of the electromagnetic spectrum and thus is almost completely surface sensitive. If in cases where the surface is completely covered with e.g. a gold paint as in the case of the Archimedes palimpsest [Bergmann 2007; Easton et al. 2011] or if the text of interest has been glued as support material face down as in a book cover a mere optical, surface sensitive method cannot deliver any useful result. These are classic cases, where XRF mapping is the method of choice.

The XRF signal contains the sum of information from the entire thickness of an object (usually a single page) at a specific point, sampling over the spot size of the primary X-ray beam. With carbon based writing supports, as parchment, papyrus or paper the X-rays penetrate the object completely with only a small portion being absorbed. The method as spot based has no intrinsic spatial resolution; the lateral resolution needs to be achieved by selective detection. Usually the lateral resolution is achieved by focusing the X-ray beam to a small spot while mapping the surface with this spot, measuring one spectrum at a time. This mapping approach is time consuming, even if every single measurement is extremely short as e.g. 100 ms and thus allowing measuring an impressive amount of 800.000 points in a day. 100 ms is a reasonable time for modern laboratory based systems or storage ring based measurement. Times of measurement per point can be reduced even down to the low ms-range, but usually the necessary counting statistics of a spectrum requires longer counting times per point. In order to reproduce a legible text from the mapping experiment, the resolution must be sufficiently high (some 150-300 dpi or better). In total, the amount of measurements exceeds a million in many cases (see Table 1) making XRF mapping a time-consuming measuring technique. A small area of 1 cm^2 in 150 dpi can be mapped in less than 10 minutes if a point takes only 100 ms, an A4 page in the same resolution and same sampling speed already takes 2-3 days, while higher resolution of 600 dpi causes the time to jump to 1½ months. Spending days on a single page may be all right for a few selected cases, but considering the tens of thousands of unread palimpsests known at this point even a day per page is too long to measure all of them. For serial investigations of e.g. codices 1 day of measuring time for each page is already too much:

The usual time for processing of XRF data is even longer for each spectrum than the 100 ms time assumed to measure it and each measured point has to be analyzed separately. When proceeding normal protocol each XRF spectrum is background subtracted and fitted with multichannel analyzer software as e.g. *AXIL*[1] [Vekemans et al. 1994] or *PyMCA*[2] [Solé et al. 2007] to identify the amount of (primarily) metals, that are contained in the object at each measured position. After analyzing all spectra the individually derived amount of metallic components at each point is used to create element maps of the measured region of the object. Those element maps or some of them may contain the hidden text. The main reason one can digitally recreate old iron gall ink writing with XRF mapping experiments is the above described imperfect removal of the ink in the palimpsest creation. Typically the metallic contributions that remain of the former ink consist of iron, copper, zinc and lead. While iron has an essential role in forming the black colored complex in all iron gall inks and thus is usually present in a relative large amount, the presence and relative amount in respect to iron of all other metals (the inks fingerprint) often allows to distinguish between one ink and another [Hahn et al. 2004; Malzer et al. 2004]. Iron, copper and zinc in highly variable stoichiometric ratios were part of almost all historic vitriol's with vitriol's being commercially traded. Lead (if

[1] AXIL code available (as bAXIL) at: http://www.brightspec.be/brightspec/

[2] PyMCA code available at: http://pymca.sourceforge.net/

present) is assumed to be incorporated through the local water used and may in the future be a mean to identify the provenience of an ink with lead isotope analysis. These metallic residuals, especially of the differently impure vitriol used in the mixing process of all historic inks is the starting point to argue for a different, a statistical approach for data processing when dealing with XRF mapping experiments of manuscripts.

Table 1. Number of XRF measurements for a given area and resolution

Resolution in dpi **Area**	100	150	300	600
1 cm x 1 cm	1.55×10^3	3.5×10^3	14×10^3	56×10^3
10 cm x 10 cm	0.15×10^6	0.35×10^6	1.4×10^6	5.6×10^6
21 cm x 29.7 cm (page A4)	0.24×10^6	0.55×10^6	2.2×10^6	34.8×10^6

ARGUMENTS FOR STATISTICAL XRF ANALYSIS

With often over a million spectra measured on a single page and measuring times of 10 ms - 100 ms (even with some modern transportable systems), the time consumed in processing is exceeding the measuring time significantly. The use of more detectors increases this problem by a factor equal to the number of detectors. This processing time problem can be addressed by brute force, just massively upgrading the hardware for processing and extensive parallel processing, which eventually will be very expensive and in cases where the equipment needs to be mobile may even become unpractical. To achieve a factor of e.g. 100 in processing speed by more and faster hardware is a big investment, even if the starting point is just a standard PC with a regular i7 processor. Thus any real time or near real time processing is not possible when keeping to the conventional XRF analysis methods, but is nevertheless urgently needed. Fast feedback during a measurement is essential to avoid time loss, especially if the measurements are performed under severe time constraints.

Those time constraints may on the one hand be due to the access time to an object granted by a library, museum or monastery or on the other hand the measurement time available due to the equipment/facility used for the measurement. Mobile equipment often needs to be shared and cannot remain at a location in-definitely or be fixed on one task, while at a storage ring facility beam-time is granted for short time periods only and with an advance time of almost a year. Those boundary conditions often cause measurement situations, where re-measuring something may become virtually impossible. Hence the first attempt needs to be a success and thus real time evaluation is essential. The applied evaluation method must be efficient enough to keep up even with fast mapping speeds (2-5 ms per point) and needs to provide results that allow evaluating a measurements level of success, ideally without the necessity of any special processing hardware upgrades. The results from standard XRF analysis factorize the signal into elemental contributions and are therefore very useful to characterize an individual ink by delivering the individual fingerprint. The situation of trying to decipher a lost text provides a different question. The signal contribution from text of interest needs to be separated from the total signal, while element maps which most of the time will contain contributions from new and erased text do not necessarily give the best contrast for reading the erased text.

There are several components that contribute to the signal measured with XRF at each position. Let's assume a palimpsest which has one layer of erased text on both sides and one layer of new text also on both sides: The measured XRF signal at each point is composed of signal from the new and the erased ink from either or both sides of the parchment, as well as signal from the parchment itself and possibly also signal from dirt or other contamination (again from either or both sides of the parchment). The contribution of the inks (and dirt) from the two sides of the parchment are typically separated by using detectors on opposite sides of the page, thus considering the respective absorption of XRF signal passing through the page [Bergmann and Knox 2009]. The XRF signal mixture that remains for each side nevertheless is at least that of the two different inks, the parchment and the contamination on that side. Splitting this XRF signal mixture into element maps (as the standard XRF analysis does) may help to digitally recreate a hidden text, but may not provide the best results. If the two inks have the same trace elements (non-iron metallic components), only with different stoichiometric contribution or if either dirt or parchment have their contribution in the spectral range, where the inks are most diverse, no element map would

supply a really good reading contrast to identify the erased text. The results one should therefore aim to achieve for best reading contrast is ideally a complete disentanglement of the total XRF signals into the original mixed components. Those initial components from the example above are: Ink A, Ink B, parchment and multiple dirt components (as sand, mold, stains). Assuming a mapping experiment with at least tens of thousands of spectra measured a statistical approach is reasonable. Comparing all measured spectra for statistical similarities and differences is a task that mathematical procedures as “principle component analysis” (PCA) and “independent component analysis” (ICA) are designed for. Using comparative measures one no longer aims for clean separated elemental contributions, but for a basis set of signals, that reappear in different intensities in many of the measured spectra, as it is shown in the schematic example in Fig. 1. Every time ink A is present, it leaves its entire metallic fingerprint; the same is true for ink B. The only difference between two measured points with the both inks present is their relative amount due to the individual ink concentration at those points. The same holds true for the different dirt components or the parchment (which can e.g. vary in thickness). Mathematically speaking the best result in means of reading contrast for the erased text, could be achieved if a basis for all XRF measurements is found, were each basis vector represents one of the physical components. This is what PCA is designed to do: Separation not by elements (which are at least 5 or 6 in each ink), but by the inks fingerprints (just 2 in this case). This way ideally one map for each initial contribution could be reconstructed. Of course this is hypothetical and there are effects attached to real samples that will make life more difficult.

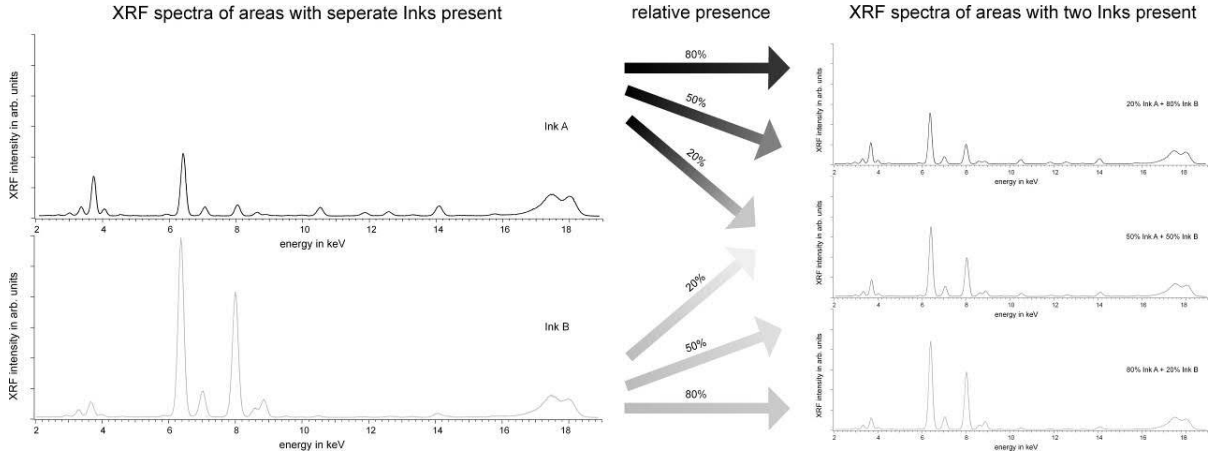

Fig. 1. Normalized XRF spectra of two Inks A and B on the left side and the recorded XRF spectra of a combination of those two inks on the right side with different relative amounts of the two inks present.

Each ink being present will contribute to the XRF signal alone in some spectra, while together with the second ink in others. These two occurrences of the ink will be separated by the PCA . When both inks are present the upper ink will absorb some of the fluorescence emitted from the lower ink. The absorption is stronger for low Z elements, since X-rays with higher energy are less likely absorbed in matter. This effect will lead to a slightly different contribution of the ink measured by the detector at this position and will create a PCA component representing the difference to the unmodified ink XRF fingerprint. For real measurements one may additionally assume noise and general inhomogeneity in all contributions that way additional PCA components representing this inhomogeneity are to be expected. Some of those contributions are less significant for PCA than others, since PCA repeatedly separates the most contrast containing contribution left in the data set. Therefore in the assumed example above among the expected first component found by PCA analysis should by the main ingredients (as the two inks and parchment) mixing into the various individual XRF measurements. Several additional PCA components are expected to address those correction terms. Hence the best readability contrast for any erased text is expected in one of the first PCA components. One very positive feature to be expected from this mere statistical approach run on a complete data set is that the computation time should be orders of magnitude shorter than that of using a multiple fitting algorithm on each spectrum separately. Due to the layered structure of the inks (one overwriting the previously erased ink) and the parchment, it is completely reasonable that some PCA components one will receive from upper layers have artificially negative contributions. For the total XRF spectrum it would be physically impossible to have negative contributions anywhere, but if an upper layer absorbs part of the signal emitted from a lower layer, this would lead

to a negative contribution in the energy spectrum representing the signal from the upper ink, since the ink removed signal of a specific energy emitted from the lower ink, before it could be recorded as XRF spectrum. With this in mind the standard PCA and ICA without any additions as non-negative matrix factorization is best suited for the task. It may be noted that non-negative matrix factorization has been applied successfully for XRF mapping of paintings [Alfeld et al. 2014]. The minimum requirement on results achieved with this new XRF analysis approach is that they should at least be of the quality and usability of that what the standard approach delivers. If that can be accomplished in much shorter time a lot is already gained. The data set used is from a XRF mapping experiment at the storage ring *DORIS III*[3] at the "Deutsches Elektronen-Synchrotron" (DESY) in Hamburg (Germany) and was initially evaluated using the AXIL program.

THE DATA SET AND PROCESSING

The measurements were performed at Beamline L of *DORIS III* at *DESY* in Hamburg (Germany) using a horizontally polarized, monochromatic X-ray beam with a photon energy of 18 keV and a flux of 10^9 photons per second. The beam was slit down to 70 µm horizontally and 100 µm vertically, creating a 100 µm x 100 µm footprint on the object that was mounted under 45° to the incoming beam. A single VORTEX "Silicon Drift Detector" (SDD) was used from the front side of the parchment, under 90° in respect to the incoming photons. This geometry setting is usually used at a storage ring to make use of the effect that there is no elastic scattering at this angle due to the horizontal polarization. This improves the quality of the specta, because the elastically scattered photons do not carry useful information, but increase the background and can lead to saturation effects. The XRF signal was pre-processed by a XIAmap multichannel analyzer and recorded using the *ONLINE* [4] beamline control software. The palimpsest (Cod. Lips. Rep. I 62, f 17 of the University Library Leipzig) was mounted on a 2D stage and moved continuously in front of the beam, measuring 2048 channel XRF spectra with 7 Hz (7 spectra per second). The stage was set to move 150 µm between two spectral readouts, while horizontal lines separated by 150 µm were measured. This way the resolution achieved was slightly better than 150 dpi. The element maps of calcium, iron, copper and zinc of the upper right corner of the page (302 lines of 721 points each) are shown in Fig. 2. Noticeable is a slightly smeared area in the copper map, where the copper had been washed out from the writing at some point in time, while no other element was affected.

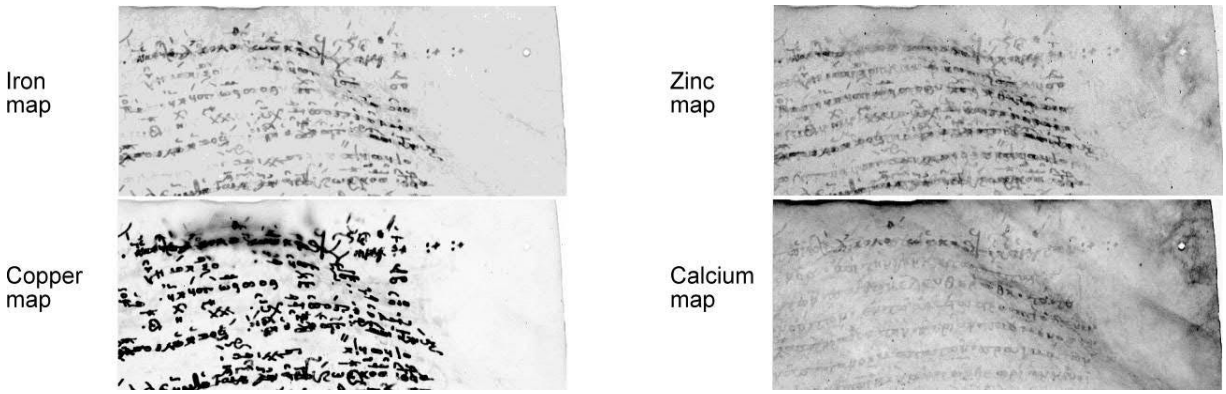

Fig. 2. Results from normal XRF mapping analysis. Element maps of calcium, iron, copper and zinc. Black corresponds to much intensity in all element maps.

XRF processing was performed on a scientific Linux PC using the *AXIL* code, always processing 4 spectra simultaneously. The results were element maps for potassium, calcium, titan, manganese, iron, nickel, copper, zinc, and lead. The lead originated mainly from a red band on top of the page and was slightly distributed equally over the entire page, but was also in traces part of the upper ink. In this case only element distribution maps of calcium, iron,

[3] *"**Do**ppel-**Ri**ng-**S**peicher" (DORIS)* was a storage ring initially build at DESY in 1974 for particle physics experiments. After the third upgrade (III) in 1993 the now called *DORIS III* functioned fully as X-ray source until it's decommissioning in 2012 (https://doris.desy.de/). It was replaced by the 8 times larger storage ring based light source the *"**P**ositron-**E**lectron-**T**andem-**R**ing-**A**ccelerator" (PETRA)*, likewise in the third upgrade stage (http://photon-science.desy.de/facilities/petra_iii/).

[4] http://hasyweb.desy.de/services/computing/online/online.pdf

copper and zinc were helpful to distinguish upper from lower text [Deckers and Glaser 2011] hence those will be used now to compare with the PCA and ICA results.

PCA and ICA processing was done on a Kubuntu[5] PC using ENVI5.5[6]. The data was saved in band sequential order and processed in ENVI. No pre-processing steps (except for binning in most cases) were undertaken before computing PCA and ICA components with the default settings. Full scene was used as input statistic information for both methods. The measured spectra were stacked in a cube such that channel maps could be generated and used as input pictures for the PCA and ICA. As a time optimization test the channel maps were first processed for all 2048 channels. Secondly all physically irrelevant channels were removed: The first 200 channels were cut, because they were below any element that could be detected by the SDD during this measurement with the air paths involved: Essentially anything in a spectrum representing elements before Potassium in the periodic table of the elements could not be real signal if anything were to be recorded there. Additionally the last 348 channels were cut, because they were representing energies above the energy of the primary photons and thus could be discarded as noise. Then the remaining 1500 channels were binned to 375 bins of 4 channels each. Finally the binning was more intensified, creating 125 bins out of the 1500 channels with 12 original channels in each bin. The results of the PCA were compared and no differences between the 3 sets were found in the main components of each of the 3 PCA runs. This was expected, since the information from a single element was spread over more than 30 channels in the original XRF spectrum of 2048 channels, due to the limited energy resolution of a SDD. Further analysis was then performed using the data cubes with 125 bins only, to maximize the time efficiency of the processing. PCA and ICA were run separately on the data sets.

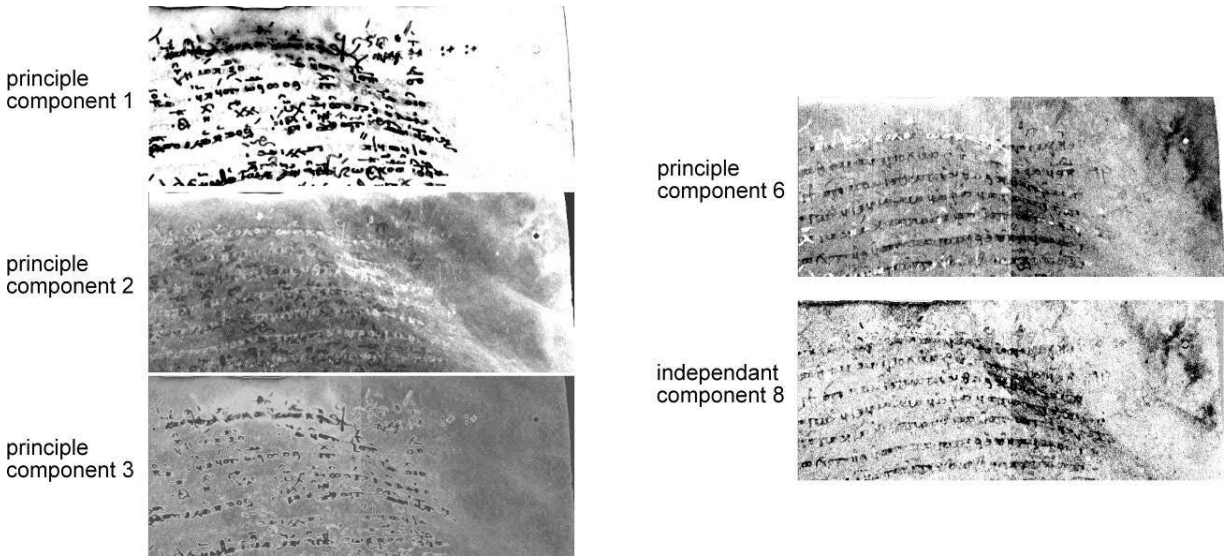

Fig. 3. Contrast maps created on basis of the components of the PCA and ICA analysis of the binned data sets. Black corresponds to highest above average value and white to lowest below average intensity in all components shown.

As expected PCA proved more useful with principal components 1, 2, 3, and 6 being of most significance in this case, while only the 8th independent component using ICA seemed to hold any useful information about the lower text. The PCA results are shown in Fig. 3. The picture from the first principle component shows a lot of the upper text, especially from the copper signal of the ink, since copper has the highest signal to background ratio with almost no copper in the parchment. Nice to see is the slightly smeared region as can be seen in the copper map in Fig. 2. The second principle component reveals some information about absorption in the measurement as explained above in the example with the two inks present at the same position. Due partial absorption by the upper ink there will be a principle component in the analysis, that has negative components (or below average in a normalized picture) for the

[5] https://kubuntu.org/
[6] harrisgeospatial.com

fluorescence absorbed by the ink. Here the contributions from the front side upper text are below average in intensity (light in color) and those from the backside are above average (darker color). The third principle component gives another picture from the upper text, this time showing mainly the differences between the copper and the iron based signal, where dominant regions of iron gives an above average signal and dominant regions of copper a below average total signal. Principle component no. 6 eventually reveals the underwriting as positive and some parts of the upper writing as negative contributions. The readability comparison standard is the Calcium map of Fig. 2 since that one was used to identify the text in the first analysis. The 8th component of the ICA gives a very similar result for the hidden text, only containing more noise and is as such less useful. The overall results look neither better than the element maps nor worse and can be used to read the erased text. The inherent feature of the PCA to produce a component, that shows which text is on which side is a bonus that can be useful, if a detector can only be placed on one side, as in the case of the used data set. The processing time for the classical XRF data analysis was 10 h for the 217.000 data points (even more when the parallel processing had not been used), while the complete data processing including binning, PCA and ICA was less than 20 minutes with most time spend on the processing of PCA and ICA. As ICA was not that successful in this case, a reduction to perform only PCA as fast analysis tool is reasonable and would get the total time down to about 10 minutes. A further acceleration of the analysis with more sophisticated algorithms is possible and will be reported elsewhere. When assuming the fastest expected scan speed reasonable today of 2 ms per point, the 217.000 points would be measured in slightly over 10 minutes[7]. The standard analysis had taken 10 hours. The expected speed gain for the analysis has been accomplished in full, while the resulting pictures were as expected slightly different, but equally useful.

CONCLUSION AND OUTLOOK

Especially PCA appears to work in favor of a quick processing alternative. The results are comparable in quality and deliver readable text quality, which allows judging the measurements success. The interpretation of the pictures in the sense of what they represent physically demands some additional input and understanding of XRF as method, the detectors used, as well as PCA, but especially in case of the PCA component 2 (in this case) the indication of whether a letter is on the front or the back of the page is a useful extra feature. For a quick and easy real time view of an ongoing measurement and for the mere identification and digitization of a hidden text PCA can be applied. Since no further assumptions or user interactions are needed the analysis method ideally qualifies for the intended application field. PCA can be performed automatically from a script using the entire spectra from a detector or optimized, when physically irrelevant regions of the spectrum and binning sizes can be given. For classical ink identification a handful of subsets from the original data can be summed up numerically and then processed with the standard XRF programs. This way the speed advantage of the statistical processing can be combined with the physical accuracy of the sophisticated analysis tools. In this example standard software was used for the PCA (IDL based ENVI), if the analysis protocol has been established and the code is run in e.g. Python or C++ further time saving can be expected, which is more advisable the more individual detectors are used during an experiment. Last but not least all processing can be done on standard hardware and platform independent, especially after the next step to implement open source code in Python.

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[7] 434 s for the mere mapping plus at least 1s for changing of each of the 302 lines (stage decelleration, lineshift and stage acceleration) gives a lower limit of 12 minutes for this mapping region with 2ms exposure per point

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