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From the Fabricae of Augustus and the Workshops of Charlemagne: A compositional study of corroded copper-alloy artifacts using hand-held portable XRF

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Chapter 1

Introduction

This thesis focuses on the copper-alloys used to make some of the more frequently found artefacts recovered from The Netherlands. A number of additional objects recovered from the Baltic States of Estonia and Latvia and also the United Kingdom are included as well for comparative reasons. I hope that this article based thesis will be of value to archaeologists interested in Roman and early medieval copper-alloy craft production, but also be of relevance to archaeological scientists interested in the development of portable X-ray fluorescence spectrometry (also known as pXRF or hand-held, HHpXRF). This is because its intention is also to address some important issues regarding the study of corroded copper-alloy artefacts with an interdisciplinary, typological and compositional approach. The following chapters present six peer reviewed articles and a book chapter discussing the composition of a large number of brooches and other personal artefacts dating from the late Iron-Age, until the end of the early middle ages. This was made possible through the application of an interesting new scientific approach using HHpXRF. The remainder of this introduction sets out the background for the research, the selection of the method, materials and equipment, the projects aims and its relevance. It then introduces the remaining chapters and gives a short account of the contribution of the co-authors.

The analysis of copper-alloys is one of the longest ongoing research projects within archaeological science. Traditionally this research, which has become known as provenance studies, has tried to link objects with distinct metal sources, from specific geographical areas. This is based on an assumption that some characteristics of a finished object (e.g. trace elements or isotope ratios) are diagnostic enough to track the raw materials back to their original geographic source (Pollard 2018, 16). According to Bray *et al.* (2015, 202), however, this approach can be demonstrated as being 'fatally flawed'. This is because this line of study tends not to take into consideration past recycling practices whereby metals from different sources could be mixed together, leading to a misinterpretation of the results; a consideration that has been overlooked in many previous studies (Pollard, 2018, 38). Traditional provenance studies can still produce meaningful results however. Objects that can be shown to have had simple life trajectories (Pollard 2018, 45), made from fresh raw materials with little or no recycling, can still produce useful results. But from the Bronze Age onwards, increased hoarding practices have been linked to widespread metal recycling, suggesting that the recognition of recycling practices in their own right could be a fruitful area of research (Radivojević *et al.* 2018). Rather than traditional provenance studies therefore, detecting change in the 'life history' of copper-alloy objects is an alternative approach, studied through the chronological and geographical nature of 'metal flows' (see Bradley 1988; Needham 1998; Pollard 2009). It is a more socially embedded approach as it explores composition through the intentional actions of crafts people, in the deliberate addition of alloying ingredients. Copper can be deliberately alloyed with tin to make bronze, or with zinc to make brass, with or without the further addition of lead. Furthermore bronze and brass can also be melted together to make an alloy called gunmetal, which then contains copper, tin and zinc, again with or without lead. Whilst gunmetal can be a deliberate alloy choice in its own right, it is frequently associated with the recycling of scrap metal, where old bronze and brass items are melted down to form new objects. All of these choices alter both the working characteristics of an object during production and its final colour (see Bayley and Butcher 2004, 14-16). Tin and zinc were mined in quite distant geographic regions (from each other) in Roman and medieval times and were probably transported widely in the form of bronze and brass ingots, before being turned into objects (see Penhallurick 1986; Boni and Large 2003). If the 'metal flow' for a region, at a particular time, shows a preference for tin or zinc dominant alloys then something could be inferred about the direction in which the raw material is travelling. The relationship between object typology and alloy can also be explored in terms of consistency (or otherwise) in choice, including the identification of chronological or geographical change and objects whose alloys deviate from the local norm. This is a meaningful approach because it explores the more socio-technological context of production in terms of human interactions with the craft (Bray *et al.* 2015, 208).

The scale of crafting and production activities for any particular period (typically explored through the typological study of ancient artefacts), are our best indicator of the complexity of a past society, especially in identifying economic change (Wickham 2005, 700). The study of ancient artefacts from the Roman and Medieval periods, primarily those of metal, also plays a crucial role in archaeology in matters of identity construction and the study of ancient mobility and migration (e.g. Lucy 2005; Kershaw 2009; Knol 2011; Ivleva 2016). The distribution of artefacts has been used as a proxy for human mobility for a long time and primarily metal costume accessories are seen as valuable indicators in this study. This is not so true of bulk goods like pottery however, that were transported in large quantities by traders and were distributed widely by economic networks, but not necessarily connected to the movement of people. Costume accessories on the other hand were made in smaller numbers and were less likely to have been shipped as bulk goods. Whilst objects must have frequently travelled on the clothes of their owners, perhaps indicating mobility or migration (e.g. Ivleva 2016), there are further complexities that must be taken into consideration when addressing identity construction. Costume accessories could well have been included in exchange events such as marriage rituals and other life cycle events. Retiring soldiers, pilgrims or other travellers, when returning to their homelands, might have brought souvenirs back with them, perhaps as "returning gifts" for friends and family, which when worn could change a persons status without them assuming the identity of the people from which the objects originated. Therefore the role of a costume accessory in creating an identity, as envisaged by the producer, could be quite different from the wearer. Furthermore objects can become venerated over time, perhaps becoming heirlooms; curated by people several generations removed from the original owners (see Caple 2010), whose meaning when worn may associate more with memory than the present day. That said, the distribution maps of find locations of personal costume accessories can still be a valuable indicator for the travel distances covered by mobile individuals and objects, and can therefore be a proxy for the extent of human contact at a given time.

From a historical perspective, interest in the transformation of the Roman world in the west, into the nation states of the modern era, has been ongoing for nearly a century (Dopsch 1937; Pirenne 1939; Duby 1974; Hodges 1982; Wickham 2009). The processes that led to the post Roman or medieval 'recovery' cannot be fully understood without a better understanding of craft production and its role in the economy of the time. The structural characteristics of the Carolingian empire, the first post Roman Empire in the west have recieved much attention in recent years (McCormick 2001, Devroey 2003, Wickham 2005, Story 2005, Devroey 2006, McKitterick 2008, Hodges 2012 and many others). The debate on the nature of the economy of this period (which extends into the Ottonian and Viking Age, ending therefore in the 12th century) is more than a century old now and various aspects have been discussed and considered more or less important according to various authors, such as agrarian production, trade, craft production and elite demand. The social organisation of production and more specifically the amount of control by the elite, of the output of the lower classes, be they peasants or craftsmen, took centre stage in the debate. The position of craftsmen, at times considered to be 'middle class' and crucial to the development of the economy, is subject to debate as well (see Calmer 2001; Verhulst 2002, 29-84; Hansen *et al*. 2015). An insufficient answered question is: what contribution did craft production make (compared to agricultural production and trade) to the economy as a whole? There is no straightforward answer to this question and this thesis cannot answer it either. Various aspects of the total field of craft production will have to be analysed and both historians and archaeologists have yet to fully tackle the problem. Archaeologists have commonly used the remains of production sites (mainly pottery) as well as finished products to analyse craft production. Traditional research used macroscopic analyses of material remains and their distribution through various regions. Modern research includes the use of scientific methods such as XRF to bring this research one step further. The socially embedded approach presented in this thesis, makes a contribution towards tackling one aspect of the problem by providing a better understanding of the choices made in copper-alloy craft production.

The objects

Copper-alloy artefacts played a very important role in identity construction and brooches (also known as fibulae) in particular are a very visible type of personal-dress accessory. They were used by ancient communities for many centuries, especially during the Roman and early medieval periods where their forms and types change significantly over time and geographic region (see Almgren 1923; Frick 1992; Weetch 2014; Ivleva 2016). For the Rhine basin (covering The Netherlands, parts of Belgium and Northwestern Germany), over 20,000 brooches have been recorded (Heeren and van der Feijst 2017, 8). This has led in turn to the formulation of many hundreds of typological classes, sorted into chronological groups. Although a late Iron Age example was included for comparison (Nauheim type, Heeren and van der Feijst 2017, 42), the departure point for the brooches in this thesis is the early Roman period, dating to the last half of the first century BC (Spoonbow, Bow on spring-tube, Aucissa series, Heeren and van der Feijst 2017, 49, 60, 94 respectively). The latest bow brooches included from The Netherlands are from the late 2nd century AD (Germanic Rollenkappe, One-piece Knee, Van Buchem 23, Heeren and van der Feijst 2017, 115, 121, 127 respectively). Then a selection of middle to late Roman period disc brooches from Estonia and Northern Latvia are presented, dating between the 2nd to 5th centuries. These disc brooches are subsequently reorganised into a new typology building on previous scholars work (e.g. Vassar 1943, 70-71; *Шмидехельм* 1955, 146-199; Laul 2001, 108-114). For those early brooches found in The Netherlands, the main question is whether regional, or civilian production could be identified versus production at military centres. This is because there are some close parallels between some brooch types and military related objects, suggesting that some were made in military workshops (Haalebos 1986, 43; Müller 2002, 29). The choice of alloy is important to this question, as brass is thought to have been produced on an industrial scale by the Romans through the invention of a new technique called cementation, and initially reserved for use by the state in the production of coins and military gear (Bayley and Butcher, 2004, 13; Dungworth, 1997, 903). Any seperation between the use of brass or bronze along typological lines would add to this debate. A 'Roman' alloy if it exists could be compared to alloys more confined to local or 'tribal' brooch typologies. The questions asked of the later Roman disc brooches found in Estonia and Northern Latvia are a little different. Apart from questioning the organisation of production, variation in regional culture is looked for through comparing compositional, stylistic and typological differences with their geographical find locations. Then the results are compared with the published evidence from outside this region, and the influences brought about by contact with the southern Baltic areas and the much more distant Roman Provinces are subsequently explored.

The next set of brooches included in this thesis are early medieval in date and the study returns once again to The Netherlands with an analysis of Carolingian/Ottonian period disc brooches, many of which are thought to bear Christian motifs, which rapidly gained popularity in the region overtaking earlier Merovingian forms (e.g. Roxburgh *et al* 2014, 23). These disc brooches were at their most popular between the late 8th to 10th centuries but gradually declined in use during the 11th to 12th centuries. The variety of forms and types grew enormously during these centuries, indicating that they played an important role in creating a variety of new identity constructions. The earliest of this type of brooch is known as the *Heiligenfibeln*, or Saints brooch, which was popular during the 9th century but may also have had a late 8th century origin (Bos 2006a, 767). These are included in this study as well as the more common *Kreuzemailfibeln* or enamelled cross type, which date to the 9th and 10th centuries (Bos 2006a, 730). The organisation of production is again looked at for these brooches, based on morphological and typological consistency as well as alloy choice. Then from this departure point, the exchange system in which they circulated is explored, with a particular emphasis on the Christian nature of their motifs. The brooch theme continues with a geographic, compositional and typological comparison of disc and equal-arm brooches (also known as Ansate brooches, see Bos 2006; Thörle 2001) from the island of Walcheren in Zeeland, with the other regions of The Netherlands. The disc brooches studied here date between the 9th and 11th centuries and a comparison is made between those found at Domburg and Middelburg (Walcheren) and those found at Westenschouwen on the neighbouring island of Schouwen-Duiveland, and then the rest of The Netherlands. Again suggestions are put forward regarding the organisation of production of the brooches found on Walcheren against its historical past. This in turn leads to discussions surrounding cultural identity within an interconnected North Sea world. A study of early medieval equal-arm brooches also takes place. This brooch type and its many subgroups are typically found in Western Europe, including Belgium, Britain, Northern France and The Netherlands (Bos 2006, 455; and Weetch in press). There are two relevant typologies for equal-arm brooches. The first is by Thörle (2001) who classifies the brooches from the Frankish areas and the second is by Bos (2006) who classifies the brooches from the Frisian areas. Thörle dates the brooches from Walcheren between the 7th to 10th centuries and associates them to the Western Frankish areas of Neustria, Burgund and Aquitania. Bos, for the Frisian coastal areas, divided the classification into early and late forms based on the early ones being the more robust of the two. He subsequently dated the early ones to the 6th and 7th centuries and the later ones to the 8th to 11th centuries. He also considered the later ones to be most numerous during the 9th century (Bos 2006, 458). A combined typological and compositional comparison was undertaken to further compare the Walcheren brooches against these two geographically different classifications. This tests the hypothesis put forward by Bos (2006, 455) that the more northerly Fresian brooches are regionally different to those from

Northern France, Belgium and Walcheren. Following this the organisation of production is discussed, in particular ideas surrounding local versus regional manufacture. The equal-arm brooches also form part of a discussion on the identity of the peoples living on Walcheren at that time.

Moving away from brooches, the next artefacts to be included are a group of zoomorphic mounts tentatively dated between the 9th and 11th centuries (see Capelle 1976, 28; Webley in press). These mounts are found in large numbers on Walcheren, and similar ones are also distributed along the southern coastal areas of the North Sea, but in much fewer numbers. The origin of these mounts, their original function and their relationship with expressions of identity, are discussed in detail through a combined typological and compositional study. The organisaton of their production is also discussed and comparisons are made to the other types of finds on the island. A number of Stirrup-strap mounts were also found on Walcheren and these are included in the thesis. This type of mount is considered to have an equestrian function and examples of certain subgroups appear both in England and The Netherlands (see Williams 1997, 105). A typological and compositional analysis enabled discussion to take place regarding local versus non-local origins. The choice of alloy could also be compared with the other find types contained in the study. Lastly, ring and dress pins are also investigated, but with a more detailed comparison to an assemblage from England (see Capelle 1976, 17; Rogers et al 2009). The pins are seperated in the first instance into two functional types. Those with shaped decorative heads and those with wire rings threaded through their heads. The wire ring type may have been attached to a chain, perhaps working in a pair with another one at the other end of the chain. The ring, which is sometimes an adjustable slip-ring type, could also have been used to attach to a belt or other personal garb. Dress pins on the other hand do not attach in the same way. They were possibly used in closing or holding together items of personal clothing (again see Capelle 1976, 17 and for comparison Ross 1991; Rogers et al. 2009). The Walcheren examples are thought to date from the 7th to 11th centuries. Pins are quite a common find around the North Sea coastal areas, and the way production was organised for those found on Walcheren is explored once again from a typological and compositional perspective. The analysis is taken further with a comparison between a contemporary group of pins from England. A discussion takes place regarding mercantile contact between the two coasts as well as local or centralised production in an interconnected North Sea world.

The methodological approach

There have been many scientific objections to the use of HHpXRF in Archaeology, especially over its reliability and validity. A major objection is that highly accurate quantitative data is not capable of being obtained on corroded copper-alloy surfaces (Nicholas and Manti 2014, 8). Furthermore it has been suggested that it is an unacceptable side of science, that it is in fact flawed, as archaeologists are too ready to accept the invalid or unreliable data coming from these machines (Speakman and Shackley 2013, 1435). Although this debate is a few years old now, there is still scepticism of analytical performance - typically at conference settings and less formal meetings. A relevant departure point for summarising this debate starts with the very important question posed by Stephen Shackley (2010), regarding the level of reliability and validity present in the fast growing use of HHpXRF. This question was triggered by his observation that its increased use in archaeology had created a divided community, locked in a heated debate that was fuelling animosity between the two sides. Shackley proposed that the problem centred around the potential misuse of this quickly appearing technology, by a community that was not prepared for it intellectually, primarily through a lack of appropriate scientific training. More specifically he suggested that it was not appropriate for archaeologists to just simply purchase a HHpXRF machine and go out shooting anything they wanted (and apply the results) without having the benefit of years of compositional analysis behind them, or an equivalent training. Therefore a basic protocol was needed (discussed in more detail in Davies et al. 1998; Shackley 2011), especially in establishing reliability and validity for analysing archaeological artefacts.

Firstly he warned that validity cannot be provided without demonstrating that the analyses are undertaken with a demonstrable understanding of the instruments settings and parameters; the results of which could subsequently be compared against readily available callibration standards. Secondly that the magnitude of error introduced by varying artefact surface morphology and size must be known and within acceptable limits. Thirdly that the reliability and stability of an HHpXRF's internal system needs to be better defined, requiring testing outside of a vendors own service parameters. Lastly that the nature of the analyses, be they qualitative or quantitative needs to be understood, particularly in the light of qualitative analyses generally being rejected, mainly due to the risk of missassignment caused by inter-observer error. Quantitative analysis is needed (for sourcing studies at least) based on emperically callibrated datasets that can provide elemental weights expressed as a % value. Shackley ends with a call to establish research protocols for this newly emerging technology and warns of the dangers of the wholesale acceptance of HHpXRF; that a solution is needed that combines the different approaches of the physical scientist and archaeologist, against a potential background of 'scientific ignorance' or indeed 'anti-scientific' sentiment.

The artefacts used by Shackley to elucidate the argument outlined above (for studies involving correct source assignment) were not made of copper-alloy but of obsidian, a naturaly occuring glass like material used by ancient peoples in the production of various tools. That said, the issues outlined earlier are valid for archaeological applications in general, and especially those made of copper-alloy. In addressing Shackleys concerns, two HHpXRF machines from different manufacturers, were tested for use in Aegean obsidian sourcing studies by Ellery Frahm *et al.* (2013). Their conclusions were that the accuracy and precision of both machines were suitable for this area of study even though both used 'off the shelf ' factory settings. Frahm quickly followed up by publishing a more detailed discussion regarding of the validity of this 'off the shelf' approach, using the sourcing of near eastern obsidian fragments as its case study (Frahm 2013).

The first issue addressed by Frahm was one of low accuracy and precision. Accuracy can be described as how close a measurement is to the 'true' value of a particular quantity. This would be important in measuring the 'true' value for the metal ingredients in a copper-alloy brooch for example. Precision, on the other hand is the ability of the machine to repeat its measurements in a precise way under identical conditions. This becomes important when you want to see discernable clusters in the compositional measurements. Frahm's argument followed that for HHpXRF machines, which are less powerful in term of wattage than laboratory based devices (a sacrifice needed in order to make them portable), any applied analytical technique, regardless of equipment type will be subject to a base level of variation (e.g. statistical fluctuations) that limit its precision.

Limitations to accuracy (such as a flawed callibration), on the other hand become a concern when attempting to compare measurements to previously published data. The second issue was that of callibration and correction. The x-ray counts (the count or intensity of the radiation of a specifc wavelength, detected by the XRF device) are turned into elemental concentration data. A correction or callibration model is needed in order to correct for a number of complex phenomena to do with the way for example X-ray emissions, absorbtion and secondary fluorescence are handled by the machine (see Shackley 2011 for more). This model is used to turn the x-ray counts into numerical concentration data that can be used by the operator. But it is unclear when and how this step of the process is done, particularly if it is done within the instrument before outputting the data, or later during database processing. This raises issues about confidence in the internal consistency of the data. The third issue was fundamental to XRF itself. The size of measurement error can be related to the size and shape of the artefacts being measured. Thin artefacts and those that do not fully cover the instruments detector window are harder to analyse as they may not experience the full energy of the electron beam. Variations in the surface morphology of an artefact can also introduce error. The ideal solution is for the samples being measured, to be polished flat, be big enough to cover the detector window and to be thick enough to be able to fully absorb the X-ray emissions. This ideal, especially in the case of copper-alloy artefacts would of course require destructive samplng of selected specimens. Frahm's experiment with two analysers from different suppliers was conducted to address these issues. The experiment was deliberately intended to test the capability of HHpXRF in 'off the shelf' conditions. That is, to accept the issues mentioned above on a range of complex artefacts and to determine if the analysers are sufficently precise enough to produce valid measurements for assigning obsidian flakes to different sources. I. E. to sort them into distinct groups. This turned out to be the case, that HHpXRF was precise enough to apply valid source assignments even though optimum conditions and 'best practice' were not followed. In conclusion Frahm suggested that the use of HHpXRF was neither intrinsicly valid nor invalid, that it is the application, the choice of question, that makes it valid or not.

The observation by Frahm that the aplication of HHpXRF could still produce valuable results, despite its sub-optimal 'off the shelf' approach, may have sat well with one side of the "divided community" but it quickly prompted a published reply by Robert Speakman and again Shackley (2013). They argued that the form of science put forward by Frahm was unacceptable, as he still ignores the real issues behind validity and reliability, by suggesting that 'internal consistency' (e.g. relying on a machine's internal callibration or factory settings) was enough to answer archaeological questions. Speakman and Shackley further argued that this was "playing at science," that HHpXRF use needs to conform to internationally published standards in order for it to be valid. They subsequently described Frahm's approach as 'silo science,' meaning that data produced by researchers working in this way was 'self-contained,' produced in a bubble, whereby outside independent validation could not take place. What they did agree upon however was that most archaeological users lacked relevant experience and tended to take a 'trust me because my results are internally valid' approach, that the users principle understanding of HHpXRF was that it was a 'black box,' that could analyse samples and subsequently generate useful looking numbers. This, according to Speakman and Shackley was poor science and suggest that these portable machines were originally designed to be used as 'black boxes,' to be used by inexperienced operators, but intended for the mining and metal-recycling industries, not archaeological research.

In this respect they were never intended as true research instruments and if they are to be used in sub-optimal conditions as proposed by Frahm, then they questioned whether publishing could be considered at all. Furthermore they called on all peer reviewers of manuscripts containing HHpXRF research to seriously scrutinise them for invalid and unreliable data.

Frahm once more responded to Speakman and Shackley's concerns in a follow up paper (2013a), asking the question are we dealing with geochemistry or archaeology? Frahm argued that the goal was to determine wether a HHpXRF analyser could sort obsidian flakes in relation to their sources. That it was not to achieve geochemical accuracy and that Speakman and Shackley were wrong to associate correlation, reproduceability and validity to this aim. He went on to explain that reproduceability does not equal accuracy and results do not equal raw data, in seperating geochemical versus archaeological avenues of investigation. Validity is also suggested as being confused with accuracy, that attempting to match 'true' values based on standards, is a measure of accuracy, not validity. Validity should be defined as the suitability of a machine's ability to measure values in relation to the context of the question being asked of it. More specifically he suggests that the archaeological approach is primarily concerned with sorting artefacts in relation to typology, also to associate them to geographic origins, before attempting to associate any patterning with past human habits or decision making. It is not concerned with the gathering of reasonably accurate geochemical data that can be directly comparable to other studies and collected into one large database. Furthermore Frahm suggested that past studies have been largely limited to the laboratory, which is problematic in itself in terms of limiting the direction of questioning, mainly due to the internal organisation of people, resources and materials. Also laboratory techniques are often limited to analysing small percentages of an overall assemblage from which interpretations must be drawn. Conversely he suggests that the growing use of HHpXRF will in fact increase reproduceability, as more independant researchers become involved. It will also allow a much higher percentage of an assemblage to be measured, which in turn will allow for better interpretation. In conclusion Frahm calls for a break away from the limitations of laboratory based paradigms and suggests that in order for HHpXRF to be successful in archaeology, users need to work within an appropriate methodology. This conclusion however is at odds with Speakman and Shackleys call to work within the 'traditional' laboratory XRF based paradigm.

The debate that I have briefly summarised is very important when considering the application of HHpXRF on corroded copper-alloys. Firstly there appears to be a sociological, Kuhnian issue at play in terms of a crisis over the emergence of a new scientific paradigm (see Kuhn 1970, 66-76), especially with its lack of acceptance within some areas of the archaeological community. The two groups could be described as those who deem themselves as having "appropriate" scientific training, who typically assume the high ground in the debate, versus those "untrained" archaeological users with lack of scientific parity. But critically there are some genuine technical issues that need to be addressed as well.

The main issue is that of corrosion depth on corroded copper-alloy surfaces (also refered to as the patina). HHpXRF is a surface measuring device whereby the analytical information only comes from the first 20μm (Feretti 2014, 1754). This shallow depth of measurement is typically well within a corroded surface layer which severely affects accuracy if the aim is to measure the absolute composition of the alloy (ibid 2014, 1756). Aside from corrosion, the surface morphology of the object, as well as the varying distance between the object and the x-ray source, are also issues said to prevent HHpXRF from producing highly accurate quantitative data (Nicholas and Manti, 2014, 8). This quantitative approach can only be achieved in fact by the destructive removal of the patina and the altered outer surface from which the patina grew; a practice that is resisted by museums and artefact owners, thus limiting the number of samples available for study. It is also based on the assumption that gaining a 'true' measurement of the objects composition is central to solving historically based archaeological questions.

The alternative as proposed by Feretti (2014, 1757) is the non-destructive route, where the artefacts are not harmed. Although measuring the corroded layer means that the results are much more variable, more measurements can be taken both in terms of a single object and also much larger numbers of objects within museum or depot collections. This approach involves working with clusters of measurements rather than individual ones. This has the benefit of being able to identify trends (or choices) in alloy use, and subsequently to identify outliers from these trends. This qualitative, or semi-quantitative approach is also in line with the conclusions put forward by Nicholas and Manti (2014, 9). They suggested that whilst HHpXRF is not capable of producing fully quantitiative results on corroded surfaces, the apparatus is still able to produce qualitative data that enables basic alloy classification and therefore provides a similar sorting mechanism to that proposed by Frahm earlier.

These technical issues imply that HHpXRF is a suitable analytical tool to answer questions regarding the basic choices craftsmen made in terms of copper-alloy production (e.g. brass or bronze), when studying large artefact assemblages. This conclusion clearly supports the argument put forward by Frahm, but in doing so opposes Speakman and Shackley's suggestion that quantitative analysis is the only acceptable avenue of scientific enquiry (2013, 1435). Thus, the use of HHpXRF in the study of corroded copper-alloy artefact has to align itself with Frahm's argument and reject that put forward by Speakman and Shackley. But this is not a sufficient place to leave it. Speakman and Shackley's concern about archaeologists 'playing at science' is very valid. There is little to stop an enthusiastic archaeologist, in posession of an HHpXRF device, measuring a corroded copper-alloy object and subsequently interpreting the measurements, incorrectly, in a quantitative manner. It cannot be assumed that archaeologists automatically have awareness of, or a working understanding of the issues mentioned above. Furthermore there are no easily accessible protocols or guidelines as yet which enable them to formulate their questions accordingly. So in this respect Speakman and Shackley's call on the reviewers of peer reviewed journals to be especially vigilant, is a valid one. This discussion is continued further in chapter 2 where a reconaissance oriented protocol is proposed as a way of positioning the application of HHpXRF (employed in a qualititative or semi quanititative manner), alongside, but not replacing, more rigourous laboratory methods.

The dataset

The methodology required that both typological and compositional data be analysed together. This meant that a database had to be created with a relatively large number of variables. Microsoft Excel was decided upon to build the database with, due to its ease of use and familiarity by all those involved in the project. A particular requirement of the database was that data could be entered very quickly in the field. This is because the time required to gather compositional data by

HHpXRF was around 60 seconds per object, meaning that large numbers of measurements, typically several hundred in a day, could be made onsite at a storage facility or museum. This fast throughput of objects created two problems. Firstly there needed to be enough objects coming out of storage to keep up with the demand, otherwise the HHpXRF device could be standing idle for long periods of time. This was not so problematic when direct access to the storage areas was available, but was potentially an issue if the process at a museum or storage facility meant that an individual staff member was responsible for moving objects in and out of storage. This problem would mainly be due to a lack of awareness of the speed of throughput of the device and hence the numbers of items that were required to be moved during a visit. The problem was reduced to some extent by communicating the large-scale nature of the activity in advance. The second problem was that of collecting typological and morphological data as the objects became available for the HHpXRF. The identification, measurement and photographing of each object (for further analysis post-visit) takes several minutes longer than needed for the HHpXRF analysis, quickly leading to a backlog in the data gathering activity. The methodology needs the presence of typological and morphological data for linking to the compositional data in order for it to work. The compositional data is relatively consistant because it is downloaded from the device either in the field or at a later date, then put through a normalisation procedure. The typological and morphological data also needs to be as consistent as possible to gain the best results. The best way to achieve this was to create a basic dataset in the field as the objects were becoming available, then to fill in the missing details after the datagathering event had taken place. This was more reliable and consistant than taking already existing data in the posession of the museums or storage depots. It was more reliable in the event of the existing data not becoming available in a timely way, or subsequently not being useful enough to assign to the compositional data. To gather this information in the field was more consistant than taking and merging a number of different external datasets, whose information would have been gathered by a wider number of individuals, leading to more variation in description and typological assignment. The quality of the data, as it was being created, could also be checked whilst in close proximity to the objects and if neccessary the artefacts could be reassessed much more easily than from a distance and some time afterwards.

The database subsequently required a merging of the following typological, morphological and compositional information. Firstly and quite key was the need for a unique identification number for each artefact (Finds ID). Furthermore this identification number had to have already been formulated by the museum or depot and be their standard way of locating and identifying the object. This is critical if the results of the project are to be reproducible or validated by other researchers in the future. Following on from this an easily identifiable name was needed for the different collections from which the objects were accessed (Collection). This could typically be the name of a museum, or other owner such as an archaeology company or metal detectorist. Then a consistantly identifiable name for each object (Object type) was required. This frequently required a choice as many artefacts are named differently in different publications, or translate differently between languages. The important factor was to be consistent in the use of a name once decided. The next requirement was for a longer description including sub group names and other distinctive features (Description). Again consistency of terminology was important for the later analysis. Two more columns were also included to aid geographic and chronological analysis (Culture name and Broad Period). The culture names could typically be Roman, Merovingian, or Carolingian/Ottonian, but with some local variation such as Migration or Germanic. Subsequently a column for assigning a broad date for an object was included, with due consideration of the dates proposed by relevant publications. A column was also added to list the name of the publication from which the typology was assigned, including the typological group or subgroup number where possible. Then some columns including morphological details were required, including measurements of length, width, thickness and diameter (recorded in mm). Because of the corroded, distorted and fragmentary nature of individual artefacts, these measurements could only be taken as a guide for inter-group comparisons. Measurements were taken using a caliper device with plastic rather than metal jaws, which was to reduce or eliminate damage to the artefact surfaces when in use. Each time a measurement is taken by the HHpXRF device a unique analysis number is automatically generated. This number (XRF no.) is critical to the success of the database. This is because the typological and morphological data is gathered seperately to the internal generation of the compositional data. As each object was measured by the device, the unique XRF number needed to be logged against the unique Find ID number so that the composition data, once outputted and transfered into a spreadsheet, could be matched to the correct typological and morphological data. The correct matching of these two numbers was key to preventing missassignment of composition to object. The next column is also important in preventing misassignment as it records the location on the object that the measurement was taken (XRF Loc.). To maintain as consistant an approach as possible, artefacts of the same or similar type were presented to the device in the same way. But this was not always possible due to missing parts of an object leading a measurement being taken from a different location. This was noted in the database. More than one location could also be required, such as measuring the front or rear of a brooch, or a corroded, patinated area contrasted with clean patina free area. When these types of situation were encountered a note was required in this column to assist in later analysis. Finally the bulk and trace elements recorded by the device could be added (each element having its own column). These are presented in numerical format and expressed as a percentage of the sum of the individual elements. The important columns contain the data for the bulk elements found in copper-alloys, these were Copper (Cu), Tin (Sn), Lead (Pb) and Zinc (Zn), followed by Gold (Au), Silver (Ag) and Iron (Fe) as contaminants or traces of surface treatment caught up in the corrosion layer. The only trace element that was found to be relevant was Mercury (Hg) as its presence associates with gold plating in a process known as fire gilding.

Aim

To contribute to our understanding of Roman and early medieval craft organisation, through the choices made in the production of copper-alloy personal items. More specifically the aim is to investigate variability in the control of copper-alloy composition, in particular the variation between bronze, brass or gunmetal, and to identify regional or chronological differences between groups of artefacts. Subsequently the intention is to gain new insights into the way production was organised, including the supply of raw materials, and to identify differing cultural associations through the choice of alloy.

Research Goals and Questions

The research goal is, by necessity limited, because basic knowledge about the production processes and organisation of craft production is still in its infancy, although in recent years an increasing amount of research is filling up this knowledge gap. The research focuses on the analysis of copper alloy objects, mainly personal belongings such as brooches, which were produced on a massive scale. The primary research goal is to analyse various aspects of the production process of these objects by analysing their chemical composition and the variable use of different base materials. The degree of variability (or standardisation) of the ratios of the metals used in production will help elucidate the technical and raw material choices available. From there, the social organisation of production and the various (exchange) mechanisms that brought about the observed distribution of objects can be elaborated upon. The main focus therefore is on production practice, then exchange and identity. The expected variability in alloy composition will be analysed in geographical, temporal and organisational terms.

Subsequently the following research questions have been formulated:

a) Is it possible to construct an interpretative framework for the use of HHpXRF on copper-alloy, particularly regarding the issues concerning its use on corroded artefacts?
b) What compositional variation is there in large numbers of similar copper-alloy objects?
c) Is there evidence for deliberate control of copper-alloy composition (perhaps between different types of objects)?
d) Is there evidence for chronological change in alloy use from the early Roman period to the beginning of the Middle Ages (1st century BC - 12th century AD)?
e) Can regional differences in composition be seen (perhaps eluding to the availability or otherwise of raw materials)?
f) What are the potential supply lines for the sources of raw materials?
g) To what extent can compositional variation, across geographical and temporal distributions, allow new insights into the social organisation of production, or the identity of those wearing the finished objects?

Relevance

It has been demonstrated above that changes in craft production can be used as a proxy for changes in economy and society. This engages with the debate surrounding the emergence of the western economy after the end of antiquity and how Europe came into being.

Chapter outline

There are 9 chapters in this thesis including the introduction and conclusion. The structure of the thesis is article based, and presents a collection of 6 peer reviewed articles and 1 editorially reviewed book chapter, all of which are either printed or in press. After the introduction, Chapter 2 presents an article on methodology, then the following chapters 3 to 7 present the results of a series of case studies that not only engage with the debate over HHpXRF, but also employ appropriate lines of typological questioning. After chapter 2 the chapters are set out as far as possible in chronological order. Chapters 3 and 4 present research on early and late Roman brooches respectively. They also draw their measurements from brooches found in two distinctly seperate geographic regions (The Netherlands and Estonia/Latvia) and from two HHpXRF machines, from two different manufacturers (Niton and Brucker). Chapter 5 returns to the Netherlands and also deals with brooches but from the early medieval period. Chapters 6 to 8 then concentrate in more detail on one particular assemblage again from the early medieval period. The artefacts found in and around Domburg, The Netherlands. The artefact types include disc fibulae, equal arm fibulae, dress pins, ring pins, stirrup-strap mounts, and zoomorphic mounts (discussed in more detail in chapter 6). Lastly Chapter 8 draws a comparison between the dress pins found at Domburg and those found in England. HHpXRF compares the alloy compositions of typologically similar pins from these two seperate regions.

Chapter 2 presents an article entitled, A Non-destructive survey of early Roman copper-alloy brooches using portable X-ray Fluorescence Spectrometry. It is authored by Marcus A. Roxburgh, Stijn Heeren, Hans D.J. Huisman and Bertil J.H. Van Os and was published in *Archaeometry*, 2 July 2018, doi.org/10.1111/arcm.12414. Archaeometry is a scientific peer reviewed journal published on behalf of the Research Laboratory for Archaeology and the History of Art, University of Oxford. This paper argues that portable X-ray fluorescence spectrometry (pXRF) is a suitable elemental measurement technique to study the production of copper-alloy artefacts. However, rather than try to imitate the accuracy and precision of laboratory techniques, it is more beneficial to deploy it in a survey role, one that attempts to model chronological and geographical changes within large quantities of artefacts. To achieve this, the effects of corrosion and the issues surrounding surface measurement were investigated to determine their affect on this type of research. Analyses on early Roman period brooches gathered in the Nijmegen region of the Netherlands were subsequently compared with published data.

Chapter 3 - Early Roman copper-alloy brooch production: a compositional analysis of 400 brooches from *Germaina Inferior.* Authored by Marcus. Roxburgh, Stijn. Heeren, Hans Huisman and Bertil Van Os. It is published in the *Journal of Roman Archaeology*, Volume 29, 2016, pp 411-421. The *Journal of Roman Archaeology* is a peer reviewed journal published by Cambridge University press and focuses the Roman world between c. 700 B.C. to 700 A.D. In this paper we present a compositional study of a large number of copper-alloy brooches, undertaken in 2014 using Handheld X-ray Fluorescence Spectrometry (HHpXRF). The brooches, which come from the area of Nijmegen, date from the Late Iron Age until the 2nd c. A.D. Our aim is to explore the ways in which artefact production was organized both in the context of Roman centres and in the countryside. The link between alloys and workshop organization is discussed before the methodology and results are presented. The results are then discussed, leading to the formation of several hypotheses regarding the organization of workshops that produced metal artefacts.

Chapter 4 - Disc Brooches of the Roman Iron-Age, from the *tarand* cemeteries of Estonia and North Latvia. Authored by Maarja Olli and Marcus Roxburgh. It is published in *Lietuvos Archeologija*, 2018, T. 44, p. 39-70, which is a scientific peer reviewed journal, publishing in the Baltic region. Disc brooches from the Roman Iron Age are very diverse in style and execution throughout Europe. Their diversity in the *tarand* cemetery areas of modern day Estonia and North Latvia is also very high, with many unique traits and some multidirectional influences being observable. Therefore, regionality in the *tarand* cemetery areas was studied through these diverse brooches in order to see whether some motifs, typological groups, or alloys were more preferred in some areas than in others. A typological and compositional approach (using HHpXRF) was adopted for this. Based on the distribution of certain groups of disc brooches, their surface treatment, and the direction of the influences, two distinct areas could be seen: Northeast Estonia and Southeast Estonia–North Latvia. The study shows how people in the *tarand* cemetery areas adopted foreign techniques and stylistic features in accordance with local preferences and used them in their local culture.

Chapter 5 - The Cross & the Crucible: The production of Carolingian disc brooches as objects of religious exchange? Authored by Marcus A. Roxburgh, Hans D.J. Huisman and Bertil van Os and published in *Medieval & Modern Matters*, vol.5 (2016) 117-132. This is a peer reviewed Journal by Brepols focusing on the archaeology and material culture in the Low Countries from the middle ages to the modern period. In this article, aspects of the production and exchange of Carolingian/Ottonian disc brooches are examined through an analysis of 281 brooches selected from collections across The Netherlands. The composition of the brooches was measured with a HandHeld-XRF device and the results showed a sharp deviation from the alloys seen in those from earlier periods. The results across all geographic areas consisted of a mixture of copper, zinc and lead, but no tin. If recycling practices were present in the making of these brooches, then you might expect an element of tin to enter the mixture, as bronze items entered the alloy-recycling stream. But this appears not to be the case. The results suggest that production was more likely organised on a regional or even super regional scale. An analysis of production at royal estates, emporia and the great abbeys found the latter to be the most likely source of these objects. Furthermore, the Christian nature of these brooches, if produced within monastic centres, may have imbued them with religious meaning, allow for a fresh interpretation of their methods of exchange.

Chapter 6 - Where Worlds Collide: A typological and compositional analysis of the copper-alloy mounts from Viking Age Walcheren. Authored by Marcus A. Roxburgh, Nelleke L. IJssennagger, Hans D.J. Huisman and Bertil J.H. van Os. It was published in *The Medieval Low Countries*, vol.5 (2018), pp.1-33. This is a peer reviewed journal by Brepols focusing on the Low Countries from the beginning of the 5th to the second half of the 16th centuries. In this article we present a combined compositional and typological analysis for a group of 90 Viking-Age mounts, found in Walcheren, The Netherlands. This new analysis is supported by the use of Handheld X-Ray Fluorescence Spectrometry (HHpXRF). The new data sheds light not only on the character of the technology available at the time, but also allows us to formulate a new hypothesis of their purpose and cultural associations within a Viking-Age, North Sea world. Similar mounts have been found in other coastal areas of the southern North Sea, but not in great numbers, suggesting that production was more likely to have been local. Their compositional and typological characteristics matched those of certain forms of Viking-age equestrian gear, but the relatively small size of these mounts allowed for the possibility that these items were more likely used on personal equipment. Although a question over the exact dating remains, the styles suggest that close ties existed between the Frisian, Viking and Anglo-Scandinavian worlds between the 9 th and 11 th centuries. This expression of identity should be considered in a context of continued intensive relations between Frisian, Viking and Anglo-Saxon.

Chapter 7 - A workshop at the edge of the world? A compositional analysis of copper-alloy finds from Early Medieval Walcheren. Authored by Marcus A. Roxburgh, Nelleke L. IJssennagger, Pim van Tendeloo, Hans D.J. Huisman, and Bertil J.H van Os. This is a book chapter (in press), published in P. Deckers and L. Ten Harkel (eds), *A Central Place on the World's Edge: Studies on the early medieval history and archaeology of the Island of Walcheren, the Netherlands*, Brepols. Over many years a large and diverse number of early medieval copper alloy artefacts have been recovered from the former island of Walcheren (Zeeland, the Netherlands). In this chapter we present the results of a combined typological and compositional study of six groups of these artefacts dating between the 7th and 12th centuries, encapsulating a period in time better known as the (long) Viking-Age. The valuable new qualitative data collected by the non-destructive HHpXRF technique sheds light not only on the character of ancient technology and the organisation of craft production, but also allows a reconsideration of the role of these personal objects in expressing social identity within an interconnected North Sea world.

Chapter 8 - A Comparative Compositional Study of 7th- to 11th century Copper-Alloy Pins from Sedgeford, England and Domburg, the Netherlands. Authored by Marcus A. Roxburgh and Bertil J.H. van Os and published in *Medieval Archaeology*, Vol 62, Issue 2, 2018. Early medieval pins are found in large quantities on both sides of the North Sea and the English Channel, and as a result are one of the few artefact types that can facilitate the exploration of cross-cultural contacts in terms of style, material and manufacture. This paper presents the results of the analyses of two contemporary groups of copper-alloy pins dating from the 7th to the 11th centuries using X-Ray Fluorescence Spectrometry. One group of pins considered here was recovered from an excavated Anglo-Saxon settlement at Sedgeford (Norfolk), while the other derives from a coastal settlement at Domburg (Zeeland, the Netherlands). We argue here, on the basis of our results, that while pin production may have been focused around major mercantile, royal or ecclesiastical centres, it was also localised in terms of materials and production methods, suggesting potentially different trajectories in each region for the development and control of specialist production.

Chapter 9 - In conclusion this chapter presents a synthesis of the articles. The discussion evaluates the methodology for the use of HHpXRF on corroded copper-alloys. It also addresses the evidence for compositional variation in large numbers of typologically similar objects. Regional and chronological differences are discussed where identified, followed by the evidence for deliberate compositonal control. Following this the availability and source of the raw materials are considered. Lastly the social organisation of production, exchange and notions of identity associated with these personal objects are discussed against the projects overall aims.

A discussion then follows regarding limits of the methodology. Its weaknesses are explored against the scientific debate concerning the use of HHpXRF on corroded copper-alloys, especially focusing on the validity of the qualititative versus quantitative approach and also the potential for misinterpretation by less experienced operators. Finally, the future development of HHpXRF is addressed, in its application on corroded copper-alloy objects.

The role of the co-authors

Prof. dr. Hans D. J. Huisman has a background in soil science and geochemistry. He worked for several years in the geochemical laboratory of the geological survey of the Netherlands, before joining the Cultural Heritage Agency of the Netherlands (RCE) as a senior researcher (soil science and degradation of archaeological remains) in 2003. He is also a professor of geoarchaeology and archaeometry (by special appointment), at the Groningen Institute of Archaeology (GIA), University of Groningen. From 2009 to 2015 he held an ancillary position as lecturer at the Faculty of Archaeology, University of Leiden. His main interests are micromorphology and also chemical research on ancient artefacts, including the changes brought about by the corrosion of copper-alloy artefacts when buried in the soil. He was involved in the development of the methodology, in particular the decision to apply the qualitative/semi-quantitative approach to the study of large groups of artefacts, and in understanding the chemical processes taking place within the formation of the copper-alloy patinas of the destructively tested Nijmegen brooches.

Dr. Bertil. J. H. van Os has a background in geology and geochemistry. He works as a senior researcher of inorganic materials at the Cultural Heritage Agency of the Netherlands. His research is focused on wetlands, archaeological artefacts, built environments and the degradation of cultural heritage in general. He has had over 25 years experience of many scientific analysis techniques such as those used in spectrometers including XRF. One speciality in particular is the application of HHpXRF on soil and archaeological artefacts. His contribution to the research presented here was primarily in the development of the methodology, especially in the use of the Niton HHpXRF device. More specifically he was responsible for outputting the raw data from the device into a numerical format suitable for use in Microsoft Excel. This also involved a quality control check of the data, for system and other errors, as it was being outputted. He was also reponsible for the initial data normalisation.

Dr. Stijn Heeren was a lecturer and post-doctorial researcher at the department of archaeology, the Vrije Universiteit Amsterdam, before taking up the position of coordinator of the PAN programme (Portable Antiquities of the Netherlands). His main research interests are rural communities, their settlements, cemeteries and material culture, from the Late Iron Age into the Early Middle Ages. This especially includes the Late Roman period (3rd to 5th centuries) in the Low Countries and metal artefacts in particular. His role in the project was to provide additional specialist typological knowledge (for chapters 2 and 3) on the Roman period brooches and to assist in the formulation of the interpretation and subsequent hypotheses.

Dr. Nelleke L. IJssennagger was the curator of the archaeological and medieval collections at the Fries Museum in Leeuwarden, whilst working on her PhD at the University of Groningen (Dept. of Medieval History). Her thesis explored the position of Frisia in, and interconnections with, the Viking Age North Sea world, through both material and written sources. This included metal-detected finds. Her role in the collaboration was to assist in the typological analysis and the formulation of the subsequent hypotheses for the artefacts found at Domburg (chapters 6 and 7).

Maarja Olli is a PhD researcher at the department of archaeology, University of Tartu where she is mainly interested in understanding regional identities in the *tarand* grave distribution areas during the Roman Iron Age. Her role in the article in chapter 4 was that of lead author, focusing on the typological analysis and subsequent interpretation of the combined typological/compositional results.

Pim van Tendeloo was a Research Masters student at the department of archaeology, the Vrije Universiteit Amsterdam. His research project under the supervision of Prof. dr. Henk Kars and Prof. dr. Hans D. J. Huisman (see above) was on the copper-alloy artefacts found at Domburg. This also included HHpXRF analysis. The dataset that was produced was subsequently donated to the bigger project of the first author. As well as kindly donating the dataset he also contributed in the interpretation of the results for chapter 7.

As well as the specific roles mentioned above, all of the authors also volunteered their time at the depots and museums assisting in the gathering of the typological and compositional data.

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