

Micro-PIXE studies on Visigothic ‘Pietroasa’ gold hoard

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Abstract

Several fragments of ancient gold objects belonging to Pietroasa ‘*Cloșca cu Puii de Aur*’ (*The Golden Brood Hen with Its Chickens*) Romanian hoard were analyzed using the micro-PIXE (Particle Induced X-ray Emission) technique. The purpose of the study was to gain some more knowledge regarding the metal provenance by determining the presence of PGE (Platinum Group Elements), high-temperature melting point trace elements (Ta, Nb), as well as traces of Sn and Te - at a micrometric scale. Several Ta inclusions (micrometric areas of composition different from the surroundings) on three samples and Ir inclusions on other samples were found. Sn, an important fingerprint for ancient South – European gold, was detected in two samples at trace level. No Te - the

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main fingerprint for Transylvanian gold - traces were found. The measurements led to some conclusions for the possible gold ore sources of Pietroasa treasury: the South-Ural Mountains, Nubia (Sudan) and/or Anatolian deposits and Roman imperial coins, but certainly not Transylvania. A remelting procedure for the gold used to manufacture these artifacts is very probable and must be also considered.

Keywords: micro-PIXE, gold, PGE, inclusions, provenance, archaeometry

Introduction

The hoard of Pietroasa, Buzay county, Romania (today the village is named Pietroasele) was discovered in 1837 on the Istrița hill by four villagers. At the time of the discovery, the hoard comprised 22 pieces, some adorned with gems. By the times when the authorities heard about the hoard finding and decided to confiscate it, 10 pieces had already disappeared, having been most probably melted shortly after their discovery. What remained from the hoard today is a tray - cut in four by the villagers that shared the gold between them, a patera with pagan (Germanic) gods representations, an octagonal basket, a dodecagonal basket, an oenochoe cup, some girdles and some fibulae. These remaining pieces of the hoard weigh about 19 kilos.

An evident characteristic of the hoard is its stylistic diversity, indicating different provenances (workshops). The patera and the oenochoe vessel were crafted in classical Hellenistic style. The other pieces are characterized by the insertion of gems into the body of the objects, a setting technique characteristic to the peoples of the Pontic steppe (Oprescu 1968). Fibulae were used in the antiquity as a brooch or safety-pin for

fastening garments, as a substitute for buttons. Usual fibulae had a bow-shaped head and a pin. Some fibulae also had pendants. Oenochoe or oinochoe is a deep wine jug, with a graceful aspect, having a trefoil-shaped mouth in order to facilitate the pouring of wine. The name of this jug comes from Greek, being originated in the word *oinos* meaning wine. A runic inscription on one of the girdles is worth to be mentioned: '*Guthâni Ocwi Hailag*', translated as '*to Odin blessed country*' or maybe '*of the Goths sacred propriety am I*'.

The thesaurus is known mostly by the name: '*The Golden Brood Hen with its Chickens*' due to the physical resemblance of the fibulae with some birds (at least the great fibula has the top shaped as an eagle).

The hoard was owned by some Germanic population, being attributed by different authors to the Visigoths or to the Ostrogoths. It was hidden in the place where it was found in 1837 most likely during the 4th century A.D. In (Giurascu 1976) is mentioned the hypothesis according to which the thesaurus belonged to Athanaric, chieftain of the Visigoths, who came in Carpathian region from their original place situated between Caucasus and Don river, in the North-East of Black Sea, pushed by the Asian barbarians. In 376 king Athanaric stopped in a place named Caucaland - possibly the actual zone of the Buzău county - from the face of the Huns' pressure (Ammianus Marcellinus).

A very interesting aspect of the Pietroasa hoard history is its sequestration in the USSR from 1917 until 1956. In September 1916 the Romanian government was moved from Bucharest to Iași subsequently to the collapse of the frontline against German and Austro-Hungarians. The National Bank of Romania was also moved to Iași, with all its deposits. The possibility of Moldavia being occupied by the Central Powers having

become a real one, the part of the Romanian national treasure that guaranteed the banknote circulation - 93.36206 tons of pure gold in ingots and coins - was evacuated to Moscow in December. Given the grave situation on the Mărășești front, the Romanian government was constrained to evacuate in July 1917 to Russia the most precious artistic values, including the Pietroasa hoard, which were deposited, together with the Romanian gold reserve somewhere in the Kremlin complex in Moscow, together with the Russian state treasure. During the Russian civil war all the Kremlin valuable deposits have been evacuated to Urals region, captured by the White Government of admiral Koltchak in the summer of 1918 and recuperated by the Red Army near Irkutsk in Eastern Siberia in the spring of 1920. After 1920, the Soviets refused to return the Romanian treasure, claiming that Romania was led by an *'imperialist'* government, and they would only return it to the working class. During the WW2, the Romanian treasure, including the Pietroasa hoard was again evacuated in September 1941 to Urals region. After WW2, in the conditions of the new communist regime in Romania, a very small part of the Romanian treasure has returned to Bucharest. The twelve pieces of the Pietroasa hoard came back to Romania in 1956 in broken and damaged wooden boxes. The hoard pieces presented scratches, marks of strokes, especially on edges, and deformation aspects on some large areas. Restoration was absolutely necessary.

In these conditions, especially from the edges of the objects, some very small (millimetric) fragments from the pieces have been removed during the process of the mechanical restoration. These fragments have been carefully preserved until now, and from them, the present analyzed samples have been selected.

The restored hoard is now exhibited at the National Museum of Romania's History in Bucharest.

The study of trace-elements in archaeological metallic objects can provide indications about the metal provenance and the involved manufacturing procedures, leading to important conclusions regarding the commercial, cultural and religious exchanges between the antique populations. Ancient metallic materials are usually inhomogeneous on a micrometric scale, the imperfect melting being revealed by the presence of segregated phases in alloys and inclusions (Tylecote 1987).

Inclusions of Platinum Group Elements (PGE) - Ru, Rh, Pd, Os, Ir and Pt - in gold were released into rivers by the decomposition of rocks and occur in placer deposits in the form of grains and nuggets of complex alloys. The melting point of PGE is higher than that of gold; thus, PGE grains remain unchanged during the metallurgical processing of the gold ore. Apart from PGE inclusions, gold alloys can contain low amounts of high-temperature melting point trace elements, such as Ta, Nb, Cr etc, other potential fingerprints for base metal deposits.

The purpose of this Pietroasa hoard study was to obtain relevant information about the metal provenance. Trace elements - such as PGE, correlated with known mines fingerprints, such as Sn, Sb, Te, Hg, can help to identify the ore source. The gold provenance of the objects can lead to further historical conclusions.

A set of nuclear activation analyses - Neutron Activation Analysis (NAA) and Proton Activation Analysis (PAA) - on fragments from artifacts from Pietroasa hoard was performed some years ago, trying to clarify which of the different historical hypotheses regarding the origin of the gold is the correct one (Cojocaru 1999). However, PAA has the disadvantage of sample activation. The disadvantage of NAA consists in the necessity of using a radiochemical preparation in order to detect platinum and iridium - the HCl digestion - implying the destruction of the neutron activated sample. The NAA

and PAA results are relevant for the major components (Au, Ag, Cu) concentrations and also for the Pt content.

In the present work, non-destructive micro-PIXE (Particle Induced X-ray Emission) analysis on fragments from the original objects was used for micro-inclusions and trace elements detection in order to refine the knowledge on this hoard, attempting to validate the stylistic classification of the hoard objects with gold provenance arguments.

Materials and methods

Sample description

Several small pieces from six different objects belonging to the Pietroasa treasury were analyzed: the large, the middle and the small fibulae, the dodecagonal basket, the patera, the central figure representing Cybele, and the oenochoe cup.

The large eagle-headed fibula is decorated with almandine and rocky crystals, and has a total weight of 867 g, a height of 27 cm, and the maximum width 15 cm – see figure 1.



Figure 1 Pietroasa treasury large fibula

The middle fibulae pair is adorned with garnets, almandines, quartz crystals and pearls, and weights 1219 g and it is 25 cm in height.

The small fibula is decorated with garnets, almandines, quartz crystals, pearls, and has a total weight of 1219 g, and is 12.5 cm in height - see figure 2.



Figure 2 - Pietroasa treasury small fibula

Despite of the Roman tradition of using fibulae for chieftains' clothes, the bird motif is typical Germanic and it is found in a lot of Gothic treasures from the Vth until VIIth centuries in France, Italy, and Spain.

The dodecagonal basket is decorated with almandines, tourmalines, garnets, turquoises, and has a total weight of 2433 g and is 11 cm in height – see figure 3.



Figure 3 – Pietroasa treasury dodecagonal basket

The dodecagonal and the octagonal baskets present similarities with a cup decorated with a gemstone with the figure of the Sassanide king Chosroes carved in it. This cup is exhibited at the Cabinet de Medailles de Biblioteque Nationale de Paris and it was a gift of Haroun-al Rashid Caliph to Charlemagne.

Patera weights 2101 g, its diameter is 25.7 cm and its height is 7.5 cm; it appears to be a round sacrificial dish, with carved figures of Gothic gods in Greek dress surrounding a seated three-dimensional fertility goddess (Dea Mother or Gea, also adopted by the Germanic tribes).

The oenochoe cup weights 1740 g and is 36 cm in height. A quite similar cup was discovered in a former Roman settlement from Kertch (on the Black Sea northern coast) – see figure 4.



Figure 4 – Pietroasa treasury oenohoe cup

Micro-PIXE measurements

The micro-PIXE measurements and point analyses were performed at the Nuclear Microprobe Facility of the Institute of Ion Beam Physics and Materials Research, Forschungszentrum Rossendorf (FZR), Germany - see some of the results in (Bugoi 2003) - using the 3 MV Tandatron accelerator (Herrmann 1995), at the Oxford microprobe facility of the Legnaro National Laboratory (LNL), Italy (Boccaccio 1996) - see some partial results in (Constantinescu 2005) - and at the microprobe of the AGLAE accelerator of the C2RMF, Louvre, Paris (Dran 2004).

At Rossendorf, a 3 MeV proton beam was used and the beam current intensity was about 400 pA. A focused beam of $6 \times 6 \mu\text{m}^2$ was rastered on $800 \times 800 \mu\text{m}^2$ areas

(128×128 pixels elemental maps). The X-rays were detected using a Si(Li) detector - FWHM of 190 eV at 5.9 keV - positioned at 120° with respect to the incident beam, and mylar absorbers of different thicknesses were employed to reduce the soft X-ray region of the spectra. The total accumulated charge for the scanned areas was of the order of 3 μC .

At Laboratori Nazionali di Legnaro, a 2 MeV proton beam generated from the AN 2000 Van de Graaff accelerator was used. The beam was focused to $5 \times 5 \mu\text{m}^2$. The maximum beam current was around 1 nA. To reduce the intensity of the peaks in the low spectral region (below 4 keV), a mylar funny filter (171 μm thickness, 3.3% hole) was employed. The maps were scanned on areas of $250 \times 250 \mu\text{m}^2$. The Legnaro Si(Li) detector had a resolution (FWHM) of 170 eV at 5.9 keV.

At AGLAE accelerator of the Centre de Recherche et de Restauration des Musées de France, located in the basement of the Louvre Museum a 3.2 MeV proton beam, extracted into helium atmosphere was used. The micro-beam (roughly 30 μm diameter) was used to scan different size areas on the samples (usually $300 \times 300 \mu\text{m}^2$ in size, but larger areas were scanned as well), while a milli-beam (approximately 100 μm in diameter) was employed in order to make some point measurements using beams of higher current intensity ($I \sim 40 \text{ nA}$). For PIXE signals acquisition, two Si(Li) detectors (low - and high-energy) were used (FWHM $\sim 140 \text{ eV}$ for both detectors). All the measurements were performed using a pinhole filter in front of the low energy PIXE (LE-PIXE) detector. For the high-energy PIXE detector (HE-PIXE) sometimes a 50 μm Al filter was used, while in other measurements a 75 μm Cu filter was chosen, in order to reduce the high contribution of gold L lines and to diminish the sum peaks of Au L lines which interfere with the signals of elements neighboring silver.

In all the cases, the gold samples were put on tape holders. Caution was taken in order to avoid the cracks present in some samples, by making the measurements in areas that were not affected by the sampling or by other previous manipulation. An optical examination of the samples with a microscope was also performed, in order to check if the inclusions in the archaeological samples were visible as grains of color different from the surroundings.

In all the above cases, PIXE data analysis was done using the GUPIX code (Maxwell 1989).

It must be mentioned that using micro-PIXE for this kind of samples, it is practically impossible to detect Pt traces due to the presence of a massive Au signal. This is mainly due to the Si(Li) detector resolution(s) and to the high differences in the expected Pt and Au concentrations (Pt at trace level, and Au major component).

Results and discussions

Alexandru Odobescu, one of the pioneers of Romanian archaeology, who published in the late 19th century a 650-pages comparative work called *Le Tresor de Petrossa* (Paris, 1889-1900), believed that the hoard dates from the 4th century and belonged to Athanaric, leader of the Gothic Tervingi tribe. He also believed that some of the pieces were forged in Byzantine workshops, but the Goths made the more ornamental items, having learned this technique from the Scythians and Sarmatians who had spread the technique across Europe, from Novocherkassk in South-East Russia to Pietroasa in Romania (Odobescu 1889).

Table 1 presents some point concentrations for some of the analyzed samples. The points were chosen in the following way: for the three fibulae, the points were

characterized by high concentrations of Ta, for the dodecagonal basket, the oenohoe cup, the patera and the central figure of the patera, the points featured a relatively high content in PGE (Pd or Ir). For the dodecagonal basket and the small fibula measurements performed at different accelerators (see the legend of the table) are presented.

A first conclusion of these microprobe experiments was that the elemental composition for the seven pieces is strongly different, which is in good agreement with the classification made for the objects taking into account only the stylistic aspects (Odobescu 1889). Thus, the patera and the oenohoe cup are in Hellenistic style, the dodecagonal basket combines Sassanide (Persian) and Hellenistic characteristics while the fibulae are in Germanic style (the bird motif). The microscopic concentrations are rather different from the ones obtained using NAA (Cojocaru 1999). This fact is not surprising at all, since it is not expected an agreement between the point (the present measurements) and the bulk concentrations of an ancient inhomogeneous alloy (Cojocaru 1999). The differences in concentrations of the point measurements performed at different accelerators also confirm the inhomogeneity of gold. The uncertainty of the elemental composition figures in the table is difficult to be estimated due to the different experimental conditions from one experiment to the other. However, one can quote an estimate for the overall uncertainty, as being maximum 1% for concentrations higher than 5%, and gradually increasing up to 15-20% for the lowest reported concentrations. To solve the differences between different laboratories values, more work is necessary in the future. The most important issue for this paper was the order of magnitude for the main components (Au, Ag) and the presence of the trace elements (some of them as inclusions). A detailed discussion of the uncertainties will be

included in another publication dealing with the methodology employed, which is the final stage of preparation.

The principal obstacle that hinders a credible provenance attribution is the generalized procedure of gold remelting in the Mediterranean area in this period.

Combining the NAA (Cojocaru 1999) and the PIXE results from table 1, for the central figure of the patera – Pt determined through NAA and Ir inclusion (Pt cannot be detected from any X-ray spectrum due to the massive presence of Au) found using micro-PIXE –one can only assume that, partially, gold ores from Anatolia - Pactolus river area where Pt and Ir have been detected (Moesta 1986) - were used to manufacture this object. However, in the patera itself (the plate), Pt was not detected (Cojocaru 1999), so, a re-melting procedure is evident; however Ir was found again as a microinclusion in the patera. Due to the high fineness of gold (~95%), it is likely that gold used to create this object has been obtained by melting some existing jewelry and Roman imperial coins of Mediterranean origin, as Romans were mastering very well the gold processing technique.

Analyzing the elemental maps for the three fibulae, inclusions of Ta (see Fig. 5, showing a Ta map for the small fibula) were found. The Ta inclusions have been found in all the three experiments (FZR, LNL, AGLAE), in different areas of the samples. This fact strongly suggests that gold was used for the first time when manufacturing the fibulae. Another finding for these samples is the presence of Nb at trace level (see Fig. 6, presenting a point spectrum on the large fibula). These metals have high melting points, and the Ta and Nb grains did not dissolve during the gold processing. As stated in (Lupei 1971), the Ta and Nb are found in tantalite - columbite type minerals, e.g. in samarskite, mineral which is found in gold ores from the Ural Mountains (Southern region around Samara). It must be outlined that in Eurasia only in the Ural Mountains

Ta and Au can be found together in different minerals. These findings confirm the fact that the Germanic owners of the hoard were coming from the region between Caucasus and Ural Mountains in the second half of the 3rd Century A.D., bringing along their precious jewelry (Ammianus Marcellinus), and that some of the Pietroasa hoard artifacts – i.e. the fibulae – were manufactured using Ural mountains gold. The Ural Mountains hypothesis is also supported by the Pt content in the large and small fibulae – see (Cojocaru 1999) – and the Os (for small fibula) inclusion – see Table 1.

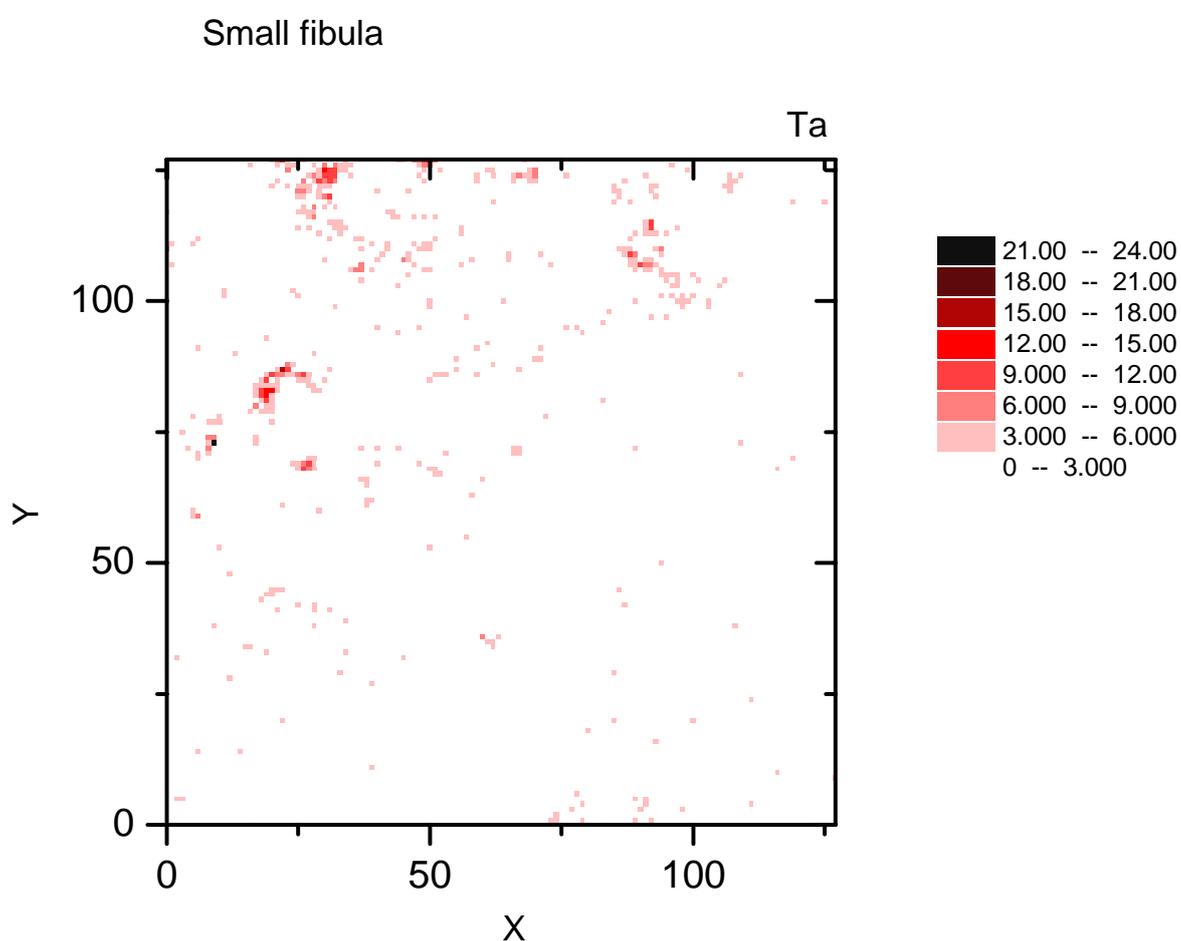


Figure 5 – Ta map on the Pietroasa small fibula sample; scanned area: 800×800 μm^2

(128×128 pixels)

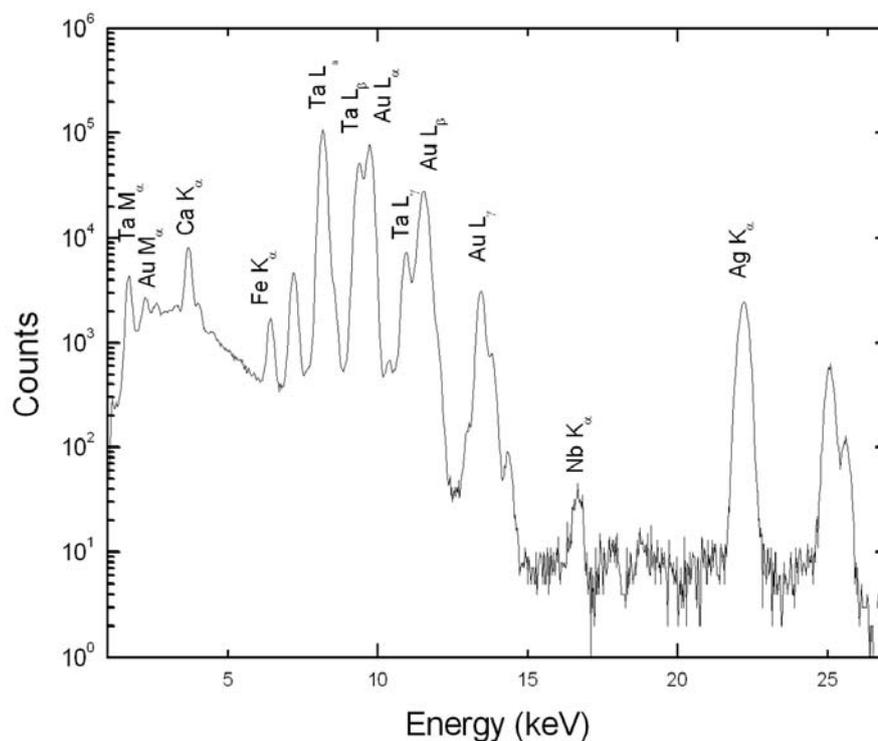


Figure 6 - Point spectrum on the Pietroasa large fibula sample - high content in Ta and Nb at trace level

As concerning the dodecagonal basket, the most important result of these experiments is the finding of Pd traces. The only accessible gold sources with Pd in the IVth Century A.D. were Nubia (Sudan) and Anatolia (Turkey) deposits, intensively used in Egypt (Alexandria) and Syria (Antiochia) workshops - see (Guerra 1999), where Pd was determined in the coins minted by Alexander the Great after the Persian Empire conquest. So, at least one source of gold was most probably in Anatolia, but also the Egyptian (Nubian) origin can be refuted. The high gold content of this sample (Au from 93% up to 99%, depending on the points in which the measurements were performed in the three different laboratories) also suggests a re-melting procedure, probably using

Roman imperial coins struck in Oriental provinces – see (Oberländer-Târnoveanu 1996).

The Sn presence - see (Hartmann 1970, 1982) - detected in the dodecagonal basket and in the oenohoe cup could be an argument for Persian provenance – see (Gondonneau 2000) – but it is necessary to also look for Pt, eventually with an analytical method other than PIXE.

Some fragments from nuggets coming Transylvania mines were also analyzed, to check whether the hypothesis of local - Carpathian Mountains - gold holds. Te (from gold telluride) is known to be an element characteristic for the native gold of Transylvania. None of the archaeological samples contains Te, so the hypothesis of Transylvania's gold provenance can be definitely rejected.

Summarizing the results, one can conclude for the analyzed objects of Pietroasa hoard we have at least three possible geographical gold sources: Southern region of Ural Mountains, Nubia (Sudan) deposits or a Persian source (most likely Pactolus river in Anatolia), a reusable gold source - various emissions of Roman imperial coins, and probably, other not yet identifiable ones used in the remelting procedure. A relevant result is the connection between the stylistic characterization and the geographical gold sources for the Germanic fibulae (Southern Ural Mountains nearby the first Visigoths 'homeland') and for the Sassanide basket (partially used Persian type gold).

Conclusions

The results obtained by micro-PIXE experiments on gold ancient artifacts, especially the inclusions findings, provided some useful hints regarding the possible provenance of fragments of the manufacturing metal. The Pietroasa hoard artifacts were again proved

to be of different origins, confirming the stylistic arguments by the different possible gold sources identified: Southern region of Ural Mountains, Nubia (Sudan) or Pactolus river deposits. The most probable remelting procedure, e.g. using Roman imperial coins must also be considered. Further analyses on other artifacts belonging to the same hoard are to be done. However, a correct answer to the question of the native metal provenance used for each artifact remains a difficult task, taking into account that a complete data bank for the composition of Euro-Asian native gold is not yet available.

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Table 1 – Pietroasa hoard samples point concentrations (see the comments below the table)

Object	Cu	Pd	Ag	Sn	Ta	Os	Ir	Au	Pt (bulk)
Central figure of patera (R)	100 ppm	nd	4.48 %	na	nd	nd	300 ppm	95.14 %	110 ppm
Patera(A)	0.33 %	nd	3.93%	nd	nd	nd	0.13 %	95.20 %	nd
Dodecagonal basket (R)	0.13 %	0.04 %	0.34 %	na	nd	nd	nd	95.49 %	nd
Dodecagonal basket (L)	1.25 %	0.35 %	4.95 %	na	nd	nd	nd	93.35 %	
Dodecagonal basket (A)	0.09 %	31 ppm	0.54 %	37 ppm	nd	nd	nd	99.36 %	
Large fibula (R)	0.13 %	nd	15.35 %	na	4.93 %	nd	nd	78.82 %	110 ppm
Middle fibula (R)	0.05 %	nd	16.85 %	na	0.16 %	nd	0.04 %	82.17 %	180 ppm
.Small fibula (R)	100 ppm	nd	8.73 %	na	39.14 %	0.45 %	nd	49.30 %	80 ppm
Small fibula (L)	0.06 %	nd	30.57 %	na	1.96 %	nd	nd	67.41 %	
Small fibula (A)	0.13 %	nd	34.80 %	nd	11.27 %	0.13 %	0.54 %	51.93 %	
Oenohoe cup (A)	3.21 %	54 ppm	4.47 %	175 ppm	nd	nd	nd	92.29%	na

nd - not detected

na - not analyzed (for Sn, at Rossendorf and Legnaro, the $K\alpha$ region above Ag was not investigated, and the $L\alpha$ are not reliable)

The last column presents some bulk results obtained using NAA and are from (Cojocaru 1999).

Nb traces were detected in the large fibula sample – see the spectrum presented in figure 6.

L – Legnaro result

A – AGLAE result

R – Rossendorf result

