# SYNTHESIS OF INGEMMET ANALYSIS REPORTS

November 2018

Lilian Cristofol Engineer in Materials Science / Characterization

Translation: Catherine Rayer

## SUMMARY

Introduction	1
1. Lot N°1 - sample 01 : rock	2
1.1 Presentation	2
1.2 Summary of Characterization Results in MEB / EDS	2
1.3 Discussion of results in the study context	3
2. Lot N°1 - sample 02 : Josephina's metal implant	4
2.1 Presentation	4
2.2 Summary of Characterization results in MEB / EDS	4
2.3 Discussion of results in the study context	5
3. Lot N° 1 - sample 03 : Annular Metal Implant of a three-fingered hand	6
3.1 Presentation	
3.2 Summary of Characterization results in MEB / EDS	6
3.3 Discussion of results in the study context	7
4. Lot N°1 - sample 04 : Metal implant of a three-fingered hand	8
4.1 Presentation	
4.2 Summary of Characterization results in MEB / EDS	8
4.3 Discussion of results in the study context	9
5. Lot N° 2 - sample 01 : Metal implant of Alberto's hip	. 10
5.1 Presentation	. 10
5.2 Summary of Characterization results in MEB / EDS	. 10
5.3 Discussion of results in the study context	. 11
6. Lot N°2 - sample 02 : Metal object into a shaped-cross form	. 12
6.1 Presentation	. 12
6.2 Summary of Characterization results in MEB / EDS	12
6.3 Discussion of results in the study context	13
7. Additional comments	. 14

#### Introduction

The purpose of this document is to summarize the results of the basic micro-analyses conducted in EDS (Energy Dispersive Spectroscopy) and conducted by INGEMMET on six samples submitted to them by the INKARI-CUSCO Institute.

The purpose here is to be able to evaluate, within the limits of the results obtained, the relevance of authenticity of the objects from which the analyzed samples were extracted, placed in the study context, namely possibly Pre-Columbian era objects.

For each sample, in this synthesis, after presentation, a condensed summary of the notable basic chemical characteristics measured in EDS micro-analyses is presented, followed by a brief discussion in the study context.

For full details of the analyzes of each sample presented here, refer to the report "ANALYSIS OF INGEMMET REPORTS".

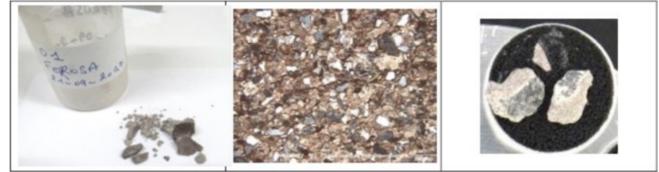
Good reading.

#### LC

#### 1. Lot N°1 - Sample 01 : rock

#### **1.1 Presentation**

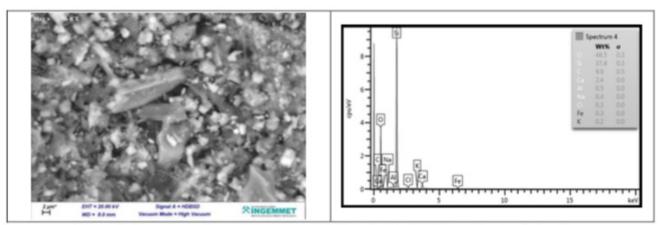
Sample 01 - Lot 1 is composed of several pieces of rock, three of which are taken for analysis :



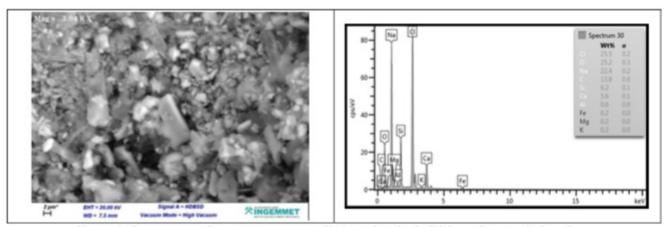
Sample 01 - Lot 1 (left) - Photos with a binocular magnifying glass of sample 01 (in the middle) - Sample 01 fragments on contact for analysis (right).

#### 1.2 Summary of Characterization Results in MED / EDS

For this sample, 3 zones are analyzed on the sampled fragments, for a total of 39 spectras carried out, with for each one a measurement of the basic mass concentrations.

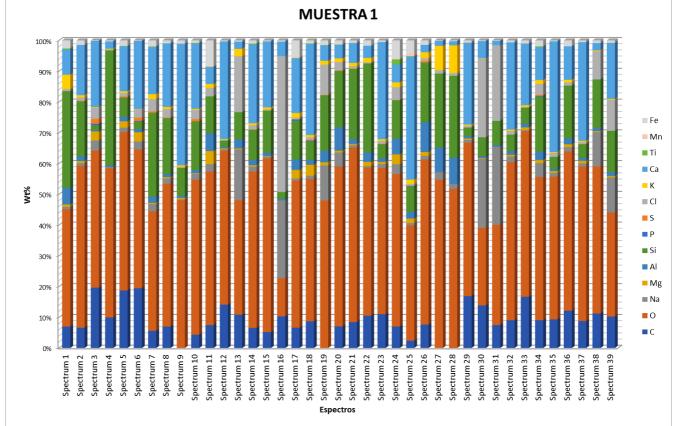


MEB image (left) and EDS acquisition spectrum (right) from sample 01 - Lot 1. The spectrum shows a siliceous chemical signature of the sample.



MEB image (left) and EDS acquisition spectrum (right) from sample 01 - Lot 1. The spectrum shows a chemical signature correlated with a halite evaporite type.

In conclusion, the set of EDS mass concentration measurements for each zone is compiled in the following summary bar chart :



Summary chart of EDS mass concentration measurements for sample 01 - Lot 1. Several composition profiles similar to mineral chemical composition profiles are noted.

In synthesis, this sample would be a sandstone-type sedimentary rock, as shown by its basic composition, which indicates the possible presence - besides quartz grains - of characteristic minerals of igneous rocks, cemented by a carbonate matrix.

The presence of micro-fossil organisms remains are noted for this sample, whose exact nature has to be determined, although it is very likely diatoms (morphology, chemistry). Furthermore, it is difficult to determine whether these micro-fossil remains are inherent to the sample or an exogenous contribution.

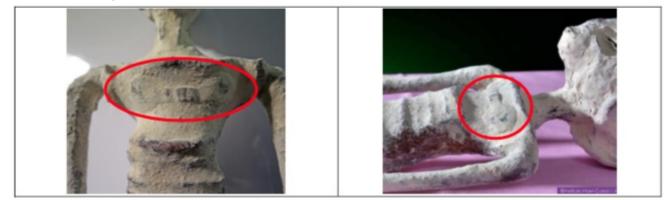
#### 1.3 Discussion of the results in the study context

In the study context, this sample matches perfectly with Peruvian geology, however a substantial information without much more investigation is difficult to obtain : the source deposit may be possible to identify, however at geological scales (several hundreds / thousands of kilometers), this would have little relevance for the precise location of a place, for example.

#### 2. Lot N°1 - Sample 02 : Josephina's metallic implant

#### 2.1 Presentation

This sample consists of several fragments extracted from Josephina's pectoral implant, three of which are taken for analysis.

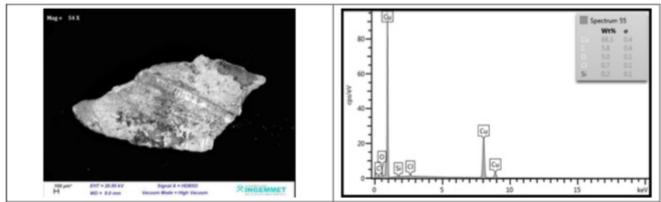


Photos of the organism in which the source object of sample 02 - Lot 1 (surrounded in red) is implanted.

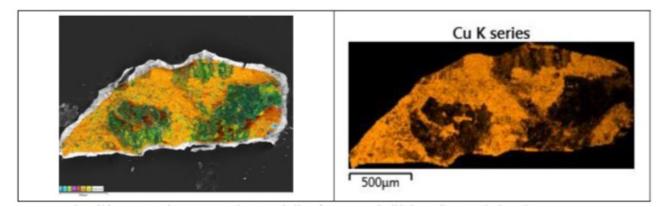
#### 2.2 Summary of Characterization Results in MEB / EDS

For this sample, 5 zones are analyzed on the collected fragments, for a total of 70 spectras carried out, with a measurement of the basic mass concentrations for each one.

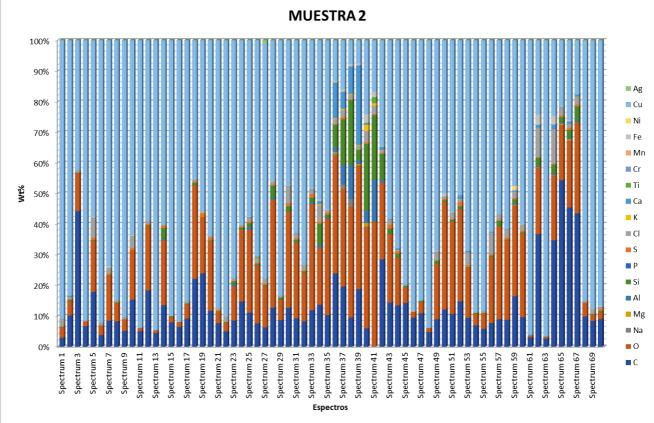
In addition, an EDS mapping of carbon, oxygen, silicon, sulfur, chlorine, calcium and copper elements is carried out on one of the three fragments.



MEB image (left) and EDS acquisition spectrum (right) from sample 02 - Lot 1. This spectrum shows a full of copper Cu zone of the analyzed fragment.



Basic mapping (C, O, Si, S, Cl, Ca, Cu) of a fragment from sample 02 - Lot 1.



In conclusion, the set of mass concentration measurements made in EDS for each zone is compiled in the following summary bar chart :

Summary chart of mass concentration measurements, made in EDS on sample 01 - Lot 1. The sample is mostly made of copper. The presence of minerals is also observed.

This sample is mainly composed of metallic copper (85% by mass on average for the purest zones), whose the partial deterioration is observed through the formation of oxidation product such as cuprite or tenorite, or of alteration mineral such as atacamite or brochantite. This copper also contains impurities (iron, sulfur, ...), which could be characteristic of the ore from which the copper used for the design of the implant was extracted. These impurities could also give indications as to the extraction method used, in particular through the presence of sulfur (extraction possible from chalcocite-type sulfides). Sulfur and chlorine levels characteristic of the alteration products observed, such as atacamite, may indicate an alteration occurring in a place with an atmosphere full of chlorine and / or sulfur, such as a coastal or volcanic environment, or in a polluted environment (factory, gas exhaust).

This sample also shows a mineral chemical signature (carbonate, silicates, salts), which may be the consequence of an exogenous supply (gangue wrapping the implant) or crystallizations in situ under certain conditions.

#### 2.3 Discussion of the results in the study context

In the study context, this sample matches perfectly in chemical composition terms (copper whose purity -perfectible- is fully compatible with the refining methods known and accessible at the time) with a Pre-Columbian object. Furthermore, the oxidation and weathering products observed involve slow processes and thus contribute to corroborating the age of the object from which the sample is extracted, provided that it has not been exposed to a very aggressive environment (atmospheric pollution). The mineral signature observed would also go in this way, in case of crystallization in situ (slow process).

#### 3. Lot No 1 - Sample 03: Annular Metal Implant of a Three-fingered Hand

#### 3.1 Presentation

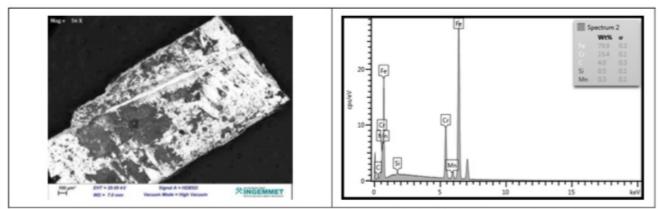
This sample is a fragment extracted from an annular implant taken from the three-fingered hand given by Paul Ronceros to the INKARI-CUSCO Institute.



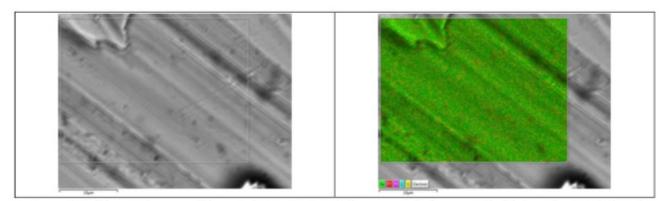
Photos of the three-fingered hand where the source object of sample 03- Lot 1 (surrounded in red) is implanted.

### 3.2 Summary of Characterization Results in MEB / EDS

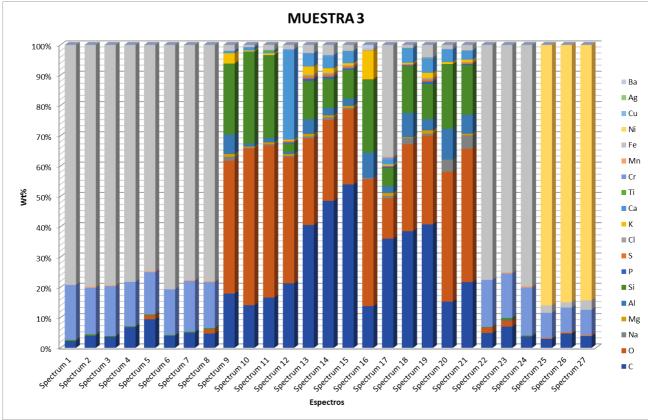
For this sample, 4 zones are analyzed on the fragment, for a total of 27 spectras carried out, with a measurement of the basic mass concentrations for each one. Elementary mapping is also performed on an area of this fragment.



MEB image (left) and EDS acquisition spectrum (right) from sample 03 - Lot 1. This spectrum shows a zone full of iron Fe and chromium Cr, as well as presence of manganese Mn.



Basic mapping with elements (C, O, Ši, Cr, Fe) of an area of the fragment of the sample 03 - Lot 1. Chromium is localized punctually, in form of small inclusions.



In conclusion, the set of mass concentration measurements carried out in EDS for each zone is compiled in the following summary bar chart :

This sample is composed of an iron-carbon alloy (iron average 78% for carbon 5%) full of chromium (average 16%), however the EDS analysis does not make it possible to decide between a steel (stainless) or a cast iron (white). If this alloy does not contain any nickel in its intrinsic composition, it is detected on the other hand very locally on one of the analysis zones in an almost pure concentration (average 85%), which would indicate the existence of a nickel passivation layer for this sample (hypothesis to be considered apart from a possible exogenous contribution, with pollution by the tools used for the extraction, for example). We would have therefore for this sample a steel or a cast iron, perhaps covered on the surface with a layer of nickel, possibly deposited by electroplating (chemical or electrolytic bath).

A mineral chemical signature is also observed for this sample, which is probably of exogenous origin (mineral gangue).

#### 3.3 Discussion of the results in the study context

In the study context (apart from the considerations of basic compositions : steel or cast iron, nickel electroplating, etc.), the biggest "problem" with this sample is that, until now, none object made of iron base alloy is existing among the Pre-Columbian manufactured objects listed.

The main reason is purely technical : civilizations of that time did not master the required techniques for extraction and working iron.

The only alternative to explain this "anomaly" in the hypothesis of an object developed by a Pre-Columbian civilization would be the conception of the source object from which the sample is extracted, from native iron of meteoritic origin (observed in other ancient civilizations), with equivalent chemical composition.

Summary chart of mass concentration measurements in EDS on sample 03 - Lot 1. Concentrations of iron, chromium, as well as elemental mineral signature and nickel are confined to a single zone.

#### 4. Lot N°1 - Sample 04 : Metal implant of a three-fingered hand

#### 4.1 Presentation

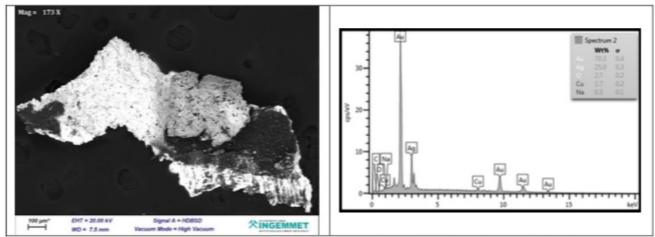
This sample is composed of several fragments extracted from an implant (disk-shaped) of one of the three-fingered hands. Three fragments are taken from this sample for analysis.



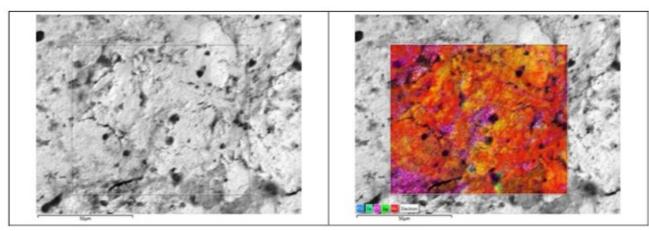
Photos of the metal implant from which sample 04 of Lot 1 is extracted.

#### 4.2 Summary of Characterization Results in MEB / EDS

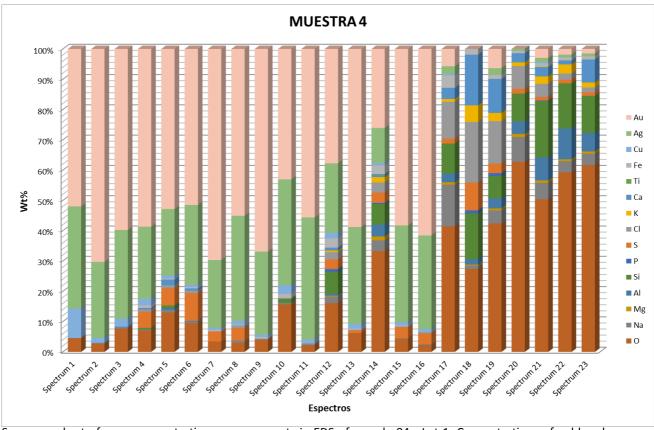
For this sample, 3 zones are studied on the sampled fragments, for a total of 23 spectras carried out, with a measurement of the basic mass concentrations for each one. Basic mapping is also carried out.



MEB image (left) and EDS acquisition spectrum (right) from sample 04 - Lot 1. This spectrum shows a zone full of silver Ag and gold Au, as well as copper Cu presence.



EDS basic mapping of an area on sample 04 - Lot 1: O, Fe, Cu, Ag, Au elements.



In conclusion, the set of mass concentration measurements carried out in EDS for each zone is compiled in the following summary bar chart :

This sample consists mainly of a gold-silver alloy (and to a lesser extent, copper), in an average proportion of about 60% of gold for 30% of silver (about 10% of copper), except hypothesis on possible technique of manufacture of the source object (see below). We

can observe the presence of other elements such as iron in inclusion form referring to the basic cartography, this could be an indication on the source of the alloy used for the manufacture of the object from which the sample 04 is extracted :

It could be a native gold-silver alloy, which is compatible with Peruvian mineralogy.

It is also possible that a type of "depletion gilding" finishing technique was applied to the object, in that case, values of the mass concentrations measured would be only representative of the near surface of the sample and the alloy probably proportionally more full of copper (type tumbaga). Here we find also a mineral chemical signature (carbonates, silicates, salts), consequence of an exogenous contribution (gangue sample), or crystallizations in situ (slow processes under certain conditions that would show the antiquity of the source object).

#### 4.3 Discussion of the results in the study context

In the study context, this gold-silver alloy matches perfectly in terms of composition with alloys used at the Pre-Columbian era. Impurities presence such as iron in inclusion also goes in this way, plausible indicator of the use of a native gold-silver alloy for the design of the object, in the absence of completely mastered processes by the Pre-Columbian metallurgists for this type alloy development. It is also plausible that a finishing type "gilding by depletion "has been applied to the source object of the sample, whose alloy would then be a gold-silver-copper alloy, characteristic of many Pre-Columbian objects and significantly fuller of copper in mass proportion, the tumbaga.

Summary chart of mass concentration measurements in EDS of sample 04 - Lot 1. Concentrations of gold and silver almost constant are noted, as well as a mineral chemical signature.

#### 5. Lot N°2 - Sample 01: Alberto's hip implant.

#### 5.1 Presentation

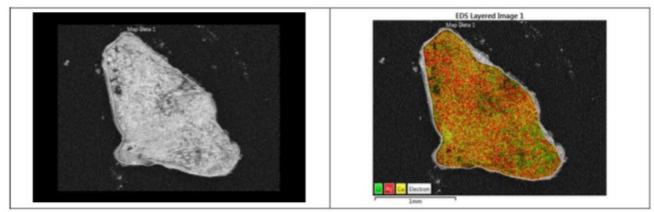
This sample is a fragment extracted from the Alberto's right hip implant, with a disc-shaped geometry referring to the photographs, X-rays and scanners available.



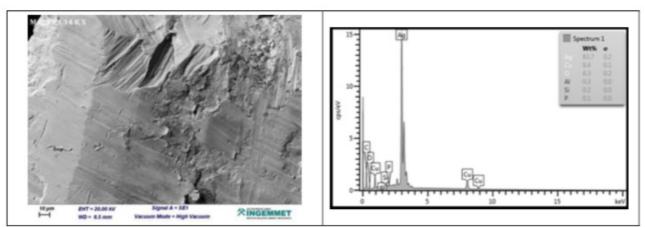
Photos of the organism where the source object of sample 01 - Lot 2 is located (circled in red).

#### 5.2 Summary of Characterization Results in MEB / EDS

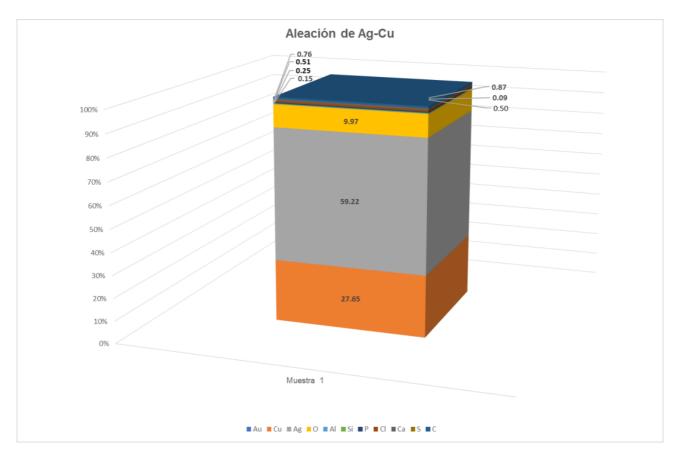
For this sample, 3 zones are studied on the fragment taken, for a total of 13 spectras, with a measurement of the basic mass concentrations for each one. Basic cartography (chlorine, copper, silver) is also presented in the introduction.



Basic mapping of sample 01 - Lot 2 (Cl, Cu, Ag). The distribution seems homogeneous on this scale.



MEB image (left) and EDS acquisition spectrum (right) from sample 01 - Lot 2. This spectrum shows a fulled zone of silver Ag, as well as presence of copper Cu.



In conclusion, the set of mass concentration measurements made in EDS for each zone is averaged and represented in the following summary diagram :

Summary diagram of mass concentration measurements carried out in EDS on Sample 01 - Lot 2. This sample consists mainly of a copper-silver alloy with traces of gold.

This sample consists of a copper-silver alloy, in a mass ratio ranging from about 10% copper to 90% silver to 10% silver to 80% copper, depending on the area analyzed. The average composition is estimated at about 25% of copper for 50% of silver. Several factors conditioned these disparities, the main ones being the alloy composition used for the manufacture of the piece (copper / silver ratio which will favor certain micro-structures), the method of manufacture used (casting, cold-heading, finishing of the part, etc.) together with the orientation of the sample during the analyzes (conditioned also by its extraction from the source object).

A mineral chemical signature is also noted (carbonates, silicates, salts), which could also be of organic origin because of the elements involved, especially with the phosphorus entering the bones composition.

#### 5.3 Discussion of results in the study context

In the study context, the composition of this sample matches perfectly with the typical compositions listed for Pre-Columbian era objects made from a copper-silver alloy. The observable micro-structure in topographic imaging seems to indicate that the source object was designed by stamping copper-silver alloy leaflets by cold-threshing, a technique also listed for many Pre-Columbian objects design. Concerning the implant bio-compatibility made from such an alloy, given the chemical activity of copper and silver depending on the context (pH, ...), the histological consequences on a living organism would be uncertain and requires the advice of a specialist.

### 6. Lot N°2 - Sample 02 : Metallic object in a cross-shaped form

#### 6.1 Presentation

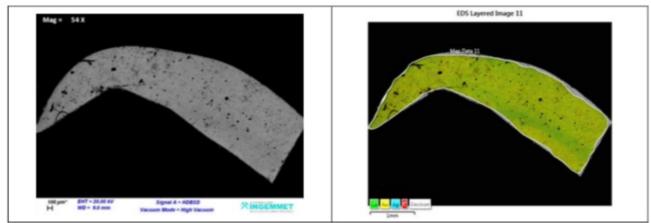
This sample consists of two fragments extracted from a metallic object in a cross-shaped form.



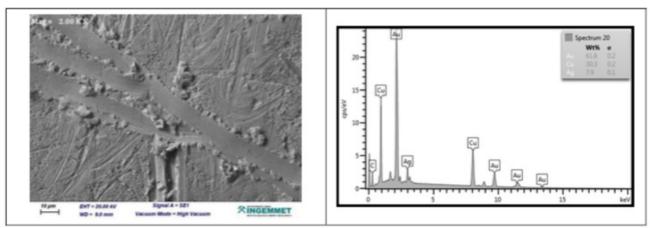
Source object of the sample 02 - Lot 2. We note the presence of possible minerals on the underside.

### 6.2 Summary of characterization results in MEB / EDS

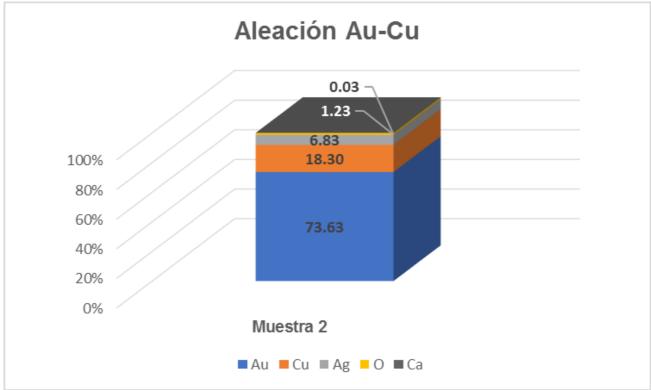
For this sample, 3 zones are studied on the fragment taken, for a total of 13 spectras, with a measurement of the basic mass concentrations for each one. Basic cartography (chlorine, copper, silver) is also presented in the introduction.



EDS elemental mapping of sample 02 - Lot 2 : carbon C, copper Cu, silver Ag and gold Au. At this scale we observe the spatial distribution of gold and copper which seems homogeneous.



MEB image (on the left) and EDS acquisition spectrum (on the right) from sample 02 - Lot 2. This spectrum shows a zone full of gold Au and copper Cu, as well as the presence of silver Ag. On the MEB image, we notice a porous surface texture on the unaltered zones.



In conclusion, the set of mass concentration measurements carried out in EDS for each zone of this sample is averaged and represented in the following summary diagram :

Summary diagram of the mass concentration measurements made in EDS on sample 02- Lot 2. This sample consists mainly of a gold-copper alloy, or tumbaga, with traces of silver.

This sample shows the chemical composition of a gold-copper alloy (with a little silver, 5 to 7% by mass on average), called tumbaga. The average gold / copper mass concentration ratio varies from approximately 8% copper, 85% gold to 26% copper and 65% gold, depending on the zones analyzed.

The analyzes also show a mineral chemical signature (carbonates, silicates), possibly induced by exogenous pollution.

#### 6.3 Discussion of results in the context of the study

In the study context, the gold-copper alloy (silver) composing this sample is characteristic of Pre-Columbian manufactured objects, it is called tumbaga. Many objects made from this alloy have been listed. It is also interesting to note that disparities in concentration measurement, are the consequence of a technique for finishing the objects elaborated in tumbaga, the gilding by impoverishment technique regularly implemented by the metallurgists of the Pre-Columbian era in order to embellish the conceived objects. The hypothesis of the application of this technique on this object is further corroborated by the porous surface texture of the unaltered zones, a porous texture observable on the MEB image produced in topographic contrast. This sample therefore has many clues attesting of a plausible ancient origin.

7. Additional remarks

LC

With regard to all the metallic samples, we can notice that none of them shows traces of arsenic (As), according to chemical micro-analyses in EDS.

This can be an important indicator as to the origin of the objects in the study context (Pre-Columbian era ) : arsenic, used for the first time by Moche culture, was sometimes added as a hardener in alloys, it can be found this way in several Pre-Columbian objects. It is also noted that this element may be present or not from the source ores used to develop the alloys, which may be a link as to the ores origins location used to design the alloys (Petersen G. Georg, William E Brooks, Mining and Metallurgy in Ancient Perú), together with the culture having designed the objects, in addition to the methods of extraction of minerals and manufacturing implemented.

The same observation can be made for the tin element Sn, for which no trace has been detected during the basic micro-analyses of the different metallic samples .

This metal, used for the bronze manufacture (copper-tin alloy), has not been used significantly by Pre-Columbian civilizations, from the end of the Moche culture / beginning of the Inca culture (that is about 1400 years ago), on the basis of the analyzes of chemical compositions available and performed on several artifacts dating from those eras (Petersen G. Georg, William E. Brooks, Mining and Metallurgy in Ancient Perú). Tin in alloys prior to this time is only present as impurity (source of ores used). This indicator, absence of tin in the various metal samples analyzed, can be a strong indicator as to the period of manufacture of the corresponding source objects in the study context (Pre-Columbian civilizations).