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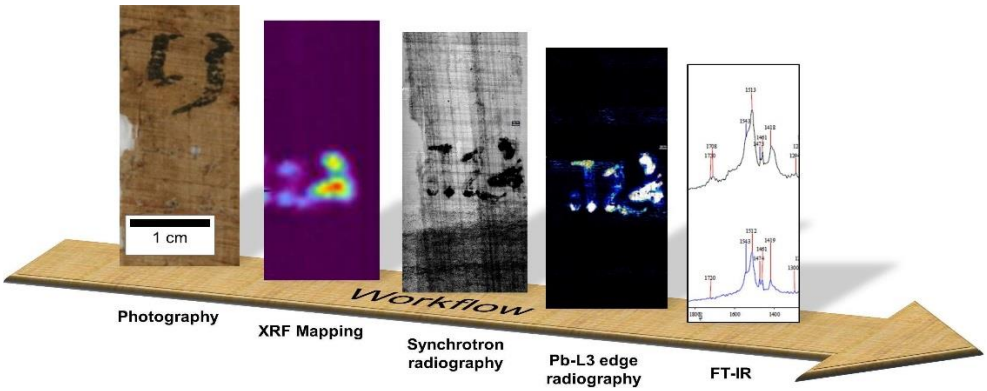
Graphical abstract



Highlights (3 to 5 bullet points, maximum 85 characters including spaces)

- Synchrotron imaging was applied to ancient papyri with ferrous and plumbiferous ink.
- XRF was used for first, quickly available information about Fe and Pb distribution.
- Absorption edge radiography revealed element-sensitive distribution.
- Lead carboxylate was identified as invisible ink by means of FT-IR analysis.

Keywords
Elephantine papyri; synchrotron absorption edge radiography; non-destructive; X-ray; FT-IR; lead carboxylate

Abstract
In the Egyptian Museum and Papyrus Collection Berlin a multitude of papyrus manuscripts is stored. Of special interest are papyri found on the island Elephantine, no other settlement in Egypt has been so well documented through texts over four millennia. But 80% of the Elephantine texts are yet to be documented and published.

As part of the "Elephantine" project, funded by an ERC starting grant, we try to get access to hidden texts. Most of the fragments are very fragile, deformed, some rolled or folded. Papyri from the Old and Middle Kingdom were typically written with carbon ink. Consequently, these fragments show no absorption sensitivity for hard X-rays. If small admixtures of high-Z elements, like Fe or Pb, are found, absorption may be sensitive enough for radiography and tomography to distinguish writing and base material. We sorted out suitable fragments and papyrus packages by X-ray fluorescence mapping. When promising high-Z elements were detected, absorption tomography using micro-CT laboratory systems or synchrotron X-rays at the BAMline at BESSY II was applied. The sensitivity can be enhanced by element-sensitive absorption edge imaging, where transmission data taken above and below the edge are compared. This technique was applied at the absorption edges of the elements Fe, Sb and even Pb, known as used ink and pigment material. These X-ray results were complemented by Fourier-transform infrared spectroscopy (FT-IR) measurements showing that the lead found is in the form of lead carboxylate. In the future, the presented workflow will be applied to folded or rolled papyri, allowing for analyzing writings without a manual opening of the fragments.

Introduction
In the Egyptian Museum and Papyrus Collection Berlin a multitude of papyrus manuscripts is stored. Of special interest are papyri found on the island Elephantine.

The objective of the ERC research project ELEPHANTINE is to write a 4000-years cultural history of Elephantine Island in Egypt. An important island in terms of military strategy, Elephantine is located in the Nile on Egypt's southern border as the center of the Aswan region. No other settlement in Egypt has been so well documented over such a long period. Its inhabitants comprise a multi-ethnic, multicultural and multi-religious community, which has left behind large amounts of written material, which provides evidence of everyday life from the Old Kingdom right up to the era following the Arab conquest. The texts are written in various languages and scripts, including Hieroglyphs, Hieratic, Demotic, Aramaic, Greek, Coptic and Arabic. 80% of these manuscripts are yet to be published and investigated [1]. Through international cooperation, the "papyrus puzzle" can thus be solved – also with the help of cutting-edge new methods from the digital humanities, physics or even mathematics. Most of the fragments are very fragile, small pieces, deformed, even rolled or folded. Therefore, it is impossible to get access to these writings by opening some of these delicate and precious fragments manually. Here, we present a non-destructive and non-invasive technique based on X-ray imaging, allowing the access to hidden texts in rolled or folded manuscripts. For additional information about the chemical form in which the detected elements were bound in the inks, FT-IR was also applied.

Research aim
As part of the "Elephantine" project we try to get access to hidden texts. A non-destructive method, that allows accessing the hidden information, is urgently needed. In particular, the inter- and multidisciplinary research (e.g. imaging technologies) has reached a high quality in recent years allowing to address this challenging task. Therefore, we developed a workflow that allows getting fast access to hidden texts: suitable fragments and papyrus packages are sorted out by X-ray fluorescence using a portable instrument, if possible by elemental mapping. Based on the XRF results, proper tomography techniques and setups are chosen, such as absorption or phase contrast tomography. For a

fast access, micro CT laboratory systems containing a conventional vacuum tube are applicable but lack in spatial resolution and sensitivity of certain elements. Synchrotron devices, as used for the present work, are less applicable but allow an energy selective tomography. Here, element-sensitive radiography was performed at the BAM*line* at BESSY II at Helmholtz-Zentrum Berlin, Germany.

The combination of these methods allows a selective access to suitable samples, which are then subjected to more sensitive measurements. Although chemical compounds are an interesting topic and contain supplementary information, we will only deal with such a technique briefly.

Material and methods

Information related to the Elephantine papyri that were selected for these measurements are described below.

Papyri fragments

Two fragments were analyzed in this study. Both fragments were taken from the Egyptian Museum Berlin and Papyrus Collection Berlin, Germany. Photographs of both the recto and the verso side are shown in Figure 1. Fragment “ÄMP B/H x 133g” (shown in Figure 1a and b) contains two different writings in black and red ink on the recto side. The photography of fragment “ÄMP B/H x 352” shows black ink, only. A tiny sample (< 1mm) was collected for FTIR analyses.

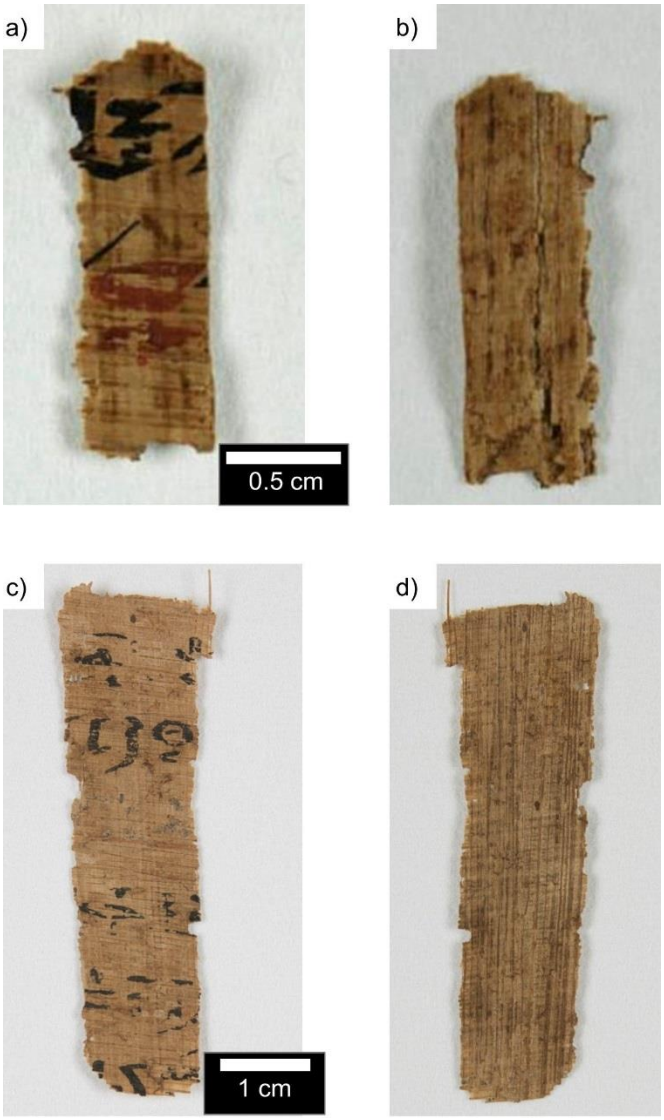


Figure 1: a) Recto and b) verso side of fragment "ÄMP B/H x 133g", c) recto and d) verso side of fragment "ÄMP B/H x 352". All pictures were taken by T. Siopi (AMP)

Portable XRF spectrometer

X-ray fluorescence (XRF) allows for a non-destructive elemental analysis of a sample. An incident beam of high energy is pointed on a sample. The emission, fluorescent X-rays, is detected by an energy-dispersive detector (8k MCA). An example is given in Figure 2c, taken with the ELIO system from XGLab. For the presented measurements, a high voltage of 40 kV was applied to the Rh-anode of the X-ray tube, and a current of 200 µA was allowed. The incident X-ray beam was collimated to 1 mm^2. The setup is shown in Figure 2a. The spot of the incoming X-ray beam is visualized by two laser points on the fragment, as seen in Figure 2b. If both laser spots merge to one single spot, then the X-ray beam is focused on the surface of the fragment. Beside single point measurements, an element-sensitive mapping of whole fragments can be performed. For mapping, only a small energy bandwidth around an element-characteristic X-ray emission line is selected for each mapping point. The peak corresponds to one specific element. Subsequently, the scanning along the fragments surface provides element-sensitive, two-dimensional information about the fragment.

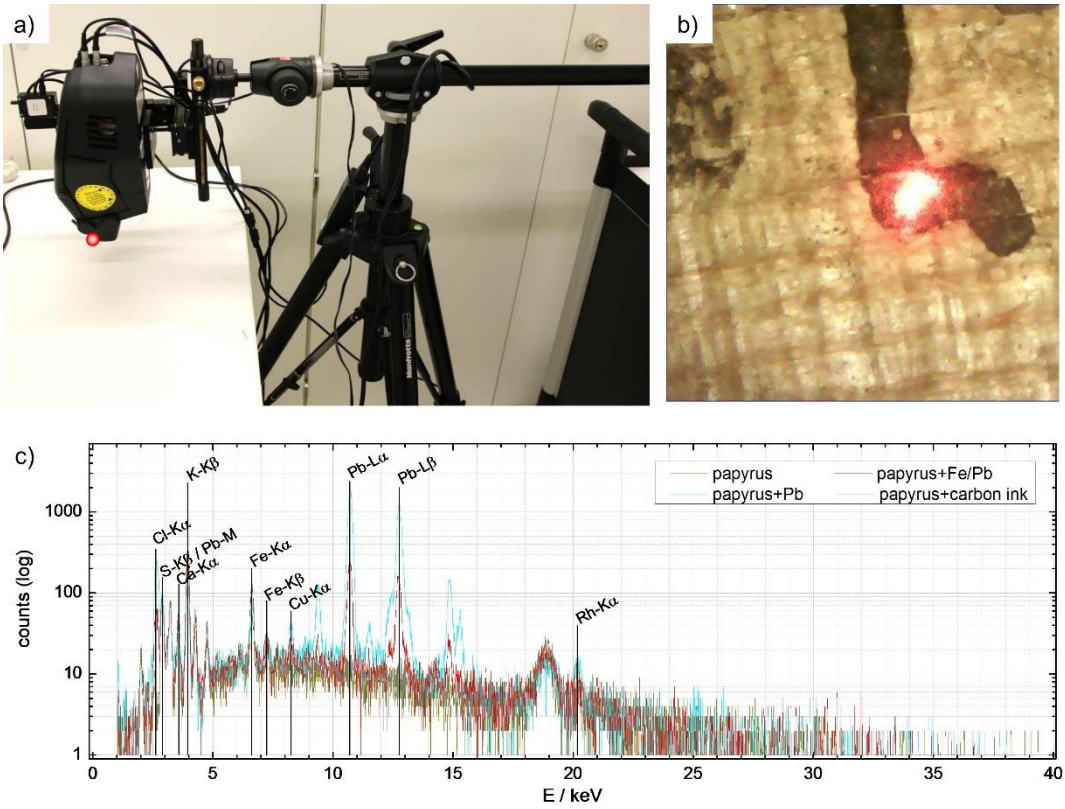


Figure 2: a) Mobile XRF spectrometer ELIO, b) laser spot on fragment and c) measured spectra. For element-sensitive mapping, a proper energy bandwidth around one peak is selected

Synchrotron imaging

Compared to laboratory X-ray devices, synchrotron X-rays exhibit a high brilliance. The most important consequences for imaging issues are the increased number of photons per time period and a real cross section and a high coherence. The latter one is important for phase contrast measurements, whereas an increased number of photons allows for fast and additional energy-selective measurements within acceptable time periods. This method may basically be considered to be non-destructive like conventional x-raying with the advantage of tailoring the useful energy of the synchrotron radiation needed for the investigation. But in modern synchrotron facilities, so-called 3rd or even higher generation sources can easily deliver intensities much higher than typical X-ray tubes, so that intensity tests are needed before starting investigations.

Hard X-ray synchrotron imaging was performed at the BAMline at the synchrotron source BESSY II at Helmholtz-Zentrum Berlin, Germany [2]. The electron storage ring was operated in top-up mode providing stable X-ray beam properties at the experimental stations. The tomography station at the BAMline allows for spatial analysis of high resolution of a multitude of samples. A sketch of the setup is shown in Figure 3a. The beam can be formed using different monochromators and slit systems. In this study a double multilayer monochromator (Si/W) was used in order to lower the energy bandwidth to $\Delta E/E=10^{-2}$ (FWHM). An energy of 19 keV was chosen for standard radiographic measurements while 7.0 keV and 7.2 keV (Figure 3b) and 12.90 keV and 13.15 keV were chosen for measurements at the absorption edges of Fe (K-edge) and of Pb (L_{23}-edge), respectively. With the versatile detector system, different magnification optics can be combined with different CCD cameras and scintillator screens. For the presented work, a 20-µm thick CWO_4 scintillator was combined with a 10-fold magnification optics and a pco4000 CCD camera, resulting in a pixel size of 440 nm.

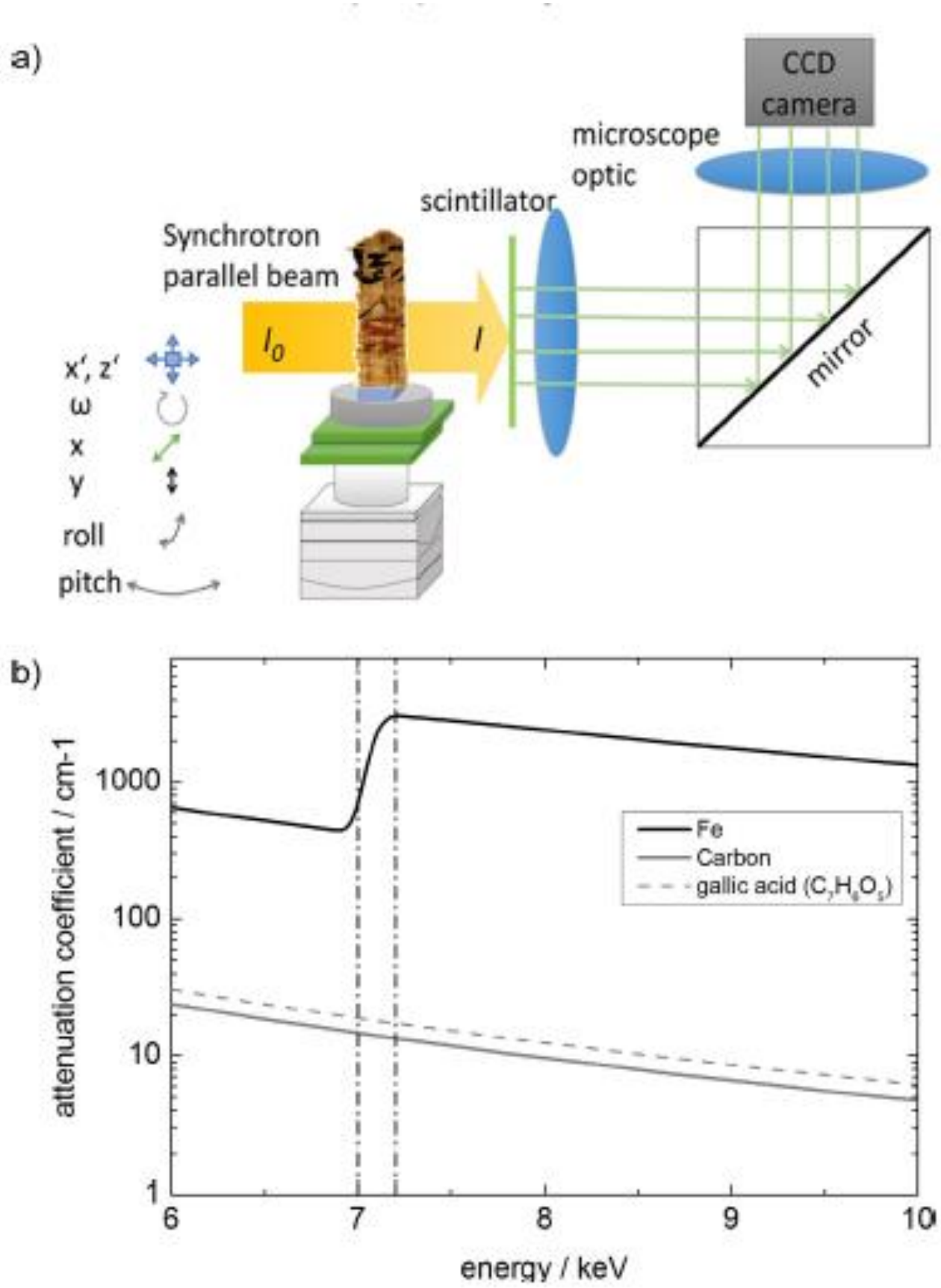


Figure 3: a) Setup of X-ray radiography. b) Two discrete energies were selected below and above the absorption edge of specific material (here: Fe); absorption coefficients taken from XOP software [3]

The transmittance, which is the ratio of the incident beam I_0 and the transmitted beam I is measured with a spatial resolution of 440 nm for each pixel of the CCD camera. A scintillator converts X-rays to visible light which is detected by the camera, see Figure 3a. All radiograms were corrected for the camera offset and normalized to the beam intensity. The division of two radiograms, one taken below and the other one taken above the absorption edge, was done ex-situ. Consequently, the thickness of the specific element (here: Fe) can be calculated. The obtained images yield a higher sensitivity for thickness analysis than the transmission radiography does. Element thicknesses of down to a few tens of nanometers can be determined. The normalization and image analysis were performed with ImageJ [4].

Raman spectroscopy

Raman spectroscopy was done using the Horiba XploRa Raman-microscope equipped with Lasers at a wavelength of 532 nm, 638 nm, and 785 nm. By selecting different filters, microscope objectives and spectroscopic gratings, different beam intensities as well as spatial and spectroscopic resolutions can be achieved. The laser powers are 25 mW (532 nm), 24 mW (638 nm) and 90 mW (785 nm). The maximum spatial resolution is 1 µm. The spectra are given in Raman shift [cm^{-1}] (shift of wavelength/frequency relative to the laser used given in wavenumbers). The calibration of the spectrum is accurate to approx. 2 cm^{-1}. Only carbon could be identified within the writings (see Figure 4).

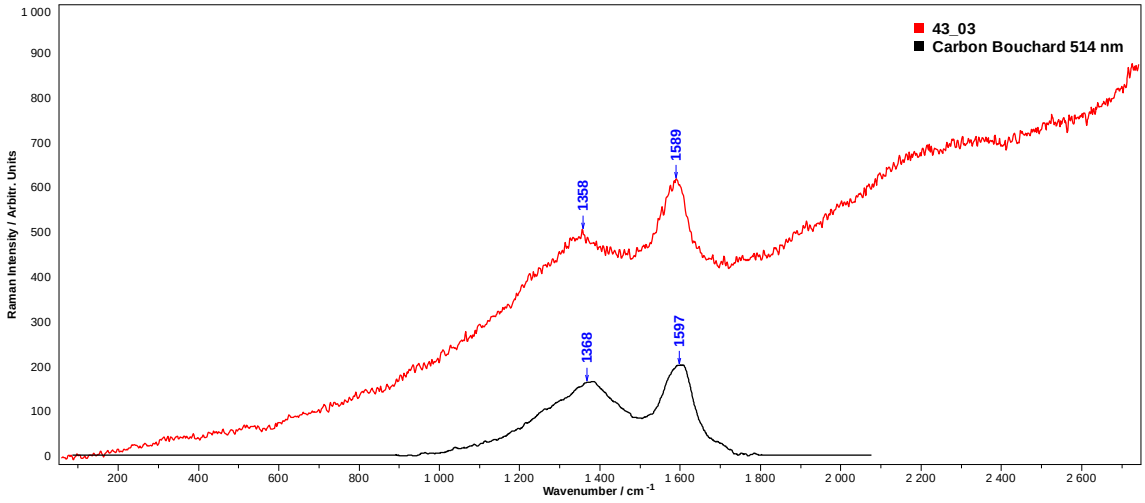


Figure 4: Raman spectra of carbon reference (black spectrum) and of writing on fragment ÄMP B/H x 352 (red spectrum)

Fourier transform infrared spectroscopy

A Perkin Elmer instrument Paragon 1000 PC was used for the Fourier transform infrared spectroscopy, coupled with an FT-IR – microscope in transmission mode in the range of 4000 - 520 cm^{-1}. The spectral resolution is 4 cm^{-1}. The calibration was done at different point as listed in *Table* 1. The sample was prepared on a diamond measuring cell from High Pressure Diamond, from Perkin Elmer in transmission.

Table 1: Peak calibration of FT-IR spectra

Wavenumber / cm^{-1}	Interpretation	Source
2919 + 2955sh	CH_2/CH_3 asymmetrical stretching	[5]
2850	CH_2/CH_3 symmetrical stretching	[5]
1512 + 1543 sh	COO^- asymmetric stretching	[6]
1461	CH_2 bending (characteristic for lead palmitate or stearate	[6]
1419	COO^- symmetric stretching	[6]
930	CH_2 rocking	[6]

The preparation of Lead(II)stearate was done on Dec 15, 1998 by C. Herm. Sodium stearate ($C_{18}H_{37}O_2Na$) and 0.306 g (0.01 mol) were dissolved in 10 mL water. Lead(II)acetate-trihydrate 0.190 g (0.05 mol) was added and the solution was brought to boiling, afterwards. A white precipitate is formed and filtered after cooling to room temperature. The residue is dried on the ambient air. The yield was 0.390 g, respectively 100%.

Results and discussion

A workflow that allows for element-selective measurements is presented in this work. It is focused on single-layered, flat fragments, since the feasibility needs to be proven before applying to 3d-objects.

As a *first step*, a mobile XRF is used in order to obtain information about the content of metals inside the fragments. XRF is a fast accessible and easily available method. Although mapping allows for spatially resolved measurements, there are certain reasons to complement this method with others. One reason is the spatial resolution of portable XRF devices, often around a few mm^2, in our case approximately 1x1 mm^2 along the surface of the samples. This may be insufficient in order to detect writings readably. A more serious issue is related to the depth resolution perpendicular to the sample surface which actually is not present. The penetration depth of the initial X-ray beam is quite high, but the emitted X-rays are of lower energy resulting in higher absorption from deeper parts of the sample. Subsequently, the majority of the detected intensity contains near-surface information. Additionally the geometry for excitation and detection is optimized for the region where the X-ray beam hits the sample well focused. Consequently, this method can be considered as a surface-sensitive technique. Consequently, this (portable) XRF is very useful for getting first qualitative information about the sample.

Exemplaric, we analyzed iron and lead here. Iron was found in every papyrus fragment studied, which is on the one hand partly attributed to the manufacturing of the base material, the papyrus, and on the other hand to the origin, the geological situation and the desert climate of the storage place as well, with an abundance of Fe containing materials like ochre or hematite in most of the archeological sites. Thus, the iron content may also vary in dependence on the age of the fragments as well as on their former and intermediate storage containment and conditions. And the material of interest, the ink, contributes to the signal, too, if made out of ferrous ink. Therefore, qualitative information about the iron content and its lateral distribution is of great interest. We can also expect lead to be used as a pigment, at least as additive in inks used in the Roman period, but occasionally found in earlier periods as well. Thus this element is worth to be analyzed.

The qualitative information, revealed by XRF, is a crucial criterion for selecting suitable fragments for time consuming tomographic methods.

The *second step* is X-ray transmission radiography. The ratio of the incident beam and the transmitted beam is detected spatially. The field of view is only 1.7 × 1.2 mm^2 in width and height. Therefore just a small part of the whole fragment was measured for these feasibility studies. Larger fragments or areas of interest must be scanned or measured with a lower spatial resolution. Figure 5a shows a photo of the fragment “ÄMP B/H x 133g”. Subfigure b) gives an impression of the superimposed transmission radiogram. Areas of high X-ray attenuation (caused by ferrous particles or other high-K elements) are shown in light gray values, areas of low attenuation are shown in dark gray values. Latter are attributed to low-K elements like carbon. In this way, structures within the fragment and the group of high-Z elements can be identified two-dimensionally. The fiber structures of the papyri base material are clearly visible for the recto side (horizontal structure) and verso side (vertical structure) of the fragment. Additional bright spots can be observed dispersed the whole radiogram. Probably these are metals, likely iron containing dust. To verify this statement, a more detailed analysis needs to be done.

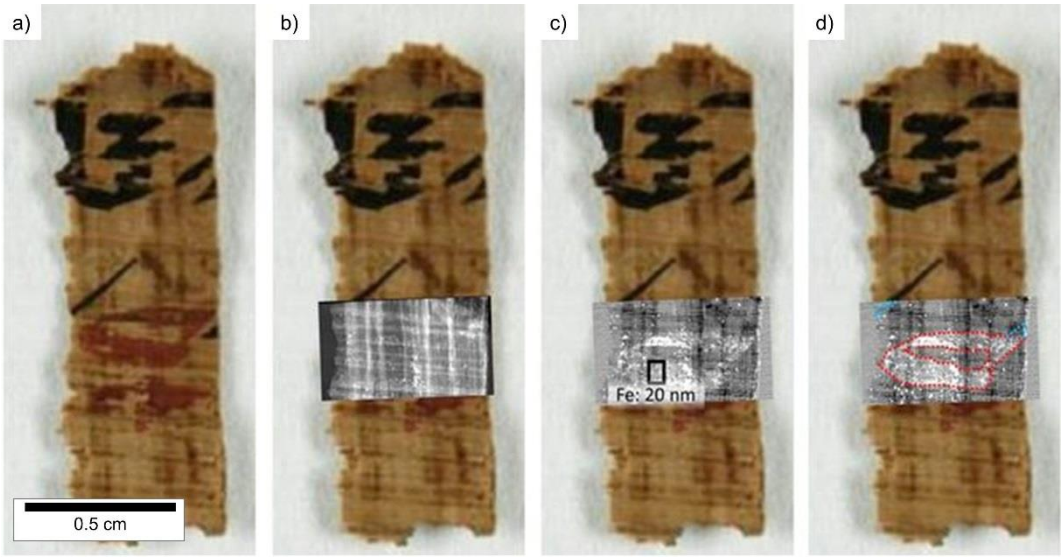


Figure 5: Fragment "ÄMP B/H x 133g". a) Photo of the fragment. Superimposed is in b) the transmission radiography, in c) the thickness of Fe and with the contours of the letter marked in red in d)

Consequently, as a *third step*, we applied synchrotron X-ray absorption edge radiography. This method is sensitive for single elements. A special element can be analyzed by selecting the corresponding energies below and above its absorption edge [7,8]. Energy edges with a sizable change of the attenuation, like K or L3 edges, are preferred. Thus the K-edge was chosen for iron, while the L3 edge was chosen for lead, since the K-edge of Pb (at 80 keV) is beyond the range of the beamline.

Figure 5c-d are showing the results for fragment "ÄMP B/H x 133g" for the absorption edge radiography for iron. Thicknesses of down to few tens of nanometer can be analyzed. As indicated in Figure 5c the absorbing effective Fe thickness is determined to be approximately 20 nm. The writings of the red ink, can also be identified in the X-ray images. The letter is emphasized in subfigure d). A direct comparison of transmission radiography and absorption edge radiography shows that the latter method is much more sensitive for the spatial distribution of the ink.

Compared to previous XRF measurements, the Fe distribution can be measured with a higher spatial resolution and, additionally, quantitatively. First enhancement allows for a more reliable letter recognition, which is hardly possible with mobile XRF devices. The measurement of the total iron amount also may contribute to the letter recognition of multilayered fragments. Hereby, self-shielding becomes a rising problem for XRF measurements. Those measurements are planned for the future.

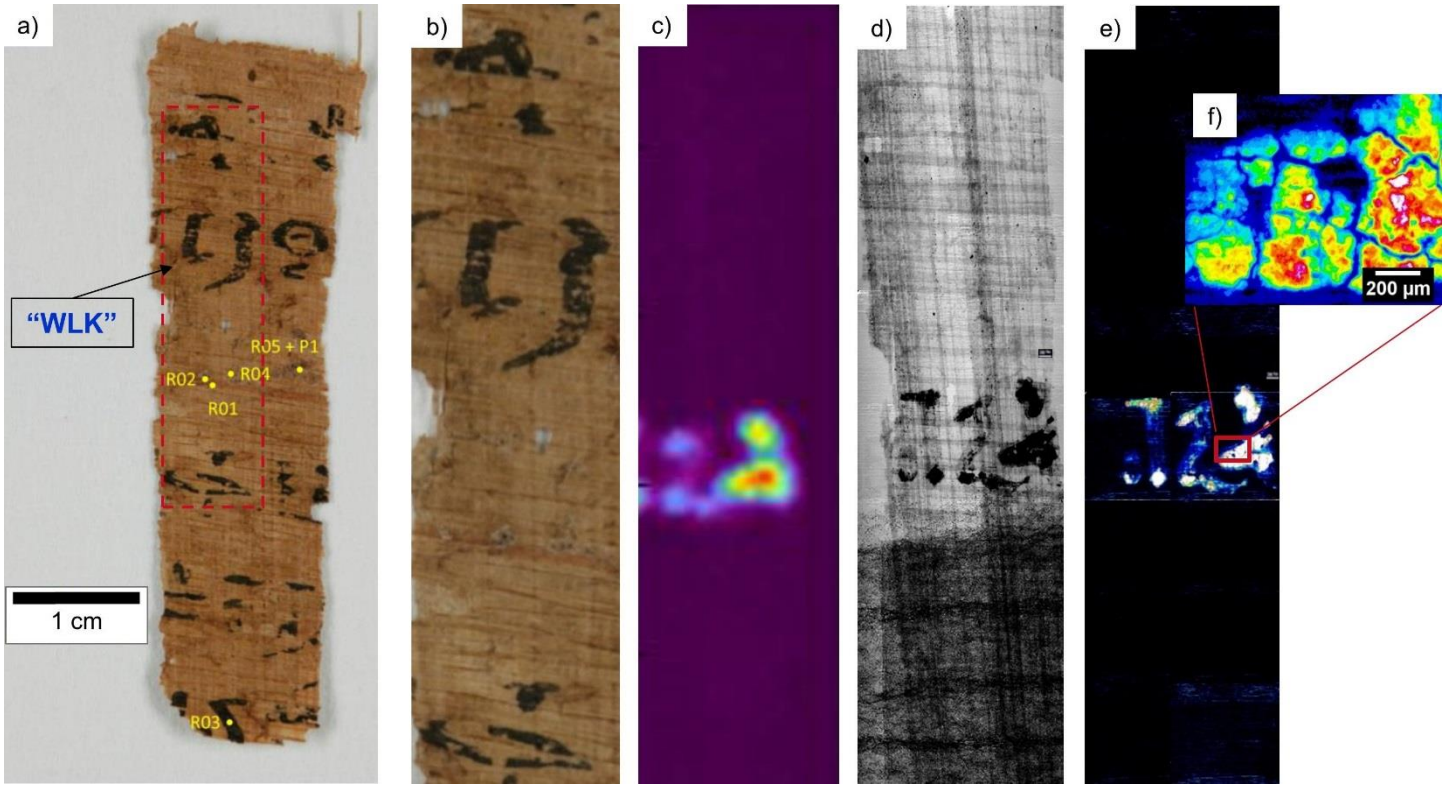


Figure 6: Fragment "ÄMP B/H x 352". a) Photo of the fragment with marked area that is enlarged in b) and indication of the Raman measurement's spots (R) and the sampling spot (P). Subfigures b)-e) show the same area. c) XRF mapping for Pb. d) Transmission radiography and e) absorption edge radiography at Pb_{L3} edge with enlarged cutout

Another fragment "ÄMP B/H x 352" with hieratic writing was analyzed using absorption edge radiography. This fragment dates back to the Greek-Roman period. It is a single-layered, flat fragment and can generally be read by naked eyes. Thus, it is well-suited by means of testing outstanding measuring methods. It is shown in Figure 6. The readable writings had been written with carbon ink. No high-Z elements were found by XRF. Their meaning is "WLK" and does not provide any reliable information at this state of knowledge. An enlargement of the fragment is shown in subfigure b). The same area was analyzed by lead-sensitive XRF mapping by filtering the Pb fluorescence spectrum around the Pb-L-line at 10.55 keV. The results are shown in subfigure c). Interestingly, a distinct signal was measured at areas without visible writing, and no signal where letters are visibly written in presumably carbon ink. A transmission radiogram taken at 19 keV is shown in d). Dark gray values represent elements of high atomic numbers. Consequently, there must be some elements of higher atomic number present. Switching the X-ray energy to the L3 absorption edge of lead revealed the information that this attenuation is caused by lead, see subfigure e). An enlargement of the absorption edge measurement is shown in subfigure f). The thickness of lead is given in false color. Cracks of the inks are visible. These are probably caused by drying processes during the long period of time. Following a suspicion that the lead may be a mixed compound containing the element antimony as well, an absorption edge radiography at the K-edge of Sb gave no indication for the presence of antimony. Additionally, arsenic sulfide compounds like e.g. orpiment could be present, too, their existence could have been escaped our detection in the absorption edge radiography. However, the XRF spectra in the region of the hidden writings showed no significant contribution (see Figure 2c).

Already the XRF on a single spot showed a remarkable intensity in the Pb L-lines without any visible writings. That is a noticeable difference to the findings reported by Christiansen [9] on various fragments of the Copenhagen collection, where the authors did not find any Pb in regions without writings (plain substrate). While XRF as well as the presented absorption edge radiography only allows determining the element apparent in the ink material and making the writing readable even when hidden, these methods do not give direct information on the chemical form in which the element is bonded in. In *step four* we have therefore applied various infrared techniques sensitive to the chemical bonds.

With Raman spectroscopy, we could confirm that carbon is the major component in the black ink (e.g. at the marked spot R03 in Figure 6a), and in the case of the very faint reddish bar in the central part of the fragment, hematite (spot R01) and goethite as the typical components of red and yellow ochre (spot R02). No substance could be identified by means of Raman spectroscopy in the region of the intense lead and therefore a small piece of substance taken from this region (at R05+P1) to be analyzed by means of FTIR.

Here the Fourier transform infrared spectroscopy was employed on that small piece extracted from the relevant region (P1). The result is presented in Figure 7 in comparison with a spectrum for a lead carboxylate (lead stearate) from the data base of the Rathgen-Forschungslabor illustrating perfect agreement. This metal-organic compound shows up in FT-IR and indirectly via the elemental sensitivity in absorption edge imaging. Consequently, the lead found in the fragment, is due to a lead carboxylate. Its use by intention as an ink for encryption seems very unlikely and, therefore, it is probably the result of a chemical transformation of a pigment originally used.

However, which was the substance originally used as pigment, we can only speculate on. It could have been minium, the red mixed lead oxide Pb_3O_4, known to be used in the Roman period, but galena, black lead sulfide, or lead white, cannot be excluded. An optically stimulated transformation might have been the underlying reason under the presence of carbon dioxide and/ or oxygen and / or occasional moisture, and over such a long period of time, even slow processes might have led to complete reactions into non-colored compounds leading to an almost invisible writing.

Finally, it should be mentioned, although a translation is presently not possible, that the symbol revealed from the “hidden” text may be interpreted as a determinative for a divinity.

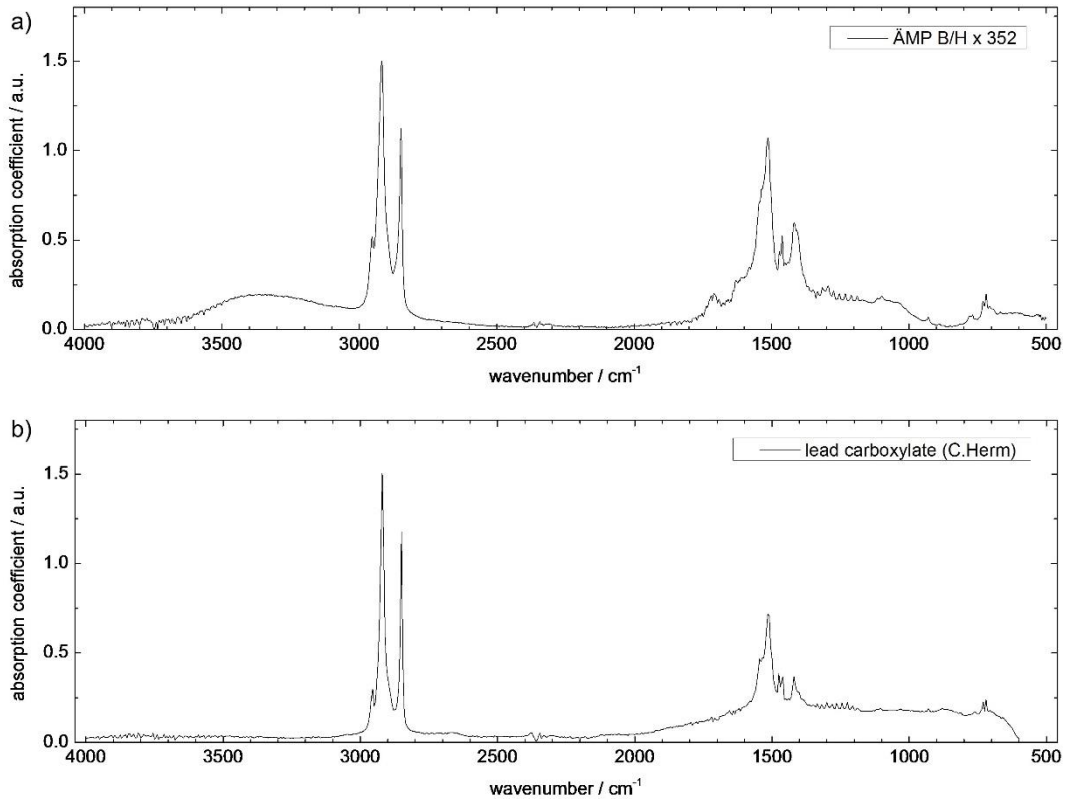


Figure 7: a) FT-IR measurement within the area of the hidden symbol of fragment “ÄMP B/H x 352”. b) FT-IR of lead carboxylate (thanks to C. Herm for providing the reference sample and spectra)

Conclusions and Outlook

Cultural heritage research is an important, omnipresent topic of human history. It offers a great opportunity to look back on the past. But it is essential to investigate historical artifacts non-destructively. This also applies to the Elephantine Papyri. These fragments are partly in a critical condition, so that a manual treatment like unrolling or unfolding should be avoided. Nevertheless, getting access to the content of these fragments is of vital interest.

In the presented work, a promising combination of mobile XRF, synchrotron imaging and FT-IR was used in order to develop a workflow that allows visualizing ink in ancient papyri. With the advantage of being able to verify the results by visual inspection, two-dimensional papyri were examined in this feasibility study. The focus was set on papyri that were written with ferrous or plumbiferous ink.

While XRF mapping turned out to be a straightforward method that allows for a rough location of iron inside a fragment, the spatial information and the sensitivity of the mobile XRF spectrometer may not be sufficient for an actual reading of the writings. Transmission radiography provides an improved spatial resolution. When inks containing high-Z elements have a sufficient thickness, an extraction of the textual information is possible. As demonstrated, absorption edge imaging improves the sensitivity by adding elemental sensitivity specifically with a very effective background suppression. By choosing well-defined energies around the absorption edge of the element in question, it delivers very detailed information about the distribution and thickness for this specific element. In the presented work, this is shown for ferrous and plumbiferous ink, but applicable to other elements as well provided the energy of the X-ray beam can easily be varied as is the case at most synchrotron sources.

Here, the combination of above mentioned methods yielded interesting results which were complemented by FT-IR measurements: lead was found in areas without any visible ink, and the chemical compound was identified as a lead carboxylate. This is remarkable, since our example presents evidence for the use of a lead compound as ink material, not just as an additive, in this historical period.

As a next step, these methods will be applied to folded or rolled fragments. Even for 3D analysis, the spatial and temporal resolution of the presented methods seems to be well suited. The only crucial issue is the availability of suitable synchrotron beamlines.

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Conflict of interests

Declarations of interest: none.

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