XRF Ink Analysis of Selected Fragments from the Herculaneum Collection of the Biblioteca Nazionale di Napoli

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Abstract

The most commonly used ink in antiquity was carbon-based, and the main element of carbonized papyrus is carbon, making conventional computed tomography (CT-scanning) of Herculaneum scrolls difficult. However, Roman and Greek inks containing metals have recently been identified in some papyri from Egypt, changing our understanding of ink technology in antiquity. This raises hope that some rolls can be virtually unrolled by CT-scanning. Here we present the results of a preliminary analysis, aimed at identifying scrolls whose ink contains metals.

Keywords

Herculaneum, XRF, ink

Introduction

Nearly 1000 papyrus rolls, carbonized during the eruption of Mount Vesuvius in 79 A.D., were discovered between 1752 and 1754 in the so-called Villa dei Papiri at Herculaneum. The charred fragments trapped in lava were at first overlooked or even discarded by the excavators, since they were mistaken for burnt wood or fishing nets.[1] However, once the importance of this discovery was acknowledged, various attempts were made to unwrap both complete rolls and fragments, the most fruitful of which was the method of Antonio Piaggio, who finally made possible the publication, from 1793 onwards, of many important, mainly Epicurean, texts contained in the Herculanean library. To

[1] Delattre 2006, 20; Longo Auricchio / Indelli / Leone / Del Mastro 2020, 53-59.

avoid putting the scrolls at risk, since 2003 efforts have been focused on virtual unwrapping using computed tomography (CT-scanning). However, this technique usually requires a good chemical contrast between the ink and its support in order to be used effectively. Until recently, the common belief was that only pure carbon ink was used in antiquity. However, recent discoveries of the addition of metals in ancient carbon ink has raised hope that CT-scanning may be used on at least some of the scrolls, provided that they contain metals. For this reason, in June 2018 a preliminary campaign of analysis took place at the Biblioteca Nazionale in Naples, where nearly all the rolls and fragments are kept, in order to analyse the metal content of a selection of fragments resulting from partially mechanically unrolled scrolls. Those scrolls in whose ink metal is detected would be good candidates for CT-scanning.

Unwrapping methods attempted on the Herculaneum scrolls

The first methods tested on the scrolls from Herculaneum were mechanical and put the objects at great risk of disintegration. They were used from the middle of the 18th century until the beginning of the 1990s. Since they have already been described elsewhere, only a brief summary of the development of such methods is presented here.[2] One of the first approaches, the so-called scorzatura totale (full peeling), was developed by Camillo Paderni. His method consisted in first moistening the scrolls in a hydroalcoholic solution, before cutting them longitudinally. The most internal layers of the two hemicylinders, containing much less text, were often destroyed in order to access the more extended inner surfaces of the outer windings of the rolls. Since Paderni was only interested in making these outer windings visible, in many cases the midollo (inside layers) survived this procedure (when the midollo was not disposed of, we talk about scorzatura parziale). In 1753, Antonio Piaggio started to work on these remainders which had survived Paderni's attempts by means of a special machine, with the help of which a significant number of midolli could be unrolled and single layers of many scrolls that remained from Paderni's attempts could be separated.[3] After separation and vertical fixing of a small portion of the external layer of such a midollo by means of silk threads, the papyrus was lowered very slowly, while the already separated part of the layer, consolidated with bands of goldbeater's skin obtained from sheep or pig bladder, was kept in the same position, so that more and more parts of the layer were gradually unrolled. Of course, what was obtained were at best only the final parts of an entire scroll and not a complete unwrapped papyrus. However, this apparatus

[2] For more details, see for example Angeli 1994; Delattre 2006, 29-39; Longo Auricchio / Indelli / Leone / Del Mastro 2020, 59-68.
[3] Blank 1999.

uncovered many substantial pieces of text and has given us by far the largest part of what we now know about the content of the Herculanean library.

Piaggio's machine remained the most successful method and was still in use in the first decades of the 20th century.[4] In addition, throughout the 19th century and until the 1960s many other methods to open Herculanean papyrus fragments were tested, without satisfying results.[5] The latest mechanical method of unwrapping, the so-called Oslo method, was developed by Knut Kleve and colleagues and applied to Herculanean fragments between 1983 and 2003.[6] The Oslo team used solutions of gelatine and acetic acid in different concentrations to consolidate the papyrus before slowly and gradually manually unrolling it. When multiple layers remained attached on the same surface, they were separated by means of an alcoholic solution. While the process was much better documented (in particular regarding the position of resulting fragments) than what had been attempted with Piaggio's machine or by other previous methods, it still had only limited success, and partial destruction and extreme fragmentation of the papyrus pieces could not be avoided.[7] As a result, there have been no attempts to mechanically unroll the scrolls since 2003 and it is highly unlikely that any will ever happen again. Instead, attention has been focused on methods to virtually unroll the scrolls, which would allow access to the text while preserving the integrity of the objects.

Multispectral imaging techniques, and especially infrared imaging, have proved extremely useful to read detached fragments on which the black ink can hardly be distinguished from the charred papyrus support.[8] Recently, infrared imaging has been combined with Reflectance Transformation Imaging (RTI) with good results.[9] However, in order to access text from internal layers, and eventually virtually unroll the scrolls, one needs light with energy sufficient to penetrate through the different layers of papyrus, e.g. X-rays used in the CT-scanning.

The usefulness of CT for virtually unrolling scrolls has been demonstrated by the recent results such as the virtual unwrapping of the Bressingham scroll, the En-Gedi scroll and modelling attempts.[10] In any case, the technique is applied to scrolls with inks that contain a prominent metallic component. However, with the Herculaneum papyri we are facing a much more complex case. First, the support consists of papyrus, which is a much more heterogeneous material than majority of the leather–based scrolls. Then, the scrolls from Herculaneum are extremely deformed due to the eruption

[4] Capasso 1991, 109.
[5] Angeli 1994, 81-85.
[6] Capasso 1991, 112-116; Angeli 1994, 85-86. Work of the Oslo team in Naples is last recorded for 2003 in the *Notiziario* of Cronache Ercolanesi 34, 2004, 231.
[7] Delattre 2004, 1364-1366; Delattre 2009, 942.
[8] Chabries / Booras / Bearman 2003. On the limits of multispectral technique, see MacFarlane / Del Mastro / Antoni / Booras 2007.
[9] Piquette 2017.
[10] On the Bressingham scroll: Mills / Curtis / Davis / Rosin / Lai 2014; Rosin / Lai / Liu / Davis / Mills / Tuson / Russell 2018. On the En-Gedi scroll: Seales / Parker / Segal / Tov / Shor / Porath 2016. On modelling attempts: Allegra / Ciliberto / Ciliberto / Petrillo / Stanco / Trombatore 2016.

and are much longer than other scrolls, such as the one from En-Gedi, which is only about 16 cm long. Due to the eruption Herculaneum scrolls are not smooth regular cylindrical rolls but are rolled in a highly irregular way, because parts were squashed under debris. Last but not least, unlike all the previous cases, where the ink contained large amounts of metal, the ink used in the Herculaneum scrolls is carbon ink, whose main chemical element, carbon, is also the main chemical element of charred papyrus. Therefore, there is very little to no chemical contrast between the ink and its support, which means that there is almost no difference in relative density for the X-rays between the support and the ink.

Some promising alternatives to conventional CT-scanning have been suggested in order to overcome this problem, such as X-ray phase-contrast tomography (CPT-scanning). Approaches based on «morphological contrast», with the help of a machine-learning pipeline, have yet to demonstrate results on inner parts of the scrolls themselves. The best results achieved so far are the detection of some letters or a couple of words.[11] In some cases, this may be all that can be achieved. However, recent research has suggested that some of the inks used on the scrolls might not be made of pure carbon. In particular, lead (Pb) was recently discovered to be present in high quantities in black ink on Herculaneum fragments from the Institut de France's collection.[12] In this case, the authors have excluded contaminated water as the source of lead and suggested that it was added intentionally. If inks from other scrolls also contain metals, there is hope that enough contrast in density between the ink and the charred papyrus support will allow conventional CT-scanning to be used, despite the above-mentioned difficulties.

The aim of the survey conducted in June 2018 was therefore to see whether metals could be detected in inks from other fragments of the Herculaneum collection. We have first focused our investigation on fragments detached from partially unrolled scrolls. In this way, we can select corresponding rolls which are good candidates for CT-scanning.

Materials and methods

Preliminarily our work focused on a group of so-called *scorze*. This choice was determined by the fact that in this way small fragments from different scrolls (in a limited number of frames) could be analyzed. In the 19th century, the *scorze* had been placed on long cardboard support containing 20-

[11] Mocella / Brun / Ferrero / Delattre. 2015; Bukreeva / Alessandrelli / Formoso / Ranocchia / Cedola 2017; Parker / Parsons / Bandy / Chapman / Coppens / Seales 2019.

[12] Brun / Cotte / Wright / Ruat / Tack / Vincze / Ferrero / Delattre / Mocella 2016.

30 pieces. Subsequently, in 1999, each *scorza* was fixed on single supports and arranged in groups (of 5-6) in single metal frames.[13] We analysed 38 *scorze* in total.

Elemental analysis was performed by X-Ray fluorescence (XRF), a technique which is commonly used for the analysis of the elemental composition of various objects in the field of cultural heritage. It has been used extensively for the last two decades by the Bundesanstalt für Materialforschung und -prüfung in collaboration with the Centre for the Study of Manuscript Cultures (CSMC) of the University of Hamburg to analyse inks.[14] X-Ray fluorescence analysis was performed with an Elio (Bruker, formerly XGLab) portable XRF spectrometer, equipped with a 4W low-power rhodium tube and a 50 mm^2 large area silicon drift detector (SDD) with CUBE technology, and with an interaction spot of 1 mm. All measurements were conducted under the following experimental conditions: spot analysis of 120s and excitation parameters of 40 kV and 80 µA.

After measurement, the spectra were processed with the Spectra (ARTAX) software from Bruker to identify the elements and determine their net peak intensities. We performed spot analyses to identify metals in the ink, both on areas where ink is visible and on areas where there is no ink on the top surface. Availability of the infrared images of the fragments was extremely helpful for the selection of the inked and non-inked areas for the measurements. The resulting spectra were compared for the presence of metal that could be ascribed only to the ink spots. However, since the fragments typically consist of several layers attached to cardboard and paper, neither the verso nor the bottom layers of the fragments could be checked for absence of ink. This fact, together with the inherent inhomogeneity of papyrus, created a great degree of uncertainty in the evaluation of the results.[15]

Table 1 lists the fragments together with the number of spots analysed in each case. For each fragment, a spectrum of the cardboard support and, whenever possible, of paper and cardboard was collected as well. The cardboard itself contains significant amounts of iron and calcium. In addition, the paper contains sulphur, potassium and calcium (see Pl. 1). Unfortunately, the paper is in some cases cut so close to the edge of the fragment that it could not be analysed for each fragment.

[13] Kleve / Capasso / Del Mastro 2000.
[14] Hahn 2010; Rabin 2014.
[15] Ghigo / Rabin / Buzi 2020.

Table 1. List of fragments with the number of spots analysed and the corresponding results.

Papyrus number (P.Herc.)	Number of spots analysed ink / no ink	P	Fe	Cu	Pb
231	5	*	X		
232	11 / 2		X		*
233#	7 / 2		<<		
234	8 / 2		*		
235	7 / 2		*		
236#	4 / 5		<<		
238	15 / 9		*		
239	10 / 5		*		
242	11 / 2			*	
245#	9 / 2		<<		
246#	7 / 2		<<	<<	<<
247#	11 / 3		<<		<<
248	8 / 2	x	<<	<<	
250#	9 / 3		<<		<<
252#	4 / 6		<<	<<	
253#	11 / 4		<<		
254	9 / 4		*		X
255#	12 / 4				
398	9 / 2		*		*

400	7 / 7		<<		*
401[#]	5 / 3			<<	<<
406	6 / 1	X	*		-
407[#]	5 / 2		<<		<<
408	5 / 2		*		*
409	7 / 2	*	*		<<
410	7 / 2	X	*		
411	9 / 3				X
412	7 / 2	X	X		*
413[#]	5 / 3			<<	
414[#]	7 / 2		<<	<<	
415	0 / 10		*		
416	8 / 2		*	*	*
418	23 / 5		*	*	*
419[#]	8 / 2		<<		
421	8 / 2	X			<<
422[#]	6 / 2				
424[#]	4 / 2				
425[#]	10 / 2		<<	<<	<<

- fragments with no conclusive results; X – element found; * - trend found; << - element more abundant on the non-inked areas

Results and discussion

To illustrate the procedure, the locations of spots analysed and the resulting distribution of lead on P.Herc. 254 are presented in Pl. 2 and 3 respectively.[16] For this fragment, four spots (7, 9, 13 and 14) were chosen on areas without visible ink, and the remaining 10 spots were taken on ink. In addition, a single spot outside of the fragment, i.e. on the support material consisting of the cardboard and paper, was analysed (support). The contribution to the signal due to the cardboard and paper was subtracted after the fitting of the spectra and prior to further processing.

Pl. 3 demonstrates the great variation of the signal due to lead: from 753 counts on the spot 14 from the area with no visible ink to 7980 counts on spot 10 that corresponds to the measurement on the letter *lambda*. We see, however, that all the spots not placed on visible ink deliver low signal values. In contrast, the spots on which the ink was the thickest and most visible (10, 11, 12) correspond to the highest intensity of lead. Despite the fact that spots 4-6 contain as little lead as the non-ink spots 7, 9, 13 and 14, we consider the correlation between the signal of lead and visible inked area in this fragment to be established.

Using the same procedure, it was possible to associate certain elements with the ink on several fragments, with different degrees of certainty. Sulphur (S), potassium (K) and calcium (Ca) belong to the most abundant elements in the support and papyrus and, therefore, could not be considered in the evaluation, leaving us with four elements of interest, namely phosphorus (P), iron (Fe), copper (Cu) and lead (Pb). Table 1 lists the results corresponding to these four elements obtained in the present study. Here, clear correlation between the inked area and an element is marked by the entry «X». When only a trend was found, the corresponding elements are marked with a star indicating that not all inked / non-inked spots delivered a consistent result. Finally, fragments have a hash symbol if no correlation between the visible ink spot and any of the elements could be found. We must stress here that the large number of entries corresponding to the trend or inconclusive results with respect to iron, copper and lead could be most probably attributed to the fact that the spots chosen to represent papyrus contained ink on one of the layers below the surface one. Though iron could be firmly linked to the ink only in three cases, we see that the number of fragments in which the non-inked area displayed a high signal of iron dominate the results. Iron presents the most complex case since the combined effect of contamination from the cardboard and the heterogeneity of the papyrus support, which also contains iron, make the attribution of this element more uncertain than any other.

Two pieces, P.Herc. 254 and 411, were found to contain an elevated amount of lead, whereby only P.Herc. 411 might have been written with an ink similar to the one reported by Brun and his

[16] P.Herc. 254 is a *scorza* from Philodemus, *On Rhetoric*, book IV (Copy A; *midollo* of P.Herc. 1673/1007).

collaborators.[17] The initial hypothesis of contamination of water by lead pipes as the origin of the lead in the ink has quickly been discarded because of the overly high amount of Pb. For the same reason, and also because of the lack of correlation between Cu and Pb, the hypothesis of contamination originating from bronze inkwells was also rejected. Instead, lead was most probably intentionally added. Tack and co-authors have suggested it was added either in the form of a pigment such as galena, lead white or red lead, or more likely as a drying agent such as litharge, which was used in antiquity as oil dryer. Because of all the events which altered the scrolls both during the eruption and since their exhumation, it is unfortunately difficult to come to a conclusion as to the nature of the lead compound. In all other cases studied here, inks might contain lead in addition to iron.

Copper in combination with iron and lead has shown a trend in P.Herc. 416 and 418.[18] It has also shown a trend as a single element in P.Herc. 242. The absence of this element in the cardboard and paper makes its identification easier than that of iron. In this case the uncertainty most probably derives from the presence of the ink under the spots measured to represent papyrus only. However, if it is indeed contained in the ink, its presence might indicate the intentional addition of the copper-based compound χάλκανθον or χάλκανθος mentioned in some ancient recipes of inks like that of Dioscorides in his *De materia medica*.[19] It is probably quoted too by Pliny the Elder for another application.[20] This would be in accordance with recent findings of copper in inks from Hellenistic and Roman papyri.[21]

Only the element phosphorus, whose low energetic X-ray photons cannot pass through a papyrus layer, can be firmly attributed to the top layer. This very property makes its presence in the ink irrelevant for the possible unrolling of the scroll using the CT method. However, in the terms of ink characterization the element is extremely important since it might point to a possible precursor of the ink. Moreover, detectability of this light element using a simple portable device indicates that it is present in great abundance and will allow the visualisation of the ink on the top layer of a scroll or a stack of the fragments. Indeed, Ira Rabin and Olivier Bonnerot have already discovered inks on papyrus pieces from the Cologne Collection and on one fragment of the Istituto Vitelli at Florence that contained text readable in the element phosphorus, in particular in a fragment from Antinoe.[22]

[17] P.Herc. 411 is a *scorza* from Philodemus, *On Music*, book IV (midollo in P.Herc. 1497). On the ink reported by Brun and collaborators, see Brun / Cotte / Wright / Ruat / Tack / Vincze / Ferrero / Delattre / Mocella 2016; Tack / Cotte / Bauters / Brun / Banerjee / Bras / Ferrero / Delattre / Mocella / Vincze 2016.
[18] It is noteworthy that P.Herc. 418 comes from the same scroll of P.Herc. 254 (Philodemus, *On Rhetoric*, book IV).
[19] Pedanius Dioscurides (1st. cent. A.D.), *De materia medica* 5, 162 (ed. Wellmann 1914, 108).
[20] According to his *Naturalis historia* 34, 123 this substance was *atramentum sutorium* or shoemaker's black. Pliny believed that this substance was used to blacken leather. This is, however, chemically implausible since no copper substance produces a black pigment upon reaction with tannin. Therefore, shoemaker's black must have contained an iron-based substance.
[21] Rabin / Wintermann / Hahn 2019; Nir-El / Broshi 1996.
[22] Detailed publication of these results will be offered in future.

Conclusion

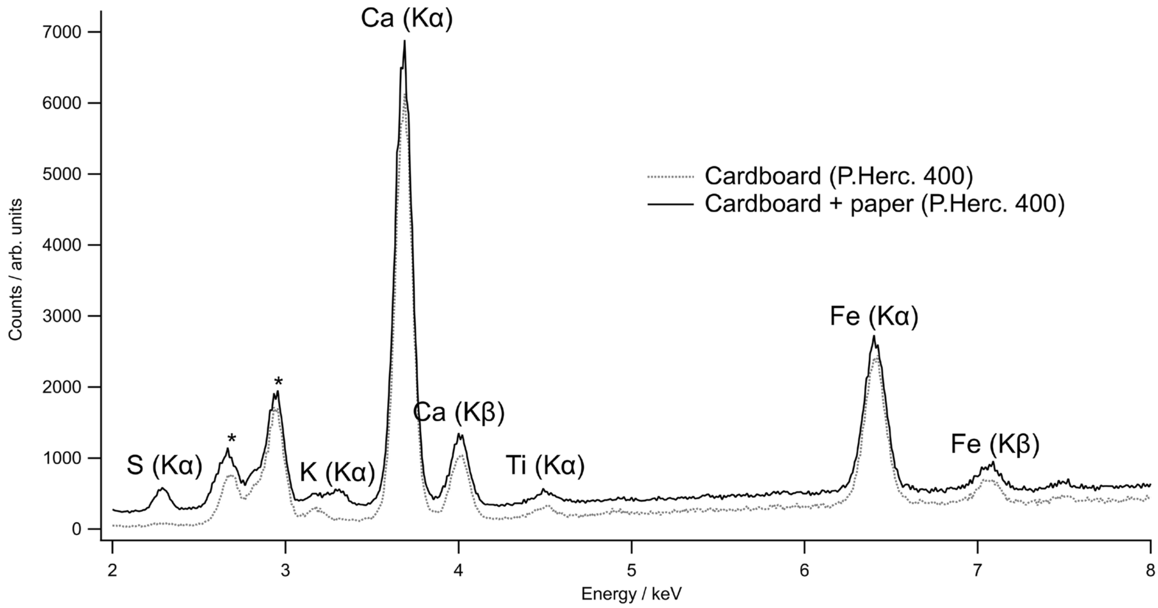
Inks of 38 pieces from the Herculaneum collection preserved in the Biblioteca Nazionale di Napoli were analysed using X-ray fluorescence technique. A number of papyri displayed detectable elements other than carbon present in their ink. In addition to lead, which had already been identified, we detected phosphorus, copper and iron, which are also likely to originate from the inks and strengthen the hypothesis that some of the inks of the Herculaneum papyri might belong to the category of mixed inks in accordance with recent findings.[23] Therefore, the whole collection should be systematically tested for the presence of the metals using imaging XRF, leading to a selection of scrolls which could be virtually unwrapped using conventional CT-scanning. Moreover, this result seems to open the possibility of using confocal X-ray fluorescence to reveal the underlying text on the stacked fragments.[24] In the case of the presence of phosphorus in the inks, imaging XRF would provide legible texts from the top layers of the fragments.

Acknowledgments

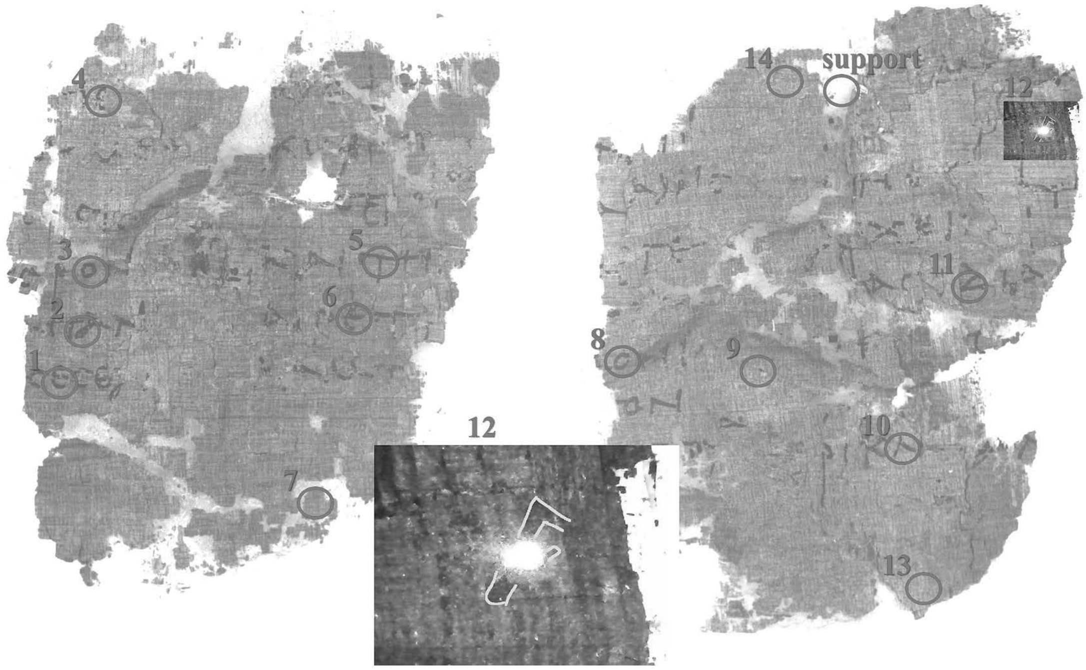
We would like to express our warmest thanks to the Arbeitsstelle für Papyrologie, Epigraphik und Numismatik at the University of Cologne for having allowed the use of its Elio spectrometer for the analysis, as well as to Sofia Maresca and her colleagues in the Officina dei Papiri of the Biblioteca Nazionale di Napoli, who gave us access to the fragments and helped us in all the stages of our work. We also thank the CISPE «Marcello Gigante» (Naples) for assistance during the measurements. Finally, we express gratitude to Thomas Ford for his diligent proofreading of the typescript.

This research was carried out at the Cluster of Excellence «Understanding Written Artefacts: Material, Interaction and Transmission in Manuscript Cultures», funded by the German Research Foundation (Deutsche Forschungsgemeinschaft, DFG), and within the scope of the Centre for the Study of Manuscript Cultures (CSMC) at the Universität Hamburg.

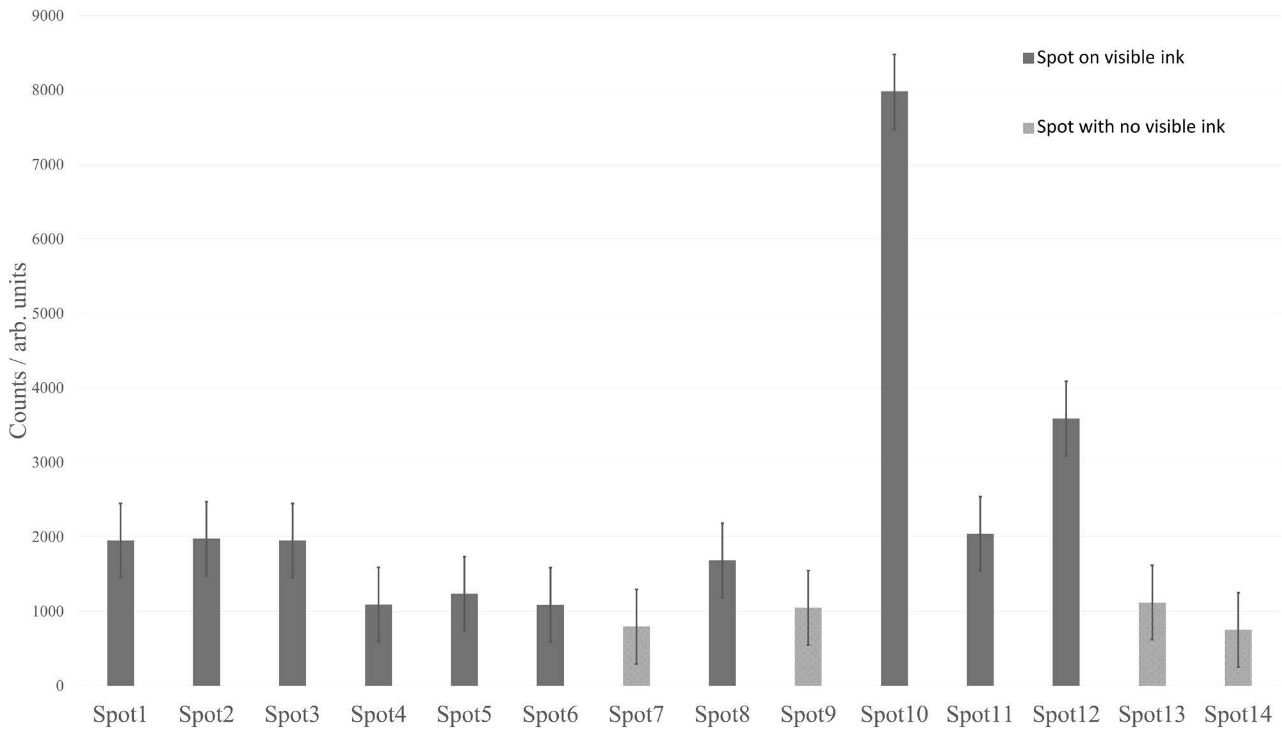
[23] Colini / Hahn / Bonnerot / Steger / Cohen / Ghigo / Christiansen / Bicchieri / Biocca / Krutzsch / Rabin 2018.
[24] Li / Finfrock / Brun / Delattre / Mocella 2020.



Pl. 1. XRF spectra of cardboard (bottom) and of paper + cardboard (top) measured on P.Herc 400. The cardboard used contains a significant amount of iron (Fe) and calcium (Ca). In addition, the paper contains sulphur (S) and potassium (K). The peaks at 2.69 kV and 2.96keV correspond to the Lα of rhodium (Rh) and the Kα of Argon (Ar) respectively. The rhodium detected originates from the rhodium tube used as a source of X rays in the spectrometer. Argon is detected because the analysis was performed in the air. Both elements are not relevant to our study.



Pl. 2. Location of spots analysed (on infrared image) and detail of spot 12 (taken from the Elio in-built camera during analysis) for P.Herc. 254. Infrared image by S. W. Booras © National Library, Naples - Brigham Young University, Provo (USA); duplication is prohibited.



Pl. 3. Distribution of Pb for P.Herc. 254. Spots 1, 2, 3, 4, 5, 6, 8, 10, 11 and 12 correspond to visible ink, while there is no visible ink for spots 7, 9, 13 and 14.

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