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INSTRUMENTAL ANALYSIS AND PROVENANCE OF ARCHAEOLOGICAL ARTIFACTS

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SUMMARY

An important aspect of archaeometric research - the application of scientific methods to problems of cultural history - is the determination of the provenance of archaeological artifacts, which demands the use of instrumental methods for multi-element analysis. Thereby the chemical composition and especially trace element contents are used as fingerprint, which can be compared with various production sites.

Relating to the most important groups of materials found in archaeological excavations - metals, pottery, and stone artifacts - examples of provenance determination in the eastern Mediterranean are described: a) Early metallurgy, b) late Bronze Age pottery, and c) prehistoric trade in obsidian.

1. INTRODUCTION

The analysis of archaeological artifacts has a history almost as long and illustrious as analytical chemistry itself. Indeed, the "father of analytical chemistry", Martin Heinrich Klaproth, renowned for his discovery of the element uranium, published analyses of Roman coins already at the end of the 18th century /1/. This first truly archaeometric work of a chemist is also the first account of a quantitative analysis of any copper alloy. Today, archaeometry is an interdisciplinary field between the natural sciences and the humanities. Broadly speaking, it can be described as the application of scientific methods to questions of cultural history. Of course, there exists a large variety of problems from various disciplines of the humanities, but most of them can be classified into areas such as prospection, dating, material identification, technology, provenance, and conservation. In the first two areas physical methods prevail, while analytical chemistry has yielded important contributions to the remaining areas. In the following I shall concentrate on the role of analytical chemistry for determination of the provenance of archaeological objects with some illustrative examples from the Aegean cultural region. In prehistory the Aegean is regarded as mediator between the early civilizations in the Near East and Central Europe. Therefore, contacts of the Aegean with Egypt or Anatolia on the one side and the Balkans and the Western Mediterranean on the other side are of special importance. Especially speed and direction of technological transfer and the extent of prehistoric trade are major issues of controversy. Archaeologists generally demonstrate cultural contacts and trade routes by typological similarities of artifacts. But unfortunately, many typological comparisons are open to discussion.

As early as in 1842 it was suggested that the chemical composition of an artifact could be used as additional criterion for classification /2/ and spread of cultures could be observed by the geographical distribution of certain materials. This method enabled Montelius /3/ to establish a sequence of principal cultural periods based on the material used for tools and weapons. Today we still use the terms Stone Age, Bronze Age, and Iron Age.

Also already in the last century, it was recognized that trace elements would be better indicators for the provenance of materials /4/. This is the "fingerprint" concept of provenance studies when artifacts are compared with raw materials from various regions. This approach requires that the raw materials are either not altered at all by the production and corrosion processes or in a quantitatively predictable way. It also requires that there are not as many sources of raw material as to make the task an impossible one. Very often, however, early artifacts are small, fragile or fragmentary and thus permission to sample them is often only granted, if the samples are very small. In addition, it is necessary for such a study to analyze a large number of samples for several elements at low concentrations, so the work seemed not feasible until the development of sensitive instrumental methods for multi-element analysis. With the advent of optical emission spectroscopy (OES) for chemical analysis /5/ the number of analyses, especially of metal artifacts rose sharply. Other important groups of artifacts studied in this way, comprise pottery and stone implements. These are also the materials found most frequently in archaeological excavations due to their durability.

2. PROVENANCE OF METALS

2.1. Lead and Silver

These two metals have to be considered together because in antiquity silver derived predominantly from argentiferous lead ores. Therefore, lead and silver were first smelted together from the ores and separated from each other by a second process called cupellation, in which molten lead is oxidized together with other impurities and separated as liquid PbO. It is evident that this greatly changes the chemical fingerprint of the ore source. Only Au and the platinum-group elements remain totally in the silver. Cupellation experiments have shown that Cu, Bi, and perhaps Sb are depleted in a predictable way /6/ and may also be used for provenance studies.

Several methods have been used for the chemical characterization of ancient silver including OES /7/, XRF /8/, activation with thermal /9/ and fast neutrons and with protons /10/. Although some of these studies discuss the provenance of silver, no attempt was made to compare the artifacts with analyses of ores so that the results were not conclusive enough. The problem is that the principal impurities in ancient silver are Cu, Au, and Pb. Since lead ores were mostly used for silver production and Cu was frequently added, only Au is a reliable source indicator. Given the large number of lead ore occurrences, many of which argentiferous, there is a danger that there are several occurrences with similar Au/Ag-ratios.

The pioneering works of Wampler and Brill /11/ and Grögler et al. /12/ have shown that lead isotope ratios could provide an additional "fingerprint" to pinpoint ore sources of lead and silver. The main advantage of this method is that the isotopic composition of lead is not changed by extraction or refining processes applied to an ore to produce a metal, nor by fabrication of that metal into an artifact, nor by any subsequent corrosion of that artifact. The lead isotope method is based on the natural radioactive decay of isotopes of uranium and thorium to isotopes of lead. In lead-ore forming solutions U and Th are in association with Pb. While these elements remain associated, the isotopic composition of the lead changes continuously. During lead ore mineralization Pb is separated from U and Th and the momentary lead isotope composition is "frozen in". Hence, the isotopic composition of lead may be unique and different for a number of mines so that the isotopic composition of lead in a silver artifact may be matched with argentiferous lead ores from ancient mining areas.

But even with two methods for comparing artifacts and ores overlap of the geochemical characteristics can occur. The number of possible ore sources is then further reduced considerably when it is investigated by dating methods if a certain lead ored deposit was actually worked in the period of interest. This requires extensive field studies and cooperation of geologists, archaeologists, geochemists, and physicists since physical methods are often involved. Almost ten years ago, such a collaboration was initiated at the Max-Planck-Institut für Kernphysik in Heidelberg to study the archaeometallurgy of the Aegean. Initially, the investigations concentrated on the provenance of silver, but lead and gold were included at an early stage and now copper has been added, too. The methods employed are NAA, AAS, MS, thermoluminescence dating of pottery and slags, and radiocarbon dating of charcoal.

An important aspect, especially with objects made of noble metals, is the danger of re-use of scrap metal. Thereby it would be unavoidable that metal from various sources is mixed and the compositional information on the provenance would be lost. At least for silver there are two periods, in which this danger seems minimal: The first is the beginning of the Early Bronze Age, around 3000 BC in the eastern Mediterranean when the first silver objects appear and the second is the Archaic period in Greece when in the 6th century BC the first silver coins came into use. The fast acceptance of silver coinage in Aegean trade resulted in a sharp rise in the demand for silver and consequently in an increase of the production within a short period.

In contrast, lead does not seem to have been re-used in pre-classical periods. Moreover, there is evidence that most prehistoric lead has not been desilvered /13/ so that more trace elements can be used for provenancing. The following two examples demonstrate that with such an interdisciplinary approach as outlined above it is possible to relate ancient silver and lead objects to certain ore deposits.

2.1.1. Prehistoric lead and silver in the Aegean. The beginning of the Early Bronze Age marks great changes in the nature of early societies. A very important aspect of change is the use of metals to a large extent for the fabrication of tools and weapons. This presupposes the ability to smelt metals from their ores, which, in turn, undoubtedly was an important force leading to craft specialization and social differentiation. In the Aegean this transition did not develop gradually but is characterized by a sudden increase of prosperity in the first half of the third millenium BC. This is indicated by the appearance of a relatively large number of metal artifacts, more than a third of which is made of silver or lead. The simultaneous appearance of lead and silver is certainly not a mere coincidence but rather an indication of the common smelting of the two metals from argentiferous lead ores. As mentioned above, this necessitates knowledge of the cupellation process. Most archaeologists think that this metallurgical technique originated somewhere in Anatolia, and consequently, early lead and silver finds in the Aegean were regarded as imports.

However, on the Cycladic island of Siphnos Wagner et al. /14/ discovered that a lead-silver mine, which was exploited in the archaic period, was already worked in the Early Bronze Age, i.e. in the same period when objects of lead and silver appear. Therefore, it seemed conceivable that the lead-silver metallurgy was independently developed in the Aegean. Analyses of prehistoric Cycladic lead and silver objects indeed show that a large part of the metals was derived from Siphnos or Laurion, in the south of Attika, but also from the north or northeast of the Aegean. Present evidence suggests that the idea of metallurgy and maybe its technology came from the early flourishing urban centers of Troy, Thermi on Lesbos, and Poliochni on Lemnos but that is was soon adopted and developed in the Early Cycladic culture /15/.

2.1.2. Archaic Greek coinage. The so-called Asyut hoard, which consisted of approximately 900 Archaic Greek silver coins, was unearthed in Egypt in 1969 /16/. The importance of this hoard lies both in its size - it is by far the largest hoard find up to now - and the fact that it contains a great variety of coins from mints in mainland Greece and Asia Minor. Although the exact chronology of the hoard is a matter of dispute /17/, it can safely be concluded that most of the coins date from the beginning of the 5th century BC. Since the introduction of silver currency in Greece is thought to have started only in the first half of the 6th century BC /16/, the danger of mixed silver from different ore sources to any large extent is probably precluded.

Another peculiarity of the Asyut hoard is that practically all of the coins are damaged by chisel strokes. This allows the removal of a small sample of about 10 mg from the inner part of a coin without lowering its numismatic value. Tests have shown /18/ that analysis of the surface can be misleading. The samples were grouped according to their concentrations of Cu, Sb, Au, and Bi using average link cluster analysis /19/. This information combined with the lead isotope data resulted in the identification of three major ore sources: Laurion, Siphnos, and an ore deposit in northern Greece, probably the Kassandra district on the Chalkidiki peninsula (Fig.1). All of these were mentioned by ancient authors like Herodotus, but only Laurion had been unequivocally relocated before this study.

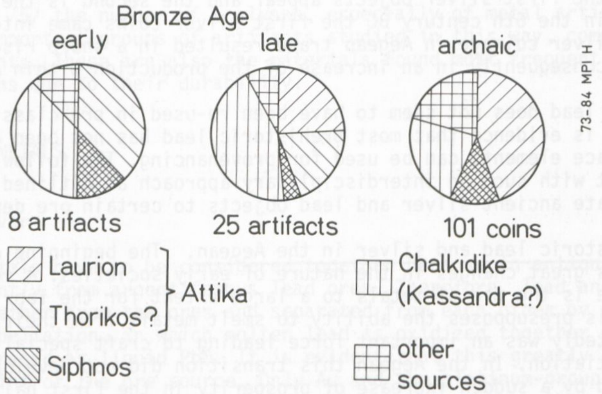

Figure 1: Provenance of ancient silver in the Aegean as deduced from chemical composition and lead isotope ratios.

2.2. Copper and Copper Alloys

Studies on the prehistory of metallurgy have focussed their attention on copper and its alloys, the arsenical and tin bronzes because they are most significant for social change at the transition from the Stone Age to the Metal Age. Therefore, in contrast to lead and silver, a large body of trace element analyses has accumulated, which can already be used for comparison with new analyses of artifacts and of ores. To avoid duplication of work, new analyses need to be compared with published analyses, and particularly with the largest corpus of analyses from Stuttgart, published by Junghans et al. between 1960 and 1974 /20/, who analyzed more than 40 000 European eneolithic and bronze age copper-based artifacts for Fe, Co, Ni, Zn, As, Ag, Sn, Sb, Au, Pb, and Bi. Therefore any new analytical programme should at least assay these 11 elements and Cu, which limits the choice of analytical methods.

In their interpretation of their analyses Junghans et al. /20/ concentrated on the possibilities of identifying compositional groups of artifacts. Their results have raised a controversial discussion on the archaeological significance of their groups and many archaeologists have remained sceptic of the successful application of this method to the provenancing of ancient copper. It now becomes increasingly clear that this scepticism is not justified and that with the use of cluster analysis better sorting of multivariate data can be achieved. However, few attempts have been made to relate copper artifacts to ore sources. The ones reported in the literature /21, 22/ were based on semiquantitative methods. This poses the question of how precise the analysis of copper artifacts should be, given the possible heterogeneity of the artifacts and the ore deposits.

Studies along these lines are now carried out at the MPI Kernphysik in Heidelberg to complement our earlier work on lead and silver in the Aegean. We apply the same approach and the same methods, which have proven successful for the provenancing of lead and silver, to Early Bronze Age copper objects from the Aegean. In a first step we compared the analyses of 47 objects from the Troad, which have been accomplished by the Stuttgart group with OES /23/, with our methods. The result is that - with few exceptions - the data from the two laboratories are comparable within a factor of two /24/. This is in the expected range since the accuracy of the OES method as used in Stuttgart is estimated to be ± 30% for most elements. Since the concentrations range over several orders of magnitude in copper artifacts, this accuracy seems adequate for classification purposes. However, compositional groups become tighter when precise analytical methods are used.

3. POTTERY

Compared with metals determination of pottery provenance is relatively new; first attempts have been reported two decades ago /25/, for two reasons. First, the main constituents of clay vary only slightly and even at the trace level variations are much smaller than in metals. Second, it is necessary to analyze as many elements as possible because, as yet, no group of elements has been singled out, which provides satisfactory group separation. This makes the use of sensitive and accurate multi-element analysis and objective classification methods such as cluster analysis or factor and discriminant analysis mandatory. Of the methods generally used (OES, RFA, NAA), NAA seems to be the best choice because it allows precise determination of up to more than 30 elements in clays /26/.

With the pottery it is usually not important to locate the actual clay beds, from which the raw materials derived. Instead, it is attempted to identify a certain workshop by its characteristic clay composition. This can be achieved by analyzing a range of pottery types from each proven or assumed production center within a geographical region and cultural period. Since the number of workshops can be large, it is evident that the number of analyses needed to establish even the principal groups of pottery in a region like the Aegean exceeds the capacity of a single laboratory. Therefore, it is absolutely necessary that analyses from different laboratories can be compared so that data can be accumulated and eventually a single sherd could be assigned to a certain workshop.

Fortunately,most groups engaged in pottery analysis by NAA use either a clay standard prepared at Berkeley /27/ or rock standards from the U.S. Geological Survey /28/. Both standards have been used to calibrate an in-house clay standard in Heidelberg. The difference between the two calibrations varied between 0.7 and 10% for 19 elements except for Cu, which differs by 34%. However, since already characterized pottery groups show a typical variation of 15 to 20% /29/, the accuracy at present obtainable in pottery analysis seems adequate to deal with many archaeological questions concerning the provenance of pottery. Ultimately, it may be desirable to improve the accuracy to the ± 5% level, but it certainly seems not necessary that absolute standardizations have to agree down to ± 1%.

From the Eastern Mediterranean there exist already thousands of pottery analyses, especially of Late Bronze Age pottery /30/. In this period, people of the Mycenaean culture seemingly traded profusely with foreign lands with the result that vast quantities of Mycenaean pottery are found practically all over the Mediterranean. Visual examination reveals a close affinity in style and fabric, implying that all share a common origin. Archaeologists have always been uneasy about accepting such a conclusion and it seems no surprise that the first attempts to use the composition of pottery for discriminating similarly looking wares were made with Late Bronze Age pottery /25/.

In an exploratory study we investigated if Late Bronze Age pottery from the seashore near Troy could be shown to derive from Mycenae. This could provide new evidence for the location of the landing site of the Greek fleet as described in Homer's Trojan War. The results of NAA of several sherds together with the known composition of Mycenaean pottery are given in Table 1. They show that the sherds S15, which were excavated in a Late Bronze Age citadel near the shore, could come from the north-eastern Peloponnese, while the remaining samples differ greatly. These sherds were found along the ancient coastal line and would therefore be most indicative for the problem outlined above.

But this example was mainly chosen to demonstrate that many archaeological questions concerning pottery provenance can already be answered with little effort because interlaboratory comparison for pottery analyses is generally good.

Table 1: NAA results of Late Bronze Age pottery sherds from Beşik-Tepe on the coast near Troy. The composition ranges of pottery from Mycenae were taken from /29/. All concentrations are in μg/g, except Na and Fe in percent.

	Mycenae /29/	S15-124	S15-180a	S15-137	Parz.625	OEZ	L17.22
Ba	382-921	426	400	404	1197	608	778
Ce	59.4-67.3	59.2	58.0	59.8	66.2	54.7	58.4
Co	27.5-31.0	24.1	23.7	22.9	39.2	43.1	22.2
Cr	221-286	220	218	218	490	502	174
Cs	5.9-9.2	11.5	11.1	10.7	8.6	11.6	10.3
Eu	1.26-1.40	1.16	1.35	1.25	1.72	1.35	1.28
Fe(%)	4.69-5.52	5.38	5.42	5.25	2.67	4.52	4.09
Hf	3.34-4.27	2.08	1.99	2.25	3.13	2.63	2.53
La	30.8-34.1	32.8	32.0	32.5	37.1	33.0	36.1
Lu	0.37-0.45	0.42	0.42	0.43	0.52	0.53	0.44
Na(%)	-	0.734	0.657	0.738	1.36	1.19	0.99
Rb	115-150	153	148	147	79.0	68.8	114
Sc	19.8-22.7	22.0	21.8	21.5	21.4	16.9	16.2
Sm	-	5.76	5.76	5.69	7.36	5.97	5.69
Ta	-	<0.6	1.13	<0.54	<0.6	1.26	1.17
Th	10.9-12.5	12.4	12.3	12.0	11.9	12.9	15.3
U	-	1.93	1.89	1.89	2.94	3.2	3.57
Yb	-	2.32	2.35	2.36	2.76	2.65	2.11

4. OBSIDIAN

Obsidian is a volcanic glass whose mechanical properties make it suitable for the manufacture of tools. In the Aegean it was a vital raw material through five millenia and large quantities of obsidian can be found at most prehistoric sites. Since there are relatively few sources of the material, it is almost ideal for provenance studies by analytical methods. However, in order to form obsidian, the lava must have a relatively narrow range of compositions. Thus, like with pottery, the main components do not seem to offer a good chance for discrimination of various sources. Nevertheless, Georgiades /31/ was able to distinguish the calcalkaline source of Melos from the alkaline source of Antiparos by wet chemical analysis, but the time and quantity of material required discouraged a more thorough investigation.

Trace element analysis has proven the most certain and general way of characterizing obsidian. OES has been employed to demonstrate the wide extent and early date of prehistoric trade of obsidian in the Aegean /32/. However, an entirely satisfactory separation between all sources of obsidian, which might have been used in the prehistoric Aegean, was not achieved. Among other techniques like fission track analysis /33/ and mass spectrometry /34/ NAA again provided complete discrimination among all relevant sources /35/. Since the authors used element ratios rather than absolute concentrations for differentiation (Fig. 2), their results can be readily compared with precise analyses from other laboratories without any problems of standardization. Also plotted in Fig. 2 are results of NAA on obsidian artifacts from the earliest level of Troy and on natural obsidian from two different sources on Melos. The latter indicate the excellent agreement of our analyses with the published ones and the former document that already at the end of the fourth millenium BC there was contact between Troy and the Cyclades. This is further evidence for early Troy's orientation to the Aegean rather than to central Anatolia.

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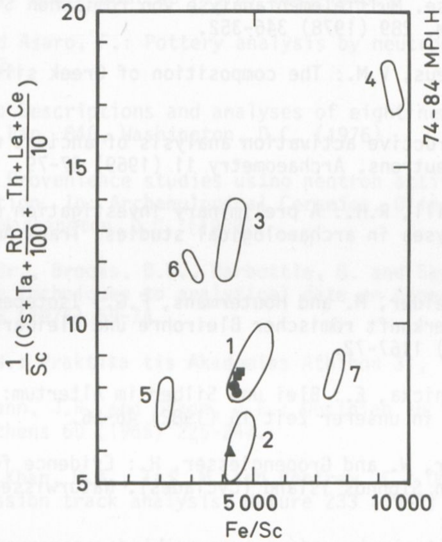

Figure 2: Element concentration patterns for obsidian sources in and near the Aegean; 1, Melos (Adhamas); 2, Melos (Dhemenegaki; 3, Giali; 4, Acigöl; 5, Carpathian; 6, Çiftlik; 7, Antiparos (adapted from /35/). Squares are natural obsidians from Melos (Adhamas), the triangle from Melos (Dhemenegaki) and the dots are two obsidian artifacts from Besiktepe near Troy (Besik-Sivritepe, 4th millenium BC; Besik-Yassitepe, Troy I level).

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