Neutron based archaeometallurgical investigation of Picenan and Roman age metal objects from the Academia Georgica Treiensis collection (Italy)

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ABSTRACT
Non-destructive prompt gamma activation analysis (PGAA), neutron radiography (NR) and high resolution time-of-flight neutron diffraction (TOF-ND) have been applied to investigate metal artefacts belonging to the Academia Georgica Treiensis (AGT) collection. 8 archaeological items have been analysed, by using the facilities of the Budapest Neutron Centre (BNC). Some of these objects mostly dating back probably to the VI-IV century B.C. and presumed to be discovered in the Marche Region, Italy. The primary goal of the analyses was to advance the correct technological and material description of the objects, providing scientific data for further and more comprehensive comparative analyses also covering the find material from the close archaeological sites.

The neutron investigations allowed determining the bulk composition, also providing either a qualitative and quantitative assessment of the phase composition and the structural properties of the constituents, or radiographic images, which would finally help to identify possible manufacturing techniques.

Additional examinations, carried out by external milli-beam particle induced X-ray emission spectroscopy (PIXE), provided quantitative analyses of major and trace elements (e.g., Fe, Pb and As) in order to recognize the constitutive alloys and to supply information on the near-surface elemental composition, complementary to the data characteristic for the bulk.

The obtained results, thought to be useful to set up a classification according to the chemical composition, and this way achieve important information related to the possible provenance of the objects.

1. Introduction

A completely fledged craftsmanship of Cu, Ag and Sb was developed in central Italy in the early/mid 4th millennium B.C., prospering until the late 3rd millennium B.C. as it was progressively replaced by tin-bronze technology. A regional metalworking style advanced in parallel by the Central Italian communities (Dolfini 2010).

Since the second half of the 10th century B.C., an archaeological reality emerged in very different regions and civilizations (e.g., Etruscan and Picenan). The Piceni (or Picentes) belong to the pre-roman culture in central Italy essentially from the IX to the beginning of the III century B.C., in practice the so called “Iron Age”. Most of the findings focus the Picenan area coincident with the Augustan Regio V (Picenum). Knowledge of this civilization is based almost exclusively on archaeological documentation arising especially from the excavation of the necropolis, but a number of villages and some workshop areas are also known. The remains emerged in the Macerata Province (e.g., Matelica) emphasize extensive necropolis already from the 9th-8th century B.C., while the remains of the built-up areas date back to the period 7th-4th century B.C. (Trump 1966, I Piceni e l’Italia medio-adriatica 2003, Archeologia nel Maceratese: nuove acquisizioni 2005).

Archaeological documentation is essential to understand this civilization as well as the various other Italic cultural facies before the Romanization. The ancient proofs coming from the Greek and Roman classic sources, in fact, are not many and the information achieved from the Picentes in their own language (usually, wrecks of short funereal inscriptions or religious dedications diversely deciphered) are not yet enough interpreted. Funereal equipment, obviously, is the major part of the emerged remains, which provide us the most consisting quantity of scientific information (Rogante et al. 2010).

AGT is one of the oldest Italian Academies, created in the XV century for the main interests of poetry and
literature. In the XVIII century AGT decided to renovate its interests, focusing on the more properly scientific, technical and socially useful problems and “encouraging rational and practical studies to improve agriculture and industry and to honour sciences, literature and arts” (Benigni 1939). The Archaeological Collection of AGT was put together by the noble family of Teloni, Counts in Treia, Italy, likely between the end of the XVIII and the beginning of the XIX century. The Collection has been recently reorganized and inventoried: it is composed of more than five hundred objects (e.g., armours, appliques, table-ware, disks, rings, pendants, fibulas, ceramics and varied instrumenta), mostly of unknown origin but practically belonging from the Prehistory to the ancient period; nevertheless, it is not excluded that some objects are dated successively (Stortoni 2000, Stortoni 2004).

Neutron analyses have recently become an increasingly significant probe for materials across a wide range of disciplines. These tools play a key role in the study of work objects belonging to the Cultural Heritage, and they are useful to characterise archaeological or industrial materials, when destruction of the objects is strictly forbidden (Rogante 2008, Rogante and Rosta 2005). Neutrons penetrate through coating and corrosion layers deep into the object without substantial attenuation. This property makes them ideal for non-destructive and non-invasive bulk analysis of both elemental and phase composition as well as for identifying bulk topology or inside content.

8 metal objects from the AGT collection have been selected for this investigation, since they seem the most intrinsically significant and presaging of scientific capacity at present unexpressed. The primary goals of our study were: the accurate description of these objects identifying the respective alloy compositions; to get useful information for the reconstruction of their manufacture, the way of usage and the potential production area, the latter in order to clarify its possible relation to the regional metalworking activity. Results were aimed also to provide the basis for comparative studies, including the comparison of the obtained data with those achieved by PGAA on Picenan bronze objects found in the Matelica and Fabriano necropoles, Italy (Rogante et al. 2010, Rogante et al. 2007). That investigation notably supported the native origin of the studied objects. This comparison, thus, will help archaeologists to better interpret the possible geographical origins of the analysed objects, as well as to enhance their knowledge on the involved manufacturing processes.

All the analyses were performed by using the facilities of the BNC, operated by the Centre for Energy Research, and the Wigner Research Centre for Physics, Hungarian Academy of Sciences. The European CHARISMA project provided the transnational access to the advanced scientific instrumentation.

2. The objects investigated.

For this analysis, 8 metallic objects from the collection have been chosen.

Object n. 1: Basin with pearl rim (inventory n. 515) Height = 4.6 cm; Ø rim = 24 cm; thickness = 0.07 cm. Basin with horizontal and reverted rim, upper decorated with embossment produced rows of bosses, profile stilly frusto-conical, straight flared walls, flat bottom.

First half VI century B.C. – early V century B.C. This object belongs to a known type, which has wide spread from Sicily to central-western Europe, but is primarily documented in Italic centre area. In Picenum there is evidence, for example, in Numana, Recanati, Castelbellino, and in the territories of Camerino and Ascoli. Claims are frequent even in the Umbrian and the Romagna areas, always in contexts dating from the first half of the sixth century B.C. and early V century. The basin with pearl hem is a type of container that has experienced wide spread and long duration throughout Europe. It was produced serially - probably in Orvieto, specialized in bronzes production, or otherwise in Etruscan workshops - by the technique of cold hammering and punching.

Object n. 2: Bronze oinochoe with cornet shape mouth (inventory n. 513) Height = 300 mm; Ø bottom 125 mm; rim’s thickness = 1.9 mm; lamina’s thickness = 1.1 mm.

Bronze oinochoe with cornet shape mouth, low cylindrical neck, convex shoulder, ovoid body, flat bottom. Missing handle and half of the body. VI-V century B.C. Object with special characteristics, difficult to place in serial production, it is perhaps an antecedent of the most known Schnabelkanne and plumpe Kanne of Etruscan production. It is a vessel used to serve in the symposia service to pour and tap into wine. If it was in association with the previous basin (object n. 1), it could provide a service for ablutions.

Object n. 3: fibula with foliated arch having a double ripple (inventory n. 446) Length = 68 mm; height = 27 mm; max. width of the arch = 12 mm.

Bronze fibula characterized by a foliated arch with double ripple, a spring with two involvements, a bracket with rectangular appendage terminating in a ringlet. Missing of the barb.

Object n. 4: fibula with enlarged bow (inventory n. 448) Length = 48 mm; height = 26 mm; width of the arch = 5 mm.
Bronze fibula with enlarged bow, spring with two involvements, elongated bracket. Missing of the barb and part of the bracket.

Object n. 5: fibula with a simple arch (inventory n. 450)  
Length = 43 mm; height = 27 mm; width of the arch = 4 mm.

Bronze fibula with a simple arch and a spring with two involvements. Missing of the barb and the bracket.

Of the three mutilated fibulae examined, only the object n. 3, characterized by a foliated arch with double ripple, provides useful elements to a typological classification associated with a specific history: based on comparisons, it can be dated to the Piceno IV A-IV B of the Lollini classification (580-470 BC), but with a continuation also in the subsequent phases. Moreover, it is likely that objects n. 4 and n. 5 belong to the same chronological horizon of the object n. 3, in an associative composition known from other Picenan necropolis in the Marche region.

Object n. 6: Small olla with shaped stamnos (inventory n. 514)  
Height 115 mm; Ø rim 115 mm; Ø bottom 85 mm; thickness 1 mm.
Olla with reverted lip and bottom folded rim, rounded shoulder, belly tapered towards the bottom, flat bottom. On the body, in correspondence of the point of maximum expansion and in the bottom conjunction point, at least five plates of bronze sheet of irregular shape are noticed, fixed with rivets in quadrangular section, which constitute the patches of an old restoration.

V-IV century B.C.

Containers of this kind were serially produced in the internal Etruria and widely distributed in Etruscan-Italic area between the second half of the fifth and the first decades of the fourth century B.C.

Object n. 7: Cylindrical rod (inventory n. 517)  
Length 305 mm; Ø 13.8-24 mm.

Cylindrical rod made by full cast, terminating with a pin at one end and with a net cut at the other. Two nuclei of indeterminable organic material (bone?) are inserted in the rod, which originally formed a single mass. Function and history of the object are not determinable.

Object n. 8: Polilicnes oil lamp (inventory n. 507)  
Height 50 mm; width 200 mm; Ø bottom 51 mm.
A not common metallic polilicnes oil lamp with six elongated and voluted radial nozzles of ogive-shape tip, of which three are decorated with different bearded faces representing the Silenus mythological figure.

3. Methodology

Most metal archaeological artefacts are composites of more than one type of metal, each sort adding its distinctive character to the whole. Composition of an artefact is also permanently associated with its function, and the major step in planning a conservation action or preservation measures is also to identify the component materials.

Appropriate analyse methods to get accurate information on composition, thus, are essential to archaeological research, since to identify the constitutive metals gives a substantial help in identifying the object. The investigations, in our case, were carried out through a multistage process at macroscopic, microscopic and large-scale analytical levels, by combining complementary non-destructive and non-invasive large-scale techniques, which needed no sample preparation. The main advantage of this methodology relies on the adoption of diverse spectroscopic techniques that, although the complexity of these artefacts, allow achieving complementary information as well as a relevant vision into the composition and the alteration phases of the investigated objects.
Apart from PIXE, the neutron-based analyses formed the most essential part of the experiment: neutron analytical methods were applied to explore the compositional or microstructural characteristics of the investigated artefact.

3.1. PGAA and NR

The bulk elemental concentrations of the alloying components were identified at the neutron capture \(\gamma\)-ray PGAA and NIPS-NORMA experimental stations. PGAA or PGNAA (Prompt Gamma Neutron Activation Analysis) is based on the detection of characteristic prompt gamma photons that originate in \((n,\gamma)\) nuclear reactions, and it allows a non-destructive analysis of elemental composition by observing neutron-capture prompt \(\gamma\)-rays. PGAA enables quantitative measurement of all the chemical elements. The detection limits depend on the neutron absorption cross-sections of the given nuclei (Révay 2009), the matrix composition and the acquisition time. The prompt gamma spectra were collected by using a Compton-suppressed HPGe detector, which has been accurately calibrated. The gamma-ray spectra were evaluated adopting the Hypermet-PC program (Révay et al. 2005). The quantitative analysis is based on the \(k_0\) principle, adopting the spectroscopic data libraries developed at the PGAA laboratory of the BNC (Firestone et al. 2007). The composition was determined using the methods described in (Révay 2009). For a detailed treatment of the theoretical bases, see (Révay 2009, Révay et al. 2004, Molnár et al. 1997, Molnár et al. 1998, Révay 2006, Révay and Molnár 2003).

Both the major components and a variety of minor or trace elements were possible to detect. The data acquisition time has varied in the range 3,300-67,000 seconds, depending on the sample dimensional characteristics, in order to gain sufficiently good statistics for spectrum evaluation. The objects have been irradiated in the cold neutron beam of \(1.2 \times 10^8 \text{ cm}^{-2}\text{s}^{-1}\) thermal equivalent flux. The cross-section of the neutron beam varied in the range 5-24 mm\(^2\), depending on the sample dimensional characteristics. No holder

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**Figure 2.** Object n. 2.

**Figure 3.** Objects n. 3 (a), 4 (b) and 5 (c).
was used, except for objects n. 3-5 (i.e., the fibulas), which were fastened with Teflon strings onto the aluminium frame before being introduced in the PGAA measurement chamber. The NIPS-NORMA station has been designed for investigation of larger objects of dimensions up to $20 \times 20 \times 20 \text{ cm}^3$ and for a large variety of experiments involving neutron capture induced prompt and delayed gamma radiation, including $\gamma-\gamma$-coincidences; $\gamma$-rays as low as 14 keV can be also observed. Additionally to the bulk composition measurements, the radiography setup of this station made possible to perform NR of selected parts and to combine imaging methods with elemental analysis (Belgya et al. 2008). NR is based on the attenuation of a neutron beam, where an object modifies the incident beam when it passes through. The sample is positioned on a xyzw sample stage and the transmitted neutrons create signals in a two-dimensional position sensitive detector. In the present investigation, the raw two-dimensional digital image was corrected for both open beam image and the dark image profiles.

3.2. TOF-ND

Materials and technological traits were studied by using the TOF-ND facility of the BRR (Káli et al. 2007). In this instrument the neutron pulses are short as 10 microseconds and they are produced by a fast double disc chopper. TOF-ND was set up for the wavelength band from 1.4 Å to 3.2 Å. The detector position was fixed at $176^\circ$ (back scattering geometry) and the total flight path of neutrons to the detectors was 25 m. At these conditions, constant $2.6 \times 10^{-3}$ Å FWHM Bragg peaks were measured in the 0.7-1.6 Å d-spacing range. The penetration depth of neutrons in copper at the applied wavelength range is in the order of 1 cm and the maximum beam size is $10 \times 2.5$ cm, consequently real bulk average results could be achieved. The analysis aimed the quantitative bulk characterisation of the phase composition and the structural properties of the metallic constituents of the alloy, averaged in a macroscopic volume. Also possible traces of the past treatment (mechanical or thermal) were observed in the crystallographic texture. To eliminate the effect of the possible preferred orientation and the background from the corrosion and crust, the positions of the valuable peaks were determined by multiple peak fit and the lattice parameters of the hcp system ($a$ and $c$) were fitted for the set of peak data, taking into account the uncertainties in the weighting.

3.3. PIXE

The PIXE measurements were carried out at the 5MV Van De Graaff accelerator of the Institute of Particle and Nuclear Physics of the Wigner Research Centre, Hungarian Academy of Sciences. The elemental composition of the base material could be determined in the upper 10 µm thick layer of the objects, as well as analytical data about the occurring surface enrichments. This method was initially proposed in 1970 by S. Johansson, who developed it successively together with his colleagues R. Akselsson and T.B. Johansson (Johansson et al. 1970). In this technique, selected spots of an object of practically any size and shape can be bombarded by energetic protons, and the characteristic X-rays produced are employed for qualitative and quantitative analysis of the irradiated volume. The spot size can be varied in the range of 0.8-3 mm, allowing to individually analyse small particularities of the samples. The sensitivities are in the range of 50-1000 ppm, they vary with the elements and depend also on the matrix composition (Gyódi et al. 1999). PIXE is adoptable in the archaeology, art
conservation and geology, to support answer questions of authenticity, dating and provenance, as well as in various other fields such as life sciences (Szőkefalvi-Nagy 1994). For a detailed treatment of the theoretical bases, see (Gyódi et al. 1999, Szőkefalvi-Nagy 1994, Maenhaut and Malmqvist 2002, Mandó and Przybyłowicz 2009, Puc et al. 2002). Spot measurements were carried out on the studied objects, and no beam scanning was performed. To elude any inadequate deterioration, the surface treatment or oxidization analysis was not performed on the investigated objects. The properly collimated proton beam of 2.5 MeV energy was extracted from the evacuated beam pipe to air through a 7.5 micrometre thick Kapton foil. A target-window distance of 10 mm was selected for the measurements, therefore the resulting beam diameter was \( \approx 1.5 \) mm. The characteristic X-rays were collected by an Amptek X-123 spectrometer. The SDD type detector of 25 mm\(^2\) × 500 \( \mu \)m active volume positioned at 135 degree with respect to the beam direction. The energy resolution was 130 eV for the Mn-K\(\alpha\) line. A 60 \( \mu \)m PC absorber was used to stop the backscattered protons, and in some cases a 0.1 mm Al filter was applied to attenuate the low energy X-rays. A beam of 2.5 MeV protons was adopted with currents around 2 nA, with measurement times varying in the range 600-1800 s. The net X-ray peak intensities and the concentration calculations were obtained by using the GUPIX program package (Campbell et al. 2000).

4. Results and discussions

Table 1 reports the evaluation results of PGAA spectra, with concentrations of all the identified elements and their absolute (+/-) and relative uncertainties. Elements of H, B, Cl and Ca are probably attributed to corrosion products and/or to contamination from the soil. The compositions of the metal objects, thus, when only the alloying components are considered, is reported in Table 2.

The results, after removing the possible elements originating from the contamination of the soil, have been normalized to 100%, therefore allowing obtaining the original alloy composition. By analysing the obtained PGAA data, the following considerations can be written.

Objects from n. 1 to n. 6 result made of tin-bronze (Cu + Sn) alloys, with Sn varying in the range 1.7-6.9 (wt %) and Ag as minor element varying in the range 0.03-0.09. Objects n. 2 and 6, in particular, show practically no minor element, and object n. 6 has a much lower content of Sn, compared to the others (objects n. 1 to n. 5). Object n. 7 results made of Cu-Zn-(Sn) alloy. This artefact, in particular, presents two sliding not-metallic inserts (see Figure 5), which can be considered as organic parts. Table 3 reports the resulted elements’ contain related to these inserts, i.e. their composition that can be related to petrified wood or bone part. In columns A, the components include Cu, Zn and Cd, which are from the brass rod. In columns B, the composition of the sole petrified part is reported, thus excluding the contribution of Cu, Zn and Cd. Next, for comparison, the result of earlier PGAA measurements on bone standards are reported.

Data of Table 3 are not enough to determine if these inserts are really made of wood or bone or other material. It could be supposed that the Ca content (3.7 wt\%) is too low for bone. The high amount of S, nonetheless, does not correspond to wood. To decide unquestionably, it is suggested to make comparisons
| el. | det. limit | 1 | 2 | 3 | 4 | 5 | 6 | 7 (1st end) | 8 |
|-----|------------|---|---|---|---|---|---|-------------|---|
|     |            | c%| el/el | abs. | unc. | c%| el/el | abs. | unc. | c%| el/el | abs. | unc. | c%| el/el | abs. | unc. | c%| el/el | abs. | unc. | c%| el/el | abs. | unc. |
| H   | 0.05       | 0.140 | 0.005 | 3.5 | 0.122 | 0.003 | 2.4 |
| B   | 0.000032   | 0.075 | 0.002 | 3.3 | 0.226 | 0.005 | 2.3 |
| Cl  | 0.003      | 0.112 | 0.003 | 3.0 | 1.73  | 0.05  | 3.2 |
| Ca  | 0.06       | 96.10 | 0.30  | 0.3  | 95.33 | 0.16  | 0.2 |
| Cu  | 0.24       | 0.08  | 0.01  | 7.0  | |
| Zn  | 0.03       | 3.6   | 0.3   | 8.0  | 4.3   | 0.2   | 3.7 |
| Ag  | 0.00004    | 0.5   | 0.2   | 5.0  | 4.8   | 0.2   | 5.0 |
| Sn  | 0.5        | 0.430 | 0.010 | 2.3  | 0.251 | 0.006 | 2.4 |
| Cd  | 0.00032    | 1.68  | 0.05  | 2.9  | 0.56  | 0.02  | 3.0 |
| Sn  | 0.5        | 92.70 | 0.20  | 0.2  | 92.22 | 0.26  | 0.3 |
| Pb  | 2          | 5.1   | 0.2   | 3.2  | 6.9   | 0.3   | 3.7 |
|     |            | 0.114 | 0.003 | 2.5  | 0.03  | 0.002 | 7.0 |
|     |            | 0.00108 | 0.00002 | 2.2  | 0.00026 | 0.00001 | 7.0 |
|     |            | 0.121 | 0.003 | 2.8  | 0.06  | 0.004 | 7.0 |
|     |            | 0.98  | 0.07  | 7.0  | 66.3  | 1.3   | 1.9 |
|     |            | 97.09 | 0.18  | 0.2  | 32.3  | 1.3   | 3.9 |
| Cd  | 0.0004     | 0.082 | 0.004 | 4.0  | 0.089 | 0.005 | 6.0 |
| Sn  | 0.5        | 5.1   | 0.2   | 3.2  | 6.9   | 0.3   | 3.7 |
| Pb  | 2          | 1.7   | 0.2   | 10.0 | 1.3   | 0.2   | 16.0 |
|     |            | 0.114 | 0.003 | 2.5  | 0.03  | 0.002 | 7.0 |
|     |            | 0.121 | 0.003 | 2.8  | 0.06  | 0.004 | 7.0 |
|     |            | 0.98  | 0.07  | 7.0  | 66.3  | 1.3   | 1.9 |
|     |            | 97.09 | 0.18  | 0.2  | 32.3  | 1.3   | 3.9 |
| Cd  | 0.0004     | 0.082 | 0.004 | 4.0  | 0.089 | 0.005 | 6.0 |
| Sn  | 0.5        | 5.1   | 0.2   | 3.2  | 6.9   | 0.3   | 3.7 |
| Pb  | 2          | 1.7   | 0.2   | 10.0 | 1.3   | 0.2   | 16.0 |

abs. unc. = absolute uncertainty of the measured value
det. limit = detection limit
el. = element
unc. = uncertainty
(#) = long measurement
with the results of complementary investigation, e.g. carried out by infrared spectroscopy.

Object n. 8 (the investigated polilicnes lamp with six radial nozzles) results made mainly of Zn (87.4), with Pb (7.8), Sn (4.0) and Cu (0.83), and for such a constitutive materials’ composition it seemed to be rare. The identification of the compositional and technological details, however, explained the date and origin of the lamp. The metallic zinc raw material as well as the two-piece mould applied in the casting revealed that this object is not an archaeological artefact from the Imperial Roman period but a possible copy from the 19th century, in style and shape reflecting the Roman lamps manufactured typically in copper alloy and pottery. Figure 7 reports NR images obtained by investigating detail of the objects n. 7 and n. 8.

Concerning the object 8, in particular, The TOF-ND data pointed out that metallic zinc was melted and cast during the manufacturing process. No significant amount of other phases was measurable (neither other metals nor epsilon brass). Apart from Zn and Cu, PGAA and PIXE analyses identified trace amounts of Sn, Pb and Fe as well.

Table 4 reports the TOF-ND results. Table 5a reports the resulted elements’ contain related to each of the objects investigated by PIXE, while Table 5b reports the composition of the low atomic number deposits on the surface of the objects 3 and 5 in mass percentage (%m/m) detected by PIXE using a 60 µm PC filter.

Figure 8 shows, as an example, the PIXE plot referred to the object n. 1.

By analysing the obtained PIXE data, the following considerations can be written.

Object n. 1 results made of a tin-bronze (Cu + Sn) alloy (examination in a corroded area), with Fe and Pb as minor elements. Object n. 2 results made of a tin-bronze alloy (examinations in black areas). Object n. 3 as well results made of a tin-bronze alloy, with about 15-20 %m/m Sn and 1.5-2.3 %m/m Pb. All of them can be classified as high tin

Table 2. Compositions (wt %) of the investigated metal objects obtained by PGAA.

| element | Object n. | 1 | 2 | 3 | 4 | 4(#) | 5 | 6 | 7 (1st end) | 7 (2nd end) | 8 |
|---------|-----------|---|---|---|---|------|---|---|-----------|-----------|---|
| Cu      | 96.3      | 95.7 | 95.9 | 95.0 | 94.7 | 93.0 | 98.3 | 66.4 | 65.3 | 0.83 |
| Zn      | –         | –   | –   | –   | –   | 3.6  | –   | –   | –         | 4.3        | 32.3 |
| Sn      | 3.6       | 4.3  | 4.1  | 4.9  | 5.2  | 6.9  | 1.7  | 1.3  | 1.5        | 3.5        | 7.8  |
| Pb      | –         | –   | –   | –   | –   | –    | –    | –    | –         | 0.007      | 0.008 |
| Cd      | –         | –   | –   | –   | –   | –    | –    | –    | –         | –         | –    |
| Ag      | 0.08      | 0.03 | 0.09 | 0.08 | 0.08 | –    | –    | –    | –         | –         | –    |

(#) long measurement

Table 3. Elements’ contain (concentration in ppm) for the two sliding inserts of object n. 7, obtained by PGAA.

| object n. 7 - A | object n. 7 - B | standards for comparison |
|----------------|----------------|-------------------------|
|                | Conc. / wt%    | abs. unc.               | Conc. / wt%    | abs. unc. | Conc. / wt%    | abs. unc. | fossil Conc. / wt% | bone Conc. / wt% |
| H              | 0.05           | 1.771                   | 0.062          | 3.5       | 0.2            | 1.54       | 1.9 |
| B              | 0.00003        | 0.00502                 | 0.00018        | 0.0098    | 0.0006         | 0.0004     | 0.000063       |
| C              | 1.8            | 0.4                      | 0.4            | 3.5       | 0.8            | 1.46       | 3.3 |
| Na             | 0.013          | 0.033                    | 0.02           | 0.64      | 0.05           | 0.18       | 0.31       |
| P              | 0.5            | 1.9                      | 0.3            | 3.7       | 0.6            | 7.6        | 5.7 |
| S              | 0.5            | 17.0                     | 0.8            | 32.2      | 2.3            | 0.74       | 0.09 |
| Cl             | 0.003          | 0.54                     | 0.02           | 1.04      | 0.07           | 0.008      | 0.03 |
| K              | 0.1            | 0.46                     | 0.04           | 0.9       | 0.1            | 0.012      | 0.03 |
| Ca             | 0.06           | 1.9                      | 0.1            | 3.7       | 0.3            | 16.22      | 10.7 |
| Cu             | 0.16           | 4.1                      | 0.2            | –         | –              | –         | – |
| Zn             | 0.0004         | 0.00143                  | 0.00006        | –         | –              | –         | – |

abs. unc. = absolute uncertainty of the measured value
det. limit = detection limit
el. = element
Conc. = concentration
wt% = weight %

Figure 7. NR detail images: object n. 8, extremity of an ogive-shaped radial nozzle (a); object n. 7, tip (b).
| Object n. | measured part     | supposed alloy by PGAA | composition                              | Cu₂O   | state                                                                 |
|----------|-------------------|-------------------------|------------------------------------------|--------|----------------------------------------------------------------------|
| 1        | bottom of the basin (2.5 × 10 cm²) | tin (normalised concentration by PGAA: 3.61 wt% Sn) | alpha-bronze 4.2 ± 0.5 wt% Sn, delta is not observable | n.d.   | Compared to the relatively low concentration, the concentration distribution is very broad, probably due to micro or macro segregation, although pure copper phase is not observable. |
| 2        | spout (2.0 × 8 cm²) | tin (normalised concentration by PGAA: 4.4 wt% Sn) | alpha-bronze 5.5 ± 0.5 wt% Sn, delta is not observable | notably present | Rather homogeneous low bronze, most likely annealed several times. |
| 3        | the whole object | tin (normalised concentration by PGAA: 4.1 wt% Sn) | alpha-bronze 4.8 ± 0.5 wt% Sn, delta is not observable | present | Very well homogenised. |
| 4        | the whole object | not investigated by TOF-ND | alpha-bronze 8.87 ± 0.5 wt% Sn, delta is not observable | present | Well homogenised. |
| 5        | spout (2.0 × 8 cm²) | tin (normalised concentration by PGAA: 6.9 wt% Sn) | Copper (0.16 ± 0.05 wt% Sn if it is the main alloyer), probably less than 7 wt % alpha bronze, containing 5 wt % Sn is present | present | Very pure copper (or the alloyers neutralises the effects of each-others). A very small peak broadening is observable. It can be caused by the different substitution atoms and the geometry of the obj, rather than stress. The presence of the second phase (alpha) is uncertain. |
| 6        | first end, first end without the tip, the tip, second end | not clear | Cu + alpha                                      |        |                                                                       |
| 7        | one arm            | Copper                  | almost pure zinc, no phase was identified |        |                                                                       |

Table 4. The TOF-ND results.

bronzes, presenting percentages above 14%. Moreover, Si and K are present on the surface. Object n. 4 results made of Cu (examination in a corroded area), with Fe and Pb as minor elements. Object n. 5 results made of Cu + minor and trace elements + light elements on the corroded surface. By using a 60 um PC filter, Sn-KA content results out of the range. By using a 0.1 mm Al filter, Sn content is around the detection level, so it can be considered as a minor or trace element. Object n. 6 results made of Cu (corroded) with Fe and As as minor elements. Object n. 7 results made of Cu-Zn-(Pb) alloy, with surface inhomogeneities. This object, in particular, presents two sliding not-metallic inserts, which can be considered as organic part with Ca and K. Table 6 reports the resulted elements’ contain related to these inserts. The "invisible elements" were not taken into account.

Data of Table 6, similarly to those of Table 3, are not enough to determine if these inserts are really made of wood or bone or other material.

Object n. 8 results made mainly of Zn, while Pb has been detected only on some areas (see Figure 9).

The presence of elements other than the main alloy constituents (e.g., Pb), sometimes, appears relevant, being able to reach even values over 1%.

The eventual changeability of the analytical results in diverse areas of a certain object is mainly due to the chosen conditions for the data achievement. Since PIXE investigates very small areas, it is very sensitive to selected surface areas and to the eventual formation of segregations, as happening particularly in Sn-rich
bronzes (Northover and Rychner 1998). Consequently, to correctly determine the metal alloy composition, we investigated mainly the not corroded surfaces. Figure 10 shows the objects n. 2 during the PIXE investigations.

Table 5a. Elemental concentration in mass percentage (%m/m) for the objects investigated by PIXE.

| object | file | notes | element | Mn | Fe | Co | Ni | Cu | Zn | As | Sr | Sn | Pb |
|--------|------|-------|---------|----|----|----|----|----|----|----|----|----|----|
| 1      | 6716 | g.s.  | 0.03    | 0.53| 0.02| 0.01| 95.34| 0.00| 0.07| 0.00| 3.82| 0.16 |
| 2      | 6717 | g.s.  | 0.02    | 0.52| 0.02| 0.01| 94.72| 0.06| 0.07| 0.00| 4.44| 0.14 |
| 3      | 6722 | black | 0.01    | 0.29| 0.00| 0.00| 84.54| 0.13| 1.27| 0.00| 13.72| 0.11 |
| 4      | 6723 | green | 0.02    | 0.33| 0.00| 0.00| 99.58| 0.04| 0.03| 0.00| 0.00| 0.01 |
| 5      | 6724 | black, very clean | 0.00 | 0.18| 0.01| 0.00| 87.05| 0.05| 1.00| 0.00| 11.65| 0.06 |
| 6      | 6706 | ***, # | 0.00    | 0.04| 0.00| 0.00| 78.64| 0.29| 0.10| 0.00| 0.00| 0.00 |
| 7      | 6707 | *     | 0.00    | 0.06| 0.00| 0.09| 75.04| 0.31| 0.20| 0.00| 0.00| 0.00 |
| 8      | 6708 | *     | 0.00    | 0.15| 0.01| 0.09| 83.35| 0.11| 0.33| 0.00| 14.64| 1.33 |
| 9      | 6709 | *     | 0.01    | 0.23| 0.01| 0.17| 75.52| 0.62| 0.42| 0.00| 20.69| 2.32 |

Table 5b. Composition of the low atomic number deposits on the surface of the objects 3 and 5 in mass percentage (%m/m) detected by PIXE using a 60 µm PC filter.

| object | file | Si   | P    | Cl   | K    | Ca   | Ti   | V    | Cr   |
|--------|------|------|------|------|------|------|------|------|------|
| 3      | 6706 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| 4      | 6707 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| 5      | 6708 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| 6      | 6709 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |

Legenda of notes
- c.s. = corroded spot; g.s. = green spot; l.s. = lower side; g.o.s. = glazed off spot
- #) = for the characterisation of the light elements on the surface
- *) = filter: 01 mm Al
- **) = filter: 60 um PC

5. Considerations and conclusions.

The preliminary autopsy analysis of the eight investigated objects, by the morphological and stylistic point of view, led up to the following provisional conclusions:

- Six artefacts (items Nos. 1, 2, 3, 4, 5 and 6) are of the Iron Age (VI-IV century B.C.), of which at least pottery (items Nos. 1, 2 and 6) were most likely produced in the Etruscan scope.
- Concerning the object n. 7, dating and nature of the artefact are ignored.
- Concerning the object n. 8, its attribution to Roman times is expected.

The analyses carried out to determine the metal alloy composition of the object n. 1 showed a very high copper content (96.3%), much higher than that of basins with pearly rim belonging to the Orientalizing period and coming from Table 5a.
archaeological found in the Marche region (Matelica and Monte Penna di S. Severino), which have a copper content in the range 87–90% and consequently a far more important tin content (until a 10.84%)\(^9\).

An alloy with higher copper content could be indicative of a voluntary search of an easier workability of the foil, perhaps connected with a massive production of serial type as that of the basins in this period (VI-V century BC). Comparison data on basins of this period could be useful to determine with certainty whether it is a specific choice of production of the metallurgical workshop or of a random composition due to various factors (e.g., by remelting of the scrap).

Even in the case of the object n. 2, a very high percentage value of copper was determined (95.7%), almost certainly linked to the need for modelling (raising) of the foil. It would be interesting to assess - if preserved - the composition of the alloy of the loop that being a melted product would probably have presented a higher content of tin.

Two of the three fibulas (objects n. 3, 4 and 5), present high values of tin (5.2 to 6.9%): for melted products, (such as these fibulas), for which it was not required a malleability for subsequent processing, the
alloy composition included a higher percentage of tin, which guaranteed performances of greater solidity and required a lower melting point.

Concerning the **object n. 6**, this type of bowl must be made by the so called “raising” technique, i.e. the progressive raising of the body starting from a single sheet, textured and shaped until it reaches the desired shape and subjected to a subsequent turning process. To be able to model and turn an object having a so complex profile, it is necessary to use a bronze composed of a high percentage of copper, as indeed is confirmed by the analyses carried out: for this object, a composition has been assessed that is very close to pure copper (98.3%). This fact, on the other hand, justifies the high fragility of the lamina and explains both the numerous old restorations applied in this case and the frequency with which restoration interventions were made on specimens of the same type.

Concerning the **object n. 7**, the analyses carried out to determine the metal alloy composition showed a high percentage of zinc (32–33%) that, associated with the copper percentage (≈46–65%) is a brass alloy. The brass was not unknown in antiquity, but usually it included zinc percentages ranging between 10 and 25%; the rare protohistorical materials found in Italy and made of brass alloy, e.g., have a percentage of 11–12% zinc. Brass, also being considered a precious metal, was used for prestigious objects of the aristocratic classes, such as armour or greaves; consequently it is unlikely that this alloy was chosen to manufacture a heavy object with an obscure functional value. These elements, combined with the fact that hardly a heavy metal object like this would remain unscathed through the Middle Ages, lead us to conclude that this object is attributable to post-classical age, probably modern.

Concerning the **polilicnes oil lamp (object n. 8)**, a preliminary optical microscopic examination affirmed that it was cast, revealing that the applied technique was not the lost wax casting, typical for ancient Roman metal lamps: the object was cast in two pieces, which were then presumably joined by soldering. The dark contours observed around the wick-holes showed that the lamp was really in use, the flame might deposit carbon residue on its surface. The captured neutron radiographic image did not reveal any hidden remains of the inner construction or the past content of the lamp. Considering the few historical and archaeological sources relevant to the metallurgical processes in the Antiquity and Middle Ages, it is concluded that until the early 19th century, metallic zinc was available only as a by-product of the zinc-rich lead ores smelting. The amount of the gained zinc, however, was practically insufficient for manufacturing large objects. In case of the investigated lamp, hence, compositional data query its authenticity raising other hypotheses about its origin. The technical details of the casting process, similarly, argue against the ancient date of the lamp. The use of the two-piece mould instead of the lost wax casting suggests a modern, 19th century product, in style and shape reflecting the Roman lamps realised mostly in copper alloy and pottery.

In conclusion, from the analysis carried out on the metal alloy it has been possible to deduce that two objects of the collection (items Nos. 7 and 8: metal rod and lamp) are not made of bronze but of a zinc alloy (brass) and therefore they are not old but attributable to the modern age (in particular, the lamp is a copy dated on the XIX century).

Concerning the objects of the Picenan age, the obtained data have confirmed the knowledge already achieved on the metal alloy composition choices in relation to production techniques: for the objects manufactured by hammering or rolling (objects Nos. 1, 2 and 6) with eventual processing by lathe, and almost certainly produced in Etruscan area, the very high copper content is probably essential for ensuring the best performance of plasticity. In the objects produced by full fusion (objects Nos. 3, 4 and 5), instead, the greater percentage of tin guaranteed greater solidity, less heat needs, improved fusibility and therefore greater control in the casting process.

It is possible, therefore, that the composition of the copper alloy is related to technical characteristics dependent on the processing rather than in choices of chronological or production areal type.

The data collected by archaeometallurgical investigations - even in the serious limitations due to the collecting provenance of the finds and then the lack of a reference archaeological context - can help integrating the database of ancient metal products dating back to the Iron Age.

The obtained results can give also a contribution in obtaining indications to create replicas of the major element compositions and in accordance with the supposed manufacturing process, as well as to analyse that as a standard to compare with the original objects.

If these techniques were applied, instead, to investigate finds from archaeological excavation methodologically controlled, the results could be more helpful to find answers to the historical-archaeological questions that the usual sources do not succeed by now to get ahead into focus (e.g., to validate the hypotheses related to the functions of the studied objects and the places of their production).

The progress of this research and the formation of an increasingly rich and reliable database will allow researchers gathering more and more interesting and original features, with potential scientific effects.

**Notes**

1. This is the F3-Type or Type 6 Albanian, corresponding to the III A 38 Bonomi Type and the "Imola-Hundersingen" Type of Krausse.
2. Quagliotti Area, tomb 143 ([Lollini 1998], Fig. 3); Davanzali area, tomb 225 ([Landolfi 1992], p. 309).
3. Tombs 10 and 35 (Percossi Serenelli 1980).
4. Tomb 3 (Krause 1996, p. 422, list 13F, n. 86).
5. (Fabrini and Sebastiani 1982), pp. 102–103, Tabs. LIx and LXX.
6. (Speranza 2001), pp. 150–153.
7. From the tomb 123 of Numana (Lollini 1979, pag. 57, Tabs. XI b - XIII).
8. (Belli 1993) p. 76–78, with notes 34–41; (Belli 2002) pp. 51–52.
9. (Milazzo 2008) pp. 251–252 and Appendix.
10. (Montanaro 2015) n. 14, Figs. 27–28; (Gatti and Ascensi 1993) p. 105, n. 8.213; Imola 1981, n. 87.71, p. 178, Fig. 2; (De Juliis 1990) p. 53, n. 52; p. 79, n. 44.
11. (Giardino 2010) pp. 190–191.

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