Tin determination in *fistulae* seals from *Conimbriga* and *Augusta Emerita*

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ABSTRACT

Most Roman hydraulic systems were made with lead plumbing manufactured by casting lead sheets which were sealed by joining the edges with a molten metal. Modern studies suggest that the Romans used distinct techniques for joining the lead pipes (*fistulae aquariae)*, such as welding (with lead) and soldering (with a tin-lead alloy). Micro-EDXRF and micro-PIXE analysis were used for micro-chemical characterization of joining areas of 13 water lead pipes collected in Roman archaeological contexts from *Conimbriga* and *Augusta Emerita*. Results indicate that two lead pipes from *Conimbriga* were sealed with a solder alloy, having a Sn content close to the eutectic point (61.9 wt.%). The remaining *fistulae* seals have relatively low contents of Sn and also of other elements such as Sb and Cu. Micro-analyses carried out along the pipes and welding regions show the use of lead with some impurities on both areas, while the elemental mapping of tin-lead solders show the presence of the α (lead-rich) and β (tin-rich) phases.

Keywords:
Roman lead pipe; Tin-lead solder; Micro-EDXRF; Micro-PIXE

1. Introduction

Residential buildings, public baths and aqueducts usually had hydraulic systems made of lead plumbing (*fistulae aquariae*). The Roman manufacture technique of these pipes consisted on the casting of rectangular lead sheets, which were bent around a cylindrical bar and hammered into the desired shape [1, 2]. The longitudinal pipe seams were made by joining the edges of lead sheets, while the joints between two pipes was usually made by overlaying an end of the lead pipe on the beginning of the other lead pipe. The sealing of the seams consisted on a moulded "box" of a cast metal, which makes up a good quality seal that is sufficient to withstand a high water pressure [3]. Upon installation the joints between pipes were sealed on site with a similar process.

In the classical literature, Pliny, the Elder, mentions that *fistulae aquariae* were joined with a Pb-Sn solder (*tertiarium*) composed by two parts of lead and one part of tin. However, early research on Roman lead pipes have found that the joining material could either be composed by lead or lead with 5 wt.% Sn or even by a tin solder with 25 wt.% Sn, although the first seems to be clearly more common, as mentioned by Gowland *in* Wyttenbach and Schubiger [4]. Seven of these weld materials analysed by Gowland were made of ordinary lead with low Sn content: 0.6 wt% for one *fistulae* from Portugal and ≤0.3 wt % for six from Switzerland. On the other hand, comparing the Sn content of the weld material with that of the pipe material the same concentration was found, except in two cases, where it was significantly lower in pipe material. Later, on the study of 21 pipes from Roman sites mostly located in Central Europe, but also including lead pipes from Caldas de Monchique and Coimbra (Portugal), two lead pipes from Switzerland were identified as being enriched with tin (~18 wt.%) [4]. The authors suggest that these results indicate mixing of lead ingots with scrap lead of high tin content (probably as a result of using a tin-rich solder on that scrap lead) at the moment of preparing the melt to cast the lead sheets. More recently, additional evidence was found on the use of a tin solder in a *fistulae* joint from an imperial residence of the 3rd century AD in Rome [5].

Regarding the western end of the Roman Empire, namely the *Lusitania* province, there are no modern analytical studies about the manufacture of lead plumbing. Moreover, despite of the apparently undifferentiated use of the welding and soldering techniques among the Romans, the practical reasons to select such distinct methods are still unknown. Furthermore it should be noted that using or not of a tin solder can have a chronological significance, if we consider that Pliny wrote in the first century AD and many of the analysed lead pipes will be one or two centuries more recent. The use of lead requires a higher melting temperature (T ~ 327 ºC), but the solidification is much faster and produces a homogeneous seal with the lead pipe itself. On the contrary, the plumber solder mentioned by Pliny has a lower *liquidus* temperature (T_{liq} ~ 252 ºC) and a wide plastic range (ΔT ~ 69 ºC), thus allowing the *plumbarius* to mould the seal by hand during cooling with the protection of a thick pad of cloth [6]. However, tin was a less common and more expensive raw material in the Roman Empire [7], so some economic constrains might also have an important role in the selection of the joining technique. This can be particularly true concerning *Augusta Emerita*, the capital of the *Lusitania* province, which was located near to important lead-rich mineral deposits [1, 8].

The present work proposes to investigate the techniques used to seal the *fistulae* at *Conimbriga* and *Augusta Emerita*, two important Roman centres in the ancient *Lusitania* province (Figure 1). A set of 16 samples was analysed by micro energy dispersive X-ray fluorescence spectrometry (micro-EDXRF) and micro particle induced X-ray emission spectrometry (micro-PIXE) to determine the composition of seams and joints, and of the lead pipe itself. These non-destructive techniques are commonly used to study the composition of ancient alloys [9, 10, 11, 12]. The samples selected for this work can thus shed some light on the use of different alloys and solders by the Roman plumbing technology.

FIGURE 1

2. Experimental

2.1. Samples and archaeological contexts

The set of Roman lead *fistulae* seals selected for study was deposited in the Museu Monográfico e Ruínas de Conímbriga (MMC), Condeixa, and in Consorcio Ciudad Monumental de Mérida (M) and National Museum of Roman Art (MM), Mérida. The collected samples comprise seams (longitudinal pipe junctions) and joints (connection between pipes). A portion of the pipe itself was included in the sample in several cases (Figure 2).

FIGURE 2

Conimbriga was an important town of western *Lusitania*, a site with pre-Roman origins, conquered in the 2nd century BC and subject to significant urbanistic, architectonic and, specially, hydraulic engineering changes from the Augustan period onwards: the construction of the first bath complex and aqueduct, *c.* 10 BC; the generalization of water distribution, *c.* AD 40-50; and the creation of a particular style of garden architecture, heavily dependent on water, *c.* AD 125-150, of which the so-called “House of water-jets” is a prime example [13]. All samples from *Conimbriga* analysed on this work belong to the structures of this “House of water jets” (Table 1).

Augusta Emerita was the capital of *Lusitania* province. It was founded by Emperor Augustus in 27 BC, being an important centre for lead consumption and trade. Probably there were local metallurgical activities connected with lead artefacts production [1]. The analysed samples from *Augusta Emerita* belong to not recorded archaeological contexts (Table 1) and, consequently, their precise chronology is unknown.

TABLE 1

2.2. Micro-EDXRF

The non-invasive character of the X-ray fluorescence spectrometry has made it particular suitable in the analysis of cultural materials. However, due to the low penetration path of the incident X-ray beam as well of the characteristic X-ray lines emitted by the sample constituents, the obtained results are highly influenced by the corrosion layer commonly present in artefacts which have remained buried for long periods. Therefore to be able to ascertain about the bulk metal composition, the preparation of samples for analysis involved the removal of the corroded superficial layer and the polishing of the area to be analysed [14]. Optical microscopy observations were used to confirm a cleaned metallic surface for analysis.

Elemental composition analyses were performed using an ArtTAX Pro micro-EDXRF spectrometer comprising: a low-power molybdenum anode X-ray tube; a polycapillary lense that generates a spot of ~70 µm in diameter of primary radiation at the sample; a CCD camera and three beam-crossing diodes that provide the control over the exact position on the sample to be analysed; and a silicon drift electro-thermally cooled detector with a resolution of 160 eV at Mn-Kα [15]. Samples were analysed using 40 kV, 0.5 mA and 200 s of tube voltage, current intensity and live time, respectively. Each sample was analysed in several spots in order to have the average composition or the element content profile in significant examples. Quantification was made with the WinAxil software using the fundamental parameter method.

In the absence of a lead-tin reference standard, the accuracy of method was assessed with the analysis of several gold-silver standard alloys [16]. The analogy was based on the analogous matrix effects for quantitative determinations of tin and silver in lead and gold matrices, respectively: the Sn-Lα mass absorption coefficient in lead – 1581 g/cm^2 is quite comparable to the value of Ag-Lα mass absorption coefficient in gold – 1644 g/cm^2 [17]. Therefore, obtained results demonstrated that the relative uncertainty of the method should be lower than 10 % (Table 2).

TABLE 2

2.3. Statistical treatment of the data

Multivariate analysis was made with STATISTICA (v.12) software package comprising the principal components and factor analysis to reduce the number of dimensions and investigate the variability of the determined elements [18]. Factor analysis was performed on the obtained dataset to compare the compositional patterns (Pb, Sn, Sb and Cu) between analysed samples. Factor loadings representative of the relative weight of each chemical element and factor scores representing the value of each sample regarding the new variables (factors) were calculated to ascertain about the identification of common sources.

2.4. Micro-PIXE

Elemental mapping analyses were carried out by micro-PIXE with an Oxford Micro-beams type set-up, using a 2 MeV proton beam generated by a 2.5 MV Van de Graaf accelerator. The X-rays emitted by sample elements were collected by a 30 mm^2 SDD detector placed at a backward angle of 45º and with 150 eV of resolution. Beam currents of 100 pA were used for all spectra and the beam spatial resolution was kept at 3x4 μm^2. The data acquisition and beam scan were controlled by the OMDAQ as described in [19].

3. Results and discussion

3.1. Elemental composition

The results of micro-EDXRF analysis of Roman lead *fistulae* seals from *Conimbriga* and *Augusta Emerita* are displayed in Table 3.

TABLE 3

Most of the seals are composed by Pb (above 97.0 wt.%) and low amounts of Sn (up to 2.80 wt.%), Sb (up to 2.35 wt.%) and Cu (up to 0.36 wt.%). It is worth noting the different composition of two of the *fistulae* seals, the seam MMC26 and the joint MMC50 from *Conimbriga*, which are composed of tin-lead alloys with two parts of Sn and one part of Pb, approximately (68.7 and 64.1 wt.%, respectively). No compositional differences were found between lead seams and joints from the other *fistulae*, which display variable amounts of impurities. Moreover, except for two samples from *Augusta Emerita*, composed of highly pure Pb (99.9 wt.%), there were no other significant differences between lead seals of the capital of the *Lusitania* province and the Roman town of *Conimbriga*.

The elemental composition of the analysed pipes (MMC42, MMC44 and MMC47) was rather close to the associated seams (MMC43, MMC45 and MMC48, respectively). All of them present high Pb concentrations (96.8 – 98.4 wt.% Pb), although some specific differences between pairs were noticeable for the minor elements (Table 3). To better understand the sealing of these lead pipes from *Conimbriga*, several micro-analyses were carried out along a cross-section that includes the pipe MMC47 and the seam MMC48 (Fig. 3).

FIGURE 3

Analytical determinations along this profile show rather small variations on the Sn content along the pipe and seam areas, which corroborate the Sn concentration obtained for each area using the micro-EDXRF (Table 3). The results indicate that the raw material for pipes and seals would be often the same. The variable Sn impurities determined on those samples can be originated by the usage of lead obtained from different ores (it seems to be the case of samples MM11 and MM12 compared with sample MMC51) or by the recycling of old lead pipes or other lead artefacts when Sn content is higher than 1 % [20]. In brief, all this suggests that the casting process of the lead sheets would often consist in a mixing of scrap lead rich in Sn with lead ingots [4].

Overall, the composition of most *fistula* seals from *Conimbriga* and *Augusta Emerita* indicates the use of lead metal to seal the pipes, resulting in a homogeneous junction between pipe and seal. However, it is interesting to note that, according to Pliny, the Elder: "pieces of black lead cannot be soldered without the intervention of white lead, (…), nor can white lead, on the other hand, be united without the aid of black lead". The same author also refers that tin was melted with lead to form a *tertiarium* with a lead-tin 2:1 proportion for joining the pipes. In fact, two tin-rich solders were found in *Conimbriga*, but with a different composition (64.1 and 68.7 wt.% - one part of lead and two parts of tin, approximately) from the soft solder mentioned by Pliny to be commonly used by plumbers. The kind of solder identified in those two pipes seems to be applied mostly for fine work [21], being a hypereutectic alloy with a much lower *solidus/liquidus* temperature than the melting point of the lead pipe (i.e. 327 ºC for pure lead).

3.2. Multivariate analysis

Principal components and factor analysis applied to the composition of *fistulae* seals from *Conimbriga* and *Augusta Emerita* comprised four variables (Pb, Sn, Sb and Cu contents) and 13 samples (*fistulae* seals) originating two factors that report about 91% of the total variance in the original data set (Table 4).

TABLE 4

Factor loadings show that factor 1 is mostly related with Pb (inversely) and Sn (directly), while the factor 2 can be directly associated with Sb and Cu (Table 4 and Fig. 4). This layout suggests that the Sb and Cu have a different origin than Sn, thus probably not resulting from lead recycling but on different ores used. The plot of factor scores shows three well defined groups. Group 1 is composed by the Sn-Pb solders from *Conimbriga*. Group 2 includes the majority of seams and joints displaying variable Sn, Sb and Cu contents. The third group has only one sample (MMC51) having the higher amounts of Sb and Cu (2.35 wt.% and 0.36 wt.%, respectively) and no Sn. This distinct composition suggests a different raw material, probably a lead ore with higher concentration of Sb and Cu. However, it should be taken into account that the "House of water jets" suffered an extensive refurbishment during the Roman time which suggests the existence of lead mixtures from different raw material.

FIGURE 4

The elemental composition of samples from *Conimbriga* also suggests that regular use of lead scrap with Sn impurities on the casting of lead sheets was a common practice. The elemental composition of the analysed Roman *fistulae* seals also indicates the use of two joining methods namely soldering with Sn-Pb alloys or welding with ordinary lead. The first was only identified in *Conimbriga*, while the second seems to be more common making use of whatever lead was available, either pure or recycled.

3.3. Elemental mapping of seals

The *fistulae* showing Sn-Pb solders (seam MMC26 and joint MMC50) and a seam with Sn impurities (MMC48) were further studied by micro-PIXE elemental mapping in order to investigate the homogeneity of those Roman seals (Fig. 5).

FIGURE 5

The edge of the pipe in sample MMC26 is clearly visible on the right side of the elemental maps, being obviously mostly composed of lead. Adjacent to this edge is the layer of Sn-Pb solder (about 740 μm thickness) showing lead-rich areas interposed with tin-rich areas. The two different phases in this hypereutectic solder therefore correspond to the α and β phases, respectively. The copper shows a homogeneous distribution without significant differences between the pipe and solder. The joint MMC50 has a similar microstructure composed of lead-rich and tin-rich areas (a spot analysis in one of the latter shows 98.5 wt.% Sn). These tin-rich solders with composition close to the eutectic alloy and low melting point (~190 ºC) have a very short plastic range (ΔT ~ 10ºC), thus not been very appropriated for plumbing, since a high plastic range allows a better moulding during cooling, thus ensuring better sealing of the pipes. Moreover, such tin contents result on a more expensive solder than the *tertiarium* since tin was more expensive than lead [8]. The use of this tin rich solder can be related to chronological issues or its use was due to a practical reason: unavailability of the common solder (*tertiarium*).

The lead seam MMC48 illustrates a quite distinct homogeneous distribution of Sn in comparison to Sn-Pb solders (Fig. 5). Similarly, the cross section of the pipe MMC47 and seam MMC48 (Fig. 6) confirms a homogeneous sealing of the pipe with molten lead indicating a proper plumbing technology of those Roman workers.

FIGURE 6

4. Conclusion

The microchemical characterisation of Roman *fistulae* seals from *Conimbriga* allows the identification of two different joining techniques: welding and soldering with a tin-rich solder, being first the most widely used. Only two solders with 68.7 wt.% and 64.1 wt.% Sn contents were found, being these amounts very different from the proportion mentioned by Pliny, the Elder (first century AD) for tin solders in use by plumbers. The remaining *fistulae* seals have low Sn contents indicating that only lead was used for seal the junctions. Moreover, in *Augusta Emerita* only the welding technique was identified. All these results suggest that the use of lead metal was a common plumbing technique for sealing the junctions of *fistulae aquariae* among the Roman world in the *Lusitania* province, even because lead is a cheap metal and the molten lead produces a quite homogeneous seal with the lead pipe, i.e. a good quality seal that can withstand a high water pressure.

The present study also suggests that the recycling of lead for both pipes and seals was very common, although the use of very pure lead, probably not recycled, is also particularly attested in *Augusta Emerita*. Perhaps, this is a consequence of the large amount of lead available as a by-product from the silver metallurgy (cupellation), which was strongly developed under Roman rule in the Southwestern Iberia.

Nevertheless, further research must be implemented in order to know if the use or not of tin solder in the sealing of *fistulae aquariae* has a chronological or regional significance. Analyses of samples from other origins and with a more precise chronology can shed light on these issues.

Finally, these microchemical techniques proved to be important and able to reach significant results on the study of cultural heritage, namely of archaeological artefacts that must be preserved in its total integrity.

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FIGURE CAPTIONS

Fig. 1. Location of *Conimbriga* and *Augusta Emerita* in the *Lusitania* province.

Fig. 2. Examples of the *fistulae* seals from *Conimbrig*a and *Augusta Emerita*: a) seam; b) joint between two pipes.

Fig. 3. Micro-EDXRF analysis of the cross section of sample composed by pipe MMC47 and seam MMC48 (top right area in figure).

Fig. 4. Plot of Factor 1 versus Factor 2 loadings (Cu, Sn, Sb and Pb) and scores (13 *fistulae* seals samples).

Fig. 5. Micro-PIXE elemental distribution maps of cross-sections of samples MMC26 (seam), MMC48 (seam) and MMC50 (joint) (scan 1060 × 1060 μm^2; colour range from blue to red, representing from low to high intensity).

Fig.6. Micro-PIXE elemental distribution maps of cross-section of samples MMC47 and MMC48 (pipe and seam, respectively) (scan 2640 × 2640 μm^2; colour range from blue to red, representing from low to high intensity).

TABLE CAPTIONS

Table 1

Identification of seams, joints and lead pipes from *Conimbriga* (MMC – *Museu Monográfico e Ruínas de Conímbriga*) and *Augusta Emerita* (M – *Consorcio Ciudad Monumental de Mérida*, and MM – *Museo Nacional de Arte Romano de Mérida*).

Table 2

Micro-EDXRF analyses of standards Au80Ag20 and Au90Ag10 (average ± standard deviation).

Table 3

Results of micro-EDXRF analysis of the *fistulae* seals from *Conimbriga* and *Augusta Emerita* (n.d. – not detected; * pipes associated to seams)

Table 4

Factor loadings of principal components of Pb, Sn, Sb, Cu contents on the *fistulae* seals from *Conimbriga* and *Augusta Emerita* (varimax normalized; bold: factor loadings with absolute value higher than 0.7).

Figure 1


TARRACONENSIS
LUSITANIA
BAETICA
ATLANTIC OCEAN
BRACARA AUGUSTA
CONIMBRIGA
OLISIPO
AUGUSTA EMERITA
N
S
W
E
Roman province limit
100 km

Figure 2

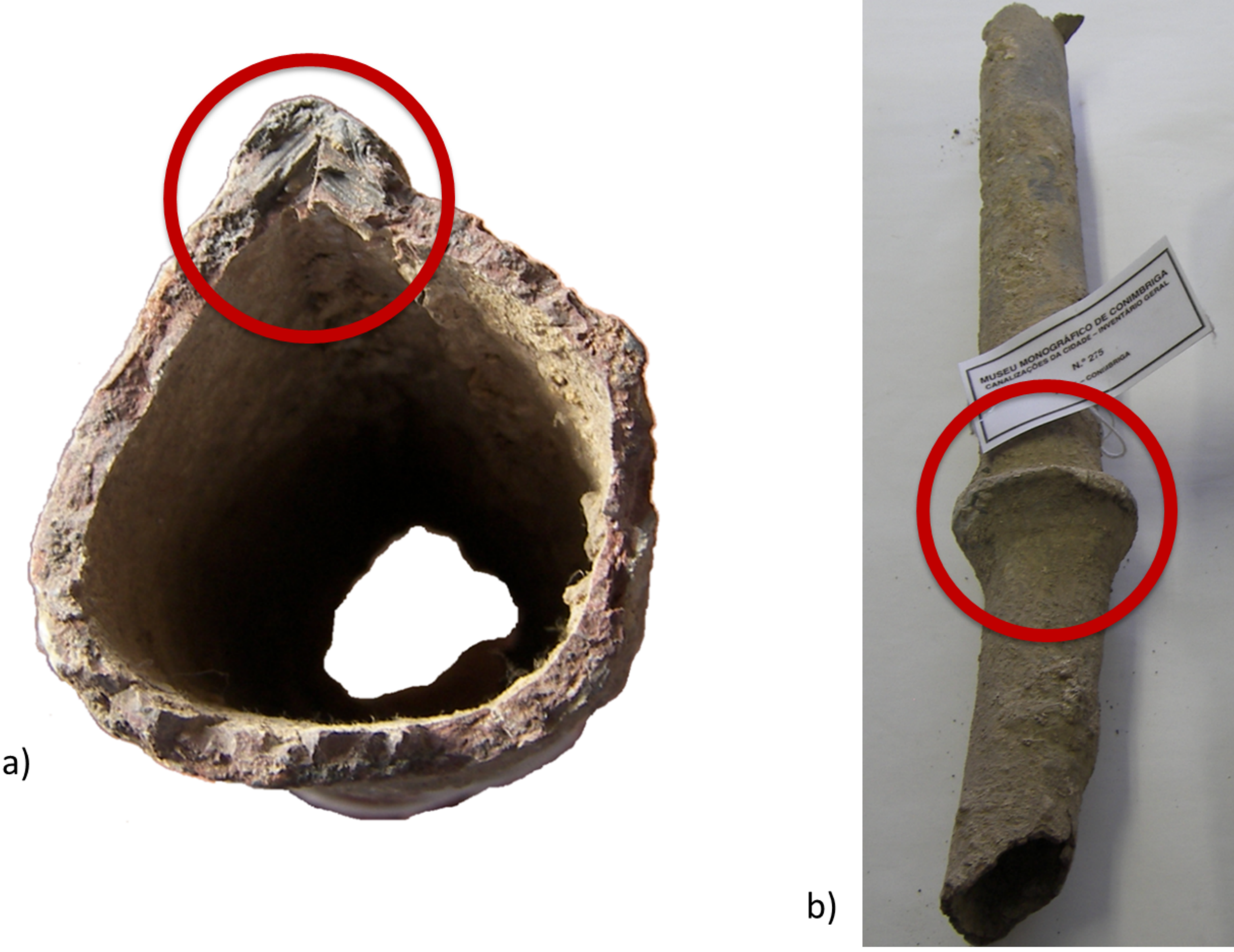

a)
b)
MUSEU MONOGRÁFICO DE CONIMBRIGA
CANALIZAÇÕES DA CIDADE – INVENTÁRIO GERAL
N.º 275
CONIMBRIGA

Figure 3

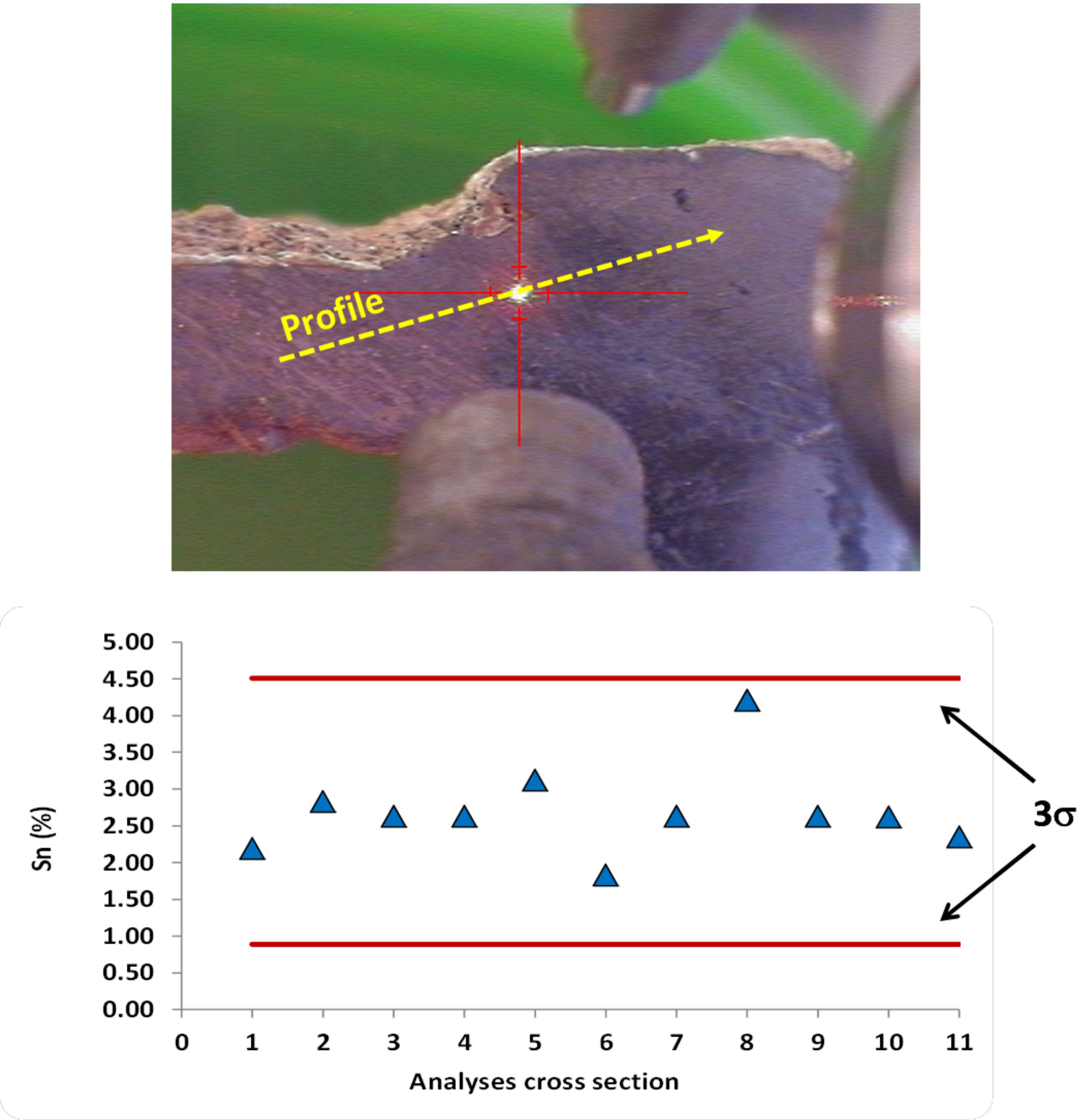
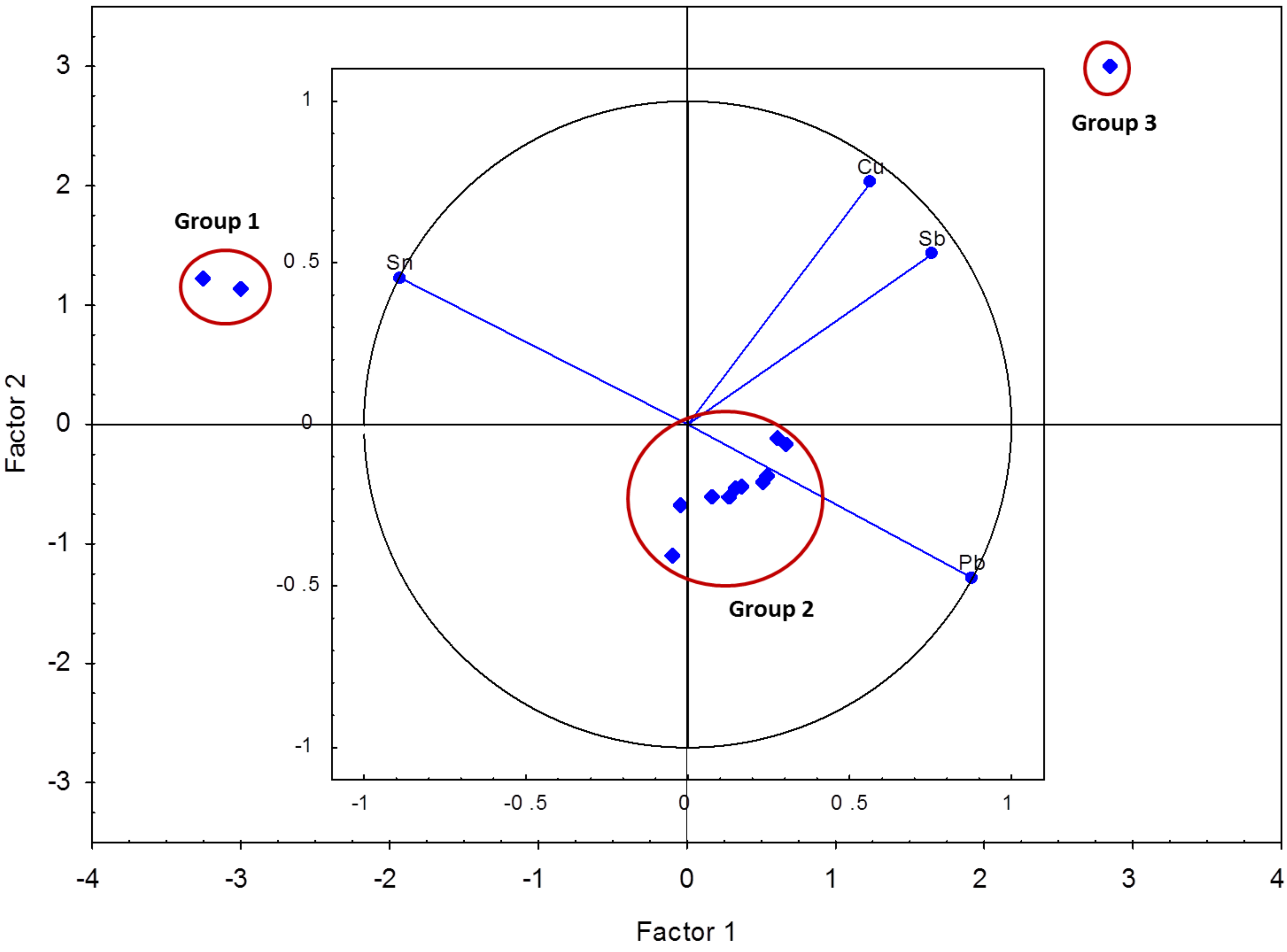

Profile
5.00
4.50
4.00
3.50
3.00
2.50
2.00
1.50
1.00
0.50
0.00
Sn (%)
0
1
2
3
4
5
6
7
8
9
10
11
Analyses cross section
3σ

Figure 4



Group 1
Group 2
Group 3
Sn
Cu
Sb
Pb
Factor 1
Factor 2
-4
-3
-2
-1
0
1
2
3
4
-3
-2
-1
0
1
2
3
-1
-0 .5
0
0 .5
1
-1
-0 .5
0
0 .5
1

Figure 5

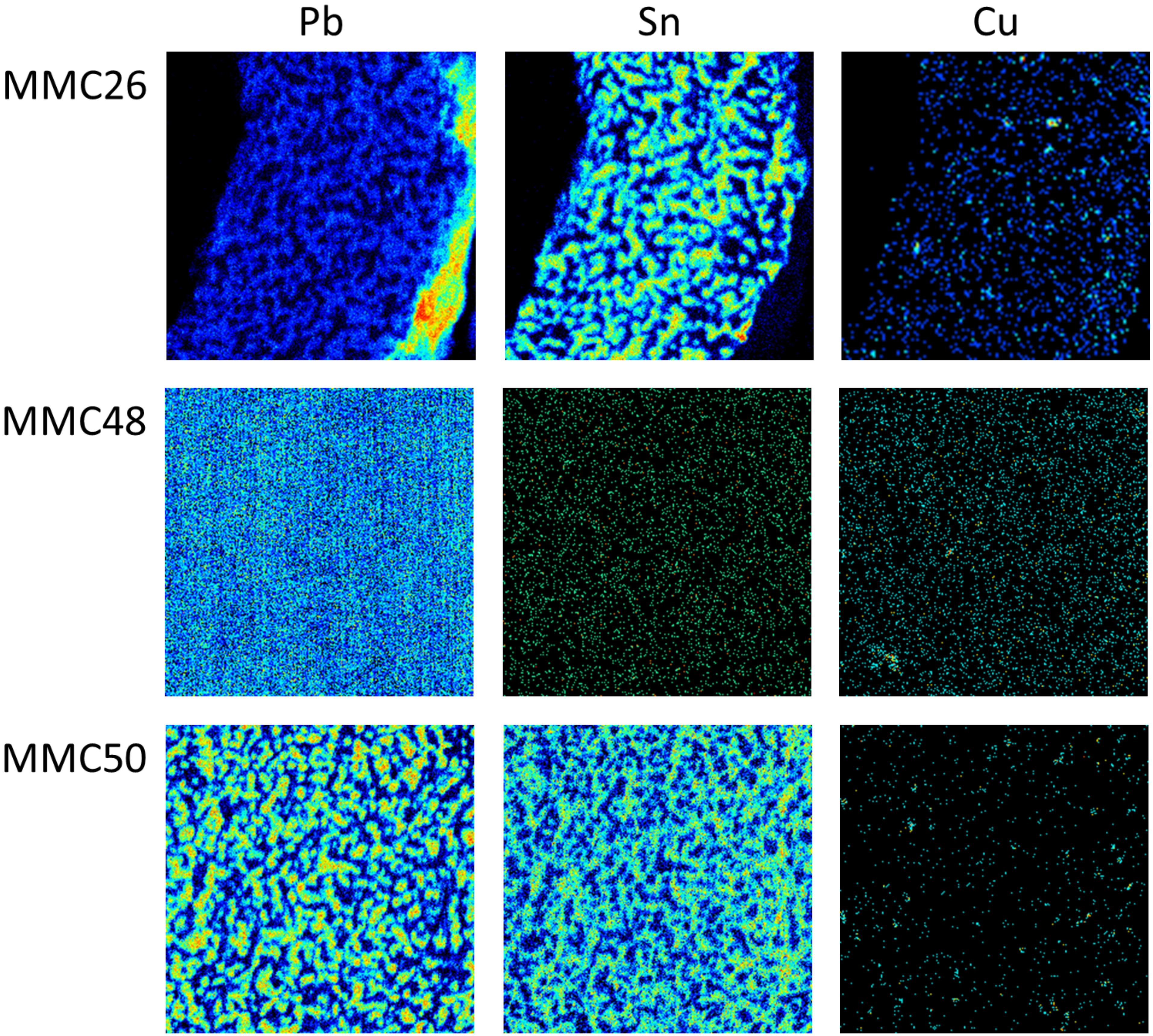


Figure 6

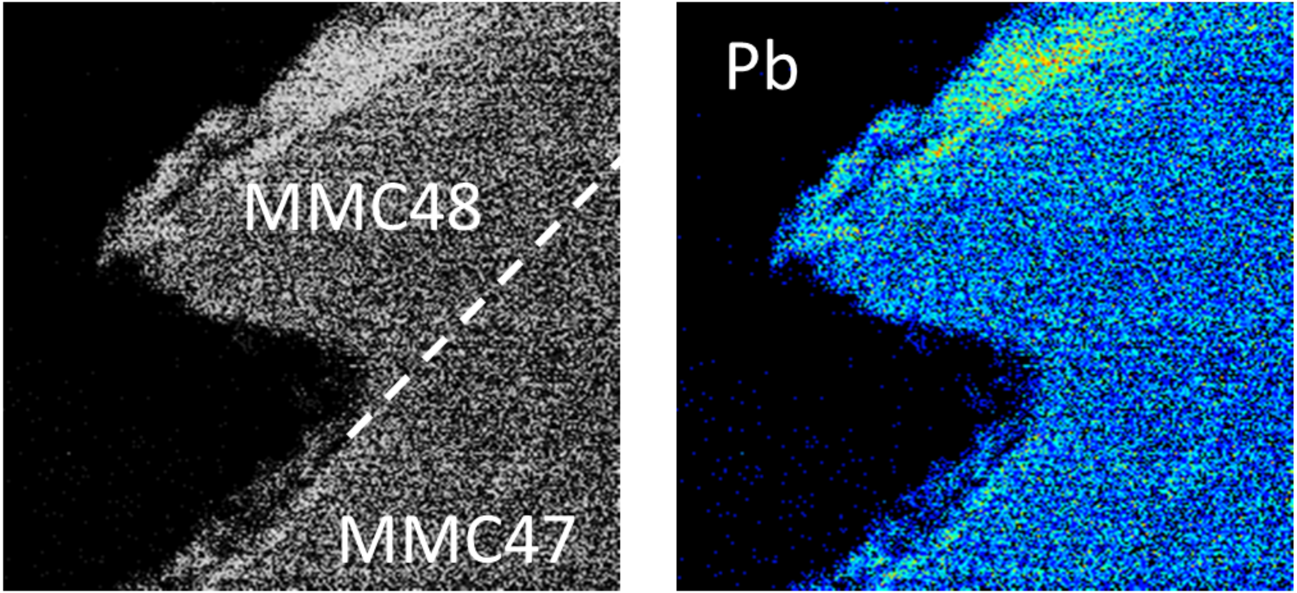


Table 1

Identification of seams, joints and lead pipes from *Conimbriga* (MMC – Museu Monográfico e Ruínas de Conímbriga) and *Augusta Emerita* (M – Consorcio Ciudad Monumental de Mérida, and MM – National Museum of Roman Art, MM).

Reference	Type	Archaeological site
MMC11	Seam	*Conimbriga* (house of water jets)
MMC26	Seam	*Conimbriga* (house of water jets)
MMC36	Seam	*Conimbriga* (house of water jets)
MMC41	Seam	*Conimbriga* (house of water jets)
MMC42	Pipe	*Conimbriga* (house of water jets)
MMC43	Seam	*Conimbriga* (house of water jets)
MMC44	Pipe	*Conimbriga* (house of water jets)
MMC45	Seam	*Conimbriga* (house of water jets)
MMC47	Pipe	*Conimbriga* (house of water jets)
MMC48	Seam	*Conimbriga* (house of water jets)
MMC50	Joint	*Conimbriga* (house of water jets)
MMC51	Joint	*Conimbriga* (house of water jets)
M2	Joint	*Augusta Emerita* (unknown archaeological context)
MM4	Joint	*Augusta Emerita* (unknown archaeological context)
MM11	Seam	*Augusta Emerita* (unknown archaeological context)
MM12	Joint	*Augusta Emerita* (unknown archaeological context)

Table 2

Micro-EDXRF analyses of standards Au80Ag20 and Au90Ag10 (average ± standard deviation).

	Au80Ag20		Au90Ag10	
	Au (wt.%)	Ag (wt.%)	Au (wt.%)	Ag (wt.%)
Standard value	80.2	19.8	90.15	9.85
Obtained value	82.1 ± 0.4	18.0 ± 0.4	90.4 ± 0.1	9.62 ± 0.06
Uncertainty	2.4 %	9.1 %	0.3 %	2.3 %

Table 3

Results of micro-EDXRF analysis of the *fistulae* seals from *Conimbriga* and *Augusta Emerita*
(n.d. – not detected; * pipes associated to seams).

Reference	Type	Pb (wt.%)	Sn (wt.%)	Sb (wt.%)	Cu (wt.%)
MMC11	Seam	98.5 ± 0.1	0.90 ± 0.22	0.62 ± 0.05	0.07 ± 0.01
MMC26	Seam	30.9 ± 1.2	68.7 ± 1.2	n.d.	0.06 ± 0.01
MMC36	Seam	98.2 ± 0.5	1.06 ± 0.32	0.70 ± 0.15	0.07 ± 0.01
MMC41	Seam	98.5 ± 0.2	0.47 ± 0.17	1.03 ± 0.25	0.05 ± 0.03
*MMC42**	*Pipe*	*98.0 ±0.3*	*1.25 ±0.09*	*0.79±0.23*	*0.09 ±0.01*
MMC43	Seam	98.4 ± 0.3	0.81 ± 0.20	0.74 ± 0.14	0.07 ± 0.03
*MMC44**	*Pipe*	*98.2 ± 0.5*	*0.79 ± 0.33*	*0.99± 0.28*	*0.05 ± 0.01*
MMC45	Seam	98.2 ± 0.4	0.70 ± 0.08	1.08 ± 0.45	0.05 ± 0.01
*MMC47**	*Pipe*	*96.8 ±0.4*	*2.80 ± 0.23*	*n.d.*	*0.13± 0.05*
MMC48	Seam	97.0 ± 0.3	2.53 ± 0.16	n.d.	0.17± 0.02
MMC50	Joint	35.9 ± 3.8	64.1 ± 3.9	n.d.	0.07 ± 0.04
MMC51	Joint	97.3 ± 0.2	n.d.	2.35 ± 0.21	0.36 ± 0.22
M2	Joint	98.0 ± 0.5	1.04 ± 0.27	0.84 ± 0.27	0.11 ± 0.05
MM4	Joint	97.3 ± 1.8	1.78 ± 0.08	1.19 ± 0.71	0.07 ± 0.01
MM11	Seam	99.9	n.d.	n.d.	0.06 ± 0.01
MM12	Joint	99.9	n.d.	n.d.	0.12 ± 0.01

Table 4

Factor loadings of principal components of Pb, Sn, Sb, Cu contents on the *fistulae* seals from *Conimbriga* and *Augusta Emerita* (varimax normalized; bold: factor loadings with absolute value higher than 0.7).

Variable	Factor 1	Factor 2
Pb	**0.987**	0.144
Sn	**-0.984**	0.167
Sb	0.303	**0.828**
Cu	0.022	**0.920**
Explained variance	**61%**	**30%**