Prompt Gamma Activation Analysis
of bronze fragments from archaeological artefacts

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ABSTRACT

The Prompt Gamma Activation Analysis facility of the Budapest Research Reactor has been adopted as a non-destructive elemental analytical technique to investigate bronze archaeological artefacts belonging to the Picenum necropolis of the Matelica site, Italy. These objects date back to the 7th century B.C. and they have been discovered during a rescue excavation carried out in the period 1994-2005. 17 fragments selected from the archaeological founds have been analyzed, together with a bronze fragment belonging to the archaeological area of Fabriano, Italy. The major components have been determined of all the analyzed objects, and some trace elements - e.g., Sb, As and Ag - have been identified. The comparison of the compositions of the different samples allowed to achieve information on the possible provenance, being useful, moreover, to set up a classification according to the chemical composition.

INTRODUCTION

The Italian town of Matelica is placed in the Marche region and precisely in the Esino River valley at a distance of about seventy kilometres from the Adriatic Sea. The Roman historian Plinio il Vecchio (1st century A.D.), in his main work, cited this town with the name of *Matilica* and put it in the *Regio VI* (corresponding approximately to the current Umbria region) and not in the *Regio V Picenum* (corresponding approximately to the territory of the current Marche region) [1]. *Matilica* was a *Municipium* at least from the half of the 1st century A.D. and it reached the greatest magnificence between the 1st and the 2nd century A.D. The period of its decline, as per various other demic diffusions into that zone, reached its peak in concomitance with the passing of foreign barbaric troops from the 5th century A.D. onwards.

Matelica is a commercial place of exchanges and crossroads of important trans-appennine communication lines from at least 3,000 years. In the last three lustra, it saw a proliferation of occasional and also planned archaeological excavations presaging of sensational findings, which oblige the experts to rewrite the history of this boundary zone between the area of Umbrian and that of Picenan influence. Figure 1 refers to the recent excavation in the Matelica archaeological site. The *Piceni* (or *Picentes*) belong to the pre-roman Culture that gravitated in this central part of Italy essentially from the 10th to the 3rd century B.C., in practice the so called "Iron Age". The remains emerged in the Matelica area emphasize extensive necropoles 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. The archaeological documentation is essential to understand the Picenan civilization as well as a lot of other Italic cultural *facies* before the Romanization: in fact, the ancient proofs coming from the Greek and Roman classic sources are not many and the information achieved from the Picenes in their own language (usually, wrecks of short funereal inscriptions or religious dedications diversely interpreted) are not yet enough studied. Funereal

Matelica Picenum necropolis, Italy

equipments are, obviously, the major part or the emerged remains, which provide us the most consisting quantity of scientific information. Also thanks to the findings of Matelica, the recovered fragments of Picenan inhabited structures are no more so rare; for their nature of typical proto-urban architectures, they present intrinsic motives of structural fragility (being mainly huts edified by using perishable elements, e.g. wooden and stramineous materials, pile pierces, raceways, fireplaces and little dry walls). The very favourable synergy developed between the territorial Local Board (Municipality of Matelica) and the Public Authority in charge for the protection and the exploitation of the Cultural Heritage (Superintendence for the Archaeological Heritage of Marche Region) has allowed, in the last years, to broaden remarkably the knowledge of this subject.

Figure 1. During the recent excavation in the Matelica archaeological site. [photo: M. Parrini]

It is possible, in this way, to express in our time new hypotheses and, therefore, to advance different socio-economical and cultural scenarios: they throw a new light on a reality that anciently was very complex and vital and of which, at the present state of the art, we maybe do not succeed to understand still enough the complicated events [2–10]. Chemical analysis of archaeological artefacts (metal objects, ceramics, polished stone tools, sculptures, etc.) has become, recently, an important tool for source identification, provenance analysis based on the determination of major- and trace elements.

The most usual analytical methods - *e.g.* X-Ray Fluorescence Spectroscopy (XRF), Instrumental Neutron Activation Analysis (INAA) and Inductively Coupled Plasma-Mass Spectroscopy (ICP-MS) - require partial or total destruction of the samples, which often is not allowed in case of valuable whole or fragmental artefacts; another method has been recently developed - the time-domain terahertz (Td-THz) imaging -, which consents *in-situ*

Figure 2. Original *situla* and its fragments analyzed by PGAA, with reference to the sample No. 1.

Figure 3. Original *cista* and its fragments analyzed by PGAA, with reference to the sample No. 6.

mapping of the internal layer-structure of the artefact [11]. Neutron investigations have recently become an increasingly significant probe for materials across a wide range of disciplines and can reveal significant properties about materials. Neutrons are becoming ever more useful in the non-destructive characterisation of materials and components of archaeological and industrial interests. For instance, the industrial applications of neutron techniques are being developed in various new sectors [12-13]. Prompt Gamma Activation Analysis (PGAA) is based on the detection of characteristic prompt gamma photons that originate in (n,γ) nuclear reactions. Every atomic nuclei, apart from ^{4}He, may undergo a (n,γ) reaction with diverse probabilities and the energies of the emitted gamma photons are characteristic for every given isotope. The intensities of the gamma peaks are proportional to the amount of a given isotope and this phenomenon consents to use a quantitative elemental (isotopic) analysis method known as PGAA or PGNAA (Prompt Gamma Neutron Activation Analysis) [12, 14 and 15]. The principles of the method have been well known for decades.

Since neutrons are able to go through deeper - as well as the surface - layers of the material, PGAA provides information on the sample as a whole without differentiating between "bulk" and "surface" composition of the investigated object: consequently, complementary analytical investigations are suggested - e.g., proton induced X-ray emission (PIXE) -, whenever an important effect of weathering is presumed on the surface. An important feature of PGAA is that it is a multi-element method: both the major components and a variety of trace elements can be detected in different types of objects, although with different sensitivities. The elements detectable with the highest sensitivities

Figure 4. Original helmet and its fragment analyzed by PGAA, with reference to the sample No. 7.

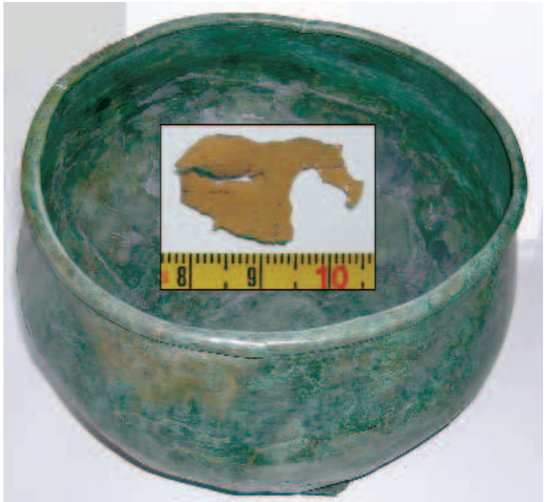


Figure 5. Original washbowl artefact and its fragment analyzed by PGAA, with reference to the sample No. 8.

are B, Cd, Sm and Gd (with a detection limit ~0.1 μg/g). C, N, O, F, Sn, Pb and Bi (with detection limits above 1000 μg/g) are the elements most difficult to identify. Sensitivities for every chemical element have been determined, using internal standardization or comparator measurements at the BRR. The detection limits depend on the composition of the investigated sample and can be enhanced by increase the acquisition time.

EXPERIMENTAL

Since bronzes are considered significant to confirm eventually the hypothesis of a local manufacturing place of the studied artefacts, the Superintendence for the Archaeological Heritage of Marche Region has appointed Rogante Engineering Office to investigate by PGAA 18 bronze fragments, 17 coming from the Matelica area and one, as a comparison, coming from the Fabriano area. This last fragment belongs, in particular, to a biconical discovered by I. Dall'Osso in 1915 [6] in the tomb No. 3 of Santa Maria in Campo site and represents the most relevant artefact either as a finding of that area, or for the purpose of the present study, because its provenance from a local metallurgical area is well known.

The 17 bronze fragments of the Matelica site form part of the following artefacts discovered in six different tombs: *situlae*, *patera*, *cista*, helmets, washbowls, lance prong coil, ring and *tripode* [16, 17]. Table 1 describes the major typological information and locations where the archaeological bronzes have been collected. Figures 2 to 8 show the original artefacts and their fragments analyzed by PGAA, with reference to samples No. 1, 6, 7, 8, 9, 10 and 14 respectively, while Figure 9 shows the sample No. 11. The investigation has been complementary to atomic absorption, atomic emission and neutron diffraction. The measurements were performed at the PGAA facility at the 10 MW BRR (see Figure 10). This instrumentation has been developed since 1996. A guided thermal neutron beam of $2.5{\cdot}10^6$ $cm^{-2}s^{-1}$ flux, supplied by the BRR, was used until 2000 for analysis [18]. The thermal equivalent flux of the cold beam, at the time of these measurements, was

Figure 6. Original lance prong and its fragment analyzed by PGAA, with reference to the sample No. 9.

510^7 $cm^{-2}s^{-1}$; successively, between January and October 2007, the first two upgrades of the neutron guides were performed and a thermal equivalent flux of 710^7 $cm^{-2}s^{-1}$ was achieved. A final third upgrade, then, allowed the use of an intensity of 1.210^8 $cm^{-2}s^{-1}$.

The neutrons are guided by Ni-coated guide tubes towards the sample position, which is approximately 35 m away from the reactor core.

The beam's usual cross section is 2×2 cm^2 and it can be reduced to 1×1 cm^2 or even to a smaller area. The prompt-gamma photons are detected by a complex detector system, which contains an n-type high-purity germanium (HPGe) main detector with a Bismuth Germanate (BGO) scintillator detector annulus to perform Compton-suppression measurements. The Compton-suppressed HPGe detector has been precisely calibrated [19] and the gamma-ray spectra have been evaluated using the Hypermet-PC program [20, 21]. The quantitative analysis was based on the k_0 principle [22], while the composition has been determined using the methods described in ref [23] and the uncertainties of the concentration values according to [24]. The spectroscopic data libraries have been developed at the Institute of Isotopes of the BRR [25].

The investigation strategy, in general, has consisted in providing information on the considered artefacts, including a technological description and a comparative analysis, with the aim to better identify their provenance and eventual relationships with known features of other production.

The bronze fragments have been basically placed into the normal sample position and irradiated in the cold neutron beam. The neutron beam cross-section was 2×2 or 1×1 cm^2, depending on the sample dimensional characteristics. Figure 11 shows the sample No. 16 fastened with Teflon strings onto the aluminium frame and being placed into the PGAA measurement chamber.

A number of bronze fragments, due to their small size, have been irradiated in vacuum to decrease the measured spectra background. A 5000 to 50000 seconds time for data acquisition has been chosen, depending on the sample size, to achieve reasonable statistics for spectrum evaluation.

Figure 7. Original ring and its fragments analyzed by PGAA, with reference to the sample No. 10.

Figure 8. Original biconical and its fragment analyzed by PGAA, with reference to the sample No. 14.

RESULT

PGAA consented to determine the major elements of Cu and Sn, while the amount of Pb was under the detection limit in all the investigated samples. Additional minor components of Fe, Co, Zn, As, Ag and Sb have been detected in some of the objects. The presence of H, Si, and Cl can be ascribed to contamination from the environment. Table 2 reports the detailed PGAA results, including the concentrations under the given detection limits.

The approximate detection limits found for the investigated bronze fragments were (in wt%): Zn: 0.5, Sn: 2, Pb: 2, H: 0.005, B: 0.00003, Al: 1.5, Si: 1.6, P: 3.1, S: 0.3, Cl: 0.05, K: 0.74, Ca: 1.8, Ti: 0.09, Cr: 0.1, Mn: 0.22,

Fe: 0.48, Co: 0.014, Ni: 0.078, As: 0.28, Ag: 0.049, Cd: 0.0009, Sb: 0.55, Au: 0.037, Hg: 0.006, supposing a 10000 s irradiation of a 0.1 cm thick sample with a 2×2 cm^2 beam. Figure 12 shows the PGAA spectrum resulting from the investigation of the sample No. 3. The Sn/Cu mass ratios have been determined, furthermore, with the aim to compare the alloying composition of the investigated bronze artefacts (see Figure 13).
This figure shows very evidently that no significant differences, except for the sample No. 6, can be individuated by comparing the bronze objects of the

Figure 9. Wall fragment of a *situla*, with reference to the sample No. 11.

Figure 10. Instrument Hall and Prompt Gamma Activation Analysis facility of the BRR.

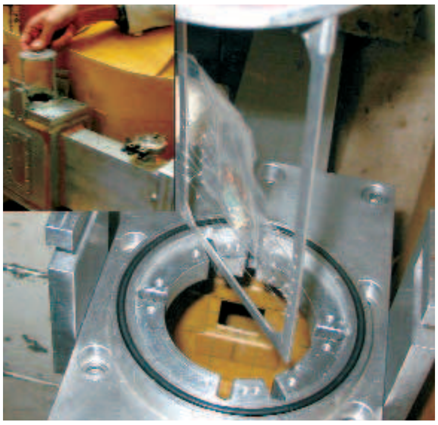
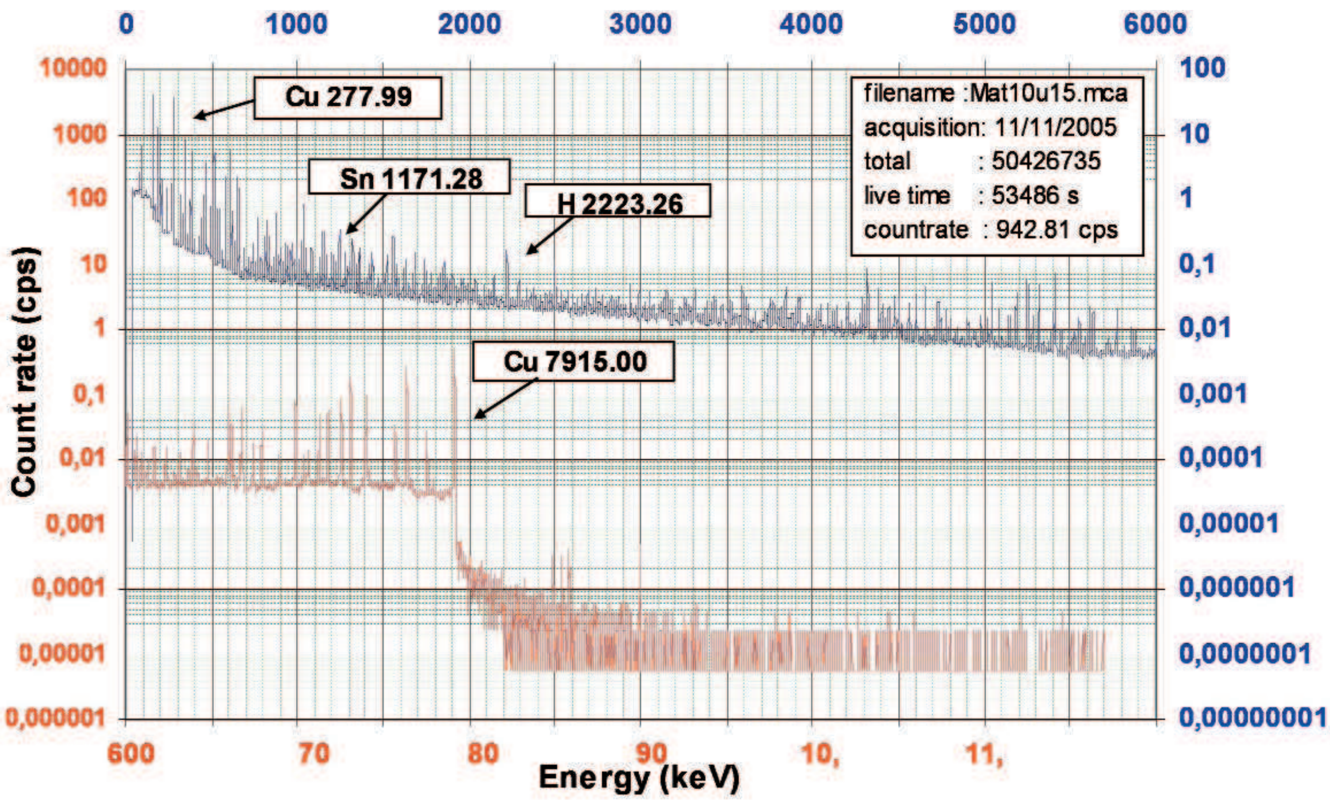


Figure 11. Wall fragment of a washbowl (sample No. 16) fastened with Teflon strings onto the aluminum frame and being placed into the PGAA measurement chamber.

Matelica archaeological site - of which the metallurgical area of provenance was not sure - with the biconical from Fabriano site (sample No. 14).
This contributes to confirm the compositional uniformity of the bronze objects between the two considered areas. The sole sample No. 6 (*cista* found in the Tomb 172 of "Crocifisso" site, Matelica), on the other hand, shows a significant difference in Sn/Cu ratios, revealing that it is almost certainly dissimilar from the other archaeological artefacts. A Principal Component Analysis (PCA) has been performed, finally, by means of "XLSTAT" program software (Addinsoft Inc., USA) [26]. PCA is a widespread multivariate numerical method which operates with linear combinations of the original composition data.
This method is extensively adopted to carry out comparative studies of archaeological artefacts; moreover, in particular, it is able to reveal various important similarities or differences between the analysed objects taking into

account their composition [27]. The chart in Figure 14 shows the results obtained from this method, which has proved to be helpful to progress the comparison between the bronze objects of the Matelica archaeological site with that from Fabriano.
These results confirm the consideration above expressed and concerning the samples No. 6 and No. 14. In this chart, also the sample No. 7 (helmet found in the tomb of Villa Clara site, Matelica) seems to be dissimilar from the others: additional investigations on the manufacturing places of the objects, thus, have



been recommended. The present work, concerning five of the investigated artefacts, has been included in the database of the Ancient Charm Project [28].

Figure 12. PGAA spectrum resulting from the investigation of the sample No. 3 (*patera* wall fragment belonging to a tomb found in Passo Gabella site, Matelica, Italy).

CONCLUSIONS

The application of PGAA has been introduced, in the perspective of future studies at 360°, to facilitate the traditional archaeological research to find answers - also very refined - to the historical-archaeological questions that the traditional sources do not succeed by now to get ahead into focus.
This concept follows the practice of the historical science of archaeology, which always uses the support of other disciplines to elaborate and to decipher correctly the dissimilar data normally collected in a stratigraphic excavation. A comparison has been carried out of Aenean objects from

Matelica and Fabriano sites, dating back to the 7th century B.C. Most of the major components and some interesting trace elements of the bulk material have been determined, together with Sn/Cu mass ratios and a PCA analysis, providing useful information for the study of the provenance.

Sample No.	Sample name	Archaeological site	Typological information
1	MAT PG 1 AC	Tomb in Passo Gabella site, Matelica	situla (wall inferior edge fragments)
2	MAT PG 2 AC	" " "	situla (upper wall fragment)
3	MAT PG 10 AC	" " "	patera (wall fragments) rep. 25
4	MAT PG 15 AC	" " "	little situla (wall fragments) rep. 18
5	MAT PG 18 AC	" " "	cista handle, rep. 14 (powder, m = 0.1198g)
6	MAT CR 28 AC	Tomb 172 in Crocifisso site, Matelica	cista (wall fragments)
7	MAT VC 30 AC	Tomb in Villa Clara site, Matelica	helmet (central element fragment) inv. 63899
8	MAT VC 31 AC	" " "	washbowl (wall fragment) inv. 63900
9	MAT VC 32 AC	" " "	lance prong coil (fragment)
10	MAT VC 34 AC	" " "	ring (fragment)
11	MAT CR 44 AC	Tomb in Crocifisso new site, Matelica	situla (wall fragment)
12	MAT CI 48 AC	Tomb 39 in Cimitero site, Matelica	helmet (fragment)
13	MAT CI 52 AC	" " "	tripode (wall fragments)
14	FAB SMC 64 AC	Tomb 3 in Santa Maria in Campo site, Fabriano	biconical (wall fragment)
15	MAT CR 80 AC	Tomb182 in Crocifisso site, Matelica	helmet (cap fragment) rep. 88
16	MAT CR 81 AC	" " "	washbowl (wall fragment) rep. 39
17	MAT CR 82 AC	" " "	situla (wall fragment) rep. 40
18	MAT CR 83 AC	" " "	cista (wall fragments) rep. 41

Table 1. Archaeological description of the investigated objects.

The absence of diversity, i.e. the compositional uniformity revealed between the artefacts discovered in the Matelica and Fabriano sites, in particular, has provided the archaeological community with a further argument to consider Matelica area as a possible manufacturing metallurgical centre independent

No.	Inventory No.	H	Si	Cl	Fe	Co	Cu	Zn	As	Ag	Sn	Sb
1	MAT PG 1 AC	<0.03	<1	0,02	<0.4	0,016	90,7	0,83	0,21	0,073	7,2	0,93
2	MAT PG 2 AC	<0.03	<1	0,01	<0.4	0,011	92,3	0,89	0,24	0,119	5,6	0,81
3	MAT PG 10 AC	0,121	<1	0,04	<0.4	<0.006	92,2	<0.8	0,61	0,128	6,9	<0.4
4	MAT PG 15 AC	0,065	<1	0,01	<0.4	0,021	88,3	<0.8	<0.2	0,085	11,5	<0.4
5	MAT PG 18 AC	<0.03	<1	0,02	<0.4	0,059	91,5	<0.8	<0.2	0,122	8,3	<0.4
6	MAT CR 28 AC	1,265	<1	0,04	0,50	0,010	74,2	<0.8	0,54	0,275	22,3	0,88
7	MAT VC 30 AC	<0.03	<1	0,02	<0.4	<0.006	84,5	<0.8	3,32	0,064	11,6	<0.4
8	MAT VC 31 AC	0,076	<1	0,02	<0.4	<0.006	87,2	<0.8	<0.2	0,092	12,7	<0.4
9	MAT VC 32 AC	0,038	<1	0,03	<0.4	0,024	90,8	<0.8	<0.2	0,052	9,1	<0.4
10	MAT VC 34 AC	0,172	<1	0,01	<0.4	0,006	83,6	<0.8	<0.2	0,059	16,2	<0.4
11	MAT CR 44 AC	<0.03	<1	0,01	<0.4	<0.006	90,1	<0.8	0,45	0,130	9,3	<0.4
12	MAT CI 48 AC	0,018	<1	0,03	<0.4	0,012	89,5	<0.8	0,23	0,100	9,7	0,40
13	MAT CI 52 AC	0,273	2,8	0,09	<0.4	0,018	82,0	<0.8	<0.2	0,094	13,8	0,41
14	FAB SMC 64 AC	0,680	1,9	1,01	0,42	0,012	83,4	<0.8	<0.2	0,072	12,9	<0.4
15	MAT CR 80 AC	0,246	1,5	0,02	<0.4	<0.006	85,6	<0.8	<0.2	0,052	11,9	0,67
16	MAT CR 81 AC	0,280	2,2	0,10	0,45	0,007	86,1	<0.8	0,86	0,160	8,7	1,12
17	MAT CR 82 AC	1,326	<1	0,05	0,67	0,009	88,6	<0.8	<0.2	0,171	8,4	0,79
18	MAT CR 83 AC	0,659	3,4	0,09	0,63	0,007	80,6	<0.8	<0.2	0,00	14,1	0,51

Table 2. Results of PGAA measurements.

from that of Etrurian (Tyrrhenian) region [29]. A significant difference between some objects has been outlined, on the other hand, which could represent a substantial indication for future discussions from the archaeological point of view. Naturally, the still slight survey at present available of data, deduced with this type of refined and innovative method of objective and non-destructive investigation, presents gaps and inevitable large margin of enhancement.
With this perspective, an increasing number of archaeological objects are being investigated by PGAA, helping also to improve this technique and to make it suitable for industrial applications too - see, e.g., ref. [30].
The progress of research and the formation of a rich and more reliable database will allow to the researchers to gather very interesting and original

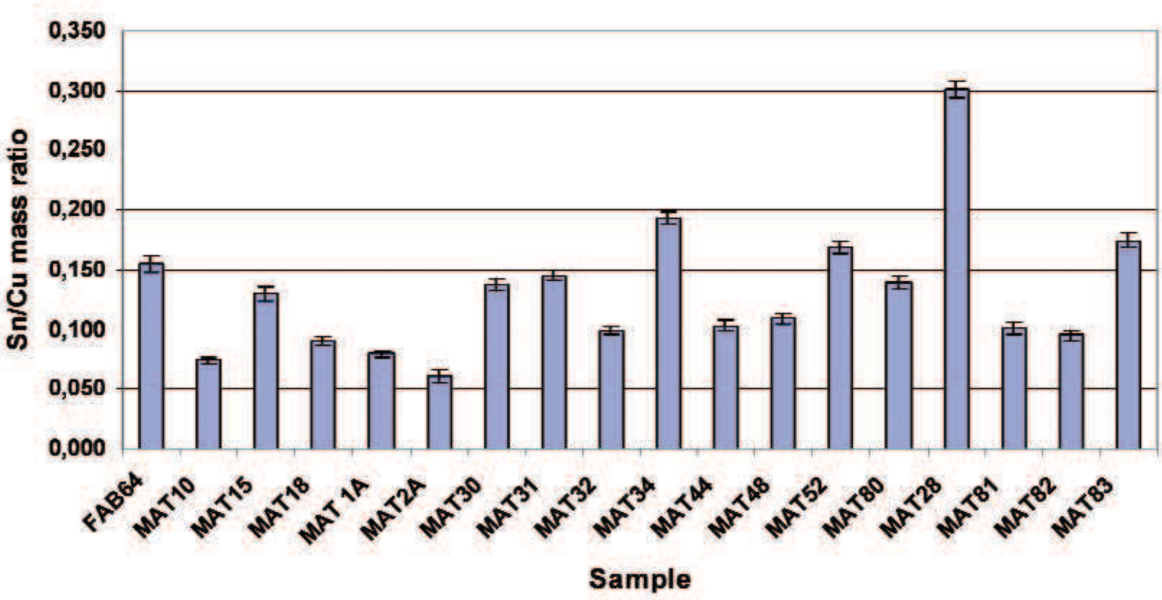


Figure 13. Sn/Cu mass ratio of the investigated bronze artefacts.

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features (in this case, concerning the metallurgical reality of the past), with potential inestimable scientific effects. "We are now in the forerunner phase, with its responsibilities and honours" [17].

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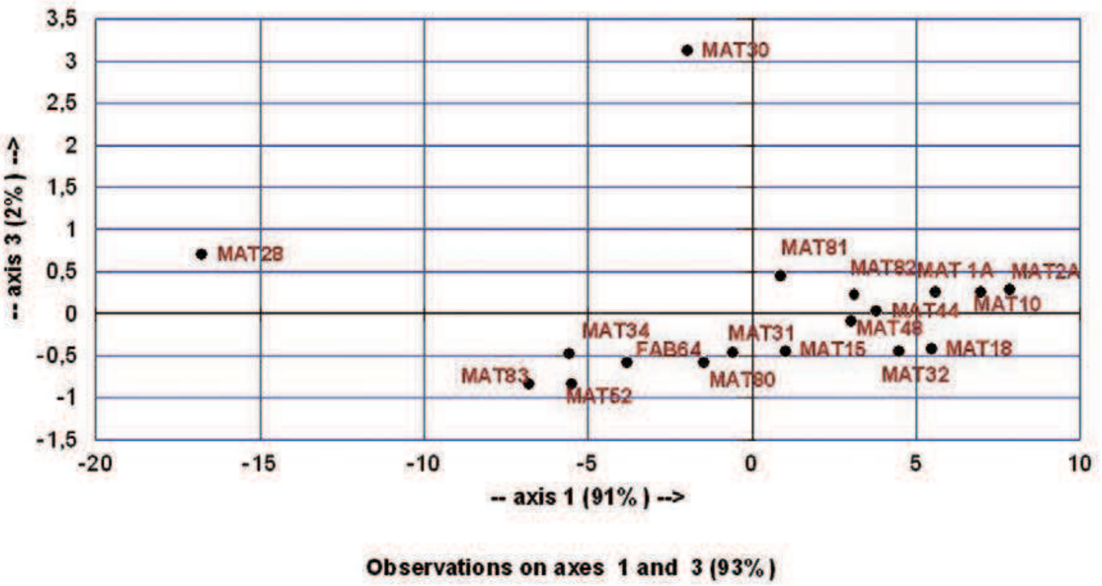

Figure 14. Principal Component Analysis of Picenum necropolis bronze objects.

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SUMMARY

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