

# Study of metal objects of Lombardian origin using X-Ray Fluorescence analysis

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# 1.- Introduction

## 1.1.- Preface and aims

The *Museo dell'Alto Medioevo* of Rome opened its gates in 1967 as part of the complex of the *Museo de la Cività*, becoming since then home to remarkable pieces from the central part of Italy, from the time period comprised between the fall of the Western Roman Empire in 476 AD and around 1000 AD. Its main collection comprises an impressive amount of metal pieces, ceramics and stone decorations from late antiquity Rome to Carolingian era, and even a treasure from the Copts. The most impressive of all these treasures might be the Room of the *opus sectile* of Porta Marina from Ostia, made with coloured marble pieces embedded into the floor and the walls, creating geometric, vegetal and animal decorative motifs.

Amongst all the objects of the collection, those from the necropolis of Nocera Umbra and Castel Trosino are specially relevant because of their high artistic value, many of them made in precious metals like gold and silver. These two are some of the largest early medieval necropolises in central Italy from Lombardian origin. Lombardian domination in Italy begins in the VI Century, around the year 568 AD, and ends in the year 774 AD with the fall of the Lombardian Kingdom by the hand of the Franks, led by Charlemagne. Ever since their discovery at the start of the 20th century, there have been many studies about these objects and the necropolises themselves, specially from the '90s up to today.

The main aim of this project is the start of a new approach in the study of these pieces using X-Ray Fluorescence. This technique is non-invasive, allowing the study of metals from both qualitative and quantitative bases. In this way, these analyses

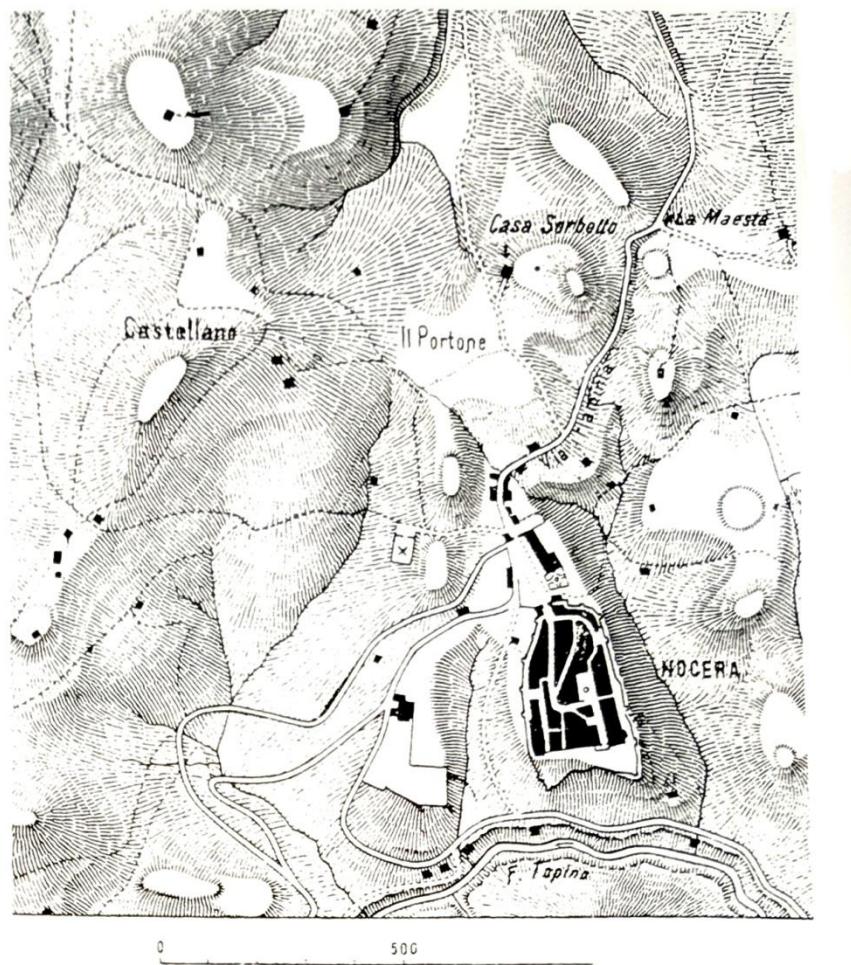
will be useful to determine the proportion between metals in alloys or to detect minor elements used in the alloys as fluxes or found as impurities. The knowledge of these minor differences in proportions of separate elements will be in turn necessary to determine with greater precision the origin of the analyzed object, aiding thus in a better comprehension of putative geographical movements and/or trading of ornamental objects in the Lombardian-controlled territory. Furthermore, another aim, which has been concomitantly carried out, has been the study of the environmental conditions in which the objects are exposed in the museum since some objects, specially the iron ones, show signs of corrosion even after having received treatment previously. This second study can thus aid for a better application of conservative methods on these pieces into museums.

## 1.2.- Description of the archeological sites

### 1.2.1.- Necropolis of Nocera Umbra

The actual town of Nocera Umbra is believed to correspond to the *Nocera* or *Nocera Camellaria*, as found in some historic documents<sup>[1]</sup>. The necropolis was used by a community of Lombards from the 6th Century AD (year 568 is the year in which the immigration of Lombards to Italy was recorded by Paolo Diacono) to the 7th Century approximately. The cemetery stopped being used around the year 630 for reasons that are still unclear. It seems that the local Lombardian community could not have been established there before the fall of Pavia under the Lombardians, so the necropolis can't be earlier from 572 and it's estimated that it was used until 610-630. A coin with the symbol of Giustinio II (565-578), found in grave 87<sup>[2]</sup>, strengthens this theory.

The necropolis of Nocera Umbra presents many elements typical of Lombardian burials, from the typology of the tombs to the funerary offerings. Tombs consist in all cases of a burial pit dug on the ground with a rectangular shape containing wooden elements. Swords and weapons were commonly present in male burials, and clothing accessories and weaving tools in female ones. The number of tombs without the funerary kits is below 5% of the total.



**Illustration 1. Old map of the municipality of Nocera Umbra and the location of the medieval necropolis near the area of Il Portone. Extracted from "Umbria Longobarda: La necropoli di Nocera Umbra nel centenario della scoperta". [1997]**

Overall, it's assumed from the study of the necropolises by A. Pasqui that the Lombard settlement of Nocera Umbra had a purely military character. That of Castel Trosino, on the other hand, must have consisted of a small group of families occupying the territory and probably had an administrative role and contact with the neighbouring Romano-Byzantine areas.

#### 1.2.1.1.- The excavation

Discovered in 1897 by the Blasi family in the location of their manor *Il Portone*. The necropolis is found in the middle of the Apennines, around 40 kilometers north-east from Spoleto, at an altitude of 548 meters over the Topino, an affluent river of the Tiber. The terrain consists mostly of karstic rock of tectonic nature, not very fertile and with scarce precipitation. These relatively harsh conditions suggest that the location was not chosen for its natural resources but rather its strategic placement<sup>[3]</sup>.

The necropolis is found in a plain valley between mountains known as "*Il Portone*" at 660m from the modern location of Nocera Umbra, and is not visible from town. This led several scholars to doubt that the necropolis actually belonged to that settlement, but no other settlements from that time period have been found in the surrounding area. The settlement of Nocera Umbra itself seemed to have small living units from different cultures connected between them. Other small high-medieval funerary enclosures dating from the VI-VII centuries have been found in the area, also displaying the presence of well-established individuals with the presence of jewels and gold utensils.

When first found, the owners unearthed 15 tombs before they contacted the local authorities. After that, further excavations were carried out, discovering 12 more tombs in the period from 9th to 23rd of September of 1897. In March of 1898, systematic excavations were carried out under the guidance of A. Pasqui. The remains of that first exploration suggest that the necropolis was unearthed on its totality, containing a total of 165 tombs. From these, 48 were identified as male burials. The necropolis seemed to be divided in at least four different areas, with close groups of tombs in each of them, which might correspond to a family or clan. Inside each area, there's a clear distinction between feminine and masculine tombs on the basis of the grave goods. No human remains are preserved. They all consist of

a human-sized rectangular pit in an east-west orientation containing wooden elements and remains, even over the anthropologic remains, which points to the use of funerary caskets. There haven't been found any marks or signs in the tombs that would help identify the individuals. There is no presence of funerary chambers and none of the tombs superpose with one another, which helped with their preservation. The dead were buried with their personal possessions which also served as a status distinction. The majority were clothing elements, with some funerary offerings: swords and battle objects for the men and jewellery and domestic items for women. Aside from Lombard tombs, some cultural remains from the neolithic period were also found: bonfire remains, flint waste and bone and ceramic fragments. Additionally, 8 tombs from the early Iron Age were found east of the necropolis. The whole area also contained remains from the Roman period, such as a water cistern and a road leading to a settlement that was probably reused and reformed later by the Lombardians.

A first publication about the discovery was made in 1918 by A. Pasqui and R. Paribeni in the collection *Monumenti Antichi della Reale Accademia dei Lincei*, consisting mostly in a catalog and a map of the necropolis. In later years there was an academic discussion regarding the cultural identity of the necropolis, either Lombardian or gothic, being N. Åberg one of the most important scholars supporting the Lombardian origin.

There's been other publications and studies regarding the graveyards, such as the doctorate thesis of Cornelia Rupp, who also published a catalog of the items in Nocera Umbra tittled "*Das langobardische Gräberfeld von Nocera Umbra. 1. Katalog und Tafeln*" and "*Umbria longobarda. La necropoli di Nocera Umbra*", with contributions from Lidia Paroli and Maria Stella Arena, as well as many others.

The items found during the 1898 excavation were first sent to the *Museo Nazionale Romano* in Rome. Later, in 1916, objects from 25 tombs were given to the city of Milan, and nowadays they are exhibited in *Rocca Albornoz - Museo Nazionale del Ducato di Spoleto*. The ones that remained in Rome were transferred in 1959 to the *Museo dell'Alto Medioevo*, where they are exposed together with the materials found in the necropolis of Castel Trosino<sup>[4]</sup>.

#### 1.2.1.2.- Interpretation

According to Rupp and based on the previous excavations, the male burials can be divided into three different groups based on the typology of their items. Moreover, these groups seem to follow a specific chronologic evolution. The first typology of burials is characterized for waist garnition with triangle plaques, lance tips of Nocera Umbra typology, shield parts, swords of Wehmörting typology, bronze pans and decorated belt plates in their kits. The second group shows complete waist guards decorated with precious metals and bone nails, shield parts (ones with sharp pointed umbo and others with round umbo) and belt guards with gems. Finally, the third typology is characterized by belt guards, some without decorations and others with just gems, axes and a higher number of Schmalsaxe swords.

Feminine burials are harder to compare because materials were used for longer periods and there are a lot of mixed typologies. However, centering on the brooches, one can still sort out different chronological phases. These phases are the following:

1st phase → Lombard brooches from the Lombardian migrants, accompanied with a few pottery remains.

2nd phase → Brooches of local production but following the Lombardian style, usually disc shaped and made of gold. The guard of the fibula is made of silver,

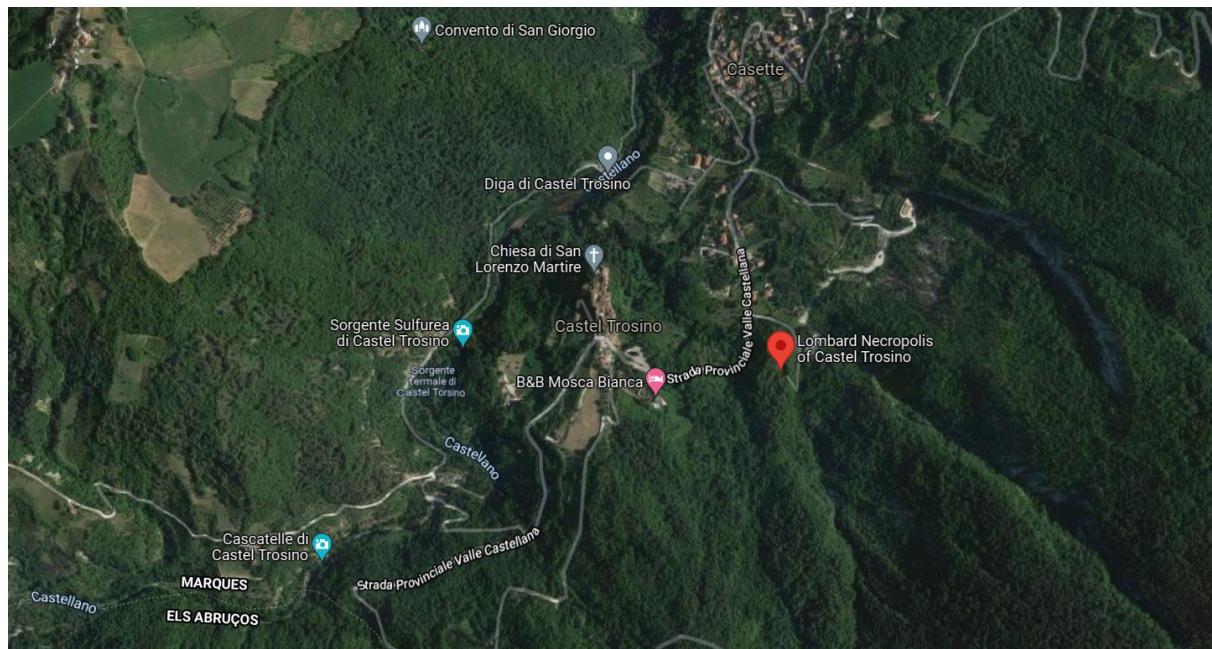
which belongs to the Lombardian tradition from before they reached the Italian peninsula. Otherwise, pottery remains are more abundant than in the 1<sup>st</sup> phase.

3rd phase → Introduction of earrings and pearl necklaces, with the greatest abundance of pottery remains.

### 1.2.2.- Necropolis of Castel Trosino

Located 4 km from Ascoli Piceno (Marche) the necropolis of Castel Trosino was excavated in a systematic way at the end of the 19th century<sup>[5]</sup>. Already in precedence (XV-XVI century) they were found in the area gold coins, weapons, bronze items and other objects made of precious metals. During the XVIIIth Century, those pieces were sold between collectionists and antiquarians, and although some records remain, the traces to their current location are lost.

The first indicator of a great funerary complex was the discovery of the tomb of a knight in 1872 in Contrada Pedata. This tomb has a very rich funerary kit, with saddle and rein gold decorations, various weapons and a dagger with gem incrustations on the hilt among other items of value<sup>[6]</sup>. Another further 50 tombs were unearthed in April 1893 in an area known as *Contrada S. Stefano*, property of the parrochy of San Lorenzo. Their structure was completely destroyed during the excavation process, with some objects stolen or spreaded on the field. At least 62 tombs with stone structures were found at the end, following the traditional style of Roman burials, however, the distinction between male and female burials is not as clear as in the necropolis of Nocera Umbra, as almost half of the male burials don't present the weaponry offerings. An article about the discovery was written the same year by Giulio Gabrielli in a local magazine. Gabrielli was also the one who promoted the excavation of the necropolis of Nocera Umbra twenty years later, taking care of regular excavations from then on.



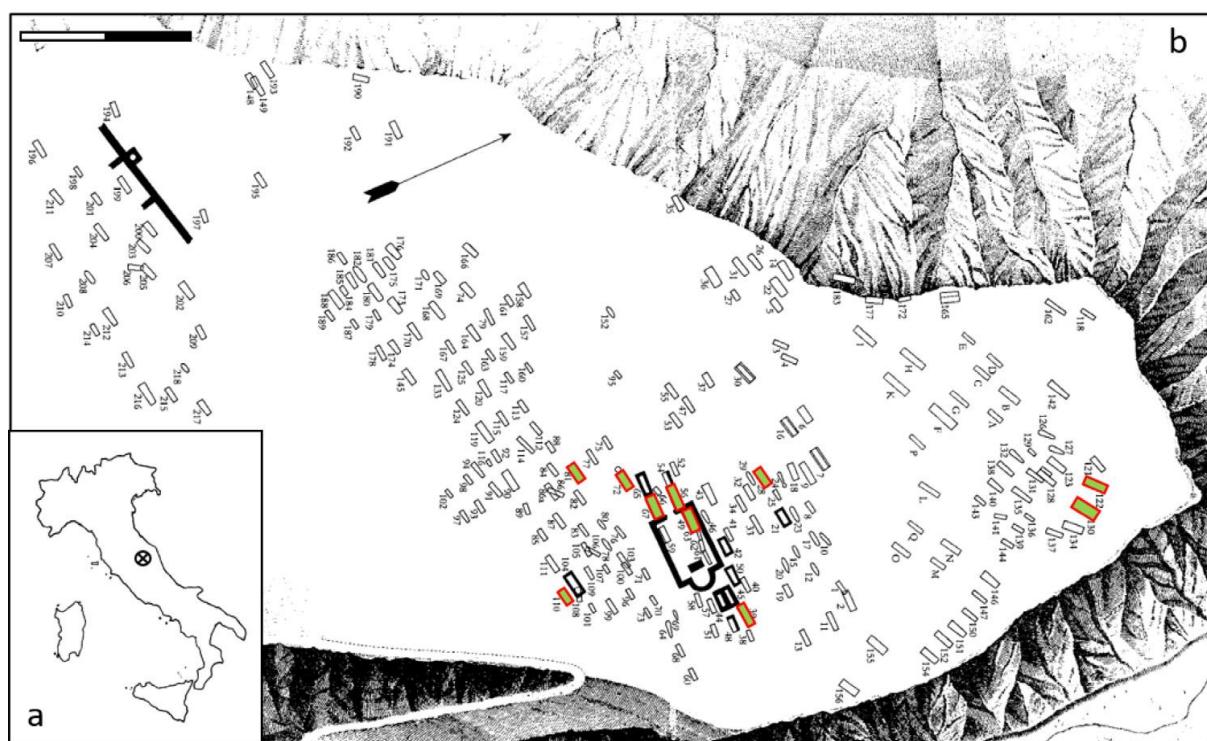
**Illustration 2. Location of the medieval necropolis of Castel Trosino in relation to its actual municipality. [Satelite image extracted with Google Maps] 2021..**

### 1.2.2.1.- The excavation

The necropolis was discovered in 1893 in an area known as Contrada S. Stefano, property of the parish of Castel Trosino. Excavations began the same year until 1896. Thanks to Gabrielli, many of the treasures of the necropolis were not stolen this time and could be preserved. He requested a fast intervention of the state, and the newly founded *Museo Nazionale Romano* sent Raniero Mengarelli as excavation director, supervised by Edoardo Brizio, Superintendent of the Emilia in that time period. Gabrielli wrote that the work of Mengarelli was fast and disorganized but also quite exhaustive.

The excavation complex comprises 289 high-medieval tombs, 270 of which are in Contrada S. Stefano, 16 in Contrada Fonte and 3 in Contrada Campo, all in a relatively good conservation state. A small area in the oriental part of the site had

suffered damage due to the agricultural work. From all these tombs, less than 50% had a funerary kit, and even less objects of value. During the excavations of the end of the nineteenth century not all the skeletons were recovered. It was made with only a sampling of about thirty skulls, however, taken almost all from the graves without a funeral kit. Today they are preserved at the Museum of Anthropology "Giuseppe Sergi" at La Sapienza University of Rome with only 19 skulls, which are currently undergoing an isotopic study of  $^{13}\text{C}$  and  $^{15}\text{N}$ [7].



**Illustration 3. Map of the location of the Necropolis of Castel Trosino (a) and its tombs (b) as they were found during the excavations. The ones highlighted are the ones with anthropologic remains.** By S. Bernardini, S. Asrat Mogesie and I. Micarelli in the article "Contribution to Longobard dietary studies: Stable carbon and nitrogen isotope data from Castel Trosino (6th-8th c. CE, Ascoli Piceno, central Italy)". [2021]

Most of the objects found in the excavation were taken to Rome, first at the *Museo Nazionale Romano* and then to the *Museo dell'Alto Medioevo* founded in 1967, while others (the kits from tomb 6, 58, 117 and, 173, the sword and shield from tomb U, a ceramic cup from tomb 48 and an amfora from tomb 168) were given to the *Museo Civico di Ascoli*[8].

In 2001-2003 started a new investigation on the site under the supervision of the *Soprintendenza per i Beni Archeologici delle Marche* for a further valorization of the Lombardian presence. In this new intervention, there was more attention put on the anthropological remains, as well as some small objects that weren't found or of no interest in the first excavation by Mengarelli, such as a seal ring found in tomb 49, the remains of a silver necklace similar to that in tomb K, glass beads and metal studs in tombs H and 90 respectively. The remains of a rural roman structure from Republican time were also found and recorded, together with a medieval settlement, although no structures from Lombardian time could be identified.

To better understand the archeological site, it might be reminded that gold and jewel-crafting and trading was controlled by the main political powers through a very centralized type of production. In this way, sources of prime materials were precious and limited, being thus objects produced from these materials available to a few. In Castel Trosino, some of the tombs include tools suggesting that they belonged to metalsmiths. Tomb 37, dated circa 600 AD, includes dressing complements and jewels aside from the scales, but no weapons. This is noteworthy, since weapon kits were still common in Lombardian funerary practices. These findings, as well as the presence of importation pottery from northern Africa, might suggest a local production of metal objects or at least regular trading ways of precious metals in the region.

In general, tombs from Castel Trosino show less presence of weapons and more of Roman-Byzantine type of objects than those of Nocera Umbra. A preliminary study suggests that the majority of disc-styled brooches come from a workshop that produced items exclusively for this place, with a unique regional typology. Some others might have been imported from Rome or other important centers.

### 1.2.3.- Previous conservation interventions

Only two major restoration and conservation processes were carried out over 600 objects from the Lombardian collection of Nocera Umbra and Castel Trosino. The first process was carried out in the 60s inside the *Museo dell'Alto Medioevo*, whereas the second one was performed in the 70s in the *Centro di Restauro della Soprintendenza Archeologica*, in Florence, and also in the *Istituto Centrale per il Restauro*, in Rome. In this way the current status of the analyzed pieces is also influenced by these restoration processes<sup>[9]</sup>.

## 2.- Materials and Methods

### 2.1.- Analyzed objects

The first batch of items in which XRF analyses were performed consisted in 17 disc brooches from the necropolis of Castel Trosino. All of these pieces were made of gold except fibula number 1735, which was made in silver. These types of objects were usually made not with a single piece, but with a large number of premade parts (plates, buttons, etc.) that were assembled together<sup>[10]</sup>. In case of plates, they were generally cold worked and hammered into their desired shape, as gold is very malleable and ductile, using a copper base or any other rigid material to give them shape. Some decoration details made with punching and chiseling or with the soldering of small gold beads were also applied in cold. The small size of these decorations showed how meticulous Lombards were in their goldsmithing.

There is not much information about the centers of production of Lombardian jewellery during their time on the Italian peninsula. It's commonly believed that the most important of these centers remained from ancient to high medieval times, being those the workshops from Constantinople and other imperial cities<sup>[11]</sup>, such as Ravenna, Roma or Milan among others. However, there have not been enough studies of the objects found to pinpoint precise places of production or said workshops.

Some goldsmithing elaboration and decorative techniques used by the Lombardians include:

- ***Fettucce flattened in obliquus waves*** → two thin gold plates are shaped up between two corrugated rollers spinning in opposite directions. This process

leaves characteristic undulated marks at 45°, compression deformations and dragging cuts in the object. It's a fast procedure that gives a particular luster and *chiaroscuro* effects. This technique is characteristic of Lombardian objects.

- **Lost wax technique** →fusion technique for the object's matrix, using a cast made from a model made of wax, thus the name. Once the mold was made, the wax was melted.
- **Soldering** →two metals put together using a third, melted filler material. They used soldents with low fusion points, such as gold-silver or gold-copper alloys. Sometimes they used copper salts to solder delicate or small parts, as it has a lower fusion temperature (600-700°C as opposed to the +800°C of gold-silver or gold-copper alloys), although it's not very frequent in circular brooches. They used smaller flames, easier to control, for precision during the soldering.
- **Boxed structure** →soldering of pre-made boxed structures over a base gold plate, creating geometric cavities on the matrix.
- **Doratura (Gilding)** →technique in which a thin coating of gold is applied over surfaces. In metal and in western jewelcrafting, it was often used on silver objects. There are many gilding processes, but the one that was most used in high medieval times was the *doratura al fuoco* with a mercury amalgam. In this process, gold and mercury were mixed in a semi-liquid mixture that was later distributed on the surface of the object. Afterwards, the metal was heated to evaporate the mercury, leaving the gold coating on the surface. This process is nowadays considered quite dangerous because of the toxic gases the mercury evaporation produces.
- **Stagnatura** →this technique consists in covering the flat surface of an object with a thin film of tin or another metal. Traces of mercury suggest the use of amalgamation. It was used to decorate brooches and decorative belt plates and to give different color variations, as well as to protect the rear of some

bronze pieces. The brass surface sometimes was decorated with punching and incisions, bringing outwards the color below.

In general, the analyzed brooches present 3 different, distinctive stylistical typologies that we have used to categorize our statistical analyses. These typologies are:

1. **TYPOLOGY 1: *Cloisonnée*** → use of metal inlays to hold gemstones or polychromous glass over a surface. Mostly seen on round fibuli and sword handles, and it's been used since the Classic era.
2. **TYPOLOGY 2: Gold-plated with filigree and gems** → same as the one before, but with the addition of gems embedded in the broch's surface. These games can be precious gems or colored glass. In the items from our two necropolis, they are distributed in a cross shape.
3. **TYPOLOGY 3: Gold-plated with filigrees and engravings, no gems** → use of metallic threads and engraved patterns as decorations, usually in circular or spiralled patterns, with no embed gems. Some items from Castel Trosino also are decorated with small granulations, which seems typical from that area as well as a star or sun-shaped decorative motif that we can see in all of our objects from this typology.



Photography 1. Brooch number 1110, an example of *cloisonnée* and typology 1.



Photography 2. Brooch number 1186, representative of Typology 2.

Items number 1442 and 1240 when looked from behind presented a main body that was not made from gold but another dark metal, probably silver. Other brooches with the same typology, such as 1239, 1277 and 1410, could have had the same body component that has been lost over time, as the hollowness of the object suggests. The excavations from Nocera Umbra found out that this typology seems to be the earliest one found

in the necropolis, although it's unknown if the same applies to the

one of Castel Trosino, since the distribution of the funerary kits was completely different. According to Lidia Paroli, this type of objects usually present platinoid<sup>[12]</sup>, an alloy of platinum, iridium and osmium, mixed with the gold alloy. This mixing appears in micro-concentrated points when there's a high density ratio during high fusion temperatures with values as high as 1700°C. This treatment prevents amalgamation with gold. It's theorized that these platinoid impurities could come from the prime source ore. Since the gold native from the Italian peninsula doesn't tend to have platinoid impurities, it's theorized that Lombards from Castel Trosino and Nocera Umbra used gold coming from mines of Spain, France, Eastern Europe, Asia Minor or the Caucasus. Another theory is that the objects were made by melting and recasting objects of Roman, Etruscan, Persian or Greek origin, and this refining of mixed materials introduced further concentrations of platinoid.

Other elements that are sometimes present are arsenic, selenium and tellurium. Silver objects tend to be an alloy of silver, copper and lead, and sometimes zinc. The lead probably comes from the mineral of origin, galena (lead sulfur), one of the main sources of silver in ancient times. It's interesting to note that almost all of them present wear signs of use, which means they were worn during the owner's life and then, at their

deathbed.



Photography 3. Brooch number 1231, representative of Typology 3.

The second batch of objects came from Nocera Umbra and consisted in:

- Two swords with an iron blade and the silver and gold decorations on the handle as well as some decorative gemstones. The sheath and the wooden parts of the handle were not preserved.
- One gold medallion with the portrait of a woman. It shows signs of corrosion and some parts have been lost, showing a hollow inside.
- Two bronze stipes in good preservation state.
- Three foldable stools made of iron. Some *agmina* decorations in silver and gold still remain. *Agmina* consists in the engraving of a ductile metal (gold, silver, copper or brass) onto another base metal such as iron, steel or bronze. Patterns are carved on the surface of the base metal and, then, the ductile

metal is inserted through hammering<sup>[14]</sup>. It's possible that the substrate metal was heated to make the insertion easier. This technique has been reported in objects from Celtic, Anglo-Saxon, Lombard and Gothic origins. In Lombardian manufactures, this technique is most often seen in iron objects, usually stools or spurs.

Lombard metallurgy is deeply influenced by ancient Roman techniques. Most remnants found in Roman sites i.e., Crypta Balbi in Rome, display similar elaboration and decorative techniques. Lombardian pieces obviously show also a deep byzantine influence. Metal matrices might have been traded for long distances during that period. As an example, a bronze matrix found in Crypta Balbi in Rome, contains decorative elements found in Adalia (Minor Asia). The hypothesis is that the brooches *ad arco* from Nocera Umbra and Castel Trosino were produced in Italy by the first half of the VIIth century, using traditional decorative techniques from Roman and Byzantine tradition<sup>[15]</sup>.



Photography 4. Sword number 36. The other one, number 380, was made on the same style and with the same elements.

## 2.2.- Environmental conditions

Objects are found in the permanent collection exhibition of the museum in two different types of display cases:

1. glass cabinets attached to the wall
2. standing glass cabinets in the center of the rooms

Both types of display cases are separated from the outside by glass panels and are equipped with inner illumination. However, these display cases do not have any equipment that monitors the conditions inside the cabinets. Likewise, exhibition rooms don't seem to have good, or at least modern, equipment to regulate the environmental conditions. Windows are located in all cases on one side of the room and cover the whole wall, and despite them having curtains, they are the main source of heat and light of the room. Since this work was carried out in the hot conditions of Roman summer in rooms that didn't have adequate climatization equipment, some windows were open, allowing for the heat and the air of the exterior to come in.

## 2.3.- X-Ray Fluorescence

The X-Ray Fluorescence (XRF) is a technique that makes use of electromagnetic radiation in the form of X-Rays to stimulate atoms of a sample, measuring afterwards the energy they emit in return. X-Rays are found in between 0.01 and 10 nm in wavelength and between  $10^{20}$  and  $10^{17}$  Hz in frequency. When the sample is stimulated, it will emit short X-Ray energy in return that will be measured for elemental analysis. The effectiveness of XRF relies on a photoionization process in which electrons of an atom are excited by X-Ray radiation. In this way, an electron is

expelled from an inner electron shell, whereas another one will have to move to fill the vacancy, thus creating an energetic signal. The energy needed to carry on this process needs to be higher than the one binding the electron to its shell. Based upon this reaction, it is noteworthy that each element has a different energetic response. Thus the emission spectra that each element will show after being irradiated by X-rays will display peaks according to the energetic level of the ones in our sample. In this manner, the appearance of a specific peak with a narrow range of energy emission will be characterized for each element, allowing thus for a qualitative analysis of the irradiated object by individually analyzing it in its constitutive elements. Concomitantly, the intensity of each signal will be proportional with the amount of each element, being thus a quantitative and specific indicator found in the analyzed object. In this way, the final graphics will show specific peaks with separate areas for each element present in the sample, although sometimes some peaks will overlap with one another.

At this moment, the XRF technique is used in the characterization of all types of objects, from metals to ceramics and glasses; paper & parchment, paintings or even stone materials) because of their deep penetration in many types of materials and their non-destructive capabilities<sup>[16]</sup>. The system is based on a source of x-rays that sends the energy towards the sample located on a substrate. A detector then gathers the energy that comes back from the sample, going through amplifying and conversion processes. The analyzer processes the data before being interpreted by the software into a spectrum containing the specific observed peaks for each element. X The XRF technique presents two modalities. The first one is the wavelength dispersive (WD) mode, which uses a monochromator, an optical device that selects a narrow band of wavelengths from a wider input of radiation or light. The second modality is the energy-dispersive (ED) mode, which relies on a detector capable of determining the energy of counted electrons. WR mode tends to be more used in laboratory equipment, while ED in portable ones. There's XRF equipment to

analyze samples in the lab, but also hand-held and portable equipment to do analyzes in situ. It's a non-destructive technique and hand-held equipment doesn't require sampling.

Centering on X-rays sources, a 35-50kv X-Ray tube is considered adequate for the analysis of most elements. Silicon Drift Detectors (SDD), Si-PIN and  $HgI_2$  detectors are currently the most used, being both thermoelectrically cooled. The SDD has a thickness of about 300-500  $\mu m$  and an energy resolution of 140 eV at 5.9 keV, which makes it optimal up to about 30 keV.



Photography 5. XRF equipment and setup used for the analysis.

The X-Ray equipment utilized in this work was composed of a silicon tube and a 10791 detector. An Aluminium filter was applied to extract the low-energy elements and the gold signal from the X-Ray tube from the spectra. Prior to the analysis the ratio of photons was tested when the applied energy of the x-Rays was slowly increased from 15Kv to 35Kv. This process allows us to calibrate the equipment

manually taking measures from the gold standards. Two types of analyses were performed on the objects; qualitative and quantitative. In both cases, each object was subjected to 2-to-5 measurements, with a maximal number applied to those that had gems attached. For quantitative analyses each measurement had a 50 second rate per measure, 35Kv at 15 ratio, but for qualitative there wasn't a standard time set, varying from 10 to 50 seconds. Measurements are then described in the manner xxxx-n, in which xxxx indicates the specific analyzed item and n the measurement number. I.e., 1711-2 means the second measurement performed on sample 1711.

## 2.4.- Dataloggers

Dataloggers are small, monitoring equipment that can be installed into rooms or display cases to survey their environmental state. It can monitor temperature, relative humidity, luminosity, and other factors of interest depending on the system and the functions installed such as distances between objects, vibration, air currents and pressure and dew temperature. The system collects the data every certain amount of time, which can later be downloaded and processed.

For this project, two dataloggers were installed. From these, one was placed in a display case of the *Museo dell'Alto Medioevo* that contained the objects subjected to XRF analysis for exhibition. The other device was installed in the same room just above the case. In this way, data from both dataloggers would be compared between them and also with the climatological data from the city of Rome. The data was extracted from the World Weather Information Service from the World Meteorological Organization. The purpose of this is to check how much environmental conditions fluctuate inside the museum's room compared to the exterior and how fast is this process. If the changes are too intense or the environmental conditions are not adequate for the correct preservation of the objects in display, it will be an indicator that we need to perform changes in the location.

The devices used were two HOBO® Onset dataloggers, configured to measure temperature, relative humidity and light intensity. Dataloggers collect information every half an hour and the overall information was collected for examination every two weeks. Before their installation, dataloggers were calibrated to compare their sensitivity, which was noted to be with minimum differences from one another.



Photography 7. Position of the datalogger inside the display case.



Photography 6. Position of the datalogger outside the display case.

## 2.5.- Statistical analysis

A statistical analysis was performed on the brooches to compare their alloy composition. To do so, they were classified into three different subpopulations depending on their typology. Subpopulation 1 consists of the brooches made in cloisonné, subpopulation 2 of the gold-plated ones with embed gems and subpopulation 3 of the gold-plated without gems. Statistical analyses were conducted using a statistical package (SPSS® Ver. 25.0 for Windows; IBM corp.,

Armonk, NY, USA). The resulting regression scores were used to run a two-step cluster analysis based on the log-likelihood distance and the Schwarz's Bayesian Criterion. This cluster analysis led each brooch to be classified in one of the three resulting subpopulations. Following this, percentages of gold, silver and copper belonging to each of the three motile subpopulations were calculated per sample and compared through a GLM/2-way ANOVA. In all analyses, the level of significance was set at  $P \leq 0.05$ . Data are shown as the mean  $\pm$  standard error of the mean (SEM).

### 3.- Results

#### 3.1.- Environmental information

The environmental analysis performed showed that levels of light remained mainly stable and equal during the observation months, with a range between 35-80 Lux at the outside of the case and 430-540 Lux on the inside. This difference is due to the inner illumination of the cases, although on days the museum was not open, the levels of light dropped to 4Lux.

Regarding relative humidity and temperature, the exterior datalogger showed huge variations between days and months. Thus, the lowest values of relative humidity was recorded on May 20th 26% RH whereas the highest was on May 17th at 60% RH. Centering on temperature, the lowest point was also on May 17th at 22°C and the highest on June 14th at 32°C. Mean daily temperature variation was of 4°C, whereas daily RH one was completely irregular (Figure 2).

Inside the display case, relative humidity followed the same variation patterns although the fluctuation was not so intense, with the lowest point the 4th of June at 35%RH and the highest the 17th of May at 54%RH. Lowest temperature point was on the 20th of may at 22°C and the highest on the 5th of July (right after we opened the case to extract the data from the datalogger) at 31°C. In general, the variation temperature along the day was 2°C but Relative Humidity variation was not regular.

The comparison of data from the dataloggers with those from Rome's meteorological stations on the same dates showed a correlation among them, with the highest temperatures registered by dataloggers corresponding to those from meteorological stations and vice versa. However, when it came to minimum temperatures, the record shows a difference of 4°C between those registered by the meteorological

stations and those by the dataloggers in the museum. The variation in temperature in the exterior of the building was much greater, ranging from an average of 29°C to 20°C in a difference of almost 10°C. In the data recorded inside the museum, there was a difference of 4°C between maximum and minimum temperatures recorded inside the display cases and 5°C outside of them (Figure 3).

In regards to relative humidity, the dataloggers showed little mean variation during the time period of our study compared to the variation measured by the meteorological stations in the city of Rome. Whereas it ranged from 45% to 47-48% on average inside the museum, it ranged between 48% to 61% on the outside (Figure 3).

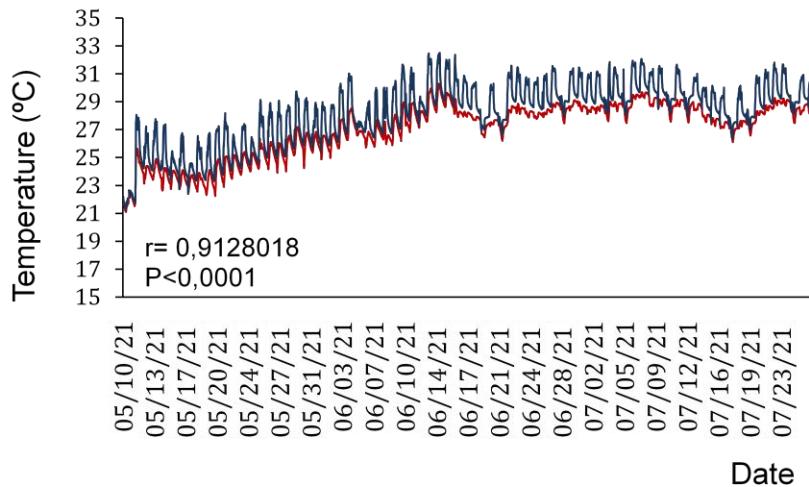


Figure 1. Daily temperature variations of simples stored in museum conditions. Figure shows daily variations of temperature inside (red line) and outside (dark blue line) of display cases in which the studied pieces were stored. Correlation values are included in the Figure.

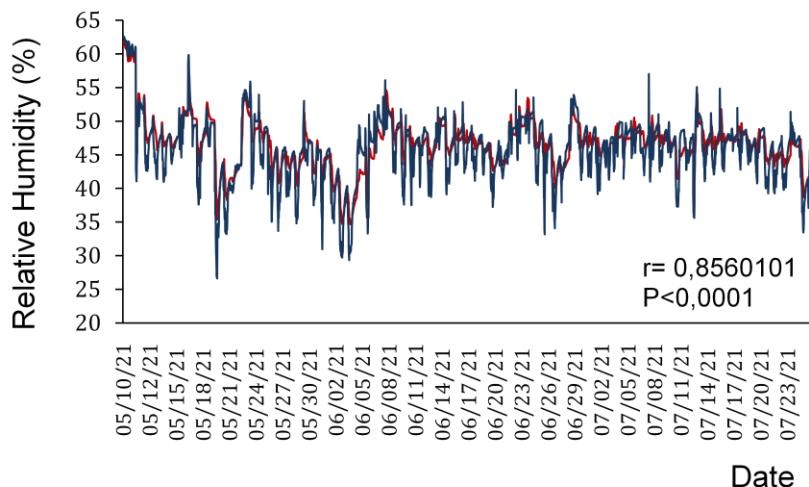


Figure 2. Daily relative humidity variations of samples stored in museum conditions. Figure shows daily variations of RH inside (red line) and outside (dark blue line) of display cases in which the studied pieces were stored. Correlation values are included in the Figure.

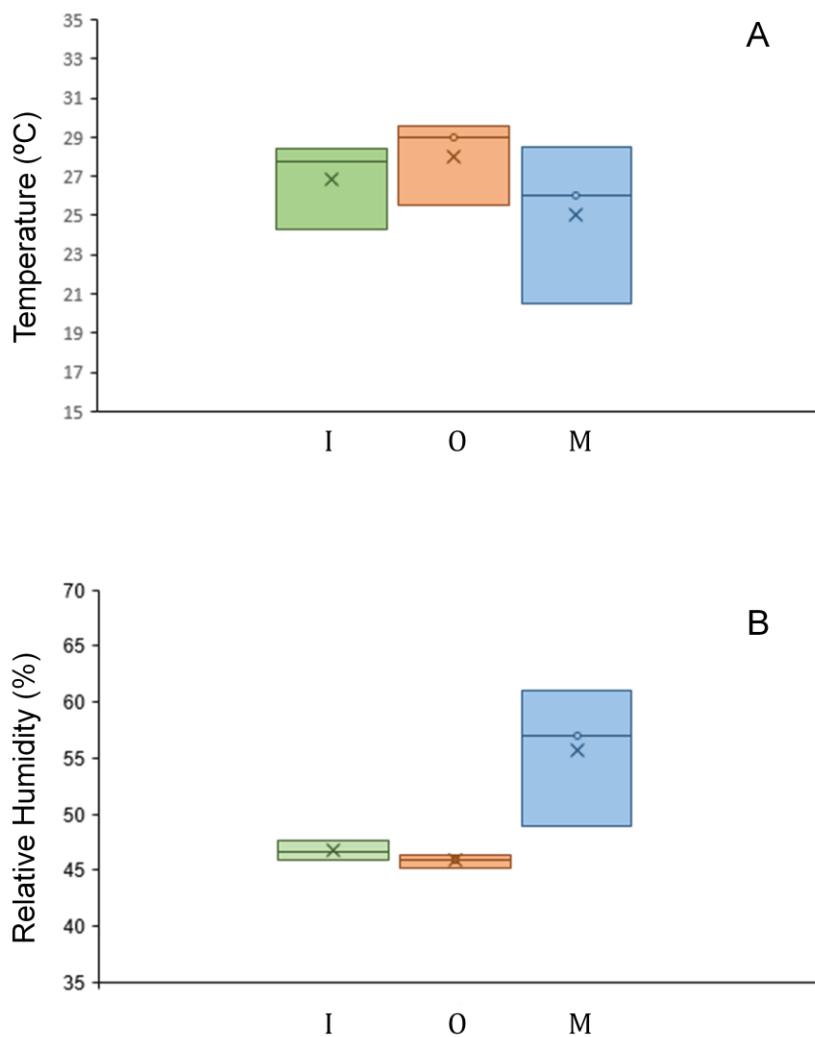


Figure 3. Mean values of temperature and relative humidity of samples stored in museum conditions during the study period. A: mean temperature. B: relative humidity. I: values inside display cases. O: values outside display cases. M: values in the analyzed meteorological station. Figure shows means (crosses inside bars) and medians (lines inside bars) of data from three

### 3.2.- Gold brooches

The XRF analysis on the brooches showed a high percentage of gold in all of them ranging between 80% and 95% with the exception of items 1186 and 1711, which have 71,20% and 74,20% of gold respectively (Table 2). Conversely, items 1186 and 1711, corresponding to Typology 2, showed a higher percentage of silver than the

other brooches. Copper levels remained below 7% with the exception of measurement 1816-2, in which is 51.9%. Measurements 1240-1, 1442-4 and 1442-5 also have high levels of silver (96.84%, 100%, 72.33% respectively). Finally, brooch 1735, has the highest percentage of silver at around 98%.

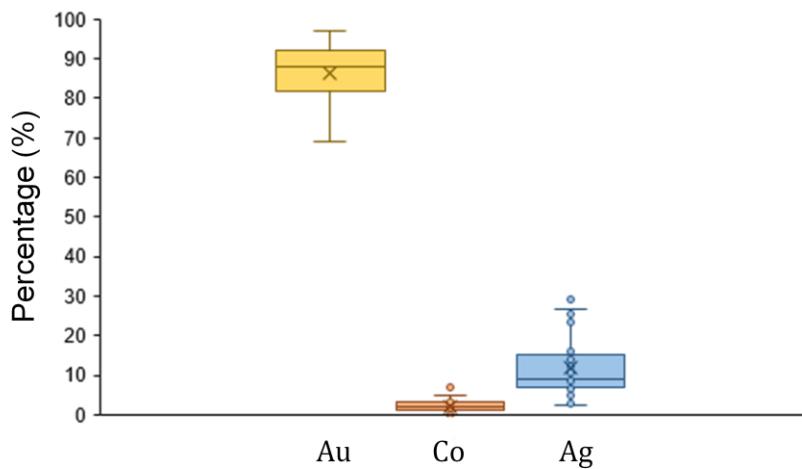


Figure 4. Mean values of gold (Au), copper (Co) and silver (Ag) of gold brooches. Figure shows means (crosses inside bars), medians (lines inside bars) and S.D.s outside the boxes.

Statistical analysis detected significant ( $P \leq 0.05$ ) differences in the percentage of silver among all typologies. Thus, whereas Typology 1 had a mean silver percentage of 6,9%, Typology 2 showed the presence of 3 subpopulations with a percentage of 25,4%, 14,6% and 4,7%, whereas Typology 3 presented 2 subpopulations with 11,1% and 7,2% (Table 4). Regarding gold percentages, Typology 1 presented a main population of 91,3%, Typology 2 three subpopulations of 73%, 82,8% and 94,5%, whereas Typology 3 had two subpopulations of 85,5% and 90,8%. (Fig. %%). No signs of iridium or osmium were found. Otherwise putative low peaks of platinum would be obscured by the much bigger gold ones as they are very close in XRF spectra.

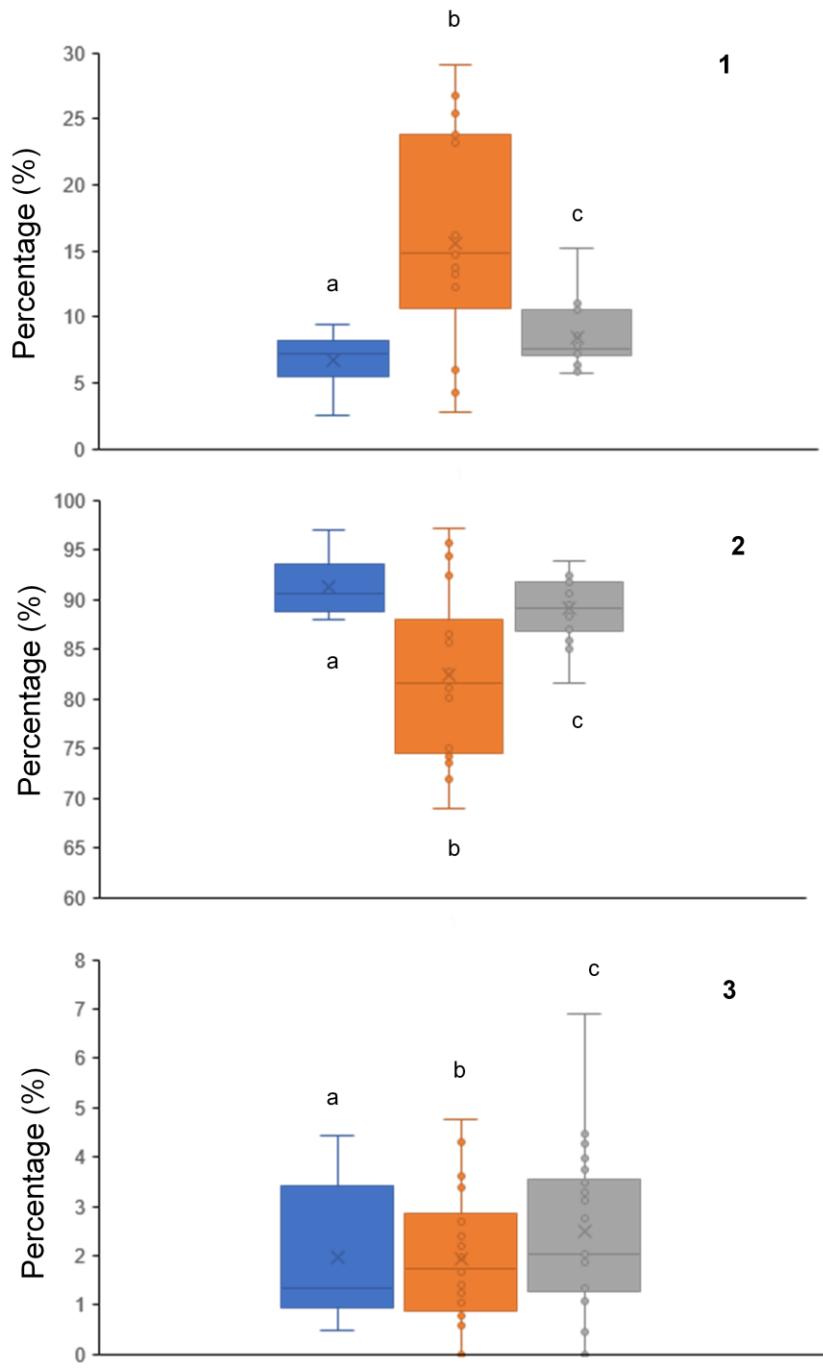


Figure 5. Mean values of silver (1), gold (2) and copper (3) and silver (Ag) of gold brooches depending on their typology. a: typology 1. b: typology 2. c: typology 3. Figure shows means (crosses inside bars), medians (lines inside bars) and S.D.s outside the boxes.

| Object | Typology | % Gold (Au) | % Copper (Cu) | % Silver (Ag) |
|--------|----------|-------------|---------------|---------------|
| 232    | 1        | 88,63       | 3,70          | 7,67          |
| 1110   | 1        | 95,45       | 0,72          | 3,83          |
| 1696   | 1        | 91,14       | 1,24          | 7,62          |

Table 1. Statistical results in percentages of gold, copper and silver for gold brooches of Typology 1.

| Object | Typology | % Gold (Au) | % Copper (Cu) | % Silver (Ag) |
|--------|----------|-------------|---------------|---------------|
| 1186   | 2        | 71,20       | 1,99          | 26,81         |
| 1219   | 2        | 82,00       | 3,23          | 14,76         |
| 1251   | 2        | 80,97       | 3,73          | 15,30         |
| 1258   | 2        | 85,26       | 1,21          | 13,53         |
| 1329   | 2        | 95,37       | 0,59          | 4,03          |
| 1410   | 2        | 92,88       | 1,33          | 5,79          |
| 1711   | 2        | 74,20       | 1,46          | 24,33         |

Table 2. Statistical results in percentages of gold, copper and silver for gold brooches of Typology 2.

| Object | Typology | % Gold (Au) | % Copper (Cu) | % Silver (Ag) |
|--------|----------|-------------|---------------|---------------|
| 1231   | 3        | 89,44       | 3,41          | 7,15          |
| 1239   | 3        | 91,44       | 1,12          | 7,44          |
| 1240   | 3        | 86,68       | 2,34          | 10,98         |
| 1277   | 3        | 87,11       | 2,69          | 10,20         |
| 1308   | 3        | 90,86       | 2,16          | 6,98          |
| 1442   | 3        | 89,76       | 3,31          | 6,92          |

Table 3. Statistical results in percentages of gold, copper and silver for gold brooches of Typology 3.

|                   | Gold (Au)             | Silver (Ag)           | Copper (Cu)          |
|-------------------|-----------------------|-----------------------|----------------------|
| <b>Typology 1</b> |                       |                       |                      |
| Cluster A         | 91,3±0,9 <sup>a</sup> | 6,8±0,6 <sup>a</sup>  | 2±0,6 <sup>a</sup>   |
| <b>Typology 2</b> |                       |                       |                      |
| Cluster A         | 73±0,7 <sup>b</sup>   | 25,4±0,6 <sup>b</sup> | 1,6±0,5 <sup>a</sup> |
| Cluster B         | 82,8±0,6 <sup>c</sup> | 14,6±0,4 <sup>c</sup> | 2,7±0,4 <sup>a</sup> |
| Cluster C         | 94,5±0,9 <sup>a</sup> | 4,7±0,6 <sup>a</sup>  | 0,8±0,6 <sup>a</sup> |
| <b>Typology 3</b> |                       |                       |                      |
| Cluster A         | 85,5±0,8 <sup>c</sup> | 11,1±0,6 <sup>c</sup> | 3,3±0,5 <sup>a</sup> |
| Cluster B         | 90,8±0,5 <sup>a</sup> | 7,2±0,4 <sup>a</sup>  | 2,1±0,3 <sup>a</sup> |

**Table 4. Results of the cluster analysis performed on the gold brooches. Clusters correspond to different subpopulations inside each typology. Superindex <sup>a, b, c</sup> refer to cluster correspondance between typologies.**

### 3.3.- Stools

The XRF analysis demonstrated that the base component of all stools was iron, with a percentage ranging from 90% in object 100 to 64% in the stool from Tomb 5. Besides iron, all objects showed slight traces of copper, zinc and tin. Stools from Tomb 5 and Tomb 17 had higher signals of copper (0,5% and 27,5% respectively) but only number 5 showed signals of tin (2,32%), which combined with copper form the alloy of bronze. The *agmina* decorations were made with silver, detected in all of the stools in a percentage range from 1,06% to 25%. Besides this, the stool from Tomb 17 also contained significant percentages of brass, as the copper signal (27,5%) and the zinc one (7,49%) showed.

### 3.4.- Swords

Both swords, 36 and 380, had a blade made of iron with traces of zinc, Percentages of sword 36 were of 97% for iron and 3% for zinc, whereas for sword 380 these percentages were of 96% and 3%, respectively. The handle is made of gold plates in an alloy of silver and bronze, with silver rods on the sheath (Figure &&&). The sheath of sword 36 was also decorated with coloured glass rich in iron, embedded with the *cloisonnée* technique. The gems on sword 36 show iron presence, used as a colouring agent.

### 3.5.- Streppes

The XRF revealed high percentages of copper (from 73% to 77%), tin (from 3% to 4%), lead (from 13% to 20%) and zinc (from 1% to 6%) with a smaller signal of iron (from 0,2% to 0,7%), forming a bronze alloy (data not shown). There were no significant differences in the overall composition taken as percentages of separate elements among all of the analyzed streppes. As the two streppes were identical and showed no structural differences, XRF

analysis was only performed in one of them.



Photography 8. Bronze strep during measurement 1 of the XRF analysis.

### 3.6.- Medallion

The medallion with the portrait of a woman gave high signals of gold on the outer surface (97%) and of copper on the inside (90%) with traces of zinc (6%).

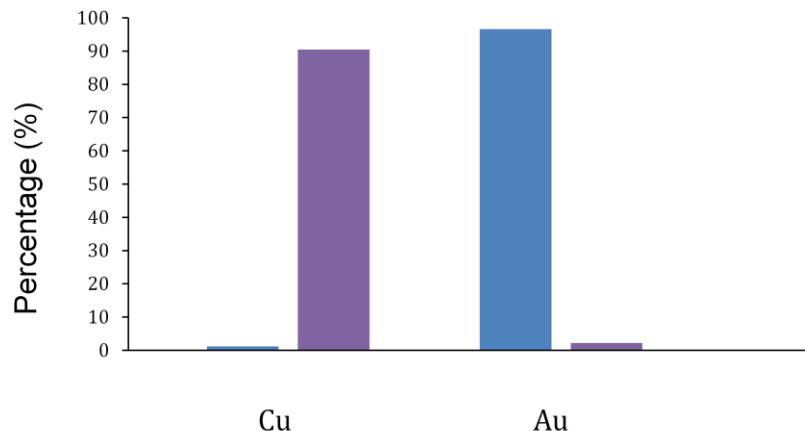


Figure 6. Percentage of gold and copper from the measurements on the medallion. Blue: measurement 1 (interior). Purple: measurement 2 (exterior).

## 4.- Interpretation and Discussion

XRF analysis demonstrated a structural difference between the gold brooches of the Lombardian tombs of Nocera Umbra. This difference may be related to the chronological evolution of Lombardian jewelcrafting and therefore tied to population changes.

As it has been demonstrated the majority of gold brooches found in the collection have a percentage of silver in the alloy between 4-11%, and only in typology number 2, the one with gems embedded on the surface in a cross pattern, does it reach percentages up to 26%. The only brooch with higher percentages than that is number 1735, which is made from silver.

Items 1186 and 1711 showed especially high percentages of silver, and they belong to Typology 2. Statistic analysis demonstrated that the bulk of brooches could be divided into different subpopulations depending on their typology: Typology 1 had a single population (1A), Typology 2, three subpopulations (2A, 2B, 2C) and Typology 3 two subpopulations (3A, 3B). Between all these, statistics evidenced that 1A, 2C and 3B had the same composition and 2B and 3A could also be grouped. Only subpopulation 2A had a unique composition.

This could indicate a correspondence in chronology between objects of different typology but same composition or same place of production. It could also be that the ore used for their fabrication was different, from a different source site or mine, or that they were made using melted gold from other jewelry pieces, thus the impurities of silver would accumulate. However, further studies are required to corroborate any of these hypotheses.

Measurements 1240-1, 1442-4 and 1442-5 also have high levels of silver (96.84%, 100%, 72.33% respectively) as 1240 and 1442 were the only two fibulas with a layer of silver on the reverse.

According to L. Paroli<sup>[17]</sup>, there should be traces of platinoid in at least some of the objects, but there were no signs of iridium or osmium, which form the alloy, together with platinum. However, any possible signs of platinum were obscured by the higher peaks of gold, since they are very close in the spectra. Therefore, we cannot corroborate that this platinoid is present in the objects using XRF, although perhaps it could be detected with other analysis techniques.

In regard to the other items, the important part of the study was the detection of elements in the alloy and although quantification of them was made, it did not follow the same standards and it should be taken as an approximative result. Regarding the stools, the decorations made in agemina technique were at first thought to be of gold and silver, however, XRF technique did not detect signs of gold, but of copper and zinc, which are the basic components of brass. It is possible that it was used as a substitute for gold because of it's cheaper cost, malleability and it's similar coloration.

The two swords showed a distinctive trace of zinc of around 3% on their iron blades. The presence of this element is uncommon in iron objects of that time period except in ores and iron objects coming from India. It is possible that the blades were exported, as both swords are rich in jewelry and definitely a display of their owner's high status. There was an hypothesis that the zinc detected might have been a result of a galvanization process to preserve the iron from corrosion, however no records of such process have been recorded. Currently, we lack the information to prove either of these hypotheses, so further investigations should be carried.

In regards to the gold medallion, the two measurements we took with XRF showed very different results, having the one from the inside approximately 90% of copper and 7% of zinc. Thus, we can believe that the object was made with a main body of copper or brass covered with a gold foil. This could make the production of objects like these much cheaper.



Photography 9. Medallion during the process of analysis. To corroded inside is clearly visible under the gold plating.

Thanks to the datalogger information, we know that the rooms of the museum do a good job keeping the temperature from dropping below 20°C, however, it does not protect it from reaching temperatures over 30°C, even though the glass cabinets attenuated the temperature oscillation between the day.

The ideal RH to store mixed metal collections is between 35 and 55%. None of the measurements we took surpass these numbers but they very much are at the limit. However, when compared to the data extracted

from the meteorological stations, we can see that the percentage of relative humidity and its variation is much lower inside the museum and in the display cabinets. On average, the relative humidity inside the cabinets is slightly higher than the one on the room due to condensation effects.

On the first visits to the museum, there was worry about some of the iron pieces of the collection bot in the exhibition and in storage as they showed signs of corrosion

despite having received preventive and conservative treatments beforehand, however the museum's conservators did some evaluations and declared that it doesn't seem to be an active process. Corrosion is the main degrading factor of metals, and its appearance is closely linked to the humidity in the environment. It would be ideal for the better preservation of these pieces to reduce and stabilize the percentages of relative humidity: introducing silica gel inside the display case is a very standard way of doing so, although the ideal process would be to either remove the pieces and store them somewhere with a controlled and stable environment, halting the degradation process, to change the cabinets where the pieces are exposed or to instal devices that would allow the monitoring and manipulation of the inner atmosphere of the cabinets.

Nowadays there are plenty of modern cabinets that can be equipped with systems to control the temperature and humidity. Glass or plastic are still the best materials for metals, since they do not conduct electricity and do not produce acidification like wood and certain types of fabric could.

## References and Bibliography

[7] Bernardini, S., Mogesie, S. A., & Micarelli, I. (2021, August 17). Contribution to Longobard dietary studies: Stable carbon and nitrogen isotope data from Castel Trosino (6 th -8 th c. CE, Ascoli Piceno, central Italy). *Data in Brief*, (38). <https://doi.org/10.1016/j.dib.2021.107290>

Cesareo, R., Ridolfi, S., & Castellano, A. (2006, March). From Giotto to De Chirico to Verrocchio: analyses of paintings and historical bronze alloys availing of portable EDXRF equipment. *Journal of Neutron Research*, 14(1), 17–27. 10.1080/10238160600672997

[11][15] Dalceggio, M. (2018). *Fibule a disco di VI-VII secolo in Italia*. BraDypUS.net communicating cultural heritage.

[10][13] Devoto, G. (1994). Osservazioni sulle tecniche e i materiali dei corredi di Nocera Umbra e di Castel Trosino. In *Arti del fuoco in età longobarda* (pp. 23-24). Ministero per i Beni Culturali e Ambientali.

Gigante, G. E., Ridolfi, S., & Ferrero, D. (2011, December 13). Diagnostic investigations and statistical validation of EDXRF mapping of the burial monument of Pope Sixtus IV by Antonio Pollaiolo (1493) in the Vatican. *Journal of Cultural Heritage*, 13(3), 345-351. 10.1016/j.culher.2011.11.003

Kladouri, N. K., Karydas, A. G., & Kantarelou, V. (2021, April 24). Bronze votive pins from the sanctuary of Athena Alea at Tegea, Arcadia, Greece, ca. 9th-7th BCE: A microscopic and compositional study using portable micro X-ray fluorescence spectrometry (micro-XRF). *Journal of Archaeological Science: Reports*, (37). <https://doi.org/10.1016/j.jasrep.2021.102975>

Logan, J., & Selwyn, L. (2019, February 22). *Storage of Metals – Canadian Conservation Institute (CCI) Notes 9/2*. Canada.ca. Retrieved November 18, 2021, from <https://www.canada.ca/en/conservation>

[institute/services/conservation-preservation-publications/canadian-conservation-institute-notes/storage-metals.html](http://institute/services/conservation-preservation-publications/canadian-conservation-institute-notes/storage-metals.html)

[<sup>14</sup>]Paroli, L. (1994). Aspetti e problemi dell'archeologia della produzione in età longobarda. In *Arti del fuoco in età longobarda* (pp. 11-18). Ministero per i Bene Culturali e Ambientali.

[<sup>12</sup>][<sup>17</sup>]Paroli, L. (Ed.). (1997). *L'Italia centro-settentrionale in età longobarda : atti del Convegno, Ascoli Piceno, 6-7 ottobre 1995*. All'Insegna del Giglio.

[<sup>6</sup>]Paroli, L., & Nepoti, S. (2006). *Archeologia Medievale*, XXXIII, 2006 (R. Francovich, Ed.). All'Insegna del Giglio.

[<sup>5</sup>][<sup>8</sup>]Paroli, L., & Ricci, M. (2006). *La necropoli altomedievale di Castel Trosino* (Vol. 32-33). All'Insegna del Giglio.

[<sup>16</sup>]Ridolfi, S. (2017). Portable Systems for Energy-dispersive X-ray Fluorescence Analysis. *Encyclopedia of Analytical Chemistry*. 10.1002/9780470027318.a6803.pub3

[<sup>9</sup>]Rotondi, M., & Museo dell'alto medioevo. (1994). Nota sugli interventi di restauro. In *Arti del fuoco in età longobarda* (pp. 25-27). Ministero per i Beni Culturali e Ambientali.

[<sup>1</sup>][<sup>2</sup>][<sup>3</sup>][<sup>4</sup>]Rupp, C. (1997). La necropoli longobarda di Nocera Umbra (loc. II Portrone): l'analisi archeologica. In *Umbria Longobarda: La necropoli di Nocera Umbra nel centenario della scoperta* (pp. 23-40). Edizioni de Luca.

Rupp, C. B. (2005). *Das langobardische Gräberfeld von Nocera Umbra*. All'insegna del giglio.

Università del Salento & Università degli Studi "Suor Orsola Benincasa". (2010, June-December). Metal finds at the Middle and Late Bronze Age settlement of Scoglio del Tonno (Taranto, Apulia): results of archaeometallurgical analyses (Soprintendenza archeologica della Puglia, Ed.). *TRABAJOS DE PREHISTORIA*, 67(2), 457-468. 10.3989/tp.2010.10050

## Annex 1: complementary tables

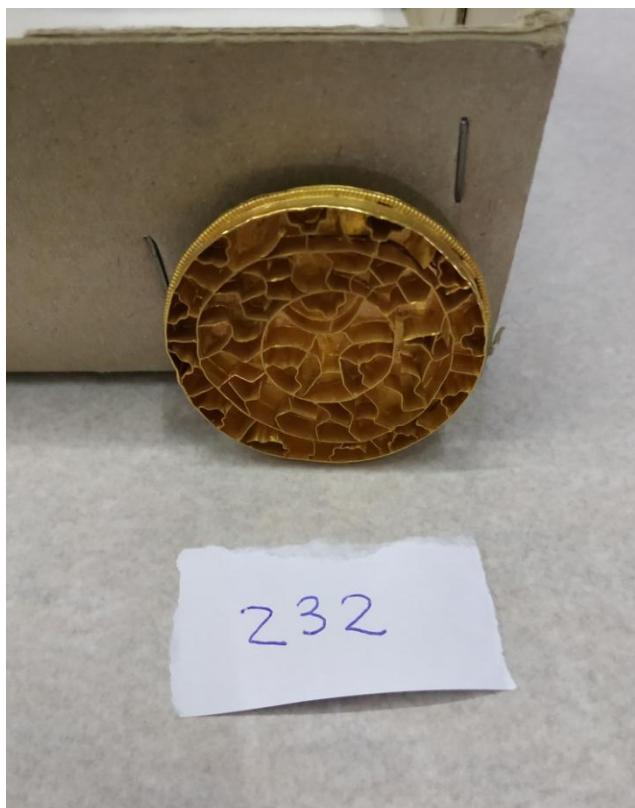
|        | Au (count) | Cu (count) | Ag (count) |
|--------|------------|------------|------------|
| 232-1  | 11943      | 410        | 1045       |
| 232-2  | 10371      | 522        | 885        |
| 1110-1 | 11612      | 57         | 298        |
| 1110-2 | 5592       | 73         | 393        |
| 1186-3 | 8329       | 151        | 3098       |
| 1186-4 | 7980       | 300        | 2822       |
| 1186-5 | 4934       | 144        | 2079       |
| 1219-1 | 11186      | 263        | 1970       |
| 1219-2 | 9254       | 432        | 1874       |
| 1219-3 | 6821       | 359        | 1170       |
| 1219-4 | 5092       | 222        | 811        |
| 1231-1 | 10655      | 124        | 737        |
| 1231-2 | 7851       | 163        | 644        |
| 1231-3 | 10321      | 499        | 858        |
| 1231-4 | 7332       | 593        | 653        |
| 1239-1 | 12370      | 62         | 1062       |
| 1239-2 | 7542       | 0          | 623        |
| 1239-3 | 9523       | 207        | 590        |
| 1239-4 | 8484       | 194        | 811        |
| 1240-2 | 8917       | 201        | 1133       |
| 1240-3 | 8732       | 334        | 1102       |
| 1240-4 | 5065       | 78         | 641        |
| 1251-1 | 10521      | 442        | 2112       |
| 1251-2 | 9590       | 563        | 1680       |
| 1251-3 | 8526       | 358        | 1583       |
| 1251-4 | 2548       | 75         | 516        |
| 1258-1 | 11411      | 166        | 1619       |
| 1258-2 | 6995       | 56         | 1106       |
| 1258-3 | 8041       | 56         | 1293       |
| 1258-4 | 7903       | 209        | 1434       |
| 1277-2 | 1967       | 75         | 367        |

| Total count | Au (%) | Cu (%) | Ag (%) |
|-------------|--------|--------|--------|
| 13398       | 89,14  | 3,06   | 7,80   |
| 11778       | 88,05  | 4,43   | 7,51   |
| 11967       | 97,03  | 0,48   | 2,49   |
| 6058        | 92,31  | 1,21   | 6,49   |
| 11578       | 71,94  | 1,30   | 26,76  |
| 11102       | 71,88  | 2,70   | 25,42  |
| 7157        | 68,94  | 2,01   | 29,05  |
| 13419       | 83,36  | 1,96   | 14,68  |
| 11560       | 80,05  | 3,74   | 16,21  |
| 8350        | 81,69  | 4,30   | 14,01  |
| 6125        | 83,13  | 3,62   | 13,24  |
| 11516       | 92,52  | 1,08   | 6,40   |
| 8658        | 90,68  | 1,88   | 7,44   |
| 11678       | 88,38  | 4,27   | 7,35   |
| 8578        | 85,47  | 6,91   | 7,61   |
| 13494       | 91,67  | 0,46   | 7,87   |
| 8165        | 92,37  | 0,00   | 7,63   |
| 10320       | 92,28  | 2,01   | 5,72   |
| 9489        | 89,41  | 2,04   | 8,55   |
| 10251       | 86,99  | 1,96   | 11,05  |
| 10168       | 85,88  | 3,28   | 10,84  |
| 5784        | 87,57  | 1,35   | 11,08  |
| 13075       | 80,47  | 3,38   | 16,15  |
| 11833       | 81,04  | 4,76   | 14,20  |
| 10467       | 81,46  | 3,42   | 15,12  |
| 3139        | 81,17  | 2,39   | 16,44  |
| 13196       | 86,47  | 1,26   | 12,27  |
| 8157        | 85,75  | 0,69   | 13,56  |
| 9390        | 85,63  | 0,60   | 13,77  |
| 9546        | 82,79  | 2,19   | 15,02  |
| 2409        | 81,65  | 3,11   | 15,23  |

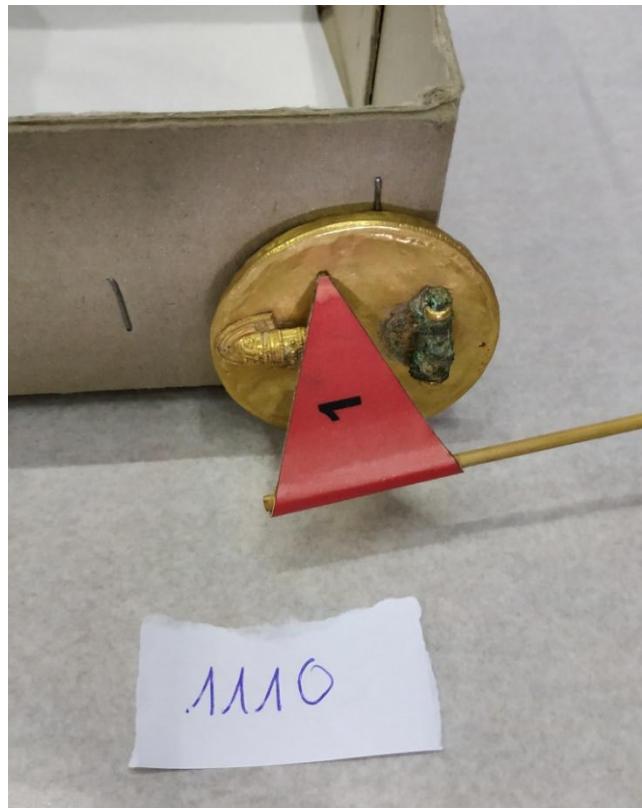
|        |       |     |      |  |       |       |      |       |
|--------|-------|-----|------|--|-------|-------|------|-------|
| 1277-3 | 10471 | 402 | 1277 |  | 12150 | 86,18 | 3,31 | 10,51 |
| 1277-4 | 8095  | 332 | 1099 |  | 9526  | 84,98 | 3,49 | 11,54 |
| 1308-1 | 8683  | 0   | 570  |  | 9253  | 93,84 | 0,00 | 6,16  |
| 1308-2 | 7141  | 88  | 600  |  | 7829  | 91,21 | 1,12 | 7,66  |
| 1308-3 | 8823  | 444 | 674  |  | 9941  | 88,75 | 4,47 | 6,78  |
| 1308-4 | 8041  | 246 | 667  |  | 8954  | 89,80 | 2,75 | 7,45  |
| 1329-1 | 11507 | 94  | 526  |  | 12127 | 94,89 | 0,78 | 4,34  |
| 1329-2 | 8285  | 0   | 241  |  | 8526  | 97,17 | 0,00 | 2,83  |
| 1329-3 | 9224  | 87  | 460  |  | 9771  | 94,40 | 0,89 | 4,71  |
| 1410-1 | 11877 | 176 | 771  |  | 12824 | 92,62 | 1,37 | 6,01  |
| 1410-2 | 2896  | 0   | 130  |  | 3026  | 95,70 | 0,00 | 4,30  |
| 1410-3 | 9399  | 169 | 606  |  | 10174 | 92,38 | 1,66 | 5,96  |
| 1442-1 | 7696  | 326 | 657  |  | 8679  | 88,67 | 3,76 | 7,57  |
| 1442-2 | 7435  | 162 | 472  |  | 8069  | 92,14 | 2,01 | 5,85  |
| 1442-3 | 8924  | 400 | 726  |  | 10050 | 88,80 | 3,98 | 7,22  |
| 1696-1 | 11374 | 139 | 842  |  | 12355 | 92,06 | 1,13 | 6,82  |
| 1696-2 | 4916  | 82  | 520  |  | 5518  | 89,09 | 1,49 | 9,42  |
| 1711-1 | 9241  | 110 | 3180 |  | 12531 | 73,75 | 0,88 | 25,38 |
| 1711-2 | 6016  | 114 | 1930 |  | 8060  | 74,64 | 1,41 | 23,95 |
| 1711-3 | 6156  | 87  | 2133 |  | 8376  | 73,50 | 1,04 | 25,47 |

Table 5. Results of the easurements taken with XRF on the gold broches. Table displaysthe counts of gold, copper and silver, the total count, and the calculated percentage of each element per measurement.

## Annex 2: complementary photographs



Annex photography 4. Brooch 232. Typology 1.



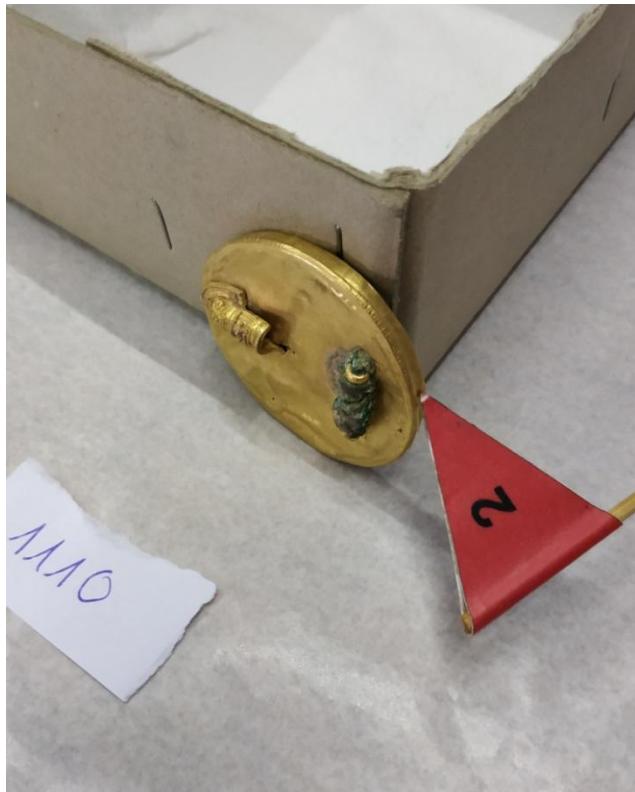
Annex photography 1. Brooch 1110, measurement 1. Typology 1.



Annex photography 3. Brooch 232, measurement 2. Typology 1.



Annex photography 2. Brooch 232, measurement 1. Typology 1.



Annex photography 8. Brooch 1110, measurement 2.  
Typology 1.



Annex photography 7. Brooch 1110, measurement of  
the orange gems. Typology 1.



Annex photography 6. Brooch 1110, measurement of  
the white gems. Typology 1.



Annex photography 5. Brooch 1110, measurement of  
the violet gems. Typology 1.



Annex photography 12. Brooch 1186, measurement 1.  
Typology 2.



Annex photography 11. Brooch 1186, measurement 2.  
Typology 2.



Annex photography 10 Brooch 1186, measurement 3.  
Typology 2.



Annex photography 9. Brooch 1186, measurement 4.  
Typology 2.



Annex photography 16. Brooch 1186, measurement 5. Typology 2.



Annex photography 15. Brooch 1186, measurement of the interior blue gems. Typology 2.



Annex photography 14. Brooch 1186, measurement of the exterior green gems. Typology 2.



Annex photography 13. Brooch 1186, measurement of the exterior blue gems. Typology 2.



Annex photography 20. Brooch 1186, measurement of the interior white gems. Typology 2.



Annex photography 19. Brooch 1219. Typology 2.



Annex photography 17. Brooch 1219, measurement 1. Typology 2.



Annex photography 18. Brooch 1219, measurement 2. Typology 2.



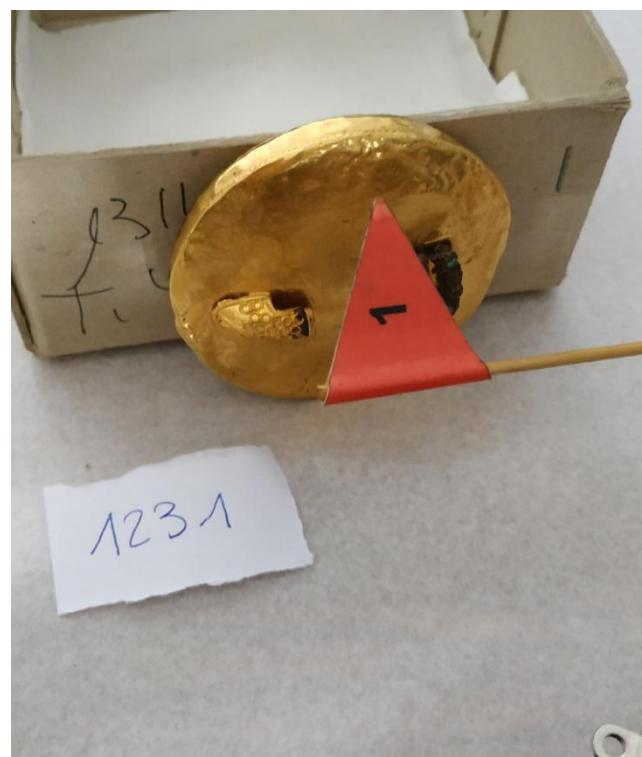
Annex photography 22. Brooch 1219, measurement 3.  
Typology 2.



Annex photography 21. Brooch 1219, measurement 4.  
Typology 2.



Annex photography 24. Brooch 1231. Typology 3.



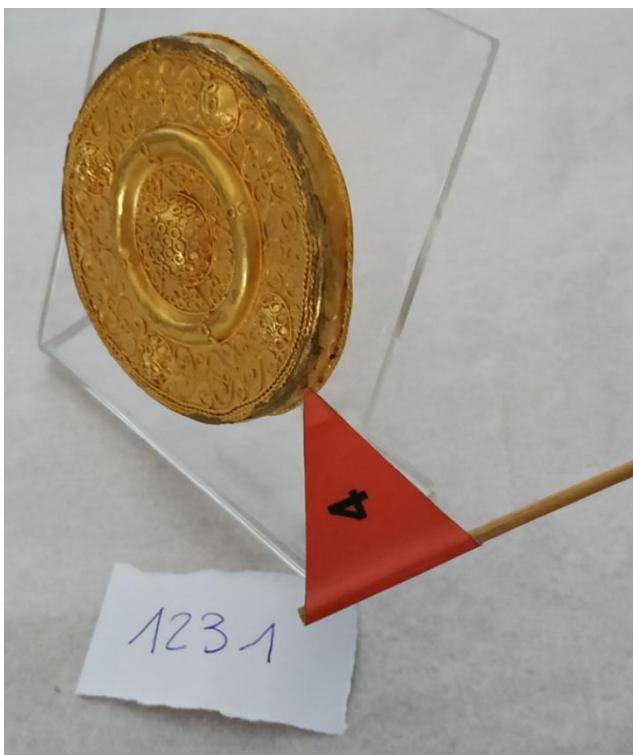
Annex photography 23. Brooch 1231, measurement 1.  
Typology 3.



Annex photography 28. Brooch 1231, measurement 2.  
Typology 3.



Annex photography 25. Brooch 1231, measurement 3.  
Typology 3.



Annex photography 27. Brooch 1231, measurement 4.  
Typology 3.



Annex photography 26. Brooch 1239, measurement 1.  
Typology 3.



Annex photography 32. Brooch 1239, measurement 2.  
Typology 3.



Annex photography 29. Brooch 1239, measurement 3.  
Typology 3.



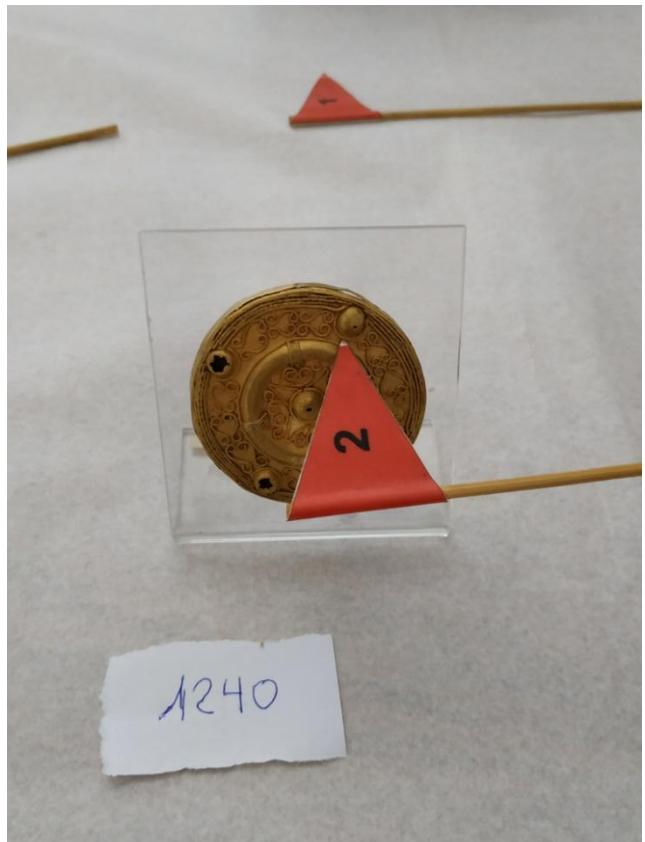
Annex photography 31. Brooch 1239, measurement 4.  
Typology 3.



Annex photography 30. Brooch 1240. Typology 3.



Annex photography 36. Brooch 1240, measurement 1.  
Typology 3.



Annex photography 35. Brooch 1240, measurement 2.  
Typology 3.



Annex photography 34. Brooch 1240, measurement 3.  
Typology 3.



Annex photography 33. Brooch 1240, measurement 4.  
Typology 3.



Annex photography 40. Brooch 1251. Typology 2.



Annex photography 39. Brooch 1251, measurement 1. Typology 2.



Annex photography 38. Brooch 1251, measurement 2. Typology 2.



Annex photography 37. Brooch 1251, measurement 3. Typology 2.



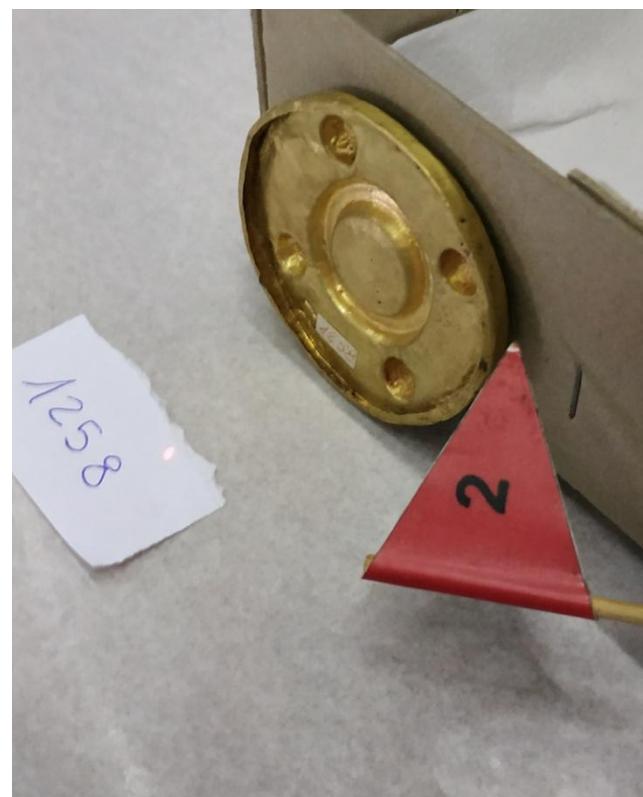
Annex photography 43. Brooch 1251, measurement 4. Typology 2.



Annex photography 44. Brooch 1258. Typology 2.



Annex photography 42. Brooch 1258, measurement 1. Typology 2.



Annex photography 41. Brooch 1258, measurement 2. Typology 2.



Annex photography 46. Brooch 1258, measurement 3. Typology 2.



Annex photography 45. Brooch 1258, measurement 4. Typology 2.



Annex photography 48. Brooch 1258, measurement of the exterior blue gems. Typology 2.



Annex photography 47. Brooch 1258, measurement of the exterior green gems. Typology 2.



Annex photography 52. Brooch 1277. Typology 3.



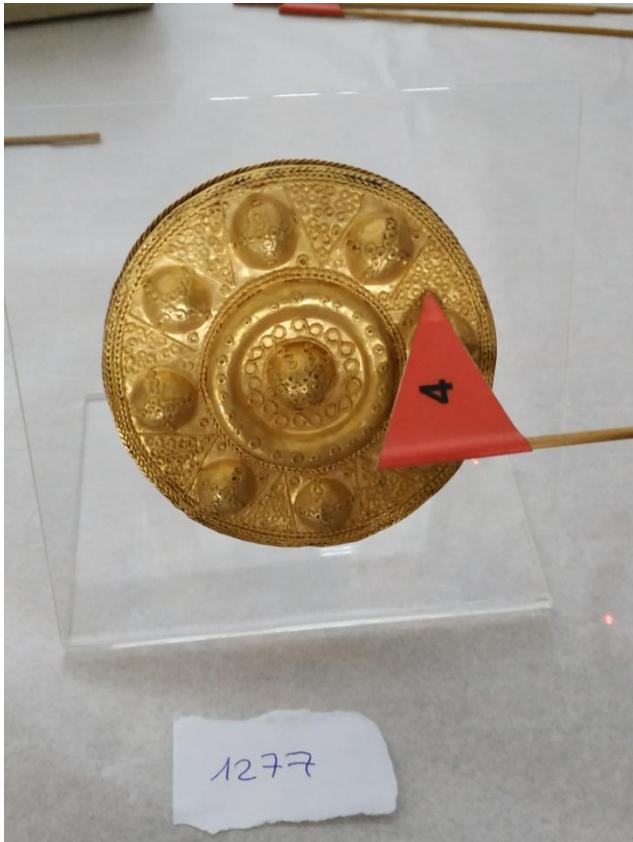
Annex photography 51. Brooch 1277, measurement 1. Typology 3.



Annex photography 50. Brooch 1277, measurement 2. Typology 3.



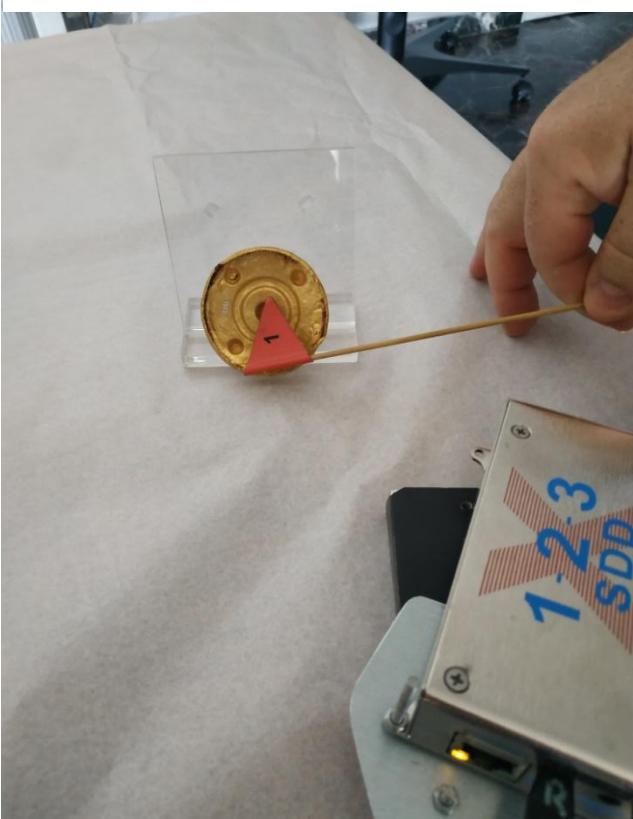
Annex photography 49. Brooch 1277, measurement 3. Typology 3.



Annex photography 56. Brooch 1277, measurement 4. Typology 3.



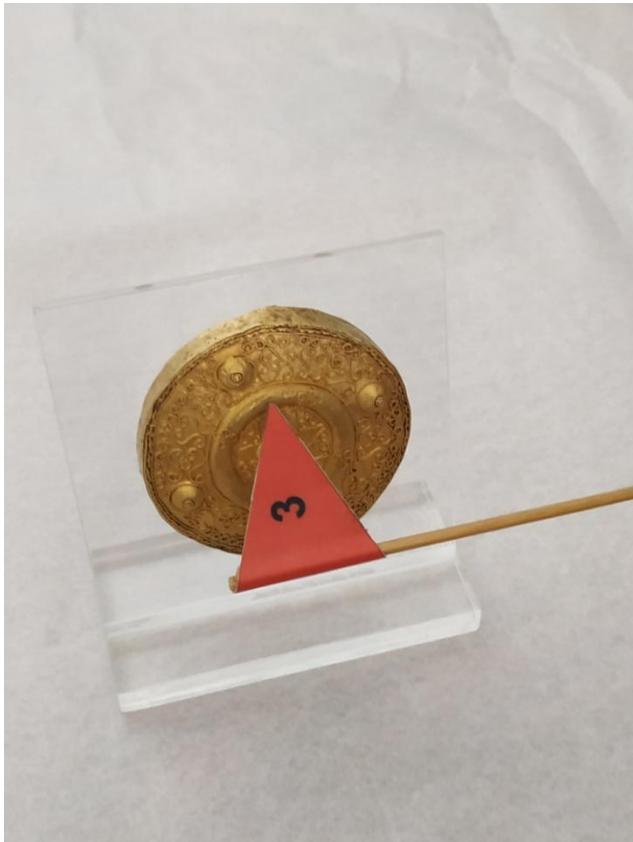
Annex photography 55. Brooch 1308. Typology 3.



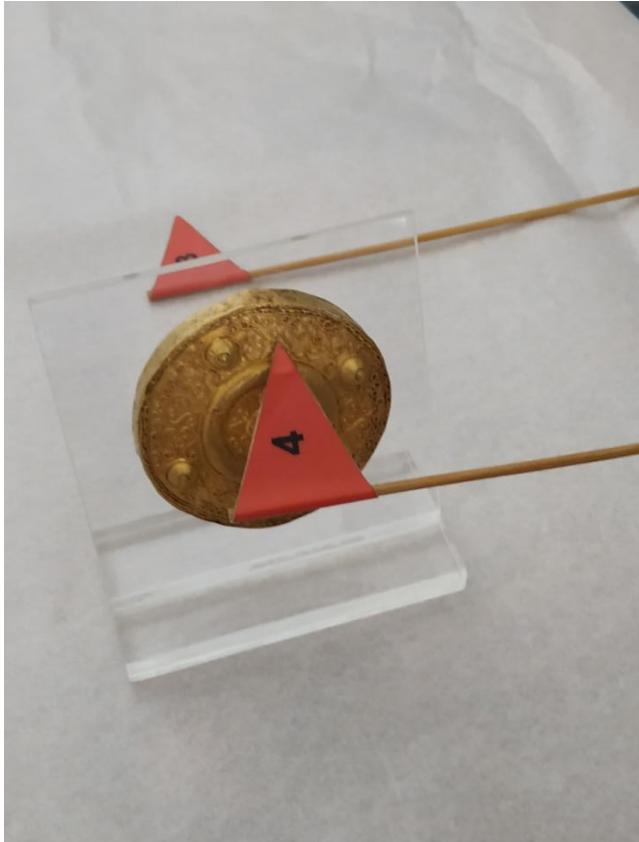
Annex photography 54. Brooch 1308, measurement 1. Typology 3.



Annex photography 53. . Brooch 1308, measurement 2. Typology 3.



Annex photography 60. Brooch 1308, measurement 3.  
Typology 3.



Annex photography 59. . Brooch 1308, measurement 4.  
Typology 3.



Annex photography 58. Brooch 1329. Typology 2.



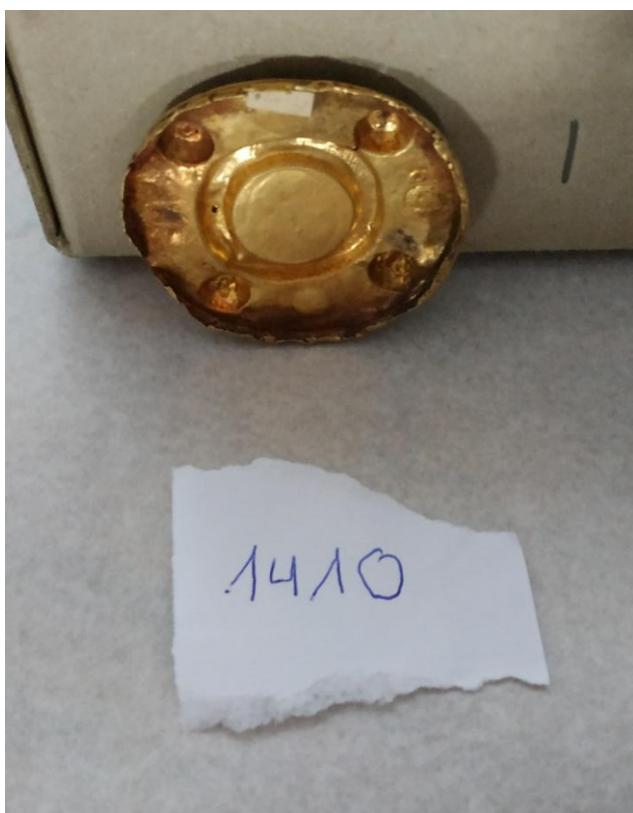
Annex photography 57. Brooch 1329, measurement 1.  
Typology 2.



Annex photography 64. Brooch 1329, measurement 2.  
Typology 2.



Annex photography 63. Brooch 1329, measurement 3.  
Typology 2.



Annex photography 62. Brooch 1410. Typology 2.



Annex photography 61. Brooch 1410, measurement 1.  
Typology 2.



Annex photography 68. Brooch 1410, measurement 2.  
Typology 2.



Annex photography 67. Brooch 1410, measurement 3.  
Typology 2.



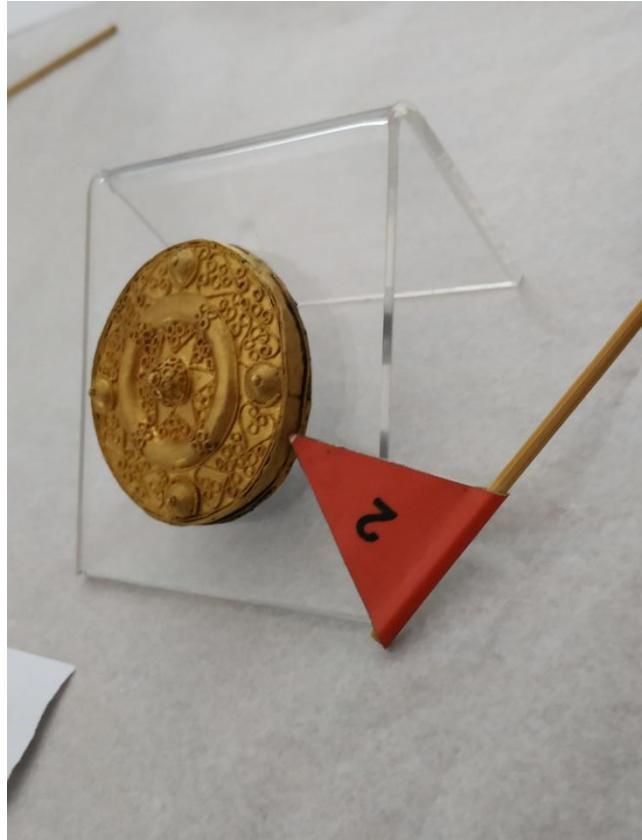
Annex photography 66. Brooch 1410, measurement 4.  
Typology 2.



Annex photography 65. Brooch 1442. Typology 3.



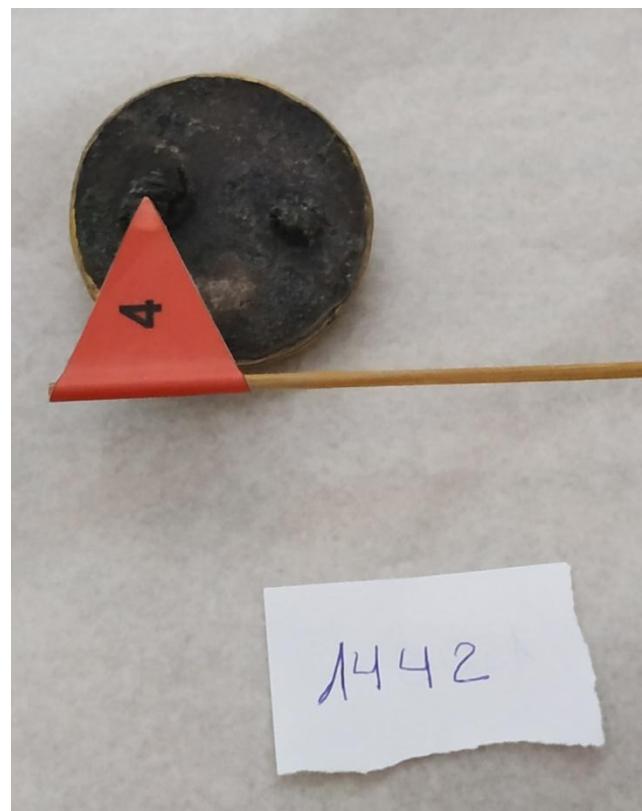
Annex photography 72. Brooch 1442, measurement 1.  
Typology 3.



Annex photography 71. Brooch 1442, measurement 2.  
Typology 3.



Annex photography 70. Brooch 1442, measurement 3.  
Typology 3.



Annex photography 69. Brooch 1442, measurement 4.  
Typology 3.



Annex photography 76. Brooch 1442, measurement 5. Typology 3.



Annex photography 75. Brooch 1696. Typology 1.



Annex photography 74. Brooch 1696, measurement 1. Typology 1.



Annex photography 73. Brooch 1696, measurement 2. Typology 1.



Annex photography 80. Brooch 1711. Typology 2.



Annex photography 79. Brooch 1711, measurement 1. Typology 2.



Annex photography 78. Brooch 1711, measurement 2. Typology 2.



Annex photography 77. Brooch 1711, measurement 3. Typology 2.



Annex photography 84 Brooch 1711, measurement 4.  
Typology 2.



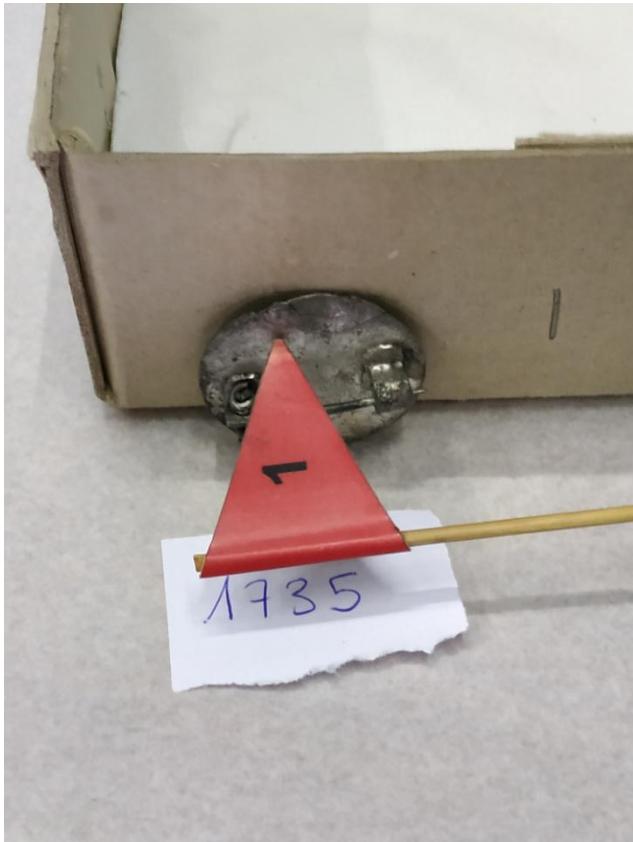
Annex photography 83. Brooch 1711, measurement 5.  
Typology 2.



Annex photography 82 Brooch 1711, measurement of  
the central blue gem. Typology 2.



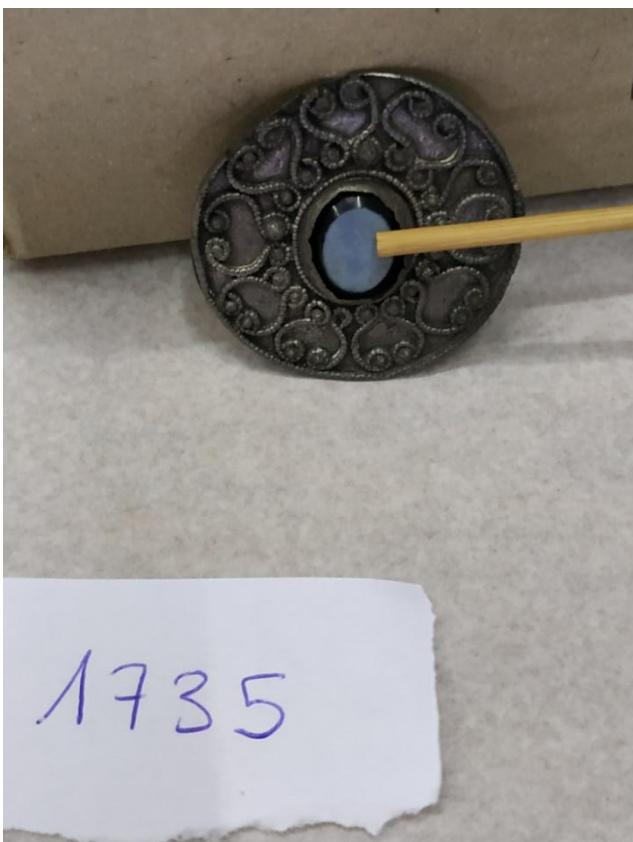
Annex photography 81. Brooch 1735.



Annex photography 88. Brooch 1735, measurement 1.



Annex photography 87 Brooch 1735, measurement 2.



Annex photography 86. Brooch 1735, measurement of the central blue gem.



Annex photography 85. Sword 380.



Annex photography 92. Sword 380, measurement 1.



Annex photography 91. Sword 380, measurement 2.



Annex photography 90. Sword 380, measurement 3.



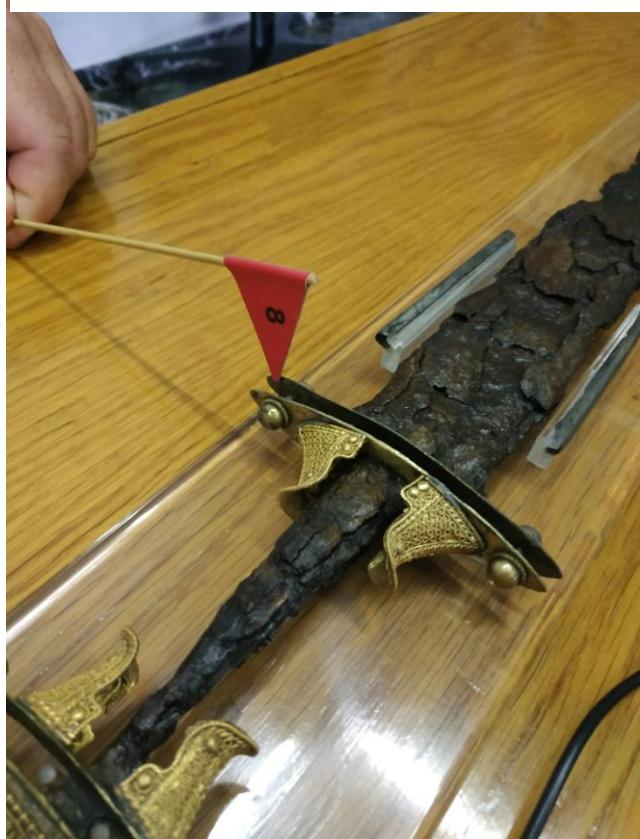
Annex photography 89. Sword 380, measurement 4.



Annex photography 96. Sword 380, measurement 5.



Annex photography 95. Sword 380, measurement 6.



Annex photography 94. Sword 380, measurement 8.



Annex photography 93. Sword 36.



Annex photography 98. Sword 36, measurement 1.



Annex photography 97. Sword 36, measurement 2.



Annex photography 99. Sword 36, measurement 4.



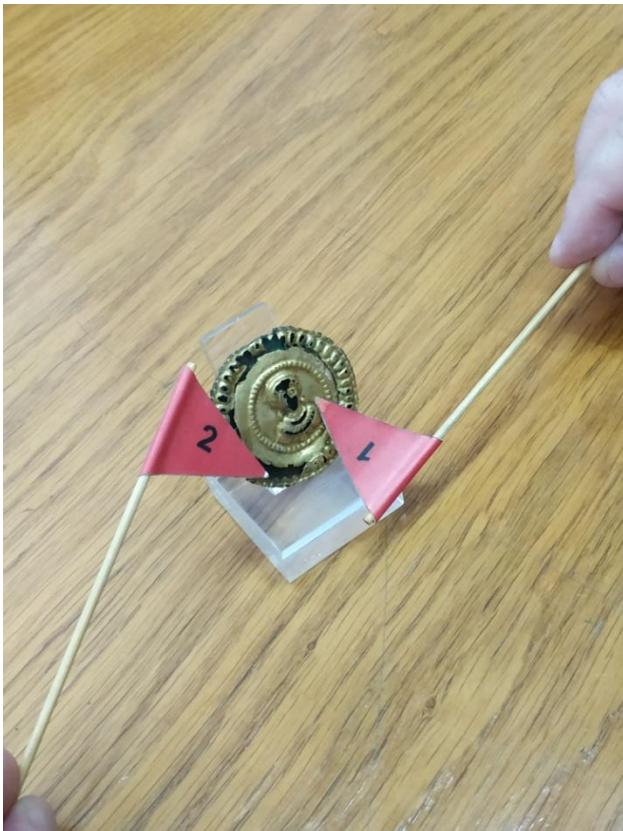
Annex photography Sword 36, measurement 3.



Annex photography 103. Sword 36, measurement 5.



Annex photography 102. Sword 36, measurement 6.



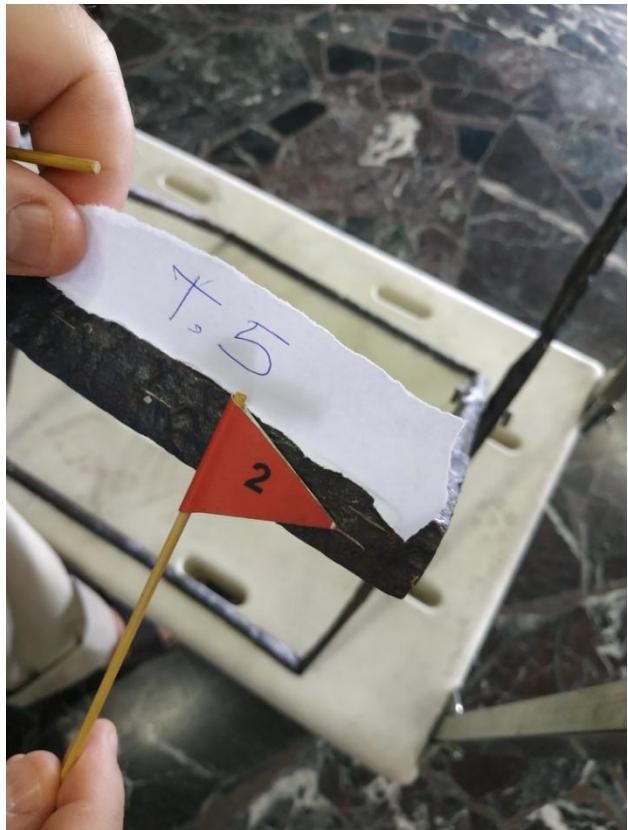
Annex photography 101. Medallion, measurements 1 and 2.



Annex photography 100. Bronze strop, measurement 2.



Annex photography 107. Stool from tomb 5, measurement 1.



Annex photography 106. Stool from tomb 5, measurement 2.



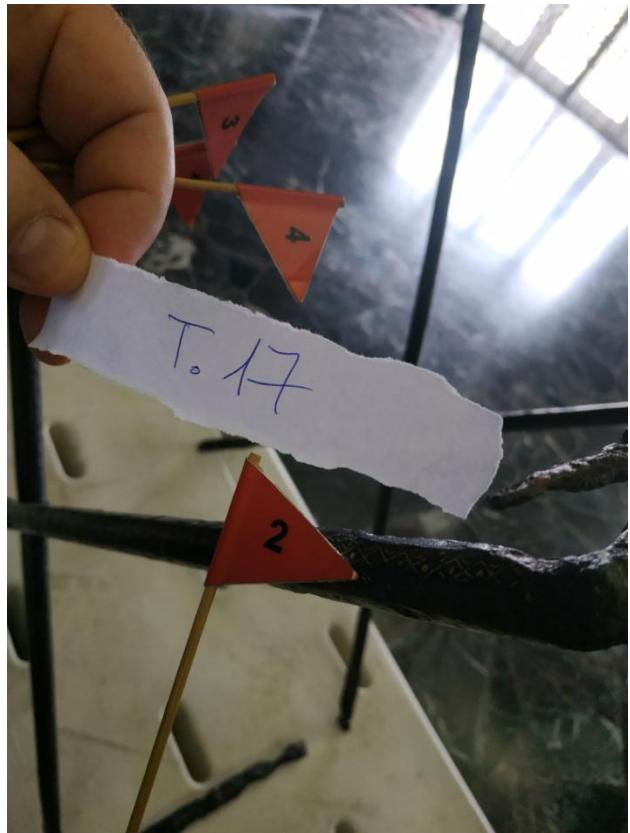
Annex photography 105. Stool from tomb 5, measurement 3.



Annex photography 104. Stool from tomb 5.



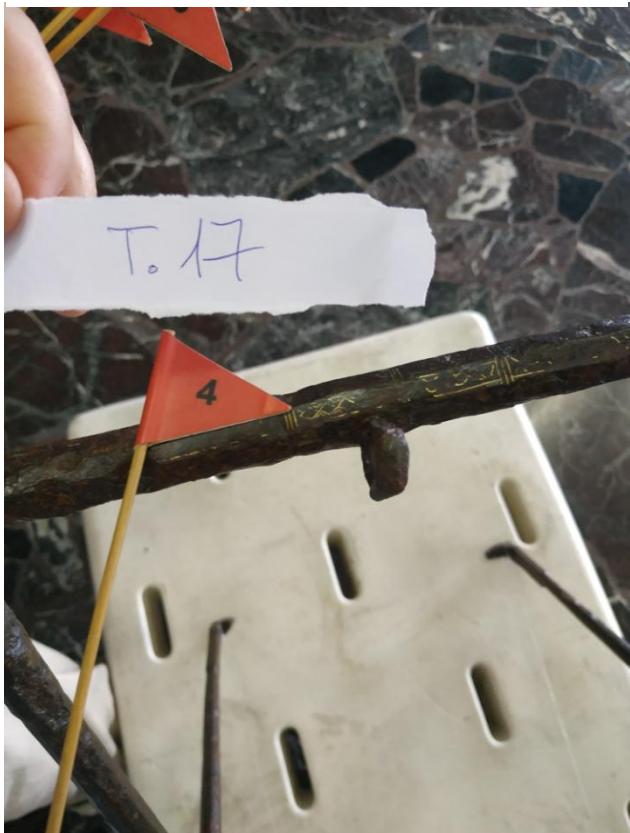
Annex photography 111. Stool from tomb 17, measurement 1.



Annex photography 110. Stool from tomb 17, measurement 2.



Annex photography 109. Stool from tomb 17, measurement 3.



Annex photography 108. Stool from tomb 17, measurement 4.



Annex photography 115. Stool from tomb 17.



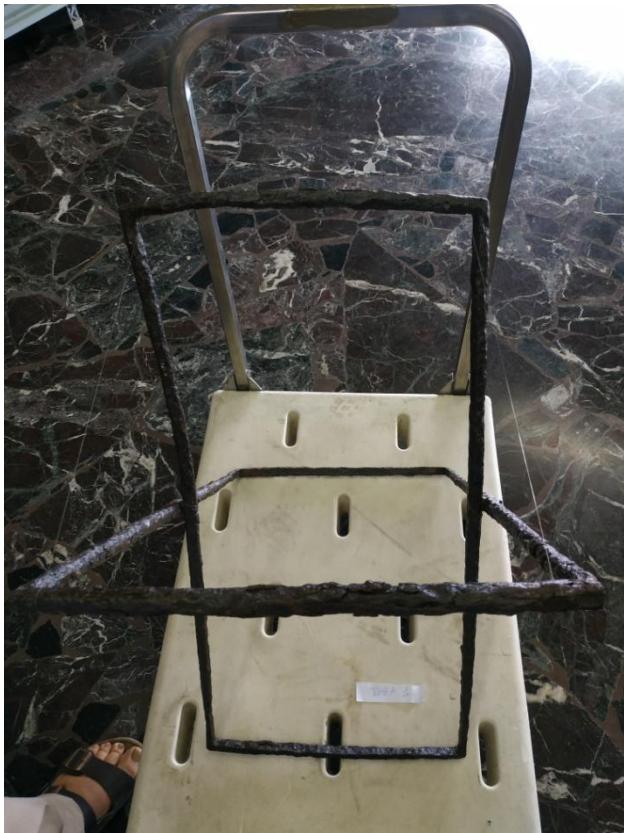
Annex photography 114. Stool from tomb 100, measurement 1.



Annex photography 113. Stool from tomb 1, measurement 1.



Annex photography 112. Stool from tomb 1, measurement 1.



Annex photography 116. Stool from tomb 1.