Compositional Studies of Ancient Copper
from Romanian Territories

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Abstract

Ancient copper objects from Romanian Territories have been analyzed by neutron activation analysis. A series of elements is determined: Au, Ag, As, Co, Cr, Fe, Hg, Ni, Zn, Sb, Sc, Se, Sn. Using mathematical dendograms some classifications and correlation have been established.
1 Introduction

The importance of analyses of archaeological objects is well known, the elemental composition coming to complete the knowledge on the archaeological objects: style, typology, place of discover, culture, dating, data got by pure historical arguments. Processing the compositional scheme of historic objects one can obtain the identification of the materials and different characterizations and classifications of the founds.

As concerns the ancient copper, the elemental structure shows first of all the bronzes: copper + arsenic, copper + antimony, copper + tin or incidentally copper + zinc. Trace elements could also help to the understanding of copper items: objects with closed elemental composition could be put in cultural or temporal synchronization. From the all the analytical methods for copper neutron activation analysis (NAA) seems to be the best single method ¹ offering enough analytical capabilities to provide a good data base. For provenance studies, which it is a very complicated problem the method to apply is the atomic mass spectroscopy: what it remained constant from the ores to the final metal objects is the ratio of lead, determined by the atomic mass spectroscopy: \( \frac{\text{Pb}^{208}}{\text{Pb}^{206}} \) and \( \frac{\text{Pb}^{207}}{\text{Pb}^{206}} \).²⁻⁵

In this paper we have analyzed by neutron activation analysis a lot of 41 Neolithic copper objects: axes, needles, daggers and others objects from the National History Museum of Transylvania, Cluj-Napoca, Romania. In Table 1 it is given the list of analyzed samples.
2 Experimental method of analysis

Sampling. First some corroded parts have been removed from the surface of copper objects, the corroded material having a totally different elemental composition from that of the body of the object. Then samples of 10-50 mg have been cut with a hard vidia knife from the object body and after that washed with different solvents: acetone, benzene, ether to avoid the impurities from the surface of the item and the protective varnish, added in the museum.

Irradiation of medium periods. Samples have been put in polyethylene foils and irradiated at the rabbit system of the nuclear reactor VVR-S, from NIPNE Magurele, Bucharest at the flux of \( \approx 1.2 \times 10^{12} \) neutrons/cm\(^2\)·sec, for 30 minutes. Copper being in majority it was strongly activated so that the induced radioactivity in the samples could be measured only after 4-5 days. Natural cooper has 2 isotopes: Cu\(^{63}\) and Cu\(^{65}\) which by the reaction \((n, \gamma)\) give the radioisotopes Cu\(^{64}\) \(T_{1/2} = 12.74\) h and Cu\(^{66}\) with \(T_{1/2} = 5.10\) min. After a cooling time of 4-5 days, in the gamma spectra of the samples, the activity coming from the photopeak of 1345.8 keV (0.0048) of Cu\(^{64}\) is small enough and permits to remark other elements, present in the cooper matrix. The samples have been measured 1000 s at a spectrometric chain using a Ge(Li) detector of 135 cm\(^3\) and an analyzer of 4096 channels coupled at a PC. The system gave a resolution of 2.7 keV at 1.33 keV (Co\(^{60}\)). We observed the elements: Au, As, Cu and Sb.

Long time irradiation: The samples of copper have been wrapped in aluminum foil and put it in a quartz phial together with metallic spectroscopic pure standards, copper and nickel and irradiated at the vertical chain of the reactor, at a flux of \( \approx 10^{13} \) neutrons/cm\(^2\)·sec, for a period of time of 40 h. After a cooling time of 2 weeks, we measured the \(\gamma\) activity of the samples at the same spectrometric chain, for 3000 s. We have determined the following elements:
Sb, Ag, Co, Cr, Fe, Hg, Ni, Se, Sn.

**Cobalt.** Cobalt was determined in the copper object using the isotope Co\(^{60}\) got in the reaction: \(\text{Co}^{59}(\text{n}, \gamma)\text{Co}^{60}\). Co\(^{60}\) is also produced by the reaction: \(\text{Cu}^{63}(\text{n}, \alpha)\text{Co}^{60}\) which is important enough in this situation, when the element copper is the major element (\(\approx 99\%\)). So that \(\text{C}_{\text{Co}} = \text{C}_{\text{total}} - \text{C}_{\text{Cu}}\), where \(\text{C}\) is the concentration. Another correction made in the calculus of the cobalt concentration in the copper samples is that the used cooper standard contains also traces of cobalt. It was determined that a standard of pure copper has a content of minimum 4 ppm of cobalt. Also in the gamma background of the experimental room it were observed the peaks at the cobalt energies of 1773.2 keV and 1332.5 keV; therefore from the respective photopeaks area it was subtracted the area given by the background, measured for the same period of time as the sample.

**Mercury** was determined from the \(\gamma\) ray of 279.2 keV and intensity (81.5\%) of Hg\(^{203}\) with \(T_{1/2}=46.60\)d. This ray is interposed with the \(\gamma\) ray from Se\(^{75}\), of 279.5 keV, and intensity 0.25. Therefore the contribution of the mercury must be extracted from the peak of peak of 279 keV: \(N_{Hg^{203}} = N_{\text{total}279\text{keV}} - N_{Se^{75}}\), where \(N_{Hg^{203}}\) is the counting rate in the peak of 279 keV, given by mercury contribution. \(N_{\text{total}279\text{keV}}\) is the total counting rate in the peak of 279 keV \(N_{Se^{75}}\) is the counting rate in the peak of 279 keV, due to the selenium presence. It was used as reference the selenium peak from the energy 264.7 keV of intensity of 0.5658:

\[
\epsilon_{264\text{keV}} \cdot s_{264\text{keV} Se^{75}} \cdot N_{264\text{keV} V Se^{75}} = \epsilon_{279\text{keV}} \cdot s_{279\text{keV} Se^{75}} \cdot N_{279\text{keV} V Se^{75}}
\]

where: \(\epsilon_{264\text{keV}}\) is the efficiency of the detector from 264 keV, \(\epsilon_{279\text{keV}}\), is the efficiency at the energy of 279 keV, \(s_{264\text{keV} V Se^{75}}\), the intensity of the line of 264 keV of Se\(^{75}\), \(s_{279\text{keV} V Se^{75}}\), the intensity of the line of 279 keV of Se\(^{75}\)

**Nickel.** The concentration of nickel was measured by the isotope Co\(^{58}\) (\(T_{1/2}=71.3\) d). Nickel was a exception by the fact that it was determined by the reaction
Ni\textsuperscript{58}(n, p)Co\textsuperscript{58}, unlike the other elements determined by the reaction \((n, \gamma)\).

In the Table 2 are given the results of activation analysis for the Prehistoric copper object, from the National Museum of History from Cluj-Napoca. The concentrations are given in ppm, and when an element was determined in a quantity larger than 10000 ppm, its concentrations was expressed in percents, using the notation of \%. The measured errors were the statistical errors and were in mean of \(<10\%\).

3 Results and discussions

First of all one distinguishes from the Table 2 the samples of copper which contain also other elements than copper, in concentrations higher of 1\%, the copper based-alloys. So that we can distinguish the items:

LG10 1.11\% Sn, LG14 2.61\% As, LG15 1.68\% As, LG22 2.82\% Sn + 2.07\% Zn, LG41 1.53\% As.

**Arsenical-copper** The presence of arsenic in the samples of ancient copper is shown in Fig. 1, the analyzed objects could be structured on 4 levels of concentrations:

- concentrations of As \(<100\text{ ppm}: \) LG1, LG9, LG11, LG12, LG13, LG24, LG31
- concentrations between 100 ppm and 1000 ppm: LG5, LG8, LG21
- concentrations between 1000 ppm and 1\%: LG10, LG16, LG22, LG37, LG38, LG39
- concentrations \(>1\%: \) LG14, LG15 and LG41

The concentrations of arsenic could be interpreted in the sense that the samples are made manufactured by the ore of type reduced, like sulfurs and not deliberated alloyed. The specified ores needs a higher degree of metallurgy level to produce the metal. The fact that the items LG14, LG15, LG41 which contain
arsenic in concentrations higher of 1% to be manufactured from a reduced ore type is possible with a probability of 3%\(^6\), in the rest of chances the arsenic being added intentionally, consciously to obtain some artifacts of copper with superior properties than those items of pure copper, the arsenical bronzes\(^7,8\). As concerning the samples LG16 and LG37, LG38, LG39 with a content of arsenic of about 1000 ppm, one can do the same discussion but the probability of bringing in close association of the object to a ore of reduced type is in this case 13%. Zvi Goffer\(^9\) gives another interpretation and method to establish the affiliation of studied objects at the type of arsenic-copper. The Sb and Ag contents of some arsenical copper objects analyzed by Zvi Goffer are directly proportional to the As content. As, Sb and Ag are associated with Cu only in sulfoarsenate ores. The presence of these elements is thus indicative that sulfoarsenate ores must have been deliberately added during the manufacturing process. For the analyzed Neolithic copper from Transylvanian territories we have represented in Fig. 2 the two dependencies indicated in ref.7: \(C_{\text{As}} - C_{\text{Ag}}\) and \(C_{\text{As}} - C_{\text{Sb}}\), considering only the objects with \(C_{\text{As}} > 1\%\). One can observe that the samples LG14 and LG15 respect the proportionality of arsenic-silver, respectively arsenic-antimony, and one could make the interpretation that in the process of the manufacturing of the axes LG14, Dragu and LG15 Hoghiz, it was added a sulfoarseniate ore deliberately, to obtain axes of higher durity at a smaller temperature. The dagger LG41 does not respect this rule.

**The axe LG10 and the chisel LG22** have about the same composition, they are tin-copper alloys, which are a superior type of bronze and they have also relatively the same values of concentrations for the others elements: Au, As and Sb, Ag and Ni. Although the axe LG10 contains concentrations of percents of zinc, the value \(C_{\text{Zn}} = 1310\) ppm is also a big concentration in comparison with the values of background for zinc, of the others copper objects, so that one
could say that also by the element Zn LG10 is synchronized with the object LG22, which contains $C_{Zn} = 2.07\%$. Zinc is reported to be present in the copper object only accidentally. In the literature are given only some metallic founds of zinc$^7$: at Dordos, also in Transylvania. The clusterisation of the 2 objects: axe LG10 and chisel LG22 on all the diagrams points out their strong association and indicates their possible common origin and manufacturing place.

As concerns the axes from Valcele, the request was to identify that the axes LG32 and LG33 belong to the Valcele hoard or are fakes, having a very different macroscopic aspect: these items have a dark brown color but the other axes from the same collection are covered by a large layer of light blue-greenish copper oxides. The elemental analyses show that the samples LG26, LG27, LG28, LG29, LG31, LG32, LG33 form a homogeneous group. The axe LG31 only contains a concentration of 74 ppm. The axes LG32 and LG33 exhibit values of concentrations very closed of the others axes from Valcele. It could answer in the affirmative that the axes LG32 and LG33 are authentics.

The 2 objects found in Ariusd the Needle LG40 and the Dagger LG41 are seemingly very different: the Needle is an unalloyed copper and the Dagger is prominent by its content of arsenic $C_{As} = 1.53\%$, already discussed above, and also by its high concentration of Ag: $C_{Ag} = 6230$ ppm. However the other trace elements: Sb, Se, Hg, Ni and especially Fe and Zn suggests the close association of these 2 objects. One could interpret the results of analyses for the Ariusd objects as follows: the needle and the Dagger seems to be manufactured from the same ore but it was added intentionally another ore with arsenic and silver.

The traces of Au and Ag in copper objects were represented in Fig. 3 It is known that there is a proportionality of the silver and gold concentrations both in the ”sulfuric” and ”oxides” copper ores$^{2,4}$ Our samples follow relatively well the proportionality $C_{Ag}$ with $C_{Au}$ with the exception of the dagger from Ariusd.
Pendantivs ”Large spiral”, ”Middle spiral” and ”Small spiral” found in Cheile Aiudului, the samples LG37, LG38, LG39 present a similar structure: they are arsenic-bronzes and they are well grouped on the elements: Sb, Ag and Ni.

Sb presents random values of concentrations in the frame of analyzed samples. It is known that the antimony it is used to replace the tin. ¹⁰,¹¹ One can notice for Sb the high values of concentration for the pendantivs from Cheile Aiudului, the samples LG37, LG38, LG39, and also for the objects LG10 and LG22.

**Dendograms.** A study of the correlation of all the analyzed objects from the point of view of the whole compositional scheme was done using the dendogram tools. ¹²,¹³ In Fig. 4 it is represented the dendogram of interdependence between the elements in the cooper samples. It comes in to view the levels of concentrations: I: Au is correlated with Ag the probability P=19.5%, II: Au, Ag, As and Ni, with P=17.0%, III: Au, Ag, As, Ni and Sb with P=14.6%. In Fig. 5 it is shown the dendogram of correlation between all the analyzed copper objects from the point of view of all concentrations. These mathematical clustering must be seen cautiously and the obtained associations must be interpreted only together with historic data: place of found, dating, culture, typology ¹⁴.
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FIGURE CAPTIONS

Fig. 1 Diagram of As content in the copper objects, by NAA

Fig. 2 Concentrations of As versus the concentrations of Sb or Ag for different ancient copper items, by NAA

Fig. 3 Concentration of Ag versus the concentration of Au for different ancient copper objects, by NAA

Fig. 4 Dendogram of interdependence between elements in ancient copper objects

Fig. 5 Dendogram of correlation of the copper objects
TABLE CAPTIONS

Table 1 List of analyzed copper objects from History Museum of Transylvania, Cluj-Napoca, by NAA

Table 2 Concentration of elements in different Neolithic copper objects, by NAA. The concentrations are expressed in ppm and for the values above 10 000 ppm, the results are given in %. The errors are generally <7%.
| Sample | Copper object | Nr. inv. | Place of found       |
|--------|---------------|----------|----------------------|
| LG1    | Axe           | P.837    | Ugru'tiu             |
| LG2    | Axe-Pick-axe  | P.838    | Mico'slaca           |
| LG3    | Axe-Pick-axe  | P.839    | Cetatea de Balt'a    |
| LG4    | Pick-axe      | P.840    | 'Sinc'ai-Pogani      |
| LG5    | Battle Pick-axe| P.841 | unknown              |
| LG6    | Axe           | P.842    | unknown              |
| LG7    | Axe-Pick-axe  | P.843    | Lacu, jud. Cluj      |
| LG8    | Axe-Pick-axe  | P.844    | region Cheile Turzii |
| LG9    | Axe-Pick-axe  | P.845    | unknown              |
| LG10   | Axe           | P.846    | unknown              |
| LG11   | Axe-Pick-axe  | P.847    | unknown              |
| LG12   | Axe           | P.848    | col. Z. Torma        |
| LG13   | Pick-axe      | P.850    | unknown              |
| LG14   | Flat axe      | P.851    | Dragu                |
| LG15   | Chisel        | P.852    | Cold'au, zona Dej    |
| LG16   | Chisel        | P.853    | Hoghiz               |
| LG17   | Battle axe    | P.854    | P'aduren(=Be'seneu)  |
| LG18   | Pick-axe      | P.855    | Col. Z. Torma        |
| LG19   | Axe           | P.856    | unknown              |
| LG20   | Axe           | P.857    | unknown              |
| LG21   | Axe           | P.859    | Col. Z. Torma        |
| LG22   | Flat Chisel   | I 2978   | unknown              |
| LG23   | Bracelet      | P.? V 9558 | Col. Z. Torma  |
| LG24   | Bracelet      | P.860    | Cata, C. Petre'sti   |
| LG25   | Needle        | Balomir  |                      |
| LG26   | Axe           | In 11    | Văclele              |
| LG27   | Axe           | 10470    | Văclele              |
| LG28   | Axe           | 10412    | Văclele              |
| LG29   | Axe           | I 10469  | Văclele              |
| LG30   | Bead          | 8445     | Decea Mure'sului     |
| LG31   | Axe           |          | Văclele              |
| LG32   | Axe           |          | Văclele              |
| LG33   | Axe           |          | Văclele              |
| LG34   | Small needle  | VI 1399=JUNGH | Decea Mure'sului   |
| LG35   | Bead          | VI 1293  | Decea Mure'sului     |
| LG36   | Necklace VI   | 1300=JUNGH 9024 | Decea Mure'sului   |
| LG37   | Large spiral  |          | Cheile Aiudului      |
| LG38   | Middle spiral |          | Cheile Aiudului      |
| LG39   | Small spiral  |          | Cheile Aiudului      |
| LG40   | Needle        | III 1789 | Ariu’sd, Covasna     |
| LG41   | Dagger        | III 378  | Ariu’sd, Covasna     |
Table 2.

| Sample | Au | As | Sb | Se | Hg | Cr | Ag | Ni | Sc | Fe | Zn | Co | Ta | Sn |
|--------|----|----|----|----|----|----|----|----|----|----|----|----|----|----|
| LG1    | 0.52 | 22 | 8.63 | 7.6 | 2.74 | <2.4 | 32.7 | 99 | 0.039 | 150 | 19.2 | – | 0.13 | 180 |
| LG2    | <0.54 | – | 2.12 | <2.4 | 1.28 | – | – | 8.27 | 68 | – | – | 15.8 | 9.3 | 0.15 | – |
| LG3    | 0.81 | – | 11.4 | 24 | – | – | 47.4 | 40 | – | – | 6.2 | 1.18 | – | – |
| LG4    | 0.69 | – | 4.8 | 1.5 | 2.35 | – | – | 21.6 | 35.5 | 0.021 | 130 | 14.3 | – | 100 |
| LG5    | 1.9 | 110 | 2.94 | 2.9 | 2.49 | – | 8.04 | 40.7 | 0.019 | 22 | 12 | – | 0.2 | 60 |
| LG6    | 0.62 | – | 43.5 | 77.4 | 2.39 | – | 119 | 48 | – | – | 39.4 | – | 2.6 | – |
| LG7    | 8.1 | – | 11.9 | <5.1 | 1.9 | – | 38.2 | 100 | – | – | 14 | – | – | 403 |
| LG8    | 0.66 | 127 | 6.72 | <5.1 | <1.7 | – | 37.5 | 42 | 0.031 | 500 | 35.4 | – | – | 390 |
| LG9    | 0.39 | 54 | 3.88 | 2 | 0.43 | – | 31.6 | 41.4 | 0.087 | 370 | 20.7 | – | – | 96 |
| LG10   | 12.7 | 2820 | 1220 | 24 | 0.8 | – | 913 | 370 | 0.198 | – | 10 | 410 | – | 1.11% |
| LG11   | 0.69 | 14 | 4.34 | 3.6 | – | – | 16.4 | 33 | 0.014 | 340 | 18 | 5.2 | 0.29 | – |
| LG12   | <0.75 | 24 | 1.24 | 2.5 | – | – | 5.4 | 47.1 | 0.03 | – | 13 | 6.1 | 0.18 | – |
| LG13   | – | 48 | 1.76 | – | 1.9 | 5.5 | 3.5 | 50 | 0.05 | 380 | 23 | 1.6 | 0.4 | – |
| LG14   | 4.4 | 2.61% | 85.5 | 37.4 | – | – | 163 | 85 | 0.04 | 540 | 28 | – | – | – |
| LG15   | 3.4 | 1.68% | 20.5 | 48.8 | – | – | 54.9 | 68.9 | 0.02 | – | 19.3 | – | – | – |
| LG16   | 9.2 | 6270 | 10.4 | 8.4 | 1.9 | – | 378 | 23 | – | – | 28 | – | 0.87 | – |
| LG17   | – | – | 2.36 | 11.7 | – | – | 10 | 53 | – | – | 29 | 8.6 | 0.97 | – |
| LG18   | – | – | 0.72 | – | – | – | 4.8 | 49 | – | 400 | 25 | 16.7 | 0.25 | – |
| LG19   | <1.5 | 10.5 | 17 | – | – | – | 39.3 | 120 | 0.05 | – | 36 | 32.2 | 2.31 | – |
| LG20   | 27.8 | – | 5.18 | 89.5 | 1.96 | – | 94.6 | 184 | 0.04 | – | 21 | – | 1.37 | – |
| LG21   | 11.7 | 550 | 18.3 | 13.9 | 1.28 | – | 256 | – | – | – | 34 | 8.8 | 0.53 | – |
| LG22   | 10.9 | 1540 | 2040 | – | – | – | 925 | 1210 | – | – | 2.07% | 7.8 | – | 2.82% |
| LG23   | 8.8 | – | 3.36 | 19.6 | 1.55 | – | 381 | 47 | – | – | 22 | 27.6 | 2.2 | – |
| LG24   | 0.32 | 19 | 3.1 | 1.4 | 0.43 | <1.6 | 17.9 | 43.9 | – | – | 31.9 | – | 0.19 | – |
| LG25   | – | – | 8.53 | – | <4.7 | 11 | 39.8 | 110 | – | 660 | 30 | – | 1.9 | – |
| LG26   | 1.4 | – | 1.23 | 14.4 | – | – | 69.5 | 47.7 | 0.02 | – | 93 | – | 0.22 | – |
| LG27   | 0.8 | – | 3.55 | <4.6 | 0.67 | 4 | 31.7 | 56.5 | 0.1 | – | 13 | – | – | – |
| LG28   | 1.4 | – | 4.37 | 13.7 | 0.417 | – | 81.5 | 63.9 | 0.03 | – | 18 | 11.3 | 0.82 | – |
| LG29   | 3.2 | – | 4.76 | 22.7 | 1.57 | – | 114 | 64 | – | – | – | – | – | – |
| LG30   | 1 | – | 6.69 | 3.8 | 1.7 | – | 53.8 | 55.2 | 0.02 | – | 26 | – | 0.3 | – |
| LG31   | 3.4 | 74 | 5.88 | 11 | 2.4 | – | 60.2 | 51 | 0.03 | – | 25 | – | – | – |
| LG32   | 1.62 | – | 11.8 | 9.3 | 1.65 | – | 97.5 | 47.9 | 0.02 | – | 19 | – | – | – |
| LG33   | 0.9 | – | 2.87 | 9.1 | 0.96 | – | 63.5 | 24 | – | – | 15 | – | – | – |
| LG34   | <0.4 | – | 1.68 | 9.73 | 2.09 | – | 6.21 | 32.4 | 0.054 | 150 | 25.3 | – | – | – |
| LG35   | 2.76 | – | 14.2 | 2.3 | 0.926 | 1.3 | 27.1 | 67.7 | 0.115 | 1140 | 16.8 | – | 0.08 | 60 |
| LG36   | – | – | 3.63 | – | 8.8 | – | 23 | – | 0.15 | 1300 | 160 | – | 2 | – |
| LG37   | 25.9 | 3600 | 548 | 181 | 8.46 | – | 307 | 330 | – | – | – | 10 | – | – |
| LG38   | 7.5 | 3250 | 307 | 71.5 | 2.5 | – | 97.5 | 419 | 0.56 | – | 140 | 2 | – | – |
| LG39   | 4.7 | 1300 | 634 | – | 55 | – | 155 | 150 | 0.08 | – | 755 | 12 | – | – |
| LG40   | – | – | 2.58 | 17.9 | 8.5 | – | 4.82 | 173 | 8.03 | 690 | 39 | 2.7 | – | – |
| LG41   | <0.9 | 1.53% | 12.3 | 12 | 4.4 | – | 6230 | 92 | – | 850 | 68 | – | – | – |
Fig. 1
Fig. 2
Fig. 3
Fig. 5
