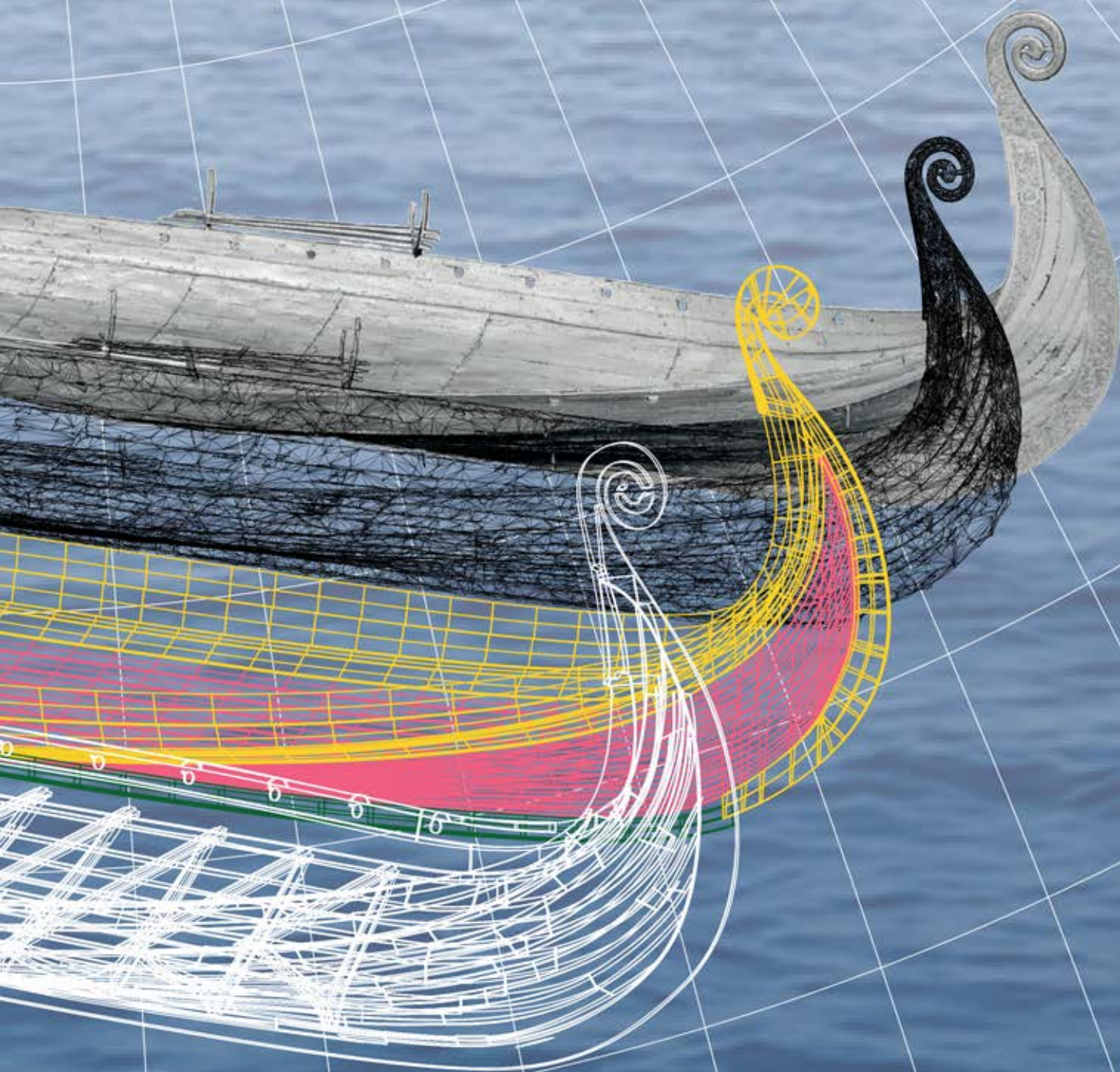


CAA2016

Oceans of Data

Edited by

Mieko Matsumoto and Espen Uleberg





Oceans of Data

Proceedings of the 44th Conference on
Computer Applications and
Quantitative Methods in Archaeology

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ARCHAEOPRESS PUBLISHING LTD
Summertown Pavilion
18-24 Middle Way
Summertown
Oxford OX2 7LG

www.archaeopress.com

ISBN 978 1 78491 730 2
ISBN 978 1 78491 731 9 (e-Pdf)

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Language Editing: Rebecca Cannell
Cover Design: Bjarte Einar Aarseth

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A Multidisciplinary Project for the Study of Historical Landscapes: New Archaeological and Physicochemical Data from the 'Colline Metallifere' District

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Abstract

In this paper we will present some results obtained by a combined multidisciplinary approach to the study of a territory located in the southern part of Tuscany, the 'Colline Metallifere' district. The area has been studied from different points of view, and there are many written contributions concerning the evolution of its human landscape over the centuries. In this paper we are focusing particularly on the use of chemical analyses on crucial historical areas. The considerable amount of information retrieved and the unquestionable value of the data obtained by portable X-ray fluorescence (pXRF) analyses are now opening new fields to the use of the portable instrument. The new opportunities offered by the 'NeuMed' ERC project, based in the University of Siena, will allow the testing of the reliability of this technique on a wider scale, in multidisciplinary research that aims to reconstruct the historical landscapes of the area.

Keywords: interdisciplinary methodology, archaeological surveys, physical-chemical analysis, pXRF, mining territories

The Colline Metallifere district: a multidisciplinary open air laboratory (L.D.)

In recent years, the Colline Metallifere district (southern Tuscany) has become a multidisciplinary study area in which a combined archaeological, geological, and environmental approach is providing new and interesting evidence for the reconstruction of historical landscapes. The territory is very well known from the numerous research projects undertaken over the years on archaeological and historical targets and mining deposits (Dallai and Francovich, 2005). The hills which give the area its name cover part of Livorno and Grosseto provinces; the territory includes the coast and the immediate hinterland of the Gulf of Follonica, an important working hub for the iron oxides (hematite) that have been exploited on Elba Island since the Etruscan period.

Ever since that period, the entire area has been historically very important for mining activities. During the medieval period in particular, the exploitation was especially focussed upon mixed sulphides deposits of copper, lead and silver; the consistent ore bodies were worked in order to obtain the metals needed for coin production.

These rich deposits and their exploitation attracted the interest of important aristocratic families, which, since

the central centuries of the medieval period, developed their control over the underground resources through fortified settlements placed close to the mining fields. The archaeological research undertaken since the 1980s and the excavation of 'mining castles', such as the well-known castle of Rocca San Silvestro or the castle of Rocchette Pannocchieschi, have demonstrated that the development of local lordships in this geographical context was deeply linked to the control and exploitation of metals that could be used for coinage (Bianchi, 2010).

Since then, a large number of research projects have been carried out with the aim of studying the possible relations between settlements and mineral resources, with particular reference to settlement patterns and the control over the cycle of production through the centuries. Extensive surveys on territorial samples have been carried out in order to define the main aspects of mining exploitation and metal production; the data collected has been processed with the aim of defining a broad picture of the territory, both from an archaeological, geological and environmental perspective (Benvenuti *et al.*, 2014).

Out of this wide territory, specific targets have been selected for a multidisciplinary research; these 'pilot' studies started with in-depth research carried out on mining fields (particularly in the mining areas of



Figure 1. Location of sites mentioned in the text.

Serrabottini and Niccioleta, close to Massa Marittima, Grosseto province). In order to get the maximum amount of useful information, a combination of underground surveys, fieldwork and XRF analyses on water-borne sediments and soils have been planned. The data collected has clarified the technical characteristics of the production and the environmental impact of ancient mines on the surrounding area. Finally, chemical analyses carried out on waste material has revealed a skilful selection operated at the mine head in order to eliminate useless minerals, zinc in particular (Aranguren *et al.*, 2007; Dallai *et al.*, 2015a; Dallai *et al.*, 2013). A combination of underground surveys, fieldwork and XRF analyses on fluvial sediments and soils explored the technical aspects of the production and the environmental impact of ancient mines in the surrounding area (Aranguren *et al.*, 2007).

The multidisciplinary approach has focused not only on territorial samples, but has also included the study of specific archaeological sites; some of which are medieval settlements located in the core of mining areas (that is the case of the church of San Niccolò, near Montieri); some are production sites (as for the alum production site of Montaleo, close to Monterotondo Marittimo); whilst others are sites based in the coastal plan, near the shore, that have been identified by archaeological surveys and remote sensing techniques and recently excavated (i.e. the Carlappiano site, near Piombino) (Figure 1). On all these specific places, as well

as on other key samples that have been discussed in previous papers (Dallai *et al.*, 2015b), a multidisciplinary approach has been first planned and then undertaken directly on site. Finally, the obtained results have been examined and analysed in order to obtain increased information. The final goal of our project is, in fact, to provide an historical and environmental landscape reconstruction based on multidisciplinary data sets; merging historical data with chemical-physical results we are gradually adding crucial pieces of information to the picture of one of the most important mining district of the Mediterranean area.

From the different case studies, by combining archaeological and physico-chemical data (pXRF in particular) we are gradually building up a solid database that is helping to define the historical outlines of the Colline Metallifere landscape. Moreover, building upon previous research experience, pXRF analyses are now playing an essential role in the research strategies of the 5 year ERC project 'NeuMed Origins of a new economic union, 7th-12th centuries: resources, landscapes and political strategies in a Mediterranean region' based in the University of Siena (P.I. Prof. Richard Hodges). The reconstruction of the historical features in the landscape becomes crucial to understand the deep changes that occurred in settlement patterns, trade routes and economical background of the area between Late Antiquity and the 12th century.

Investigation methods (V.V., A.D.)

Physico-chemical analysis of different environmental matrices is becoming a fundamental tool for multi-scale archaeological prospections. From the enrichment or depletion of certain elements or molecules in soil, stream sediments, groundwater etc., their spatial distribution and their statistical treatment gives the chemical fingerprint of a territory with possible genuine correlations with ancient human activities (Oonk *et al.*, 2009; Anguilano *et al.*, 2010).

Recently, pXRF has been used for the quantification of major and minor elements in soils, stream sediments and artefacts, and it emerged as the elective technique for *in-situ* analysis. In fact, the pXRF instrument can be directly used on the surface of untouched environmental matrices and immobile artefacts. It can be also used for laboratory analysis with treated (dried, milled, sieved) samples. The major features of pXRF analysis are the non-destructive nature of analysis, the speed of operations, the capability of on-site measurement, and the immediate availability of analytical results (Potts and West, 2008; Shugar and Mass, 2012). The protocol can be used in multi-scale investigations (*in-situ* and medium-large territorial scale) with both predictive and descriptive goals using slightly different techniques.

In-situ studies were conducted collecting data using a pre-determined grid frame of 1 m² (1 m × 1 m) areal elements. Inside each areal element the concentration of a chemical species was given by the average of three single pXRF measurements (Dallai *et al.*, 2015a). Depending on the required sampling density, the number of pXRF sample points in one of areal element could be much higher, often following the concurrent excavation of single units. In this context the measurements done directly on archaeological finds or archaeological structures (architectural elements, furnaces, walls, mortars, slags etc.) can also be included, which is a great help for their functional characterisation.

In the medium-large territorial scale the sampling density is lower and usually follows the geomorphological elements of the territory. The fluvial stream sediments were proven to be the most useful environmental matrix for studying the chemical anomalies due to ancient industrial settlements. This because the streams and rivers are the collector of the contaminated material originated by one or more different sources within their own drainage basin. In particular, the production activities exploited in this mining area can be traced in detail from the extraction of the mineral ores to the smelting and refining of the metals.



Figure 2. pXRF laboratory station.

On the other hand, the ancient mining field can also be investigated spatially and directly. In this case, the concentration of elements in geo-referenced soil samples gives the chemical fingerprint of the area.

Both laboratory and on-site pXRF analysis were performed using an Olympus DELTA-premium handheld pXRF analyser, equipped with a 40kV tube, a 'large area SDD detector, accelerometer and barometer for atmosphere pressure corrections for light elements measurements. The laboratory measurements were done with the instrument mounted on a fixed station (Figure 2). To ensure the quality of the data obtained, operating protocols was used (EPA, 2007). All laboratory samples were collected in the same spot as the on-site measurements; they were dried at room temperature, sieved at 125µm and placed in the appropriate sample holders. Data were acquired with the 'Soil Mode' (3-beam) of the instrument which utilises Compton Normalization for low concentrations (PPM to 3%) of elements in light matrices. The result for each sample was the average of the three measurements. Chemical data was finally geo-referenced together with the archaeological information in a GIS application (QGIS) used to produce distribution maps.

Medium-large territorial scale (V.V., A.D.)

In this work we present the results obtained in two sites that have recently been investigated. One of them was the ancient Serrabottini mining area, and the other is the Monte Gai site; both of them are close to the medieval town of Massa Marittima. Here, the major activity was the extraction and processing of mixed sulphide ores for the production of copper. The chemical data collected in mining dumps areas confirmed the major

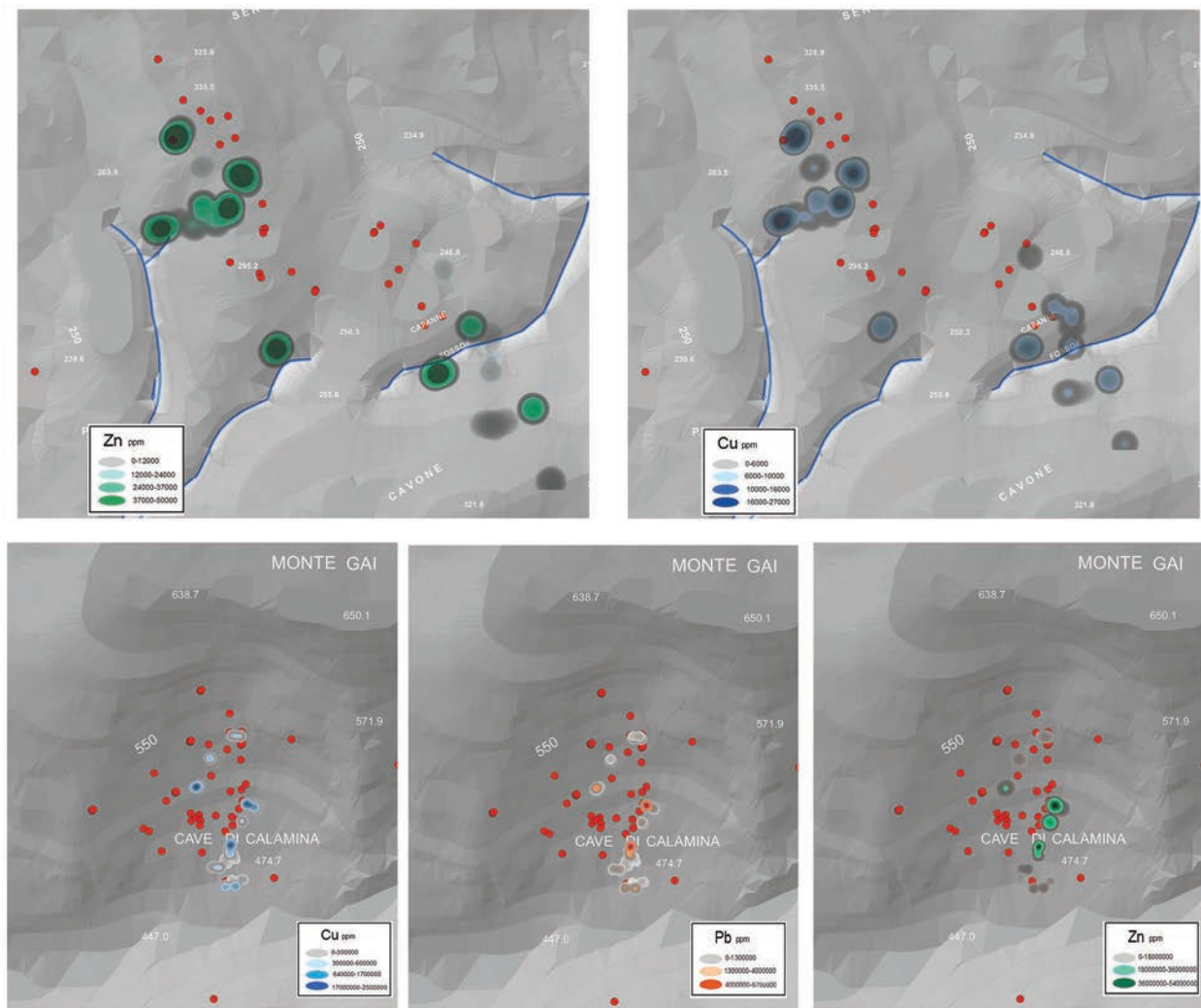


Figure 3. Serrabottini (Massa Marittima, GR). The chemical data collected through pXRF analyses on ancient and modern mining dumps have revealed the main goal of the ancient mining exploitation (particularly Cu), as well as the contamination of the soil.

goal of the exploitation (copper production) as well as the extent of heavy metal pollution and its diffusion. In the Figure 3, the concentration of Cu, Zn and Pb are reported on the map of the site, showing the position of the large mine tails dumps that are located in a strategic position with respect to the extraction sinks (red circles). The data also revealed that the diffusion of the contamination is quite restricted to the sites and its spreading is poorly enhanced by the weathering of mining residues.

Regarding the Serrabottini site, a k-mean clustering analysis was also performed. In Figure 4, 'A' reports one of the ten different correlation diagrams obtained by the k-mean clustering, in which different colours represented samples that were grouped by chemical similarity.

It was noteworthy that by observing geo-referenced data on the site map (Figure 4B), a spatial separation of the clusters emerges. In fact, Cluster-1, with higher concentration of Cu, Zn and Fe was mainly restricted in two small areas with respect to the Cluster-2 data. This fact could be explained with the hypothesis that in these two areas a different production activity (namely roasting of mineral ores) was accomplished. Cluster-3 was constituted by only two samples with very high lead concentrations.

This result can also be explained by considering that during the first phase of the metallurgical process for copper production, the roasting of mixed sulphide ores, induced a large loss of the most volatile metals: arsenic, antimony and lead; while zinc was not completely lost in this phase. To eliminate almost all impurities and zinc, after the roasting, a preliminary phase of smelting

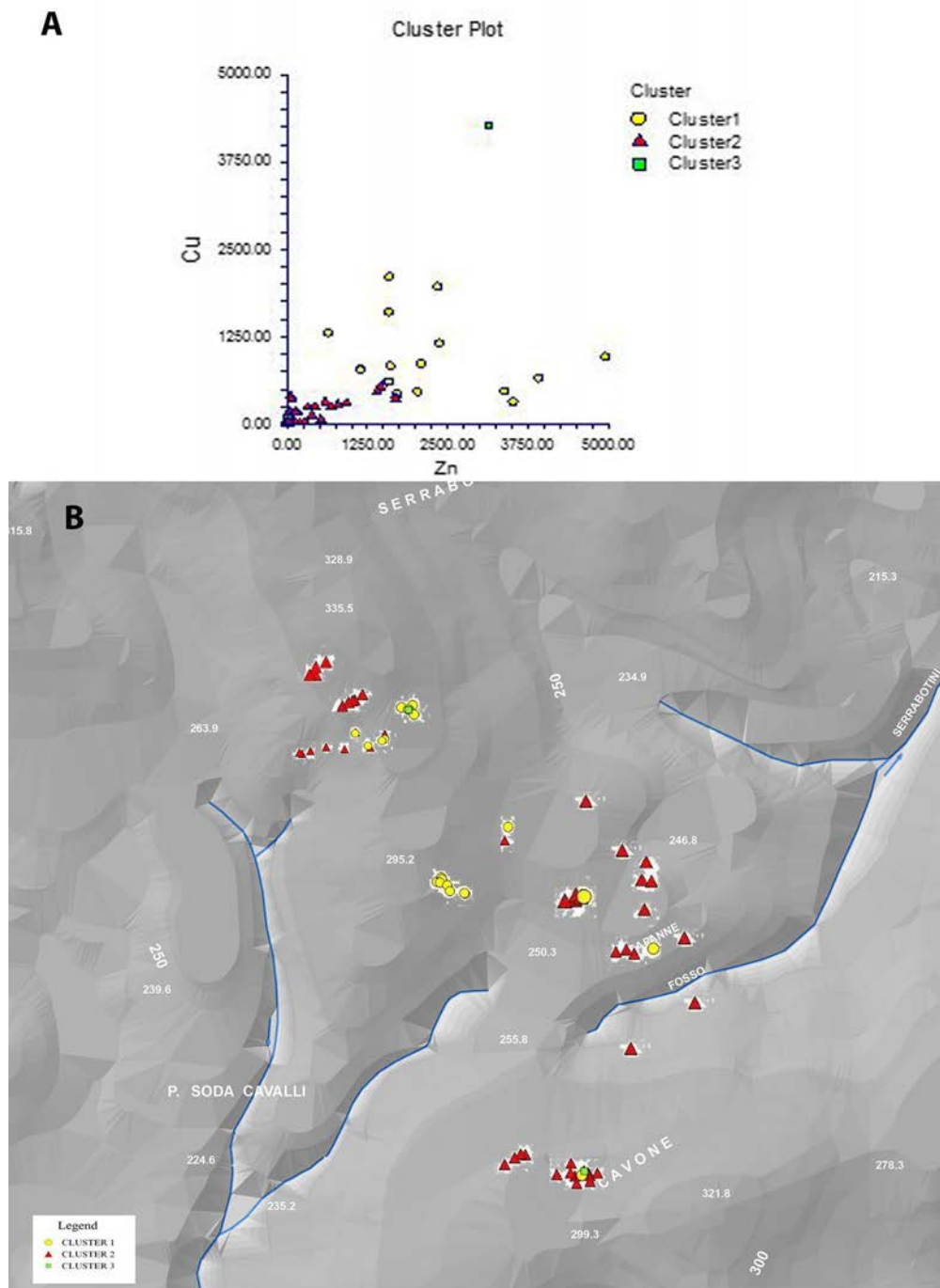


Figure 4. Statistical treatment of the data (K-means clustering analysis). In Figure A the correlation diagram of Cu-Zn; in Figure B, the georeferenced data on the site map.

produced a copper matte that was roasted a second time before the final smelting.

In-situ analyses and results (L.D., A.D., V.V.)

Regarding *in-situ* investigations, here we present three key sites showing relevant data, with interesting connections to the archaeological evidences: the ‘Canonica di San Niccolò’ site (Montieri – GR); the ‘Allumiere di Monteleo’ site (Monterotondo Marittimo – GR) and the Carlappiano site (Piombino – LI).

‘Canonica di San Niccolò’ (Montieri – GR) (L.D., A.D.)

Montieri district has been one of the most prominent silver, lead and copper mining areas of the Colline Metallifere. It was certainly at the heart of a complex system of ore-working and mineral production, probably connected to the activities of the neighbouring castles. Its’ importance is documented by the presence of a mint which, between the end of the 12th century and the first half of the following century, struck coins on behalf of the Volterra bishop.



Figure 5. The intra-situ pXRF analyses at the Canonica site; the sampling grid.

NW from the village, on the slopes of the so called ‘Poggio’, a systematic set of analyses have been carried out on the archaeological site of the Canonica di San Niccolò (a parish church), a peculiar church building with six apses, with adjacent spaces and buildings dedicated to different activities. The documents attest the existence of the site from at least 1133, while the dig provides evidence of an initial occupation of the terraced site probably in the period between the 9th and 10th centuries. The final abandonment of the site occurred just before the 15th century (Benvenuti *et al.*, 2014).

At the Canonica site, a very large sampling grid frame of 1 m² was applied, covering a large part of the excavation area (AREA 3000 and AREA 2000) (Figure 5). The chemical elements for this type of analysis considered as significant ‘tracer’ elements, useful to identify archaeometallurgical production, are Pb, Fe, Cu and Sn.

Simple statistic parameters for Fe, Cu, Sn, Pb data regarding the Canonica site showed lower values for AREA 3000 with respect to AREA 2000, both for averaged and peak values (Table 1). This fact indicates that different activities were carried out in the two parts.

		Fe (mg/kg)	Cu (mg/kg)	Sn (mg/kg)	Pb (mg/kg)
Area 2000	average	43601.2	64.7	19.7	69.4
	st. dev.	10101.1	73.3	23.5	50.2
	min	12161	10	1	19
	max	97993	898	239	371
Area 3000	average	36234.4	37.7	19.3	57.9
	st. dev.	5821.2	11.5	17.6	36.0
	min	11567	5	1	20
	max	59584	66	81	346

Table1. Comparison of simple statistic parameters for Fe, Cu, Sn, Pb data obtained in the Canonica area (Montieri – GR).

AREA 3000 had principally an agricultural function whilst AREA 2000 was a production area where different types of craft activities were conducted. In this area the excavation campaigns had already identified the remnants of forging activities and other production evidence.

A more accurate analysis was obtained by observing the spatial distribution of the metal concentrations (Figure 6). In the western part of AREA 2000, the higher concentrations of Pb and Fe confirmed the presence of a forge, while in the centre, high values of Sn and Cu



Figure 6. Results of intra-situ pXRF analyses. The ring circumference is proportional to the concentration of the considered chemical element. Analyses have highlighted high concentrations of Sn (A) and Cu (B) on the centre of the area 2000.

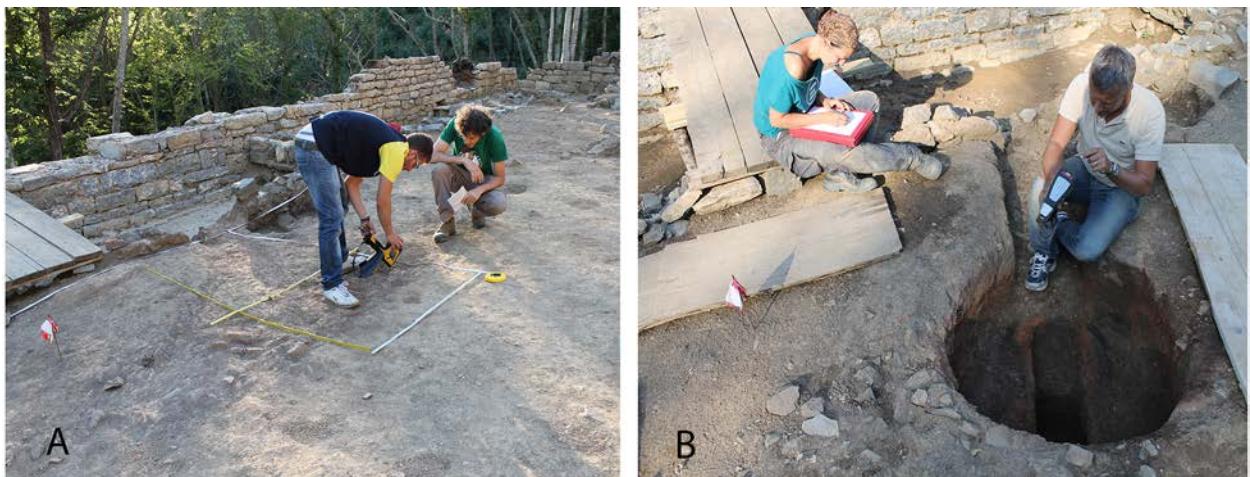


Figure 7. pXRF analyses have been used for both descriptive and predictive goals. In Figure A the intra-situ analyses before the discovery of the bell-kiln remains (Figure B) (credits: LTTM, University of Siena).

identified the presence of an activity linked with the use of an alloy of these two metals. The subsequent dig campaign discovered the remains of a bell-kiln located exactly in the area of the pXRF anomaly (Figure 7, Dallai *et al.*, 2015a). No evidence was found regarding the anomalous presence of Pb related to the silver production.

In this case, the *in-situ* analysis has confirmed and demonstrated the reliability of this technique and the descriptive and predictive power for the study of archaeological sites with a production 'vocation'.

The reported study performed at the Canonica of San Niccolò, was a part of a wider project started few years ago in order to understand the productive history of

the small medieval village of Montieri, one of the most important silver extraction sites during the Middle Ages. Previous investigation took into account the metal concentrations in the stream and river sediments of the area. Using the approach on medium-large territorial scales, a wide range of information about ancient mining, roasting and smelting activities was obtained for the area around the village (Dallai *et al.*, 2015a). Contrary to previous work, the rivers around the Canonica did not present evidence of metal contamination. In summary, the combination of physico-chemical and historical analysis have been definitely useful to understand the nature of the metallurgical activities undertaken near the village of Montieri, as well as to detail the nature of working activities that took place at the Canonica site. In this case, the absence of metal processing evidence



Figure 8. The battery of the early-modern alum furnaces at the Monteleo site. The red ring indicates the area where pXRF analyses were conducted.

excluded its direct involvement in the silver economy, which was the most particular feature of the area during the medieval centuries.

'Allumiere di Monteleo' (Monterotondo Marittimo – GR) (L.D.)

The Monteleo alum works are located in the NW reaches of the Monterotondo Marittimo municipality and extend along the two banks of a local stream, the waters of which played an essential role in numerous steps of the production process (Dallai, 2014). From an archaeological point of view, the discovery of this site and of a non-metallic working cycle in the Colline Metallifere district dating to the late medieval period, such as the alunite one, has enlarged upon the already detailed evidence of pre-industrial extraction activities in the area, which until now referred almost exclusively to the cultivation of mixed sulphides.

The raw material worked in this site is alunite, an aluminium potassium sulphate ($KAl_3(SO_4)_2(OH)_6$) that is practically insoluble in the natural state. Nevertheless, it is possible to transform alunite into high quality alum through a process of roasting, maceration, leaching and crystallisation known since medieval times and described in detail in two renown 16th century technical treatises: *De la Pirotechnia* by the Sienese, Vannoccio Biringuccio published in 1540, and *De re metallica* by Georgius Agricola, published in 1556: to date, detailed reconstructions of the technical procedures and tentative functional interpretations of known material finds have mostly been based on these descriptions.

There are currently only a few but significant case studies on archaeological sites linked to the production of alum from alunite, and Monteleo is one of these. The excavation, begun in 2008 by the University of Siena,

is providing important new information on adopted technologies. Most of the structures which have been brought to light may be ascribed to the so-called 'Renaissance phase' of the site, i.e. to between the end of the 15th century and the first half of the 16th century, a period of documented renewal of alum mining and processing in numerous areas of Tuscany (Boisseuil and Chareille, 2009) (Figure 8).

Despite the written sources, the archaeological investigation revealed a longer history on the site, with evidence of production activities starting much earlier than the 15th century. The remnants of wall structures in irregular limestone blocks bonded with soil, most likely relating to a furnace, refer to this earlier period and were obliterated by the 16th century masonry of the alum works. Radiocarbon dating performed on different charcoal samples have proven that the furnaces occupying the terrace, as well as other productive structures and forges identified on the site, were used in the late 13th century.

In-situ pXRF analysis (i.e. analysis planned inside a single archaeological site), were conducted during the excavation campaigns in order to understand the kind of production related to these ancient remains. Whilst on the Canonica site the pXRF analysis was conducted principally on the soil, at Monteleo this technique was also applied to the remains of the productive structures, as well as on finds (slags and metal droplets). The pXRF analysis, in particular has identified former metal production involving copper and silver sulphides. Data analysis showed the presence of high anomalies of Cu (2000 mg/Kg) and Fe (3000 mg/Kg) in specific and restricted areas of the site, linked respectively to a copper furnace and a forge (Figure 9). The pXRF analysis of several metallic fragments and residues from several stratigraphic units show high concentrations of S, Ca, Cu, Fe, Pb, Sn, Zn and Ag; copper is always present with high concentrations (60 wt%) (Dallai and Volpi, 2015).

Carlappiano (Piombino – LI) (V.V.)

While the previous examples proposed have shown the high potential of analyses performed on archaeological excavation and territorial samples, new research perspective are now open to a multidisciplinary approach within the frame of the ERC Advanced European Research Project *Neumed*, mentioned above. The project, started in October 2015, has selected the first targets on which archaeological excavation as well as environmental analysis have been planned and the Carlappiano site is one of these. Prior to the dig, remote sensing (i.e. magnetometer, drone and historical aerial photographs analyses) as well as pXRF measurements have been carried out, providing promising initial results. The site is located on a dune close to the shore



Figure 9. At Monteleo (Monterotondo Marittimo, GR), a late medieval alum productive site, pXRF analyses and archaeological data have identified a former metal production involving copper and silver sulphides. In Figures A and B, the archaeological remains of the late 13th century productive phase. In Figure D, the pXRF analyses indicates the presence of high concentrations of copper (credits: LTTM, University of Siena).

line, on the boundary of what once was a coastal lagoon, not far from the city of Piombino. From historical aerial photos, a dry area of about 8 ha surrounded by a dark almost circular sign was clearly visible. The analysis of 19th century historical cartography allowed us to recognise a hydrographic system composed of two different streams, which met exactly at the height of the dune, very close to the site, and lead to the sea with a seemingly large mouth.

The pottery and finds collected on the surface offered a wide chronological range: some were dated to the Bronze Age, many others could be referred to the Roman periods (imperial and late antique), and to the early medieval (ca 9th AD) and central medieval centuries (13th–14th) (Dallai *et al.*, 2003; Marasco, 2013).

Analyses using pXRF were performed on the Carlappiano area, by using both on-site and laboratory methods. The on-site analyses were conducted both inside and outside the anomaly previously observed by aerial images (interpretation of vertical historical

coverage, 1938–2015). For this investigation, a particular sampling strategy was developed. Six transects, with North – South direction were investigated, having one measurement every 20 meters; some measurements were taken also in the area surrounding the site. The chemical elements analysed were As, Cu, Zn, Fe, Ca and Pb (Figure 10). It is noteworthy that the element distribution showed the presence of two different soil compositions, and that the separation line between them corresponded almost perfectly to the aerial anomaly boundary. A hypothesis is these differences could be related to the function of the settlement that the archaeological excavation has linked to salt production (Dallai *et al.*, in prep).

Conclusions (L.D., A.D., V.V.)

The use of pXRF on archaeological contexts is gradually gaining credit, given to the amount of case studies that are providing new evidence for the reliability of this technique. The multidisciplinary approach developed in the Colline Metallifere project and the data collected,

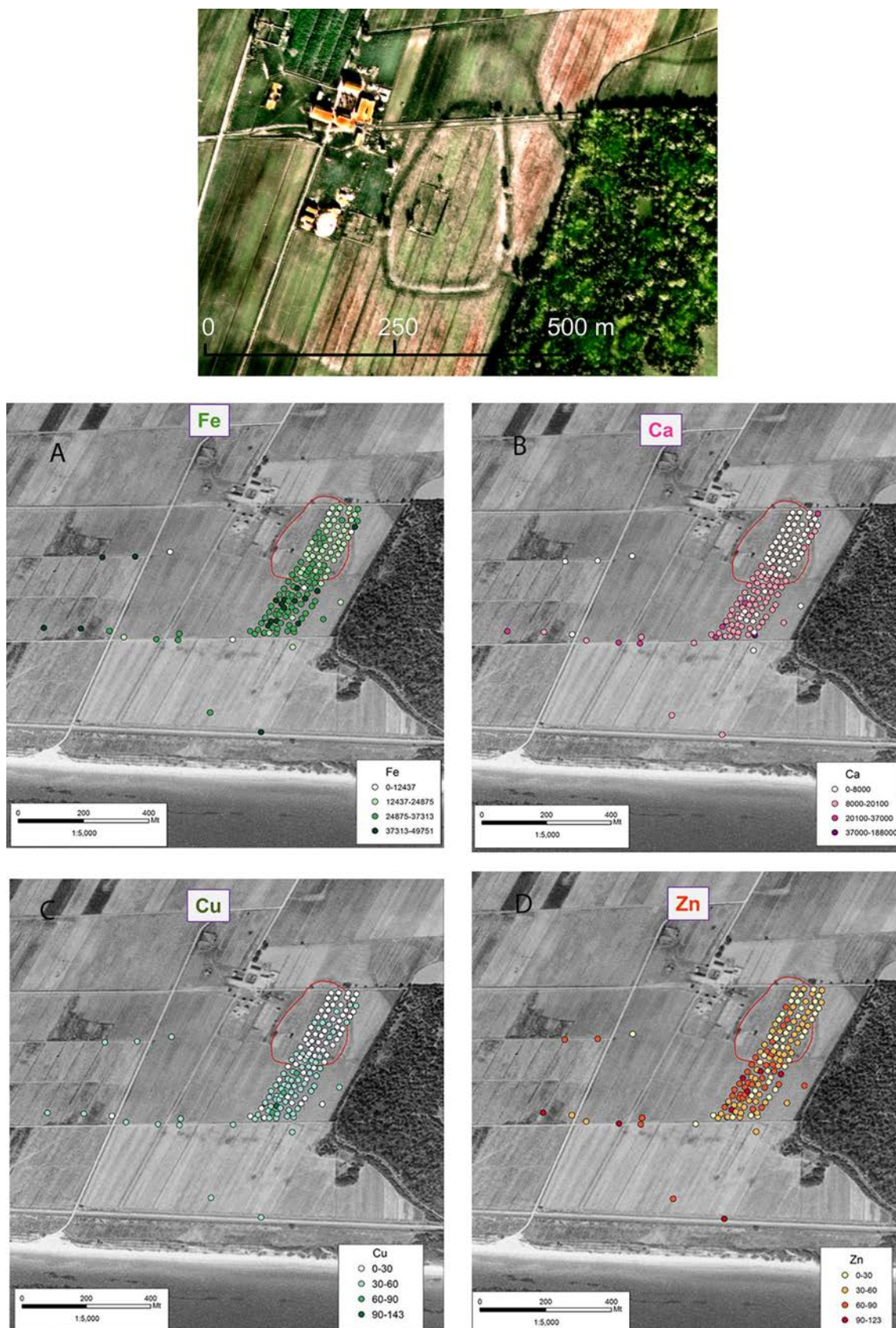


Figure 10. On the Carlappiano site (Piombino – LI), the archaeological dig has been anticipated by a multidisciplinary strategic approach, in order to map archaeological remains and relic landforms. These were visible both on-site (air photo interpretation of vertical historical coverage, 1938–2015) and in the surrounding area. In particular pXRF has highlighted how the roundish shape corresponds to an effectively different soil composition. In Figures B-E: Fe, Ca, Cu and Zn concentrations and in the legend are the concentration range considered for each element.

that we have briefly presented, demonstrate that pXRF can greatly help in understanding the nature of activities carried out on ancient sites, as well as defining the use of productive structures and entire territories. Moreover, the proper use of pXRF techniques and the careful evaluation of the analytical results based on the co-operation of different research expertise, can not only describe the environmental and historical evidences, but can be used as a powerful predictive tool which can drive the research.

Through the combination of different datasets, including those that can be obtained from pXRF analyses, the reconstruction of historical landscape can definitely be more detailed; the amount of measurements that can be performed by the user-friendly pXRF has to be considered as an advantage on wider scale projects, like the one we have described. From the consistent experience gained with the multidisciplinary study of the mining territories of the Colline Metallifere we can now experiment with the potential of pXRF on different kinds of sites, such as the coastal ones selected by the NeuMed ERC project, along with Carlappiano, which is provided the first, promising case study.

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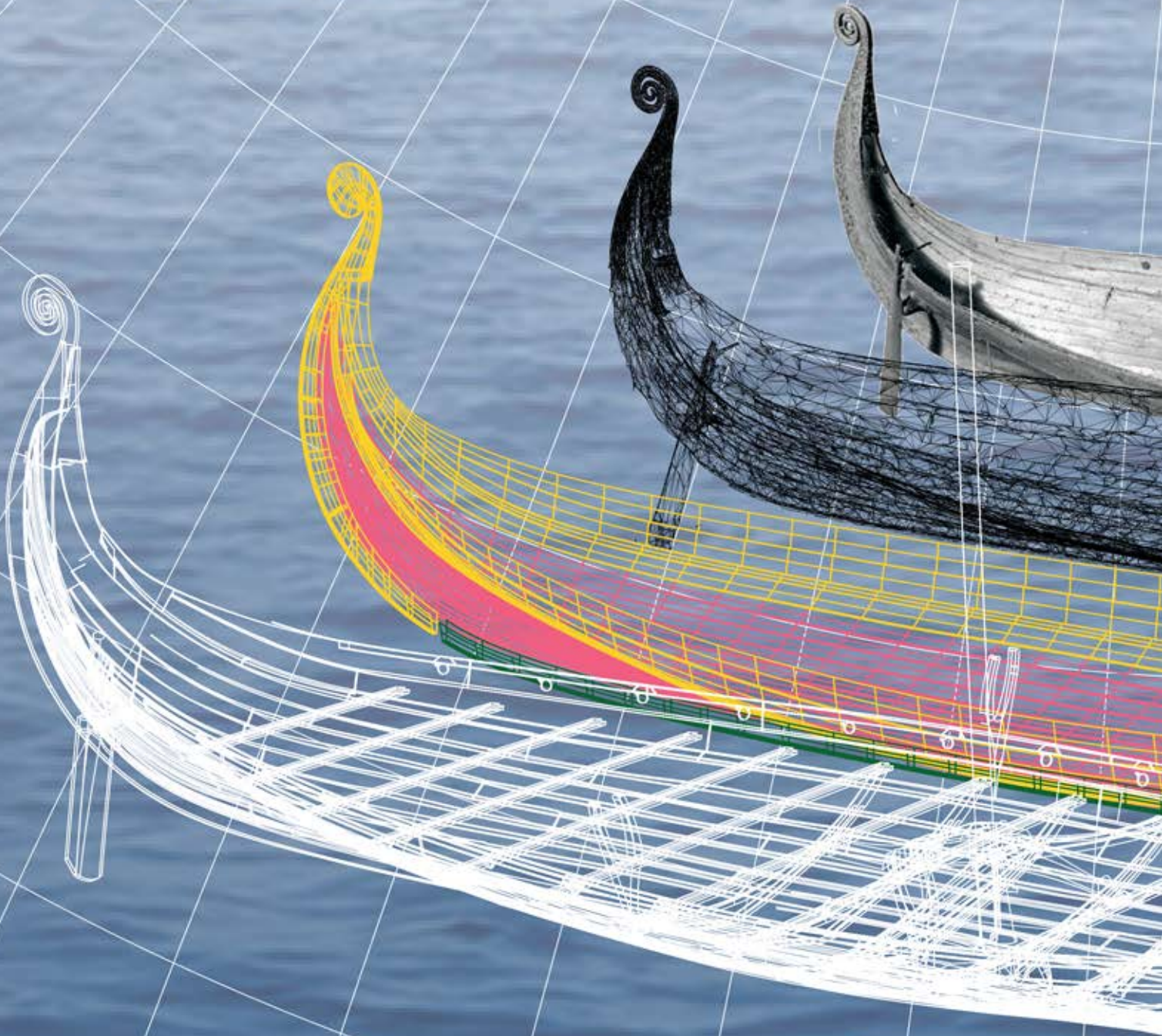
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ISBN 978-1-78491-730-2



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