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NUCLEAR ANALYSES OF THE PIETROASA GOLD HOARD

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ABSTRACT. *By means of nuclear analyses the concentrations of Au, Ag, Cu, Ir, Os, Pt, Co and Hg were measured in the 12 artifacts of the gold hoard discovered in 1837 at Pietroasa, Buzãu County in Romania. The concentrations of the first four elements were used to compare different stylistic groups assumed by historians. Comparisons with gold nuggets from the old Dacian territory and gold Roman imperial coins were also made. A good agreement was found with the oldest hypothesis, which considers that the hoard is represented by three styles appropriated mainly by the Goths.*

Introduction

The hoard discovered in 1837 at *Pietroasa*, Buzãu county in Romania by two ignorant peasants entered into the historical literature and the history of art under the name of *the golden brood-hen with its chickens*, and it is considered to be an outstanding set of archaeological pieces, the best known hoard in Romania, due both to its fine artistic quality and to the myths created around it. Out of the 22 pieces discovered initially, the 12 existing today, weighing about 19 kg, can be grouped as (i) *vases*: Tray (1), Patera (2) with a central figure, probably Cybele (3), Octagonal Basket (4), Dodecagonal Basket (5), Oenohoe Cup (6), and (ii) *jewelry*: Large Fibula (7), a pair of Middle Fibulae (8, 9), Small Fibula (10), Pierced Worked Girdle (11), Simple Girdle (12) and Girdle with the Runic Inscription *gutani hilag* (13).

The treasure had a dramatic history (see Ref. 1). The biggest artifacts were crushed and flattened two times (in 1837 and in 1875) in order to be more easily transported and two times they were restored (1867 in Paris and 1884 in Berlin). So, most of the gemstones as well as the accurate and smooth aspect were lost. In 1916 the hoard was evacuated in Russia and it was finally returned only in 1956.

The lack of an archaeological context and the singular character of the hoard have complicated and delayed its chronological integration, ethnical attribution, and establishing the workshop(s) where the artifacts were created. Using traditional methods to study objects of art and archaeology, a chronological period between the last quarter of the 4th century and the first half of the 5th century has been assigned. Thus, the *Pietroasa* hoard has been attributed either to the Visigoths (Athanaric, AD 375) by de Linas (see Ref. 1), Odobescu[2] and Harhoiu[3], to the Ostrogoths (the death of Attila, AD 454) by Horedt, [4] or to the Romans (to the general Gainnas, *c.* AD 400) by Rusu [5]. But one must also mention Burda's hypothesis [6] that the hoard belongs to the period of early Romanian feudalism. Finally, in a recent paper by Oberländer-Târnoveanu et al.[7] it was supposed that Roman imperial gold coins issued between 284 and 324/337 AD were the source of metal used for this treasure.

The *Pietroasa* hoard has been characterized by some historians such as Odobescu[8] and Florescu[9] in three stylistic categories: *(i)* pieces of Graeco-Roman tradition (1 - 3, 6), *(ii)* pieces in Celto-Germanic style (11, 13) and *(iii)* pieces produced at the initial phase of the polychrome Germanic style (4, 5, 7 - 10, 12). Some other historians[10] suggested only two stylistic groups: *(a)* the pieces 1 - 3, 6, 12, 13 and *(b)* the pieces 4, 5, 7 - 11. Finally, "the unity of the processing technique and of ornamental motives" was seen by Burda[6] as a proof of "its manufacturing into a unique autochthonous center place".

The present work was performed with the purpose of throwing some light over these numerous assumptions, in order to see which of them could be confirmed or refuted by major and trace element analysis, and also to look for some connections between the metal used for artifacts and gold both from Dacian gold mines and from Roman imperial gold coins. For this purpose, one compares the concentration of the elements analyzed with the three styles and also the artifacts with gold nuggets collected from various places over the Romanian area.

Experimental

Neutron activation analysis (NAA) was the main experimental method used in this work, but also proton activation analysis (PAA), energy dispersive X-ray fluorescence (XRF) and the streak method (SM) were also used in order to obtain additional information and confirmation.

For NAA and PAA, at least one sample was cut off with a special steel knife from every artifact of the hoard. This was done usually from a normal angle edge by two cuts resulting in a sample of an approximately bi-tetrahedral shape and a weight varying between 0.2 and 17 mg. The rear side of the artifact was usually preferred, at a place, which seemed to be original.

Neutron activation analysis

Each sample was carefully washed in pure alcohol, weighed and wrapped in aluminium foil. The samples processed in this way were irradiated, together with flux standards, for 31 hours in a thermal neutron flux of $7.2 \cdot 10^{12}$ n·cm $^{-2}$ ·s $^{-1}$, Φ_{th}/Φ_{epi} = 30.5, at the VVR-S reactor of IPNE-Mãgurele. The samples were cooled for 30 days after irradiation to achieve a convenient ^{198}Au ($T_{1/2}$ = 2.695 d) activity. The sample was then counted using a gamma spectrometer with an approximately 100 cm^3 Ge(Li) detector and a 4096 channel analyzer. The resolution of the detection system was 1.9 keV for the 1.332 MeV gamma-energy. Generally, instrumental neutron activation analysis (INAA) method was used but sometimes (for iridium and platinum), in order to increase the detection sensitivity, radiochemical neutron activation analysis (RNAA) was preferred.

In RNAA method the solution of the digested sample was evaporated to dryness and taken up with 3 ml of 3 M HCl. The resulting solution was extracted with 10 ml ethyl acetate. The organic phase was discarded. The extraction step was repeated once with 10 ml ethyl acetate. Preliminary experiments showed that 90.6 % of iridium was going in the aqueous phase together with 87.0 % Pt and 88.3 % Os, but only 1.6 % Au. Finally, if necessary, the aqueous phase was evaporated to the volume of about 1 mL and absorbed as an 8 M HCl solution (10 mL) on an anion exchange resin column (Dowex 2 x 10)[11, 12] Iridium was practically unretained by the column, 96 % from it being found in the 8 M HCl and the following 10 mL 6 M HCl eluents. These eluents are mixed together and counted with a γ-spectrometer. If the whole procedure is followed the total recovery for iridium is 87 % and only 0.1 % for gold. Some silver (about 13 %) is also going in the final solution and it reduces a little the detection sensitivity of iridium.

The element contents of the sample were usually determined by means of the monostandard technique.[13] This method is quite reliable, demands less effort in execution, and has fewer error sources than the relative method.[14] The relative method was used for Cu, Ir and Pt determination. The samples were usually flattened in order to diminish the self-shielding effects of neutron flux and decaying gamma-rays so that estimated corrections[15] are lower than 5 %.

In order to include analyses for short-lived isotopes, especially copper, each sample underwent a short irradiation at the same reactor (Φ_{th} = $2 \cdot 10^{12}$ n·cm $^{-2}$·s $^{-1}$) using the rabbit transport system. The isotope ^{66}Cu ($T_{1/2}$ = 5.14 m, E_γ = 1.039 MeV) was counted. In this method each sample (*s*) was irradiated together with a standard of metallic copper (*c*) using the program: t_{irr} = 1 min, t_{cool} = 3 min, t^s_{count} = 500 s live; 2) t_{cool} = 12 min, t^c_{count} = 500 s live. It should be noted that even after such a short irradiation with the rabbit system the gold activity is too high to measure the sample directly. Thus a filter of lead was interposed between the detector and sample. The 2.5 cm thick filter provided an attenuation of 412 keV gamma-line (^{198}Au) by a factor of about 300 but only a reduction of approximately 6 times for the 1039 keV gamma line of ^{66}Cu.

Proton activation analysis

A proton beam of 11 MeV drawn out in air and collimated at a diameter of 1 cm was used. At this energy, excepting Li and B, only the (p,n) channel is open.[16] Flattened gold samples together with standards were fastened to the surface of a metallic drum which was rotated in the front of the beam. The axis of the beam was perpendicular to the drum surface. A proton current of 0.5 μA and an irradiation time of 6 hours were used. Standards were produced by melting the known amounts of a homogeneous mixture of gold and platinum metallic powders in the proportion of 1000:1 respectively, and rolled at the same thickness as the samples. The assumption that platinum is homogeneously distributed both in the analyzed samples and standards was made. One can hope that by this method, besides the three main components: gold - ^{197}Au $\Rightarrow$ ^{197m}Hg ($T_{1/2}$ = 24 h)/ ^{197}Hg ($T_{1/2}$ = 65 h), silver - ^{107}Ag $\Rightarrow$ ^{107}Cd ($T_{1/2}$ = 6.5 h) and ^{109}Ag $\Rightarrow$ ^{109}Cd ($T_{1/2}$ = 435 d) and copper - ^{63}Cu $\Rightarrow$ ^{63}Zn ($T_{1/2}$ = 38.4 m) and ^{65}Cu $\Rightarrow$ ^{65}Zn ($T_{1/2}$ = 244 d), one could also quantify: tin - by ^{120}Sb ($T_{1/2}$ =

5.8 d), ^{122}Sb ($T_{1/2}$ = 2.72 d), ^{124}Sb ($T_{1/2}$ = 60.2 d) and tellurium - by ^{126}I ($T_{1/2}$ = 13 d), ^{130}I ($T_{1/2}$ = 12.3 h). The three tin isotopes were clearly seen in silver coins.[17] The interest in detecting Sn and Te consists in the fact that these elements, together with Ag, Cu and platinum group elements (PGE), seem the most promising for determining the provenance of gold.[18]

From the six natural isotopes of platinum only ^{194}Pt gives a high yield in the Pt(p,n)Au reaction and it is not produced by any other 11 MeV proton reaction, resulting in ^{194}Au ($T_{1/2}$=39.5 h). Calculations show that the gamma-spectrum of proton-irradiated gold (mainly due to ^{197m}Hg) can be drastically reduced by using a proper filter. A cadmium foil of 2 mm thickness was used in this case in gamma-spectrometry. The only other two major components, Ag and Cu, do not give high contributions to the gamma-ray spectrum. In this way one can hope to see some potentially existing products of PGE, especially Ru and Pt but also Pd and Os. The other two, Ru and Ir, seem rather insensitive.[19] However, none of them were detected in the present work except platinum.

X-ray fluorescence and streak method

Although through XRF and the streak method (SM) only the surface composition can be determined and in spite of the fact that only Au, Ag and Cu could be seen, these methods were used especially for studies of homogeneity, for preliminary examination of the artifacts and for checking on the possible presence in the objects of such elements as Cd, Pb and Bi which are practically insensitive to NAA. Many measured points of the original parts of every artifact, including the region near to NAA sampling, showed that they are practically homogeneous as far as the Ag/Au and Cu/Au ratios show. More details of these methods are given in Reference 20.

Natural gold

In order to check the hypothesis regarding to the indigenous provenance of the hoard[6] 15 nugget samples from Romanian territory were analyzed.[21] Three of them came from various placers and the others belonged to primary deposits of the Romanian Western Mountains from ancient and newer gold-mines. The analytical

methods used were the same as for *Pietroasa* hoard samples with the following exceptions: *(i)* Determination of the platinum concentration in nugget samples was carried out by preirradiation separation of platinum from its matrix.[22] *(ii)* By INAA only a trace of iridium was seen in the sample from Valea Pianului. Excepting that one, the iridium concentrations were generally less than 0.4 $mg \cdot kg^{-1}$.

In order to obtain a higher sensitivity for iridium two nugget samples (Valea Arieşului and Ruda gold-mine) were analyzed by RNAA. The results of the natural gold analyses, which are important for the present comparison, are given in Table 1.

Table 1. Element concentration in gold nuggets[21]

Deposit	Source	Au (%)	Ag (%)	Cu ($mg.kg^{-1}$)	Ir ($mg.kg^{-1}$)	Pt ($mg.kg^{-1}$)	Hg ($mg.kg^{-1}$)
Placer	Valea Oltului	92.4	7.54	≤230	≤0.4	460	834
	Valea Ariesului	94.2	5.43	446	≤0.01	nd	1091
	Valea Pianului	89.5	10.28	330	0.64	248	673
Primary	Rosia Montana	73.7	22.63	-	≤0.4	222	70
	Bucium-Izbita	84.0	15.96	188	≤0.4	-	337
	Baia de Aries	88.3	11.67	≤150	≤0.3	-	190
	Zlatna	78.3	21.62	≤160	≤0.4	-	347
	Ruda-Brad	84.3	15.60	186	0.044	-	491
	Valea Morii	74.7	25.19	409	≤0.4	-	546
	Musariu-Brad	77.6	22.30	≤220	≤0.4	-	-
	Musariu-Brad	83.5	16.40	≤150	≤0.4	-	-
	Valea Morii	90.4	9.55	396	≤0.4	-	-
	Runculet-Straja	86.6	13.35	242	≤0.4	-	-
	Rosia Montana	93.3	6.63	282	0.41	-	212
	Bradisor-Brad	83.1	16.82	≤200	≤0.4	-	-

nd = not detected
- = not measured.

It must be noticed that INAA does not permit to measure a copper concentration in a gold matrix under 150 - 200 $mg \cdot kg^{-1}$. But this method, especially RNAA, remains the most sensitive method to quantify iridium. The experimental detection limits, taken as three standard deviations, for this element is about 0.4 $mg.kg^{-1}$ and 0.01 $mg \cdot kg^{-1}$ for INAA and RNAA, respectively, in comparison with 7.5 $mg \cdot kg^{-1}$ for PAA method.[19] For the other trace elements: Co, Os and Hg the INAA experimental detection limits are around 0.1, 1 and 4 $mg.kg^{-1}$. For platinum in our conditions the PAA experimental detection limit was about 10 $mg \cdot kg^{-1}$, but concentrations as low as 2 $mg \cdot kg^{-1}$ have been reported [7] by this method.

Results and discussion

The provenance is quite an important problem. It is probably true that gold was sometimes only melted and, in this case, Ag, PGE, Sn, Te and Cu seem the most promising elements for provenance determination.[18] Some authors[23] tried to correlate ancient gold objects with their associated gold sources on the basis of the PGE inclusion composition, but the question is disputable as was made clear by Ogden[24] and Meeks.[25] They have shown that the inclusions in a single object exhibit too wide a range of composition to be a basis for characterizing the source of gold. The only firm conclusion, which can be drawn from the presence of PGE inclusions in gold, is that the metal originated from placer deposits. But on the basis of alluvial nugget analyses (Table 1) one can probably draw the conclusion that the reciprocal statement is not valid (no inclusions were found in Romanian gold nuggets of placer origin).

But in any case, even the high refinement techniques for gold, such as amalgamation or cupellation, known in ancient times, allowed the noble metals, and only them, to remain unaffected, offering the best approach for provenance studies. The use of PGE solid solution for provenance studies would seem questionable as long as PGE inclusions and solid solutions coexist in the same object. In fact PGE inclusions seem to represent a phenomenon, which is not frequent. Indeed Meeks and Tite[25] found only an average density of at most eight inclusions per gram of gold in very thin (200 μm) ancient gold strips. If one relies on this figure, the probability of finding an inclusion in a small sample (≈ 10 mg) will be smaller than 0.2%, which is practically negligible. Nevertheless, two inclusions have been found in our samples (no. 4 and 12') with about 74 and 34 μm diameter, respectively, and an approximate composition of 73% Ir + 27% Os (osmiridium). They are visible through a magnifying glass. This was the reason why samples 4 and 12' were left outside the cluster from Fig. 1.

The results are given in Table 2 and shown in the plot of Fig. 1. Excepting the samples 4 and 12', the iridium concentration in the bulk gold was measured. These concentrations were found, within the limit of error of about 10 %, to be practically homogeneous inside the same object. The sample pairs 2 and 3 and also 10 and 10' in Fig. 1 illustrate this. But the homogeneity of objects for major components was

checked in more detail by means of XRF and SM by measuring 3 - 6 points from every object spread over the whole artifact.

Table 2. Concentration (in $mg \cdot kg^{-1}$)of elements in the *Pietroasa* hoard

Obiect	No	Inv.No.	Au %	Ag %	Cu %	Ir	Os	Pt	Co	Hg
Tray	1	11426	97.9(3)	2.03(2)	0.27(3)	0.9(2)	nd	70(4)	2.0(2)	24(2)
Patera	2	11427	99.0(5)	0.85(4)	0.15(2)	1.0(2)	nd	nd	1.1(1)	34(3)
Central figure of Patera	3	11427	98.8(3)	0.65(5)	0.52(6)	0.8(2)	nd	110(5)	5.0(3)	25(2)
Octagonal basket	4	11428	93.1(7)	5.94(8)	0.97(5)	209(12)*	66(8)	200(8)	2.2(2)	nd
Dodecagonal basket	5	11429	88.7(3)	10.22(6)	1.11(5)	19(1)	nd	-	3.0(2)	nd
Oenohoe cup	6	11430	85.6(8)	9.48(6)	4.93(8)	5.0(5)	nd	-	9.4(5)	76(5)
Large fibula	7	11431	93.5(5)	5.07(5)	1.39(4)	10(1)	nd	110(8)	3.9(3)	nd
Middle fibula	8	11432	96.2(5)	2.86(3)	0.89(7)	36(2)	7(2)	180(6)	10.0(8)	65(5)
Middle fibula	9	11433	90.9(3)	8.02(9)	1.06(6)	9(1)	nd	-	6.0(8)	63(4)
Small fibula	10	11434	92.7(4)	5.34(4)	2.03(9)	1.9(2)	nd	80(4)	8.3(5)	55(4)
Pierced Worked Girdle	11	11435	78.4(3)	11.38(5)	10.3(2)	0.8(2)	3(1)	90(5)	-	180(9)
Simple girdle	12	11436	82.1(2)	14.47(3)	3.44(16)	8(1)	5(1)	-	7.8(5)	nd
Girdle with runic inscription	13	11437	95.1(3)	4.14(5)	0.77(6)	1.5(2)	nd	80(4)	7.1(4)	27(3)

nd = non detected
- = not measured
• = inclusion+solid solution.

The Cu/Au *v*. Ag/Au clustering (Fig. 1b) is connected with the possibility that the formula of the alloy might differ from one workshop to the other.

Table 3. Comparision of the average concentrations in the *Pietroasa* hoard and gold Roman imperial coins

Object	Method	Au %	Ag %	Cu %	<Pt $mg.kg^{-1}$
Gold Roman imperial coins (284-324)*	PIXE [7]	98.4(1.4)	1.07(1.02)	0.31(0.91)	
	PAA[7]	98.8(1.1)	1.11(1.00)	0.15(0.10)	40(38)
Pietroasa	NAA	91.7(6.2)	6.19(4.11)	2.13(2.68)	115(45)

The standard deviation is given in the brackets.
*Without the coin no. 29 from the Table (Ref.7 p.149) which seems to have a rather strange composition.

In Table 3 a comparison is given between the average concentrations of elements measured in Reference 7 in gold Roman imperial coins issued between 284 and 324 AD and the average values in the *Pietroasa* treasure. In the bracket the standard deviation is given. It is clear that the figures differ drastically.

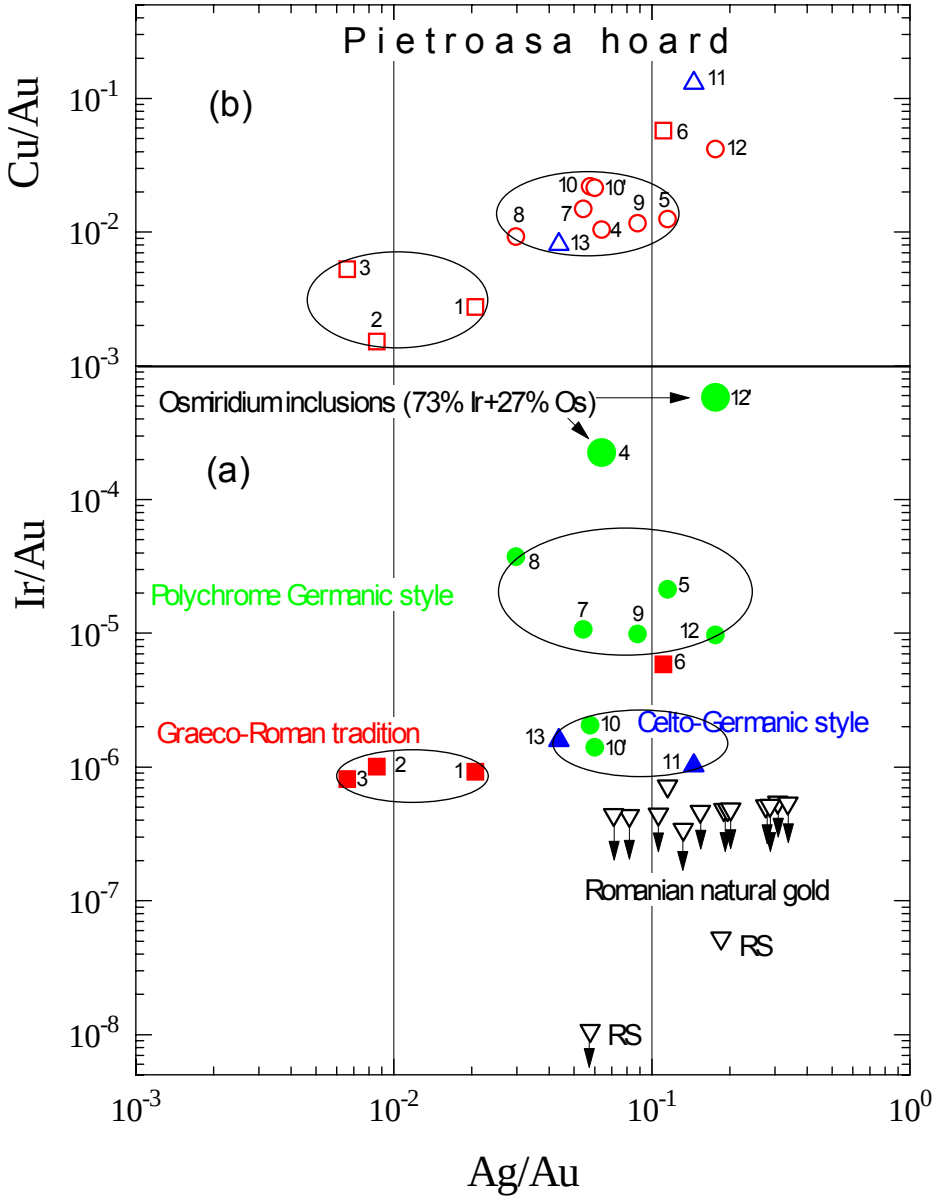

Fig. 1. Ir = f(Ag) (plot **a**) and Cu = f(Ag) (plot **b**) relative to gold concentration for the artifacts belonging to the ***Pietroasa*** hoard. Red squares indicate samples from the Graeco-Roman tradition, blue triangles - the Celto-Germanic style, green circles - the Polychrome Germanic style. The large green circles are for samples, which contain PGE inclusions. Inverted triangles show Romanian natural gold samples. The downward arrow shows that only the upper limit of the concentration was found.

Conclusions

The most important conclusions of this work are:

1. Generally, the three styles are clustered from the point of view of Ir/Au, Cu/Au and Ag/Au concentrations. There are a few exceptions as follows:

(a) The Oenohoe Cup (6) differs drastically from the compositional point of view from the Graeco-Roman style pieces among which historians placed it. One can suppose that it was manufactured in another workshop.

(b) The Small Fibula (10 and 10') has compositionally, in accordance with Fig. 1a, more similarity with the Celto-Germanic style than those from the Polychrome Germanic style. This conclusion is not valid for the plot Cu/Au = f(Ag/Au) where it stays in the expected cluster. But even if the Small Fibula could have been framed compositionally in the Polychrome Germanic style it is obvious that the manufacturing technique is quite different in comparison with the other objects of the hoard, which belong to the respective style.

(c) The two artifacts belonging to Celto-Germanic style, namely the Pierced Worked Girdle (11) and the Girdle with Runic Inscription (13) are rather distinct in the plot Cu/Au = f(Ag/Au), their copper concentration differing by more than an order of magnitude.

2. It can be noted that the plot from Fig. 1 does not confirm the division into two stylistic groups.

3. At least from the iridium concentration data, the assumption of a Dacian provenance of the hoard raw material is highly improbable.

4. From the results on natural gold samples with respect to copper it is improbable that such a high Cu concentration as in the *Pietroasa* artifacts can be found anywhere in nuggets. Therefore it is more normal to suppose that gold was deliberately alloyed with copper. But this does not change the conclusions on the three clusters.

5. The presence of mercury and the failure to detect lead in the artifacts could show that amalgamation was used, at least partially, in the extraction of gold from the ores. It is interesting to notice that mercury is missing only in four artifacts, all from the Polychrome Germanic style.

6. The hypothesis that Gea (Cybele) was posteriorly added to the Patera cannot be confirmed from the present experimental data.

7. The Gainnas hypothesis is not confirmed by the present measurements since the pieces are not uniform compositionally. Romans were able to refine gold up to a level of 99.9 % and to strictly control its fineness. [26]

8. The hypothesis that Roman imperial gold coins were used for manufacturing *Pietroasa* artifacts is not in accordance with the elemental concentrations found and displayed in Table 3. Even if copper would be added, alloying with silver is less probable and is impossible for platinum.

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