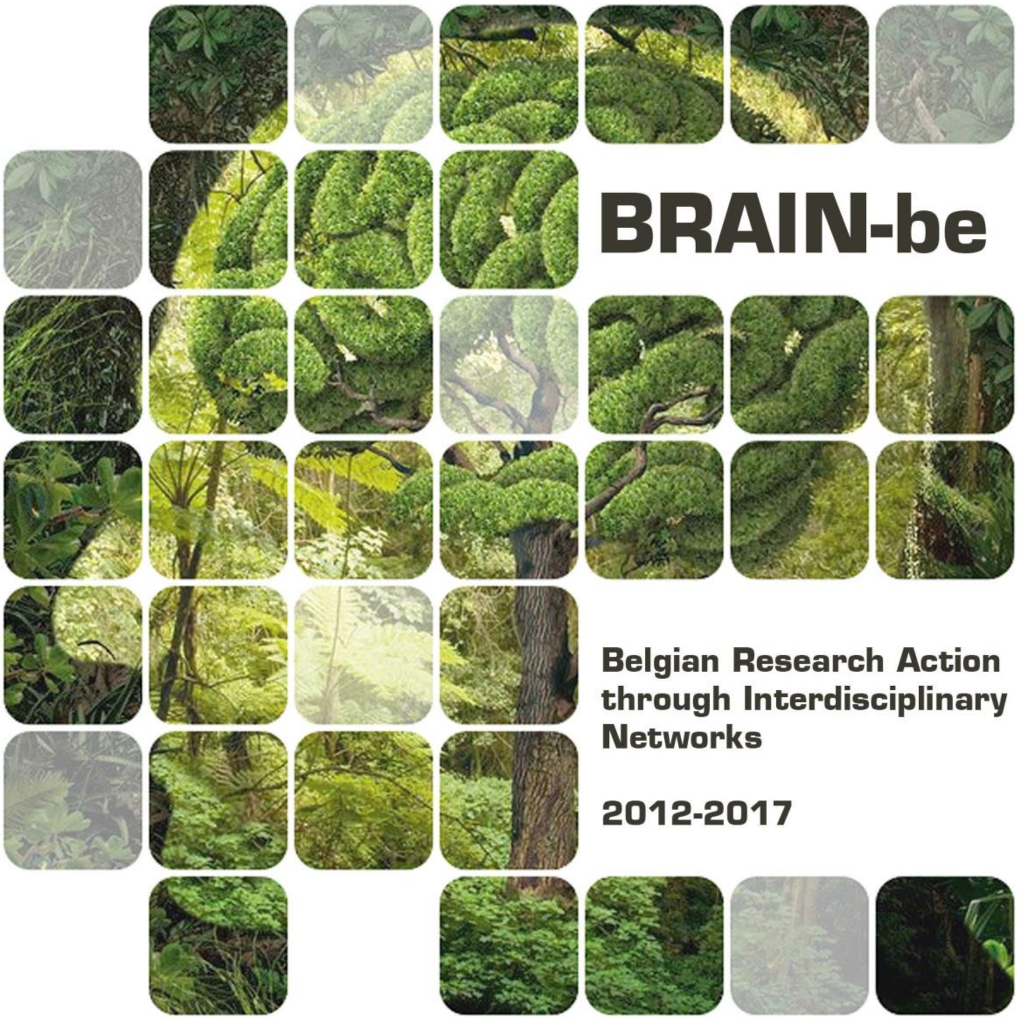

EACOM

Egyptian and African Copper Metallurgy

BR/143/A3/EACOM

Luc Delvaux (Royal Museums of Art and History / Antiquity)

Axis 3: Cultural, historical and scientific heritage

NETWORK PROJECT

EACOM

Egyptian and African Copper Metallurgy

Contract - BR/143/A3/EACOM

FINAL REPORT

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ABSTRACT

Context
The BRAIN-BE EACOM project began in the first semester of 2015 and lasted until the last semester of 2019. The project aimed to **re-contextualise and increase the qualitative value** of the material linked to copper metallurgy in the Royal Museums of Art and History (RMAH), the Royal Museum for Central Africa (RMCA) and the Department of Earth and Environmental Sciences (KU Leuven) through a multidisciplinary study of copper manufacturing processes in Ancient Egypt and in sub-Saharan Africa. The aim is to gain a more accurate image of the context of early production and use of copper as well as to increase the scientific and societal impact of the studied collections, drawing on three research axes, archaeology, ethnography and experimental archaeology, as well as archaeometry.

Objectives
The main goals of this project were to **re-contextualise and increase the qualitative value** of the material linked to copper metallurgy in the Belgian federal scientific institutions, and to increase the scientific and societal impact of these collections. The idea was also to gain a better understanding of the context of early production and use of copper, through a multidisciplinary study of the copper *chaîne opératoire* (C.O.) in Ancient Egypt and in the rest of Africa, based on the aforementioned federal collections. This meant the development of a more accurate image of the metallurgical processes, using multiple sources of information drawn from the archaeological, historical and ethnographic record pertaining to past and present societies, supported by experimental reconstructions of ancient processes and archaeometric analysis. In doing so, our aim was to shed light on some of the *sleeping beauties* in the Belgian federal collections and offer significant contribution to the history of early copper metallurgy and its role in the history of African societies.

Conclusions
The development of a common framework, organizing the *chaînes opératoires* of copper metallurgy, pertaining to primary and secondary metallurgy, had the expected benefit. It allowed for a much needed re-organisation and re-contextualisation of the collections, using the various sources, from ethnography to archaeology and experimental archaeology, in an integrated narrative. It also provided a common ground needed for a fruitful pluridisciplinary collaboration. Using ethno-historic data available at the RMCA allowed Partner 1 & 2 to develop an analytical framework, which was improved with experimental and archaeological inputs from all the partners. One of the usual benefits of the development of such an analytical grid is to be able to ask the right questions, making way for particularly fruitful analytical results.

In practice, for each collection, all the artefacts related to copper metallurgy (ceramics, waxes, ore fragments, copper-based artefacts, etc.) needed to be collated in one coherent set. Objects had to be re-contextualized, first, through the reconstruction of the original lots to which these artefacts belonged, lots that were dispersed throughout Europe at the time of their discovery and, second, through external data from several disciplines, archaeology, archaeometry, ethnology and experimental archaeology.

Thanks to our research, we are now able to provide for the selected artefacts: an archaeological context, a technical description, an archaeometric study, an attribution to a specific *chaîne opératoire*, documentation in focus stacking, µCT-scan and three-dimensional photography, a study by experimental protocols and data for the museum databases.

Due to the wealth of the RMAH and RMAC collections, both in their scope and diversity, there was much to be gained by studying, jointly and comparatively, the two collections. The identification of the social and technical context of these *sleeping* collections considerably increased their qualitative value. This also meant bringing to the public attention a different view on past societies by revealing the techniques behind the objects and by emphasizing the importance of seemingly unspectacular artefacts, offering deep insight into everyday life and the organization of extinct cultures.

Besides published material in peer-reviewed journals, our output, not subjected to copyright non-disclosure policy, is now partly available online on the zenodo server at the following address: https://zenodo.org/communities/eacom.

Keywords

Copper - Archaeometallurgy - Archaeometry - Methodology - *Chaînes opératoires* - Experimental archaeology - Egyptology - Old Kingdom - Middle Kingdom - New Kingdom - African archaeology - Central Africa – DRC – provenance study.

INTRODUCTION

In Egypt, as in Sub-Saharan Africa in general, Copper has played an important role, both as raw material for the production of ornaments, artwork and tools and as a medium of exchange. In many regions, copper was considered as a valuable metal and almost exclusively used for adornments or symbols of status. It was also used as a store of values and regularly used as a medium of exchange (Herbert 1984). In Central Africa, this metal was closely linked to power and the rise in its production from the 9th century AD onwards has been linked to the development of hierarchical societies and major kingdoms in the area. For example, control of the copper supplies would have been a significant factor in the success of the Kongo and Loango kingdoms, major polities of west Central Africa (Hilton 1985; Martin 1972). Most of the locally produced copper used in and around the Congo Basin came from two main deposits, the Niari Basin (South Republic of Congo) and the Copperbelt (South-east DRC, Fig. 1).

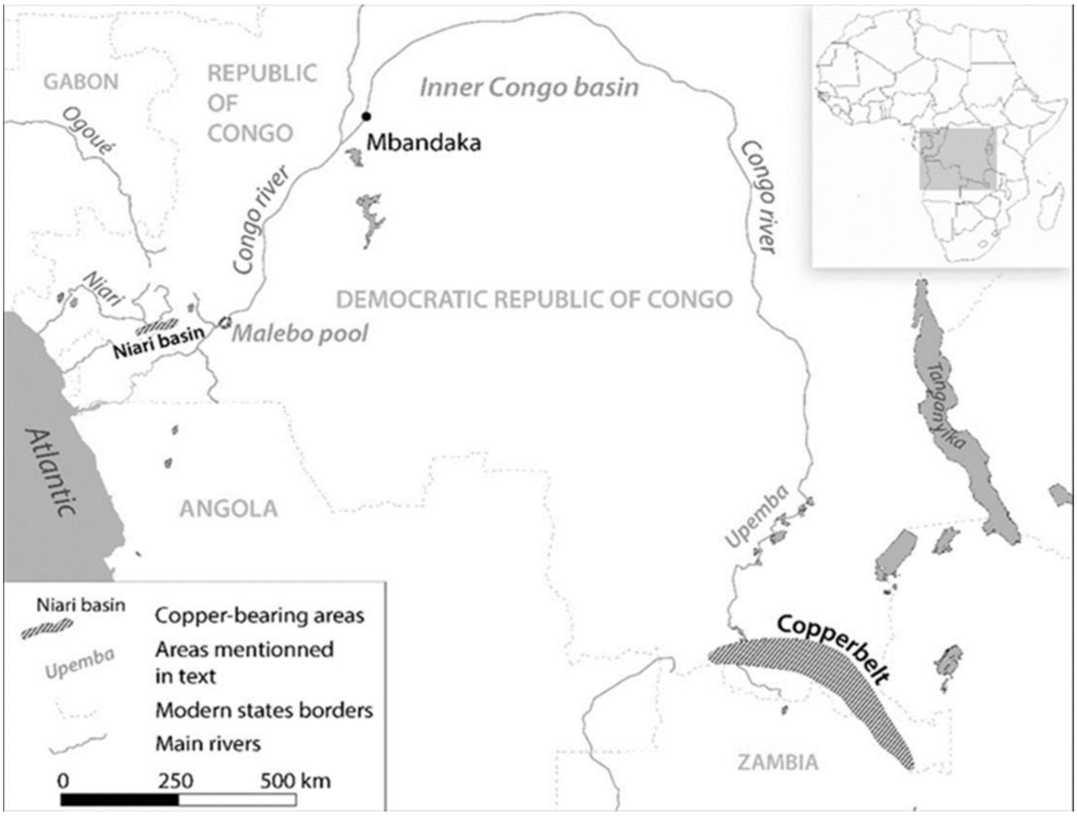


Figure 1: Location of the main geographic area of Central Africa mentioned in text (© N. Nikis 2019)

In Ancient Egypt, as in many economic sectors, metallurgy demonstrated its importance by the scale of the creations it enabled (construction of the pyramids, temples, funerary monuments, decorations...), the exploitation of resources, the metallic artefacts produced and the rise of an entire civilization. The latest work carried out by Pierre Tallet and his team opens up research perspectives that reassess the importance of the exploitation of the Sinai and the eastern desert, where metallurgy is the primary resource sought after. This increase in metallurgical activity accompanies an Egyptian state that is growing stronger, which is being built up over the centuries and which implements economic exchanges with its neighbours in order to carry out grandiose projects. Without the contribution of copper and its alloys nothing would have been possible.

1. STATE OF THE ART AND OBJECTIVES.

Egyptian metallurgy holds an ambivalent place in Egyptology. It is both constantly cited for the masterpieces produced and at the same time we know little of its many practical aspects (Scheel, 1989, Farag, 1981, Garenne-Marot, 1985). Our ignorance includes, for example, the respective roles of the state and the private sector in its production, information on the specific operating chains (Ogden, 2000), its diachronic evolution, sources of supply (Nigro, 2014), the mining systems, or the period when alloys were voluntarily introduced (Kallfass & Hörz, 1989). In fact, technological and archaeometric aspects are totally absent from art history discourses (Gouda et al., 2012). Until recently, the numerous metallurgical production centers have been neglected for essentially structural reasons, with only few notable exceptions (Rademakers 2015, Rademakers et al., 2017, 2018b, Rehren and Pernicka 2014, Rehren and Pusch 2012).

The whole of the RMAH's metallurgical Egyptological collection remained largely unexploited until the beginning of the project (Mathis et al., 2009 / Riederer, 1984). The collection presents a wide range of ancient Egyptian products: tools, vessels, figurines, jewellery, etc. Part of the collection was acquired by purchase but most of the artefacts can be contextualized thanks to the excavation reports of the Egypt Exploration Society, or the British School of Archaeology in Egypt (some of the great names in Egyptology are associated with the constitution of the collection; for example: Petrie, Garstang, Reisner...). This means that we are able to define solid archaeological and historical contexts. This long process of contextualization then allows us to use the archaeometric data to define a mining zone by isotopic origin (in connection with all the research carried out by Petrie 1906; Rademakers 2017, 2018a, 2018b, 2020, in preparation-b; Tallet 2012a-b, 2016, 2017, 2018, forthcoming; Verly et al. forthcoming a, in preparation a) or to define the type of alloy used. These two approaches then make it possible, using our holistic method, to suggest a diachronic technological vision. This vision is completed and greatly enhanced by the excavation of emblematic archaeological sites related to metallurgy (Egypt: Qantir, Ayn Sukhna, Amarna (Tallet et al. 2011; Verly et al. 2017, submitted) / Sudan: Kerma, Dukki Gel (Verly et al. in preparation b-c-d) and Israel: Timna). Finally, experimental archaeology based on the artefacts preserved in the collection and excavated structures makes it possible to propose specific *chaînes opératoires* to suggest a history of the techniques used (Verly and Longelin 2019). For the first time, hundreds of artefacts have been described separately (between 10 and 15 pages each) scientifically increasing the value of the collection and the acquisition of technological knowledge. All this research has created a synergy with three other Egyptian museums (Copenhagen, Ny Carlsberg Glyptotek; Frankfurt, Liebieghaus Skulpturensammlung and Bonn Egyptian Museum (Auenmüller & Fitzenreiter, 2014)) to collaborate in archaeometry and archaeology. These three museums have different but complementary artefacts in their collections. This approach has also greatly increased the technological and archaeological understanding of the RMAH artefacts (Verly et al. forthcoming b-c).

The development of metallurgy in sub-Saharan Africa follows an independent path from Europe and the Middle East. Evidence for iron metallurgy is as old as, or older, than evidence for copper and archaeological information does not allow so far to determine whether iron metallurgy was independently invented in one or several points of Africa or imported from other regions (Alpern 2005). In Central Africa, it is thus highly likely that earlier copper smelters and smiths had first metallurgical knowledge in producing iron. This assumption is supported both by the dating of the early metallurgical evidence – earlier traces of copper smelting are dated around the 4th/5th century AD (Anciaux de Faveaux & de Maret 1984) in the Copperbelt while evidence for iron smelting has been dated between the 800 and 400 BC in north Central Africa (Childs & Herbert 2005) - and by studies suggesting that manufacturing techniques applied to iron have been transferred to copper (Childs 1991).

Copper manufacturing processes remained largely unstudied in Sub-Saharan Africa. Yet, identification of technical processes offers not only information on the way objects were made – allowing for example to reconstruct the *chaines opératoires* and to contribute to the history of techniques – but also allows, as a social phenomenon (Lemonnier 2010; Coupaye 2015), to reconstruct past social spaces, communities of practices and diffusion of knowledge and ideas (see for example, contributions in Roddick & Stahl (2016). In the case of museum collection, working on manufacturing techniques allows to add several layers of information to the object’s history but also, by knowing in detail their technical and material characteristics, to implement conservation procedures.

In sub-Saharan Africa, anthropological and archaeological studies of pottery and iron *chaine opératoire* has already a long history (see for example Gosselain & Livingstone Smith 2013; Coupaye & Douny 2009; Robion-Brunner 2010). Conversely, a combination of different factors – the scarcity of copper mining areas in sub-Saharan Africa, the early end of production because of the colonization and the lack of interest for the archaeology of the last two millennia before the 1960s – have prevented a similar development of ethno-archaeological and anthropological works on copper.

The archives and objects collected during the colonial period offer nevertheless an invaluable documentation on the manufacturing, exchange and use of copper. Literature and RMCA collection hold, among others, some very detailed accounts of copper smelting, casting in open mould and subsequent deformation processes and wire-drawing processes, backed-up by film, photography and related tools and by-products that can be used as reference material for technological studies on copper. Ethnographical and historical data have been for a long time the main sources of work on copper production (Herbert 1984; Volavka 1998; Ndinga-Mbo 1984) and archaeological evidence on copper production has indeed remained limited until the 2010s. In the Copperbelt area (DRC), in addition to the survey and small-scale excavations around Lubumbashi that provided the earliest date for copper smelting in the Tenke Fungurume mining area (Arazi *et al.* 2012), information on copper production mainly relied on the excavations of Michael Bisson in Kipushi and Kansanshi (Bisson 1976; Bisson 2000). They allowed to outline the evolution of the production between ca. the 8th c. AD and the 17th c. and to highlight some increase in the production first during the 9th and again during the 14th c. that have been related, in each case, to major change in the regional socio-political context (Bisson 2000). In the Niari Basin, only three copper metallurgical sites were excavated before the last decade but due to conservation issues (Lanfranchi & Manima-Moubouha 1984; Manima-Moubouha 1988) or to weak dating (M. Bequaert; material partly described by Clist 1982), they were poorly contextualised.

The excavations of more than 300 graves in the Upemba depression in the 1950s (Nenquin 1963; Hiernaux *et al.* 1971), 1970s (de Maret 1985; de Maret 1992) and 1980s (Childs *et al.* 1989) have provided more than 2000 copper and 1000 iron artefacts. Copper items include both manufactured objects, mainly jewellery, and ingots, giving the best regional insight of the way copper was used between the 8th and the 18th c. AD. Manufacturing processes of some of the copper and iron objects have further been investigated and the results, among others, allowed to show technical relations between the two metals (Childs 1991).

From 2013, a new project, conducted by Nicolas Nikis in the framework of his PhD thesis (2013-2018) in collaboration with the ERC funded project *KongoKing* (ERC Starting grant

20101124 to Koen Bostoen, UGent) was dedicated to the archaeological investigation of the mining areas of the Niari Basin. The material gathered during the nine months of fieldwork made between 2013 and 2015 thus provide new data to expand the research on the copper smelting processes in this part of Africa.

METHODOLOGY

Introduction

The interdisciplinary approach and the collaboration between three leading Belgian scientific institutions (RMAH, RMCA, KUL, ULB) allow the development of a joint methodology and analytical framework for the study of copper metallurgy. The project draws upon interdisciplinary research and expertise confronting and examining data collected from several fields. Methodologically, the first step was the development of a joint analytical framework to be used in the investigation of the manufacturing processes of the objects. The second step involved the inventory and collation of collections along the guidelines set in the joint analytical framework. The third step was to develop new reference tools to clarify or improve the initial analytical framework, based on the specific case studies investigated in the different areas and at different periods (see: Scientific results).

Questions arising from the observation of the collections pertain to their nature (overall composition), their origin (source or provenance) and the way they were transformed/manufactured. In addition to information provided by the archaeological and historical sources, combined with experimental archaeology, these questions were investigated through the use of various analytical methods. Analysis techniques included: surface observation and identification of macrotraces (binocular observation, SEM, multispectral imagery, focus stacked photography), qualitative assessment of elemental composition of metals (XRF), quantitative compositional analysis of major and trace elements (ICP-OES and Q-ICP-MS), lead isotope analysis (MC-ICP-MS), and metallographic examination to reveal the physical structure and components of the object. The KUL partner (P3) was in charge of archaeometric analyses. Whenever possible, the methods used have been non-invasive in order to avoid damaging the artefacts. When invasive methods proved indispensable, all possible measures were taken to insure a minimum amount of damage to the material.

Analytical method selection

Handheld XRF (X-ray Fluorescence) analysis is a non-destructive technique used which can be used to assess the elemental composition of metal artefacts (e.g., Shackley 2011). X-rays of a chosen energy are generated by the instrument, which excite the various elements present in the artefact. These elements, in turn, generate secondary X-rays, of which the energy is measured. The specific energy of these secondary X-rays allows the identification of the elements present in the artefact, while their quantity provides an indication of the abundance of these elements. The measurement can be conducted directly on the surface of the artefact, in the course of less than one minute up to a few minutes. This makes it an excellent technique for fast qualitative assessments of alloy compositions in metal artefacts. The downside of this methodology, however, is that only surface compositions are determined: in the case of untreated ancient artefacts, this is typically a corroded/patinated surface, which does not correspond exactly to the true metal composition. The bias introduced by corrosion is highly variable and cannot be readily corrected for. Furthermore, the irregular surface geometry impedes good calibration of the measurement results. The non-invasive approach is therefore necessarily qualitative. For this reason, handheld XRF is deployed for explorative analysis of the museum collections, enabling a complete qualitative overview of copper alloy compositions to be obtained for the entire collections in a non-invasive manner.

For the quantitative determination of elemental composition, a destructive technique is required. Hereby, a sample is taken from the artefact (typically around 0.1 g), which is weighed and dissolved completely in acid. This dissolved sample can then be used for a variety of analysis techniques: ICP-OES or Q-ICP-MS for elemental analysis, and MC-ICP-MS for isotope analysis.

ICP-OES and Q-ICP-MS or MC-ICP-MS, differ fundamentally in their measurement procedure and thus provide distinct information for a sample. In both cases, the dissolved sample is introduced into an ICP (Inductively Coupled Plasma) torch, which causes the sample to dissociate into its constituent elements (atomisation).

In this high energy state, the elements[1] undergo excitation, whereby electrons jump up to higher valency shells. When these electrons fall back to their ground state, energy is released in the form of electromagnetic radiation. The wavelengths of the emitted radiation are characteristic of the elements which are excited (and thus present in the sample), while the intensity of the radiation is proportional to the abundance of those elements in the sample. Thus, the measurement of these two properties of the emitted radiation allows the elemental composition of the sample to be determined: this is precisely what is measured by ICP-OES: Inductively Coupled Plasma – Optical Emission Spectroscopy.

---

[1] Ionisation (charging) of the elements is suppressed during ICP-OES.

This emitted radiation, however, is specific to the element level only - it is not possible to differentiate between isotopes of the same element. ICP-OES is therefore used to determine elemental compositions ranging from the percentage down to the ppm (parts per million) level.

In the case of ICP-MS, the sample is equally introduced into an ICP torch (causing atomisation). The elements are then further ionized to obtain charged particles. Next, the ions are introduced into a controlled magnetic field which causes a separation of the ions' trajectory based on their mass-to-charge ratio. Thereby, different isotopes of the same element are separated along their trajectory through the magnetic field.

In the case of a single detector ICP-MS (Q-ICP-MS: Quadrupole Inductively Coupled Plasma Mass Spectrometry), the individual ions (isotopes) are measured sequentially by varying the magnetic field over time. It is therefore possible to measure (lead) isotope ratios with a single detector ICP-MS. However, due to minor variations in sample abundance being introduced into the ICP torch, significant errors are obtained when using this method for isotope determination. Rather, Q-ICP-MS is used to determine trace element compositions in the ppm-ppb (parts per billion) range. Q-ICP-MS is therefore only used for particular geological applications, while ICP-OES is generally sufficient for trace element analysis of copper alloys.

Multi-collector ICP-MS (MC-ICP-MS), however, measures all relevant isotopes simultaneously (up to 7 detectors are placed at the end of the trajectory for each ion/isotope). This eliminates the problem of variability mentioned above for single detector ICP-MS, and allows 10 to 100 times better precision to be obtained for isotope ratios. This precision is essential in order to make meaningful distinctions between samples: if the error is too high, the uncertainty interval becomes larger than the difference between samples. MC-ICP-MS is the state-of-the-art method for determining isotope ratios.

ICP-OES and MC-ICP-MS thus provide two fundamentally different and complementary data-sets: elemental composition and lead isotope ratios, respectively. Neither of these datasets alone is usually sufficient to allow a distinction between possible (geological) provenance regions, as different mining sites may have similar characteristic element abundances or lead isotope ratios. Combined, however, these data have proven to greatly increase the opportunities to distinguish between potential ancient mining sites - the main power lying in the exclusion of potential sources when they are incompatible with elemental composition and/or lead isotope ratios. For this reason, the combination of these two methods has been the standard approach to copper provenance for almost 4 decades now (e.g., Pernicka 1999, 2014).

Metallography remains an important analytical method. Metals are made up of minute crystals with distinct shape, structure alignment, and size. The factors controlling the microstructure of metals include, among others, their composition and the thermomechanical processes during object manufacture (Bayley 1982; Scott 1991). Metallographic examination, a destructive method, allows thus to determine manufacturing techniques. The procedure implies cutting small blocks of metal from the object, mounting them in epoxy resin blocks, and polishing them with progressively finer grades. The polished surfaces are examined under a microscope in reflected plane and polarized light in both unetched (in order to highlight porosities, inclusions and intergranular elements) and etched (revealing the granular state) conditions. At the Centre de Recherches et de Restauration des Musées de France (C2RMF), Paris, a Zeiss AXIO IMAGER.M2m reflected-light optical microscope equipped with a digital camera driven by AxioVision SE 64 software was used to capture and record observed features.

Metallographic examination was conducted in the course of the authentication of the early presence of the wire-drawing technique in Central Africa. It was an additional tool to the ones used in surface investigation (e.g. Scanning Electron Microscopy). Samples were taken from archaeological wires from copper jewellery from Late 9th century burials from the Upemba Depression in south eastern Democratic Republic of Congo (DRC), host in the RMCA collections. The microstructures of these samples were compared to that of attested drawn wires from the RMCA ethnographic collections to see if the wires from both origins had gone through the same deformation processes.

The aim of this approach was to make a first selection. Knowing the elemental compositions of the alloys, this first analysis made it possible to complete the lack of information for some of the artefacts in the collection. We were thus able to directly focus our efforts on the most convincing artefacts for further research. This approach also made it possible to determine whether there were any dating errors.

Development of an analytical framework.

Ethnographical comparisons were used as a reference tool to build a general framework for copper production. The Heritage section of the RMCA has a long expertise in these matters with the works conducted on the pottery-making process by A. Livingstone Smith and O. Gosselain. Furthermore, the large collection of ethnographic archives in films, photographs and descriptive reports on the copper production and transformation in the Congo kept in the RMCA constitute an extraordinary wealth of information on, for example,

- Traditional technical processes in relation to copper metallurgy from the reduction of copper ores to the casting and hammering of the metal
- The characteristics of clays used for the ovens and on the traditional ways to improve these clays
- The importance of the type and quality of the fuel
- The way the immediate environment is exploited for maximum efficiency in the process
- On the simplification of technical processes that do not entail a loss of performance

Of course, the direct transposition of the traditional know-how and techniques of the *mangeurs de cuivre* of Katanga to the archeometallurgical processes used in ancient Egypt is senseless. But a number of similarities were identified between the structures and tools found in ancient Egypt and those used in the Katanga area during the 19th-20th century. Ethnographic parallels also offer a unique insight in the intangible aspects of copper working, aspects that can only, at best, be hypothesized in the material remains of ancient civilisations: the human effort put into this type of craft, the notion of work rhythm, sometimes set by music or sounds, the extreme attention given to the choice of primary material or societal taboos.

Using this ethnographic data as a guideline, an analytical framework was developed so that each object in the RMAH and RMCA collections could find a place in the process of copper metallurgy (LGM; P1 GV; P2 RMCA & NN, P4 ULB).

This framework divides the production of copper objects along the following lines: *Raw material procurement* (mining, beneficiation), *primary metallurgy* (roasting, smelting, refining), *secondary metallurgy* (shaping, finishing). The idea is that the concept of a single *chaîne opératoire* for copper metallurgy is not completely adapted, as the process of transforming ore into objects, down to recycling, involves a series of disjointed *chaînes opératoires* (see Fig. 2 below).

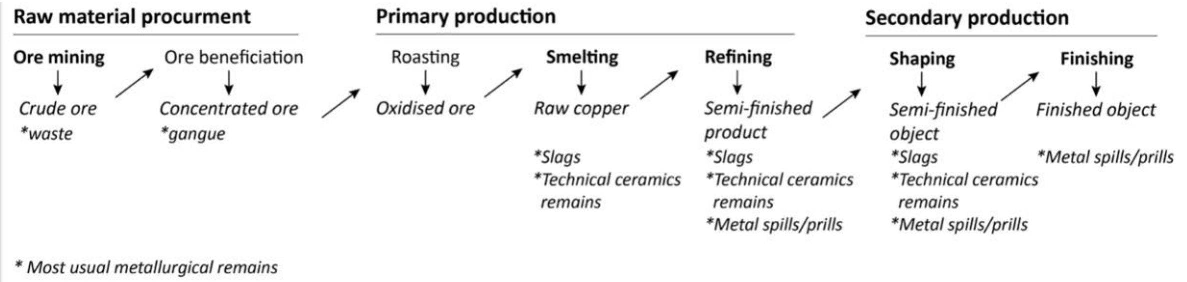


Figure 2: Schematic manufacturing process of an unalloyed copper object as usually observed in Sub-saharan Africa. This figure is based on the use of oxide or carbonate ores, sulphide being rarely ever used south of the Sahara before the late 19th century (N. Nikis 2018).

This analytical framework has structured the research all along the project, providing a theoretical division that allowed to compare different *chaînes opératoires* for the same steps of the metallurgical process or objects related to the same sequence of steps.

In addition, a **table of shaping techniques** (see Annexe 1) was created to synthesise the processes related to the manufacture of objects (LGM; P2 RMCA). The techniques are presented and explained using examples of each specified technique drawn from the African field, both ethnographic and archaeological. The English part was revised -for the syntax and spelling- by Ellen Howe, emeritus conservator at the *Metropolitan Museum* (MET), New York; she added examples from her own expertise with African collections in the course of her career at the MET. Such an overview copper shaping techniques/African examples displaying these techniques is something that has never been done in a systematic way. In relation to the analytical framework and the forming techniques table, mentioned above, a recap was done of objects and tools within the RMCA collections (LGM; P2 RMCA) that could be potential examples for to vividly illustrate the diversity of manufacturing techniques applied to copper. (*See part 5. DISSEMINATION AND VALORISATION*).

Assessment, collation and study of the data in FSE collections

The data available in the 10,000 RMAH files and the files of the RMAC and the excavation archives make it possible to locate and certify objects from different archaeological sites. This study of the material made it possible to:

1. Fully evaluate all the material relevant to the project, excluding purchases and acquisitions without provenance

2. Assess the state of conservation and take preventive measures if necessary

3. Rearrange the storerooms according to the type of material and its relevance to the study project

4. Making digital recordings using advanced imaging (high-resolution photography, focus stacking, micro-CT scanning and 3D imaging)

5. Make the material available online to the scientific community and save it in virtual format

6. Recontextualization of ESF collections by virtual reconstruction of ancient assemblages

7. Updating the museum's online databases

Recovery of material scattered throughout European museums from the same contexts of discovery as the objects conserved in the RMAH and RMCA. It was necessary to trace the "origin" and identity of each artefact in order to reconstruct its history and restore its qualitative scientific value.

The use of µCT-scan allows the study of unique non-metallic antique artefacts. The goal is to perform non-destructive analyses in order to thoroughly analyze each artefact. In addition, this very special type of analysis allows to visualize them in three dimensions, layer by layer. This methodological approach makes it possible to build up a database (which is a first in the world) as a basis for experimentation and understanding of this ancient technology (lost wax technique). The database was completed thanks to µCT-scan: layer composition, number of layers, distribution, dimensions and application method.

Re-contextualisation of the FSE collections by virtual reconstruction of former assemblages
Retrieval of material dispersed through European museums coming from the same discovery contexts as the artefacts held in the RMAH and RMCA. The « origin » and identity of each artefact had to be traced in order to reconstruct its history and restore its qualitative scientific value.

Interpretation of the FSE collections following the steps of the metallurgical process
With the data compiled in the two previous phases, work has focused on studying the diachronical evolution and the regional variations of each step in the copper metallurgical process – from extraction to the finished object. This *chaîne opératoire*-based approach allows the best coherence in the understanding of the process. To this aim, archaeological, ethnographical and archaeometric data have been gathered and confronted with the material in the collections, allowing for their reinterpretation and re-contextualisation. Indeed, a re-examination of the artefacts with scientific standards and proper identification of their place within the *chaîne opératoire* has led to a better historic and archaeological contextualization of the objects.

Experimental archaeology
Numerous experimental campaigns were also carried out by G. Verly (P1 GV) in primary and secondary metallurgy in order to better understand the archaeological data and to better excavate future archaeological structures. Indeed the archaeological data are both in museums (in the form of artefacts) and on excavation sites (in the form of structures and artefacts).

Each archaeological discovery is an enigma that raises many questions. Without the methodological input of experimental research combined with analysis, it is impossible to bring new interpretations and specific new *chaînes opératoires*.

A completely new methodology has been written to make this research tool valid as a scientific tool, quantifiable, protocoled and usable for archaeology (Verly & Longelin 2019).

Smelting: After several years, the smelting restitutions made it possible to establish a first *chaîne opératoire* of smelting based on the excavation data from Ayn Sukhna for the Middle Kingdom. This type of smelting process was under study. The new experiments basically proposed a completely new *chaîne opératoire* and a new excavation method. By combining archaeometry, we even managed to focus attention on isotope changes that are unknown to date.

Melting: After several years, the melting restitutions made it possible to establish a first melting *chaîne opératoire* based on the excavation data from Ayn Sukhna for the Middle Kingdom. This type of melting process was not yet well known before the experiments. Thanks to our methodology, technical material analyses and spatial-technical data from Ayn Sukhna, we have improved our excavation techniques, made new interpretations of metallurgical scenes and collaborated in research work in glass technology.

Lost wax technic: Following the example of the experiments protocoled for the smelting and the melting, the restitutions of the lost wax technique have also made it possible to make significant progress in the establishment of the *chaîne opératoire* for the New Kingdom and the Late Period used for the creation of figurines. A completely new interpretation of the layers of clay pasta has been made, inducing a new technological understanding of production. Collaboration with the Bonn Egyptian Museum was an important success factor, associated with the analyzes of the University of Ghent. Since, all the three-dimensional production of the RMAH collection have been re-evaluated according to new technological discoveries (full body, open core and lost core).

Archaeo-metallurgical experiments were made to explore some hypothesis concerning the use of tools or the detail of reconstructed processes. F. Rademakers further participated in experimental smelting campaigns (Melle (FR), Aubechies (BE)), whereby new data acquisition on the process parameters took place, such as continuous furnace temperatures at multiple locations, hearth colorimetry, fuel and ore consumption. Most importantly, this has allowed the establishment of a detailed temperature profile through time within these furnaces under different operating conditions. From these results, the experimental protocol was further refined for continued experiments till the end the project.

P3 (F. Rademakers) has actively collaborated on this experimental field work (Ayn Sukhna copper smelting technology) at Melle and Aubechies. He has taken part in the development of an integrated protocol for the archaeometric study of the experimental products, as well as running many of the experimental smelts together with p1 (GV). The confrontation of museum material with archaeological data thus led to the realization of important operations of experimental archaeology.

Verly, G. September 20-21, 2014: rediscovered wax
Purpose: de-waxing, bellows temperature rise and bronze castings
Technique: early Iron Age (Rhenish furnace), Etruscan clay pots

Verly, G. September 27-28, 2014: smelting of malachite for copper production
Purpose: crushing of malachite, reduction of ore to charcoal and horse dung
Technique: Ayn Sukhna, Middle Kingdom (smelting furnace, charcoal, donkey dung, malachite, sorting stone)

Verly, G. October 4-5, 2014: smelting of copper balls in a crucible furnace
Purpose: to melt the copper balls in a clay crucible, to increase the temperature by forced ventilation (two pipes and anthropic blast), to hold the crucible with a stick and to cast copper chisels.
Technique: Ayn Sukhna, Middle Kingdom (furnace, melting crucible, pipe, charcoal, wooden handle, mould)

Verly, G. April to November 2016: lost wax technique
Purpose: control of clay paste layers based on archaeological data, dewaxing, casting of 40 moulds: Osiris, Osiris with bronze foot of open-core and lost core artefacts
Technique: Ayn Sukhna, Middle Kingdom (furnace, melting crucible, pipe, charcoal, wooden handle, mould)

Verly, G. April to November 2016: lost wax technique – copper tools
Purpose: control of clay paste layers based on archaeological data, dewaxing, casting of 40 moulds: Osiris, Osiris with bronze foot of open-core and lost core artifacts
Technique: Ayn Sukhna, Middle Kingdom (furnace, melting crucible, pipe, charcoal, wooden handle, mould) / Timna, LBA (mines experimentation)

Verly, G. April to November 2018: creation of the metallurgical workshop to study lost wax techniques

Purpose: to test a dewaxing kiln according to archaeological data and to experiment with pottery protocols based on the technique of the archaeological evidence of Qubbet el-Hawa (Museum Bonn)
Technique: Qantir - KomTuman - Kerma, Middle Kingdom (dewaxing and melting furnaces) / Qubbet el-Hawa, Late Period (moulds QH 207)

Verly, G. 15 June - 6 July 2019: creation of the metallurgical workshop to study the production of metal plates
Purpose: to test a "de-waxing" kiln according to the archaeological data of Kerma.
Technique: Kerma, Classic Kerma (cross ovens)

Verly, G. 15 June - 8 July 2019: International Conference ICA 2019 (ICA2): Contributions of Experimental Archaeology to Excavation and Material Studies
Organization of the second EACOM international conference at the Sorbonne and MSH Paris-Saclay universities and at the Melle experimental platform: https://metallurgy-ica.wixsite.com/ica2/

Verly, G. 3-20 June 2019: creation of the metallurgical workshop to study lost wax techniques
Purpose: to test a dewaxing kiln according to archaeological data and to experiment with pottery protocols based on the technique of the archaeological evidence of Qubbet el-Hawa (Museum Bonn)
Technique: Qantir – Kom Tuman - Kerma, New Kingdom (dewaxing and melting furnaces) / Qubbet el-Hawa, Late Period (moulds QH 207)

Verly, G. 25 September - 2 October 2019: International Conference ICA 2019 (ICA2): Contributions of Experimental Archaeology to Excavation and Material Studies
Organization of the second EACOM international conference at the Sorbonne and MSH Paris-Saclay universities and at the Melle experimental platform: https://metallurgy-ica.wixsite.com/ica2/

Composition of museum artefacts

At the outset of the project, a compositional overview of the collections to be studied had to be established. A preliminary explorative chemical study was conducted using non-invasive handheld X-ray fluorescence (HH-XRF). This technique has enabled the qualitative elemental composition of all copper alloy artefacts in the collections, identifying their overall alloy composition as well as detecting compositional variability within the artefacts themselves. This preliminary data has been gathered in a database for each object and is integrated in the object descriptions of the respective museums. At the RMAH, a total of 500 artefacts were analysed by HH-XRF at the RMAH and 115 artefacts at the RMCA. At the RMAH, a total of 218 samples have been taken. The majority of these (195) concern copper alloy artefacts. The remaining 23 samples are organic materials (wax) related to the production of copper alloy artefacts.

Finally, the organic materials have been analyzed using GC-ICP-MS (Gas Chromatography Inductively-Coupled-Plasma Mass Spectrometry) to characterize their composition. The results of this study are yet to be published, but represent the largest study of technical wax use in ancient Egypt so far.

The results of the preliminary HH-XRF have been instrumental to the selection of artefacts for further, more detailed study. Combined with the information from the other partners (research on the collections' history and acquisition), artefacts have been selected for invasive sampling. The aim has been to obtain a holistic diachronic and geographical perspective on the technology and provenance of copper in Central Africa and Egypt. These samples were selected to represent the broad archaeological variation which is offered by the federal collections.

Raw material procurement

The different steps related to the acquisition of the ore were considered in different ways. First, partner 3 (NN) investigated the nature of precolonial mining activities in Katanga. Although, evidence in the archaeological and the historical record are patchy, this led to an assessment the type of ores which would have been potentially accessible to ancient metal workers (Nikis 2018a). A crucial aspect of the project has been the characterization of these ores in the case of the Niari Basin, Republic of Congo. Samples from different production remains (including ore, slag and copper) were analyzed (ICP-OES, MC-ICP-MS and ICP-MS). Second, archaeological excavations were undertaken on Egyptian mining in Timna (GV, FR, Verly et al. 2019). Furthermore, work on copper and lead ore in the RMAH collection has been conducted, expanding the database of ores mined in Sinai and the Eastern Desert (Rademakers et al. 2018c, in preparation).

Primary metallurgy

Copper smelting processes in Central Africa have been mainly investigated according to two lines of research: (a) the confrontation of the collection with the archaeological and geological data and (b) the geochemical investigation of metallurgical remains and copper artefacts, including some of the objects host in the collections.

(a) Archaeological surveys and geological data (ALS, P2, NN, P4 and TDP, P5)

In the Niari Basin, most of the comparison data come from the new archaeological and geological survey made around the main deposits of the region, the Mindouli, Mfouati and Boko-Songho mining area (Fig.3) between 2013 and 2015 (Nikis *et al.* 2013; Nikis & Champion 2014; Nikis & De Putter 2015; Clist *et al.* 2014). These new researches allowed for a characterisation of sources and artefacts, as well as for providing absolute dating for certain operating processes and certain type of objects. This is a typical case were old, poorly documented, collections generate new research, which, in turn, improve our knowledge on museum collections. The data and material collected during these fieldworks allowed to recontextualise finds from old archaeological and surveys and excavations. They also helped geological samples held in the collection of the RMCA and poorly studied. In turn, the RMCA collection allowed to expand the material available and insert the 2013-2015 research in a broader context. The pottery collection is particularly useful to reconstruct the sociocultural context the smelters were involved in and thus, insert the *chaine opératoire* in the broader society.

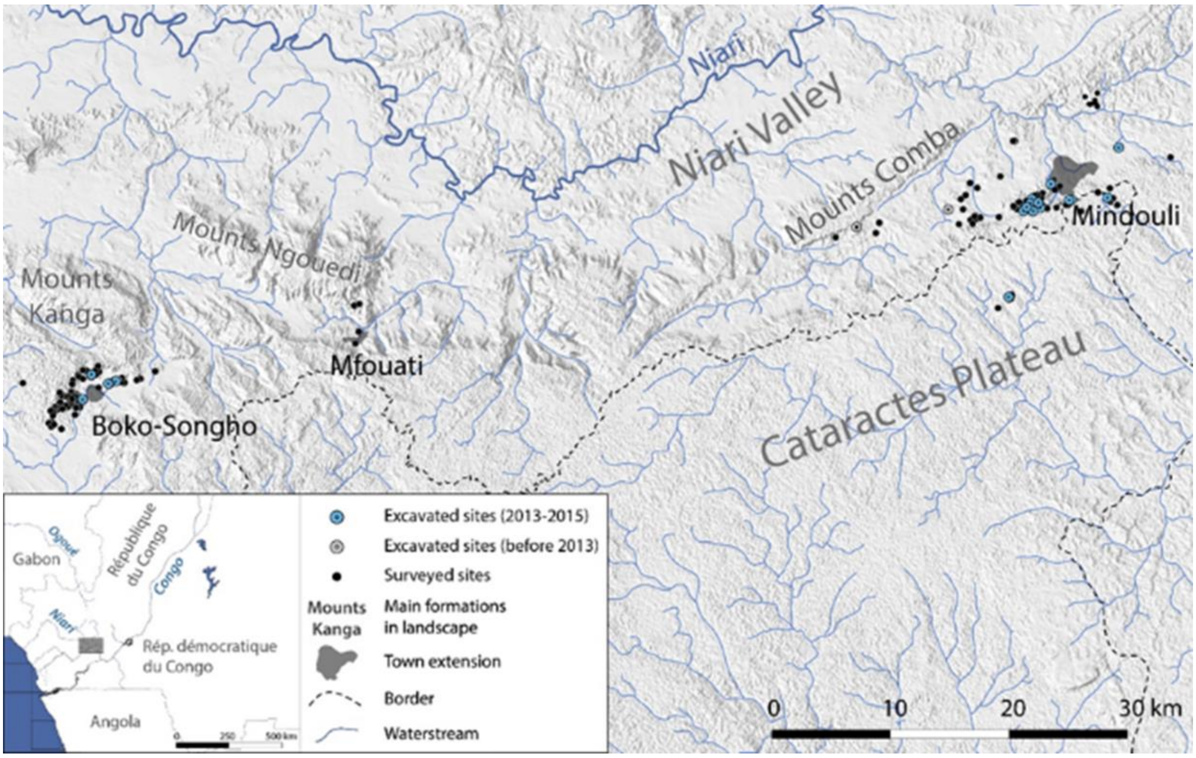


Figure 3: Sites investigated in the Niari basin mining area (© N. Nikis 2019).

In addition, a shared geological and archaeological fieldwork has been conducted in 2015 in the framework of this project (Nikis & De Putter 2015). It had as double objective to improve the understanding of the geological formation of these poorly known deposits – and, incidentally, to better contextualise geological samples already in the RMCA collection - and to document the use of the copper ore in archaeological sites, with further views on copper sourcing analyses throughout Central Africa (see infra, geochemical characterisation of copper).

No new fieldwork has been conducted in the Copperbelt and investigation mainly focused on the copper cross-shaped ingots produced in this area (Fig. 4). However, archaeological and historical data available show that these ingots were a primary product of the regional copper smelting (Raes 1987; Bisson 1976; Arazi *et al.* 2012). It has been thus assumed that these ingots can be used to trace the distribution of copper produced in Copperbelt. All information available regarding these ingots, including some unpublished artefacts from the RMCA collection, has been gathered to outline the distribution of the different types of those copper cross-shaped ingots over time and space. Understanding the way these objects were exchanged and used – especially at which point they were used as a status symbol or currency or further transformed in other objects – provides some insight on the form under which the consuming sites were receiving copper and on the economic spheres in which copper-producing areas were involved (Nikis & Livingstone Smith 2017). Since ingots are generally the main raw material used to make objects, this kind of study allows thus to fill in the gap between the primary and secondary metallurgical processes. In addition, all radiocarbon dates available to date copper metallurgy in Africa (mainly from de Maret 1985; de Maret 1992; Bisson 1976) has been recalibrated using most recent radiocarbon calibration curves and seven new radiocarbon dates were made on remaining samples from Pierre de Maret 1974's excavation. This allows for a reappraisal of the chronology of the Upemba depression and of the copper cross shaped ingots (see OSM in Rademakers *et al.* (2019).

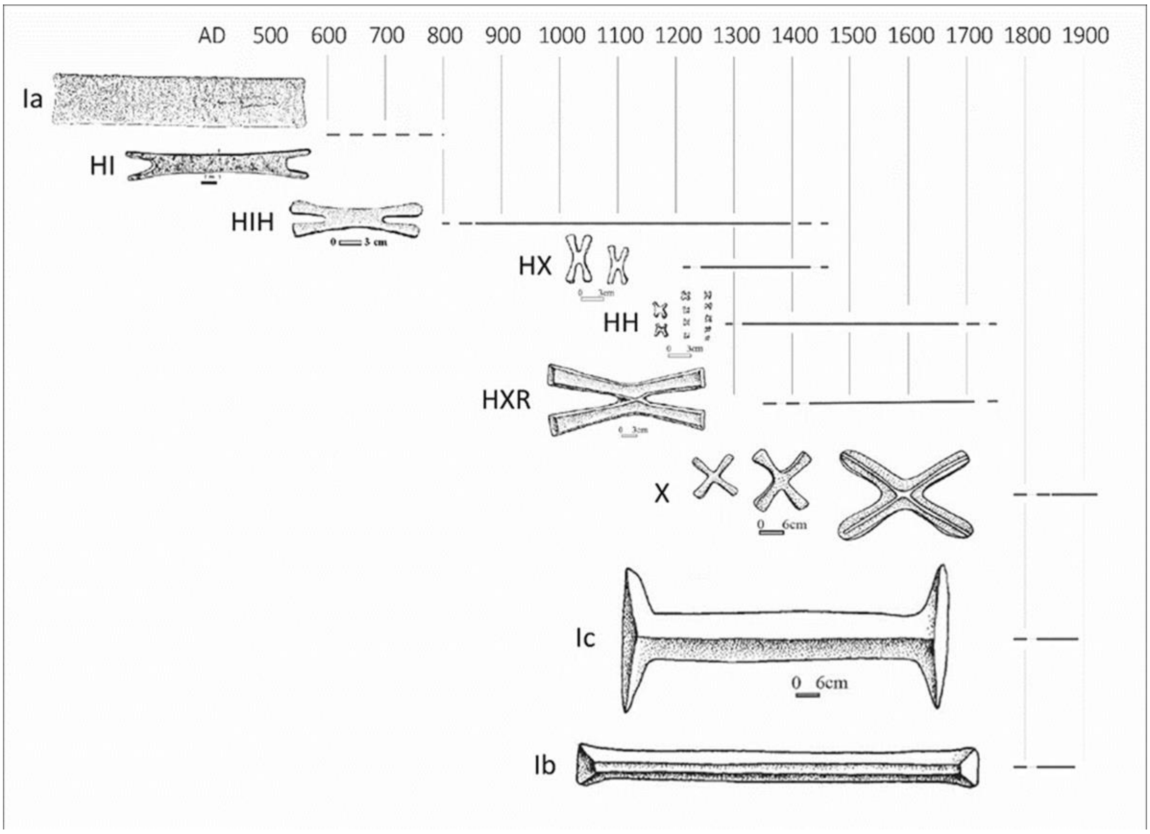


Figure 4: General chronology of the ingots produced in the Copperbelt (N. Nikis, from de Maret 1995 (revised)).

(b) Geochemical investigation of copper (FR, KUL P3, NN ULB, P4 and TDP RMCA, P5)

The second line of research was the geochemical investigation of the copper produced in the Niari Basin and Copperbelt (see also FR, P3 contribution).

In the Niari Basin, chemical and lead isotope analyses were made on fifty-one samples of ore – both collected in deposits and in archaeological sites -, slags and copper objects (Rademakers *et al.* 2018a; see also Annexe 2). They were both coming from the copper production sites excavated between 2013 and 2015 and from the RMCA collection. A second round of analyses has been done on forty-five copper ingots from the Copperbelt area, mainly copper cross-shaped ingots found in the Upemba depression (Rademakers *et al.* 2019; see also Annexe 3). In both cases, the aim was to provide, on the one hand, chemical data to supplement archaeological evidence regarding the manufacturing processes and, on the other hand, lead isotope data to set up a reference framework for provenance studies in Central Africa. While in the Niari Basin, the archaeological evidence has been interpreted against a relatively well-known geological background, there is a lack of geochemical information about ancient mining in the Copperbelt and analyses can only be compared to the few geological data available from base deposits and sulphide minerals only. This situation prevents to highlight clear links between the objects and specific deposit or mining area.

Secondary metallurgy in RMCA collection (LGM & ALS; P2 RMCA)

A specific methodology was designed for the study of the RMCA archaeological copper objects: from the ethnographic accounts and collections to the archaeological objects

Archaeology is a science of analogies and there is a long tradition within the archaeological discipline of exploring the present in order to explain the past. That is, to find plausible explanations to past technical problems by observing how such technical problems may be or may have been solved. Observing techniques in a “traditional environment”, that is performed without the use of modern equipment, provides useful material to propose, test, validate hypotheses on past techniques, gestures and cultures. There is a tradition in the archaeological team of the RMCA to confront recorded contemporary *chaînes opératoires* (C.O.) in the manufacturing of pottery -and especially the early stages of the elaboration of pots (rough-out techniques)- to the ways ancient potteries may have been made. This allows determining the gestures that have been involved in past manufacture processes. It allows going even further; for example, to observe if the techniques have been preserved or have changed over times in a specific region. Coupled with the history of population movements, traded roads and other historical trajectories, it is a useful tool to reconstruct changes in the course of regional history.

In the case of metal and especially copper transformation techniques, there are, within Central Africa, but extremely few living traditions. And this, notwithstanding the fact that Central Africa is rich of a long tradition in the exploitation of minerals (both iron and copper) and transformation of metal. The contact period and then colonial times have made metallurgical traditions disappear. Still there exist recordings of past defunct traditions. Recordings done up to the 1950s; some being re-enactments of past *savoir-faire*. A review of the ethnographic archives relative to copper transformation techniques -photographs, films, missionaries and civil servants (from the exploratory and colonial periods) accounts, tools, etc.- was done. This, in order to document as extensively as possible different manufacture techniques. This was meant, as in the case of C.O.s of pottery technology, to give a technical referential and an analogical background for the interpretation of archaeological artefacts. The record of tool marks, thanks to surface observation and identification of macrotraces (binocular observation, SEM, multispectral imagery, focus stacked photography), on finished objects helped to build-up a technical reference framework to interpret analogous fabrication marks on the archaeological objects and document how ancient may be the occurrence of the technique in the archeological material.

Verification of hypothesis and conclusions

Some additional data were collected to answer specific questions in relation to technical choices made, through fieldwork in Central Africa (RMCA/ULB) and at the ancient copper processing sites identified in Egypt, in parallel with the experimental work (RMAH). This has proved necessary in order to complete data sets or to understand specific artefacts or structures for which the interpretation has not been possible either because of the site taphonomy, or because the necessary data was not recorded at the time of excavation.

On the Egyptian material, working hypotheses were checked by archaeometrical analyses by P3 (KUL) and by experimental reconstitutions undertaken at the Archéosite of Aubechies (Belgium). Archaeological excavations at several key metallurgical sites in Egypt (Ayn Sukhna, Qantir, Amarna, Buto), Sudan (Kerma) and Israel (Timna) by the RMAH completed the data.

Dissemination of results and valorisation of the FSE collections on a national and international level

- A project website has served as an interface between the project network, stakeholders and end-users, whether among the scientific community, policy makers or society in general. Unfortunately, this website has been hacked at the beginning of the last year of the project and the data have been irremediably lost.
- A number of scientific and wider public publications has been produced in the form of scientific articles in peer-reviewed journals, museum publications and catalogues.
- A permanent exhibit in the Egyptian rooms of the RMAH on copper working in Ancient Egypt, featuring never before exhibited objects, ethnographic films and the reconstitution of an Egyptian copper working workshop.

SCIENTIFIC RESULTS AND RECOMMENDATIONS

Assessment, collation and study of the data in FSE collections

The collections available at the RMAH and the RMCA were collated, the information available on these collections regrouped and the data made available both within the network and to the scientific community, using the tools already in place in these two institutions: museum databases and online catalogue.

At the RMCA (LGM, P2) the technical data pertaining to copper objects was added to the central museum central system (The Museum System, TMS). TMS is available only to curators of the RMCA, but feeds the two main databases available for visitors (Okapi, Gazelle), partially accessible on the internet (MRAC Collections; https://www.africamuseum.be/fr/research/collections_libraries/human_sciences/collections/database). In addition, some of the objects from the archaeological site of Sanga (Upemba depression) are parts of the highlighted collections (https://www.africamuseum.be/en/discover/focus_collections/display_group?groupid=347).

All artefacts at the RMAH relevant to the project (about 600 items) have been collated, photographed and an exhaustive inventory has been completed. This inventory has been included into the museum database MuseumPlus. The artefacts have been the object of a complete reappraisal of their status and their storage condition has been assessed. Necessary conservation steps have been taken where necessary.

The analysis of the Egyptian metallurgical collections of the RMAH has been done by P2 (KUL - Frederik Rademakers), in collaboration with partner 1 (RMAH). All RMAH objects were subjected to preliminary chemical analysis using (qualitative) hand-held XRF (HH-XRF). These results have served as the basis for sampling the RMAH artefacts to be subjected to quantitative chemical analysis and lead isotope analysis. These results have been published or currently being published, essentially in the JAS (see for instance Rademakers et al. 2018c, in preparation).

Additionally, 100 samples of production remains of the experimental work on the Ayn Sukhna copper smelting process (P1 GV) were analysed for chemical composition and Pb isotopes. Also mounted sections have allowed to investigate the micro-structure and -texture of these various ores and slag, to further our understanding of the smelting process.

Research on the ancient moulds of the Ägyptisches Museum Bonn, including analyzes of the waxes and clay pastes used was completed at the UGhent completing the collection of µCT-scan.

Investigations on RMAH collections

After a preliminary study, the artefacts chosen for further study from the RMAH were processed. Each object was studied within the methodological framework established at the beginning of the project in order to understand its operational function. Thanks to the excavations and experimentations, we now have a relatively clear idea of the various *chaînes opératoires* for parallel metallurgical processes from different times (Old Kingdom down to Third Intermediate Period and Late Period) and places (throughout the Nile Valley and surrounding regions). The variations depend on the geo-political context and the technical background at various times in history. These permits, for a same type of object, to recontextualise it in its *chaîne opératoire*, taking into account the holistic context as well as the particularities of use.

Investigations on RMCA collections

To inventory at length past *chaînes opératoires* (C.O.), work was done on two past manufacturing processes: the Konga greaves manufacture and the wire-drawing technique.

a) The Konga greaves manufacture.

All steps and tools of the C.O. in the manufacture of *Konga* greaves have been recorded through photographs and film rushes done in the 1950s, sketches and letters from a civil servant. Along that the tools were examined -and more specifically the wooden patterns used to make the imprint in the sand open-mould, the finished artefacts that bear the marks of the manufacture process: that is, the outer surface smoothed due to the moulding and later finishing; the inner surface displaying the typically as-cast surface of an open-air mould, left unworked. The compilation and synthesizing work was the occasion to show how rudimentary were the tools in action in the manufacture of such an impressive finished object. It also showed that most of the toolkit was unlikely to be recovered in the archaeological record (some were wood trunks or branches, stones) and thus, to what extent a large part of such information was lost (see below: Konga greaves).

This compilation and synthesizing was done by three means: a. an inventory of the tools in relation to this C.O. and related C.O. (in relation to the elaboration of an open mould done with the imprint in sand of a wooden pattern); b. a collect of all archival material and photographs around the technique and c. drawings.

This meant a precise inventory of the wooden patterns; done on object cards, which, besides measures and condition report, recalled data on the collector, the place of collection together with the objects collected at the same time. This was accompanied by photographs done to record the characteristic elements of each object. It was a mean to show the diversity of practices within the same technical register, that is, the creation of an open mould by mean of the imprint in sand of a wooden pattern. The collect of all archival material implied sketches and ethnographic notes from M. Burhin, who then, in 1936, was a colonial administrator in the Equateur region (District de la Tshuapa, Territoire de Boende), photographs, together with the context of those, and film rushes. This led to the discovery thanks to Julien Volper, curator of the Ethnographic collections, that there existed within the RMCA film archives some rushes of re-enactments around the making of these greaves. Those were buried among other rushes that had been recorded on totally different subjects. Although these rushes do not give the entire line of processes in the making of *Konga* greaves -they are only excerpts-, and are not organized in the correct line of steps, they provide some interesting elements on the type of bellows used, the motion of them, the making of the mould with the wooden pattern, the handling of the crucible, the pouring of the molten metal and the removal of the cast plaque. Finally, drawings were done. They were meant, by intensifying in coloured sketches the gestures and the tools recorded on the photographs, to explain the technical process going on at each step of the line of processes and highlight the scarcity of the preserved tools in the archaeological record.

b) The wire-drawing technique

About the wire-drawing technique, The RMCA host a very interesting collection of dies and vises from DRC, Rwanda and Burundi. The same procedure as with the Konga greaves was thus run on those: a. An inventory of the tools in relation to this C.O. and related C.O.: object. cards of the wire-drawing devices -drawing dies and vises (grips); tables of related materials (collected materials given to the museum by the same donator at the same time); b. Collect of all archival material and photographs around the technique; c. Drawings.

The collect of archival material and photographs around the wire-drawing technique was done on even more data than with the *Konga* greaves. This is thanks to Jean-Félix de Hemptinne, bishop of the Katanga province of Belgium Congo from 1932 to 1958, fascinated by the know-how of the technique as it was performed by the Yeke smiths. There are photographs of smiths performing wire drawing during an observation conducted in 1924 in the village of Nguba in Katanga. Some of the photographs taken in 1924 (the year 1925 is sometimes cited) are preserved in the archival collections of the RMCA (Inventory #: EP.0.0.5817 to EP.0.0.5821). Others photographs together with film rushes were done in the 1950s during a reenactment initiated by the same Mgr de Hemptinne. The 1956 photographs have recently been republished (Liesenborghs et *al*. 2009). The movies *Kanu* and *Nsambo* (the latter also recorded the wire-drawing process) made by Herman Philips under the direction of Henri Liesenborghs are included in the recent publication. The RMCA also curates a sketch drawing signed “Swinnen” that explains the post-and-lever wire-drawing process along two sets of dies and vises that were once used in this process from this same Katanga region.

As in the case of the Konga greaves, drawings were made for the Yeke wire-drawing process sequences observed and recorded under Mgr de Hemptinne’s guidance. The drawings -by intensifying in colored sketches the gestures and the tools recorded on the photographs - highlight the gesture, the technical operation going on and the precise functioning of the tools (Garenne-Marot 2019).

c) The archaeological collection.

About the archaeological collection of the RMCA, the work was meant to valorise the enormous data provided by the excavations of more than 200 tombs from five major sites located in the Upemba Depression (DRC) excavated in the years 1957, 1958, 1974 and 1975. The wealth and diversity of the burial goods that span more than thousand years with the presence among these artefacts of an impressive array of copper (and iron) material, principally jewellery, instigated this interest. Approximately 2000 copper objects (some with iron parts) were excavated, a of these are held in the RMCA either as permanent collections or for study and curation; the remaining items are in the *Musée national de Lubumbashi* (belonging to the *Institut des Musées nationaux* of the DRC). In the course of the project tools were elaborated that are now being used within the RMCA archaeological collections but could as templates be adapted to other collections.

- Elaboration of tools. Catalogues of graves within each cemetery.

The catalogues of the graves within each cemetery allow to view on a *Microsoft Excel* formatted spreadsheet, organizing data in an easy-to-read and understandable format (playing with the sizes of the cells), items recovered within the same burial together with other contextual information (site, grave number, excavator, date, bibliography, etc.). The recorded objects are those in metals but the format allows to add further columns to the same spreadsheet to record other funerary goods such as potteries, ivory items and any other deposits and to add to the workbook additional formatted sheets for additional recordings (such as the graves that have not yet been included, having no metal objects). It allows checking rapidly what types of objects were found together. For each object, metal object, the C.O. of manufacturing processes is (or will be) indicated and data regarding elemental, isotopic or metallographic analyses are recorded. The 3D-scannings of some of the objects which were done in 2016-2017 are also indicated. The functions “find” and “filter” unable one to rapidly find the proper recording within the workbook.

- Typology model based on the recording of shaping processes.

Typologies are foremost tools in the interrogation of archaeological material data. And this is especially true when considering the impressive array of material culture that came out of the graves from the Upemba Depression. So far, however, typologies have been essentially devoted to two types of material unearthed from these graves. First the pottery one, and this very early on, in order to elaborate seriations of the graves and thus establish a relative chronology to which calendar dates had then been applied. Second the emblematic copper ingots -the *croisettes* (see *supra*) display marked transformations in their standardized shape and size over the last periods of the cemeteries that led to the construction of earlier typologies.

The burials in the Upemba Depression cemeteries, DRC, have yielded a profusion and diversity of copper and iron material unmatched so far for Central and Southern Africa that offer great analytical opportunities. A large amount of the copper and iron objects date to the Kisalian period (ca. 2nd half of 8th century-2nd half of 12th century), that is to cultures predating the occurrence of *croisettes* in the Upemba graves. Thus, the larger part of metallic material, the copper and iron adornment objects, and to a lesser degree, tools and weapons, were largely neglected in terms of general classifications and elaboration of typologies. Typologies of metal objects from an archaeological corpus are rare. Such classifications tend to focus on a specific artifact type that changes markedly over time and space and is therefore useful for seriation. The copper and iron objects from the Upemba graves are largely jewelry, objects that are more sensitive to the effects of fashion, and moreover are recovered from closed contexts.

A new typology was set up on this metallic material for which the C.O.s of forming/manufacture processes were at the core (Fig. 5). The idea behind that was that the steps recorded in the C.O.s for artefacts in such a complete state as the ones recovered from the Upemba Depression cemeteries could then be applied to material of less good preservation from other sub-Saharan African sites. The form and the function of objects of which a sole fragment had been archaeologically recovered may be lost but not the marks of the forming processes. Both iron and copper artefacts were taken into account in doing so: both had been shaped using comparable forming (plastic deformation processes) techniques and some artefacts mingled elements from both metals.

In establishing this typology model based on the recording of shaping (forming) processes, glossaries were elaborated to precise (both in English and French) the adequate terms to designate each object, each forming technique and the C.O. (line of processes) used to manufacture the objects. An *Excel* spreadsheet was set to record the object according to this new typology. The recording is done in the following progression:

-1st level, *the base shape*: the wire, the rod, the ribbon, the sheet and the plate. Those are the forms that will then be transformed into finished objects and those are often found on sites when, of the finished object from which they were the base element or part of, only fragments are unearthed. These basic forms are a stage in the transformation of preforms. These preforms could be either ingots –that is molten copper cast in open moulds (such as *croisettes*)- or what had been directly recovered from the smelting operation: nodules or prills. This stage of preforms, however, upstream of the basic forms, belongs to another evaluation register, more difficult to highlight by a simple naked-eye observation. In the context of a typology of forming processes, taking the basic form as the starting point for an evaluation of object-making techniques provides a safer and immediately more functional recording framework.

- 2nd level, *simple forming processes*. *Forming process 1.* These are the techniques used to transform these base shapes: bending, twisting, several strands twisting, braiding, weaving, spiral winding. These plastic deformation techniques are potentially applicable to all base forms. The C.O. in the manufacture of the object might stop at this stage. This is the case with leg rings and also with beads when they are examined individually.

- 3rd level, *complexifications*. They are at different levels. It can be at the level of forming. For example, a base shape worked by a first deformation process (*forming process 1*) may undergo a new deformation (*forming process 2*): In the case of spiral coiled wire beads, only one step of shaping is required (forming process 1): spiral coiling. In the manufacture of *cordelettes* (see Fig. 5) two degrees of forming are necessary: first, the spiral coiling of a wire (as before), then the wrapping of this coiled wire on a fibrous core. Ethnographic documents that refer to the manufacture of *cordelettes* show that these steps were carried out concurrently (see fig. X10, above; the copper wire was wrapped on the fiber with a very skillful turn of hand). A distinction must however be made between the manufacture of beads of coiled wire and that of *cordelette*s; these are two very different mental patterns of operating chains. Complexifications can also occur at the assembly level. The case of copper ring chains must be mentioned here. Same thing with the strands. Several strands are twisted together to form what looks like a big rope.

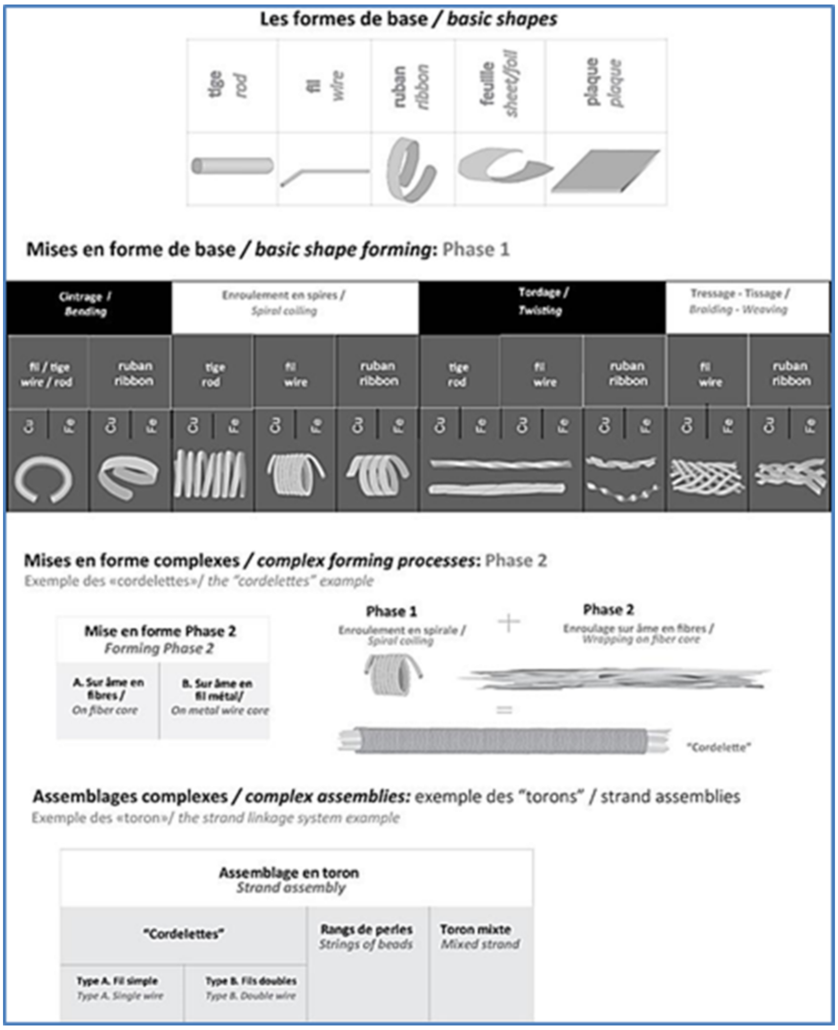


Figure 5: Steps in the recording of the C.O.s (chains of actions) in the modus operandi of shaping processes. 1. Base shapes. 2. Simple forming processes. 3. Complexifications: complex forming processes: the example of 'cordelettes'; complex assemblies: the example of "strands assemblies" (Graphics: L. Garenne.-Marot, MRAC Tervuren ©).

Primary metallurgy

a) General investigations in Primary metallurgy (RMAH & RMCA)

This study was conducted as follows:

1. Archaeometric analysis campaign was conducted on the entirety of the metal related collection, using p-XRF.

2. Cross reference mapping of the archival data (archaeological excavations from which the objects came, plans…) and the objects (provenance, period, type, composition). Cross reference mapping of the archival data (archaeological excavations, plans…) and the objects (provenance, period, type, composition). The objects with a clear

archaeological context were selected for phases 2 and 3 of the analysis campaign. A geographical mapping has been finished. Each artefact was studied according to its archaeological significance, its technological characteristics and its contribution to the knowledge of the metallurgical process. We have set up the first publishing model to create a reference series for the publication of the Egyptian collection. This will allow to publish each artefact studied by EACOM. Finally, an internal database has been implemented to centralize bibliographical and archaeological data.

3. Objects with a clear archaeological context were chosen for phases 2 and 3 of the analysis campaign.

4. The copper alloy artefacts were analyzed by the KU Leuven for their chemical composition using ICP-OES (Inductively-Coupled-Plasma Optical Emission Spectroscopy) and their lead isotope ratios were determined using MC-ICP-MS (Multi-Collector Inductively-Coupled-Plasma Mass Spectrometry). These data have provided a detailed insight into the production technology and provenance of copper in early Egypt. A first part of the data (Pre-Dynastic to Old Kingdom copper) has already been published in the Journal of Archaeological Science (Rademakers et al. 2018c), revealing the early exploitation of a wide variety of ores from Sinai and the Eastern Desert and an active selection of arsenic alloys – issues never explored in such depth hitherto. Data on the Middle Kingdom and New Kingdom copper alloys is currently being prepared for journal publication (incl. presentation at international conferences: Rademakers et al. 2019c). These analytical data provide a ground-breaking new reference framework for the study of copper production technology and provenance in ancient Egypt and constitute by far the largest dataset on its kind. The EACOM project has turned the RMAH collection into the world’s largest reference for future Egyptian copper studies.

5. At the RMCA, two main assemblages have been studied. Firstly, the museum’s collection of croisette ingots from the Democratic Republic of Congo has been analysed. This consisted of the technological and provenance study of 45 croisette ingots by ICP-OES, ICP-MS (Tervuren) and MC-ICP-MS. This study has been published in the Journal of Archaeological Science (Rademakers et al. 2019a) and represents the first ever investigation of copper provenance in Central Africa. It has already been cited by colleagues in the field as a ground-breaking study, laying the foundation for future research.

6. The second main archaeometric study concerned the copper production system of the Niari Basin, Republic of Congo (see Case study below). 51 samples of different

production remains (including ore, slag and copper) have been analysed (ICP-OES, MC-ICP-MS and ICP-MS (Tervuren)) to provide the first ever characterization of copper production in western Central Africa. This study has been published in the journal Archaeometry (Rademakers et al. 2018a). Combined with the data on the D.R.C. croisette ingots, this project has thus established the largest database for Central African archaeological copper composition currently available, turning the RMCA copper collection into a world-wide reference for future provenance and technology studies.

b) Case study: Copper smelting in the RMCA collection.

In west Central Africa, the 2013-2015 fieldwork allowed to survey more than 100 sites and 20 of them have been excavated. Four main periods of production have been identified so far and they have been divided in 8 different metallurgical traditions (Fig. 6). The detailed study of these different traditions, including their broader sociocultural contextualisation based on the pottery analyses, has been developed in the N. Nikis PhD thesis (Nikis 2018a). Only main results related to the copper smelting processes and their implication for the study of federal collections will be examined here.

Before the 14th century, sites have only been found in the Mindouli area and conservation issues do not allow to properly reconstruct and contextualise smelting processes. Because of the erosion, the only metallurgical remains are a few bases of pit furnaces that do not provide a lot of information. Between the late 13th century and the mid-15th century, information is still limited to the Mindouli area but are far more abundant. A dozen sites, grouped into the Misenga tradition, are sharing very similar pottery and metallurgical remains. One of the main features of the Misenga production sites is the production of copper bars – some of them being ornate. The eponym site was firstly excavated in the 1950s by Mauritz Bequaert. The material is curated in the RMCA collection and include among other a dozen copper bars and ingots (Fig. 7).

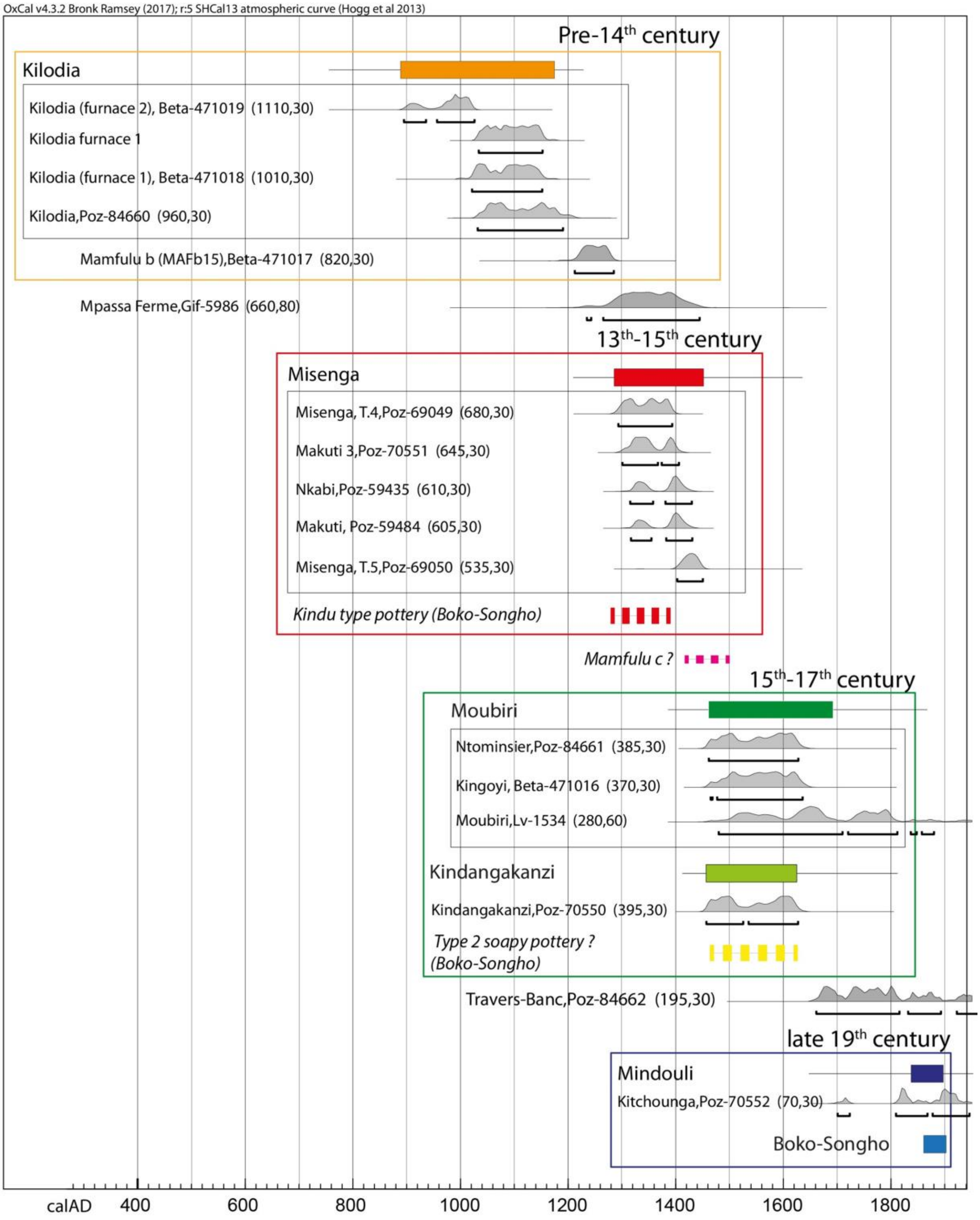


Figure 6: Chronology of the copper production in the Niari Basin based on available data (adapted from Nikis 2018)

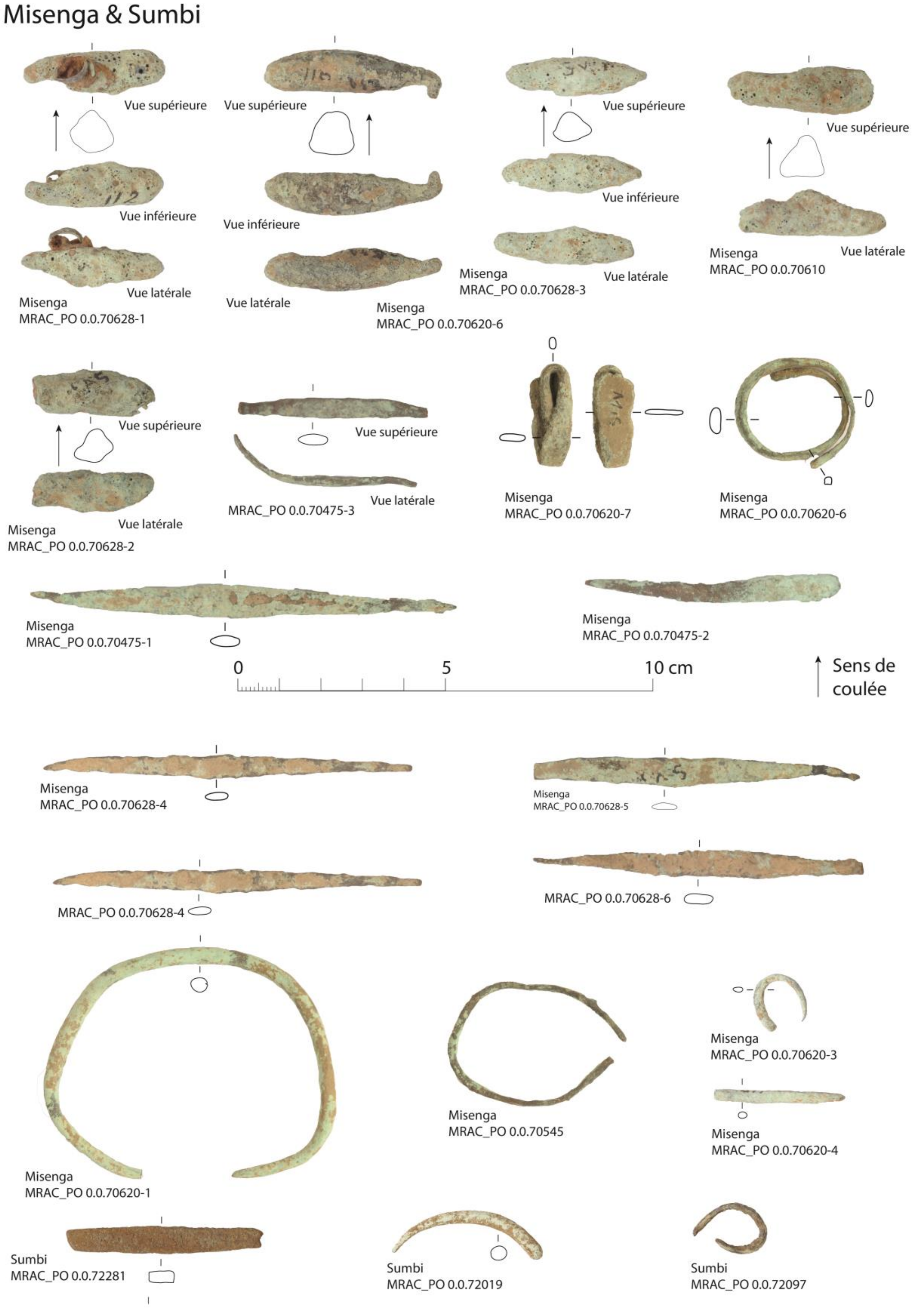


Figure 7: Copper objects from Misenga and Sumbi, M. Bequaert excavation, 1950s, RMCA collection (© N. Nikis 2018).

The 2013-2015 new excavations allowed to reconsider the chronology of the occupation – previously considered as a post-15th century settlement based on comparison with other regional site (Clist 2012) – and to include it in a broader tradition. In addition to new proper radiocarbon dating, metallurgical activities that occurred on this site are also better understood. Indeed, metallurgical remains excavated these last years and the assemblage of copper objects from both the recent and old excavations allowed, together, to insert copper ingots and bars in a *chaine opératoire* (published in Nikis 2018b). The ingots are cast in open sand mould and then further transformed by hammering into bars, that can be decorated (Fig. 8). Bars, that always display the same elongated shape seem to have been standardised in two sizes, one around 8 cm, the other around 15 cm. Discovery contexts and standardised shape are suggesting that these bars could have been used as currencies (Nikis 2018b). Geochemical data further indicate that very similar smelting condition has been used to produce copper at that period (Rademakers *et al.* 2018; see also geochemical results in Annexe 2). These results are complementing observations made on the archaeological metallurgical remains such as the fairly standardised shape of the crucible. They suggest standardised metallurgical processes between the different sites that would reflect a well-established and organised copper manufacturing economy.

Figure 8: Manufacturing process of Misenga copper bars (N. Nikis 2018).

Recent archaeological research has also allowed to enlarge the assemblage regarding the Misenga pottery tradition. New data and old collections have together been compared to other pottery groups in the wider region. The pottery comparison body is mainly held in the RMCA collection (described, among others by de Maret 1972; Clist 1982). It allows to highlight that Misenga pottery is sharing woven-inspired motifs with other regional pottery tradition. This kind of motifs seems to be systematically associated with Kongo cultural areas. It thus would indicate that the Misenga smelters have played a role in the development of the Kongo Kingdom (Cranshof *et al.* 2018).

While the different copper production identified between the 15th and 19th centuries are not documented in the RMCA collections, the archaeological investigation and geochemical analyses of the late 19th-century copper smelting tradition have allowed to recontextualise

RMCA collection objects collected in the 19th and early 20th century. It is particularly the case for copper ingots (fig. 9a) collected in the Mbandaka area, more than 1000 km away from the Niari Basin, and conserved in the geological collection. The combination of RMCA archives and geochemical analyses allow to trace back these ingots to the Boko-Songho area production. Metal from the Mbandaka ingots and from the metallurgical remains of Boko-Songho are indeed displaying a similar amount of lead (around 20%) and lead isotope ratio (Rademakers *et al.* 2018). Similar ingots have been collected in the Manyanga region, a major 19th-century market on the Lower Congo River, and in the area of Boma, near the mouth of the river (fig. 9b-d). This indicates that the Boko-Songho copper was still traded to major exchange hub of the time, despite the concurrence of European brass. This situation strongly leads to reconsider the previous assumption (Dupré & Pinçon 1997) of a marginalisation of the local copper production because of the arrival of a cheaper imported metal.

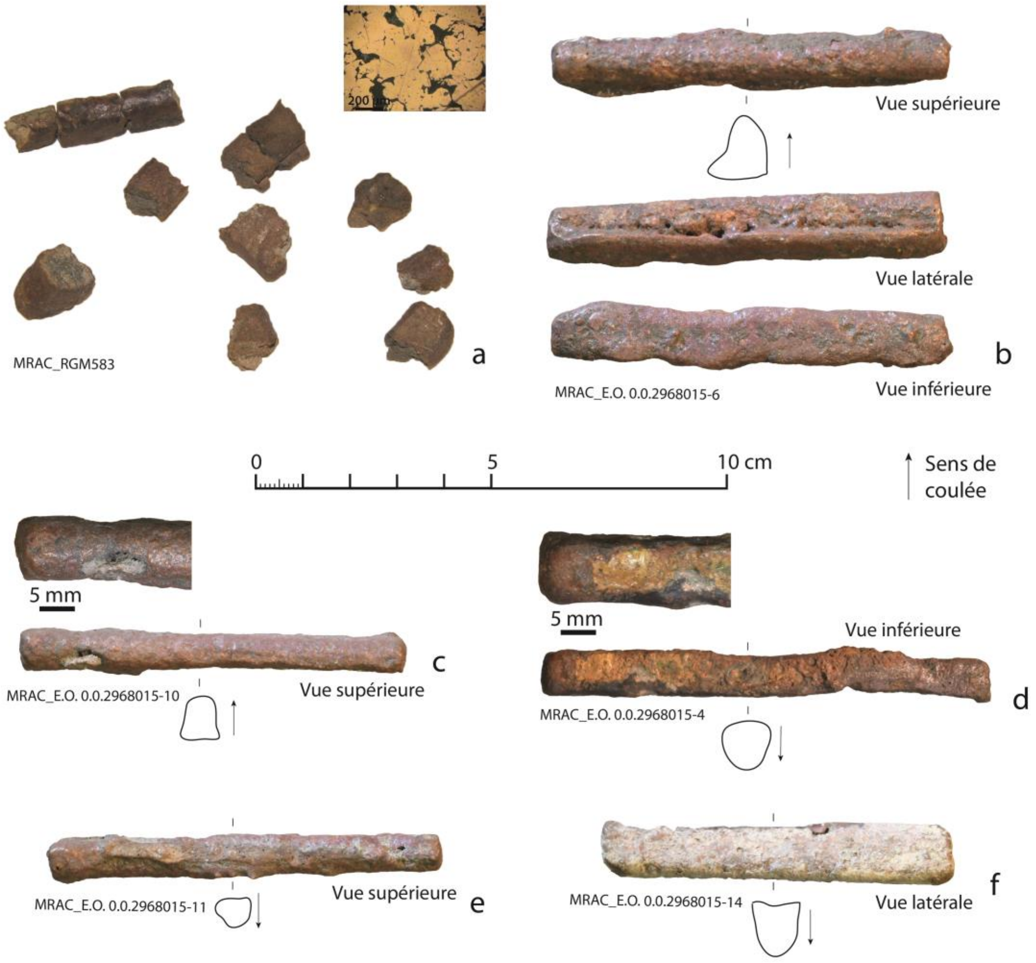


Figure 9: ingots collected in the Manyanga area with it cross section displaying inclusion of lead (a) and similar ingots collected in the Matadi area on the Lower Congo River (b-f), RMCA collection (© N. Nikis 2018).

The geological survey of the area allowed to better understand the structure of the mineralisation and to propose a schematic section of the deposits. New mineral samples have been collected and entered the geological RMCA collection in order to supplement samples from the same region. Moreover, the survey confirmed that the variation in the mining techniques observed in the 19th century (see details in (Nikis 2018a)) were related to the shape of the mineralisation. Conversely, this geological survey supplemented by the lead isotopes data showed that the same formation has been exploited during the different period and that the variations observed in the manufacturing process are more likely related to sociocultural factors than to the geological context.

In the Copperbelt, the mapping of the different information available regarding the copper cross-shaped ingots between the 9th and 19th centuries allowed to outline the distribution of the different types and their place of productions (for details, see Nikis & Livingstone Smith 2017). It particularly highlighted the early links between the Copperbelt and the region south of the Zambezi – since the 9th century CE – but also the role of the socio-political context in shaping specific economic spheres. Indeed, while the same type of ingots (HIH, see fig. X1) were produced in the Copperbelt between the 9th and late 13th century, from the 14th to the 18th century, the region is divided in two areas where different type of ingots are produced and exchanged (fig. X9): HXR in south-east Copperbelt and distributed south of the deposits and HH and HX ingots in Central and West Copperbelt, distributed north of the deposits. While the border separating the two areas moved westward, a similar situation occurred in the 19th century: the Copperbelt was again divided in two main areas producing different types of ingots that were traded in different networks. Major polities of the area seems to have shaped specific social spaces that had an influence on the copper production and trade, but also on the salt production and trade and the pottery manufacturing processes. Drawing on the observations made on 19th-century ingots, it has been hypothesised that the specific ingots distribution between the 14th and 18th centuries would also point to the existence of two economic spheres, north and south of the Copperbelt, shaped by rising polities…

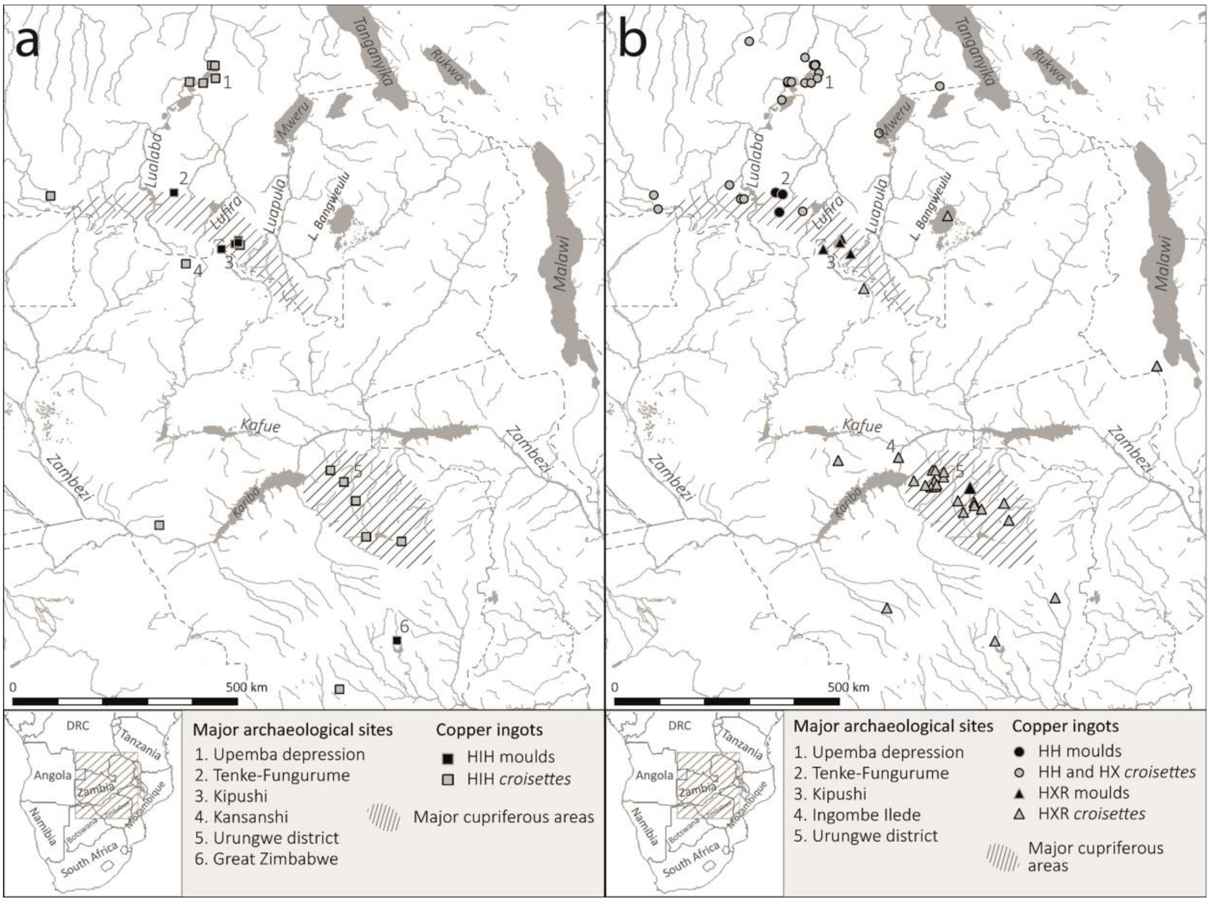


Figure 10: Distribution of copper cross-shaped ingots. (a). 9th-14th century, distribution of HIH ingots. (b) 14th-18th century, distribution of HXR (south) and HH and HX (north) ingots. (from Nikis & Livingstone Smith 2017).

Further information has been provided on the 14th-18th-century period in the Upemba depression by the geochemical analyses – lead isotopes and chemical composition by ICP-MS and ICP-OES (see supra, methodology and Annexe 3) of the copper-cross shaped ingots. A chronological trend draws from the lead isotopes and chemical results between the earlier Kabambian A ingots - HX and large to medium HH, dated between 14th and half 15th century – and later Kabambian B ones – small to very small HH, dated between the late and early 18th century. The first group seems to have been cast from a non-radiogenic ore while the second group probably come from a radiogenic ore and Cu/Co type mineralisation. A shift in copper supply would have occurred around the mid-15th century between the Kabambian A and Kabambian B period and it could be related to socio-cultural change in the area, as suggested by other elements of the material culture (Rademakers *et al.* 2019).

Chemical analyses have also highlighted the very pure composition of the copper cross-shaped ingots, suggesting the use of high-grade ore smelted in relatively poorly reducing atmospheres. Indeed, only trace elements only trace elements are present in most artefacts, generally at concentrations below 100 µg/g and many below 10 µg/g. Higher concentrations of silver, arsenic, cobalt, iron, nickel, lead, antimony, tellurium and zinc occur in particular artefacts (see results and boxplot in Annexe 3). Even if it was initially not deliberate, this kind of composition is further ideal for the manufacturing processes based on mechanical deformation that were mainly used in Upemba depression (see *infra*).

More generally, the geochemical analyses have shown that Niari Basin (Rademakers et al. 2018) and Copperbelt (Rademakers et al. 2019) can be distinguished on their chemical and lead isotope composition, opening the way to provenance studies at the scale of Central Africa. Both areas are displaying different lead isotopes ratios while Niari basio artefacts tends to have higher contents of trace elements, particularly Pb that almost absent in Copperbet artefact. At the contrary, *croisettes* have in general higher contents in Co and Ni than Niari objects (see results, Annexe 2 & 3).

In summary, these different studies show how new methods and new data can actualise the knowledge we have about ancient collections. In turn, these “old” collections, once properly contextualise, are an invaluable source of information to expand the geographical and temporal perspective of very localised research.

In this case, all the information put together allowed to display a dynamic and complex history for the copper production and trade, challenging the long-lived colonial discourse of a landlocked Central African and of societies using static primitive manufacturing processes.

The EACoM project further set the basis of international collaborations that are still ongoing. The geochemical analyses led to exchange of data and good practice with the Université Toulouse-Jean Jaurès (Sandrine Baron) and University of Arizona (Jay Stephens and David Killick). The results generated by our research has been central to the archaeological interpretation of one of the papers of the later team (Stephens *et al.* 2020).

The renew interest in copper smelting process and trade in Central Africa partly generated by the project has also led to new collaboration with the McDonald Institute for Archaeological Research, University of Cambridge when one of us (NN) has started a new project about the copper trade networks – including the metallurgical knowledge diffusion – in Central Africa with Prof. Paul Lane (Ward Oppenheimer Professor of Deep History & Archaeology of Africa) and Prof. Marcos Martinón-Torres (Pitt-Rivers Professor of Archaeological Science).

Secondary metallurgy

a) The grave T.53 of the cemetery of Sanga (Upemba Depression): a case-study for to recontextualize data of a sealed context and to validate new typology based on "forming processes"

The grave T.53 from the cemetery of Sanga, Upemba Depression (DRC) was excavated in 1957. It is a "monument" in itself. Since the early years of 1970, a reconstruction of the burial has been on display in the galleries of the RMCA. It was meant to show the wealth of ancient Central African burials best exemplified by the profusion of pots surrounding the corpse (materialized by a fake skeleton) and its rich copper and iron adornments (Fig. 11).

The renovation of the museum was an opportunity to study anew the material of this grave. It meant unearthing all possible documents from the time of its discovery, of the registration of its furnishing in the inventory books, of the first conservatory treatments and conditioning of the artefacts and publication in 1963. The story of this grave could be told anew with its gaps and uncertainties (Fig. 12).

The metallic material of the grave was submitted to a thorough study and this burial taken as a case-study. It was an example of the array of copper and iron jewellery that could be found within one single grave and of the scope of the diverse techniques that had been utilized in the manufacturing of these items. It gave thus the opportunity to test the newly elaborated typology based on forming processes (Garenne-Marot 2018, AARD presentation; Garenne-Marot 2019).

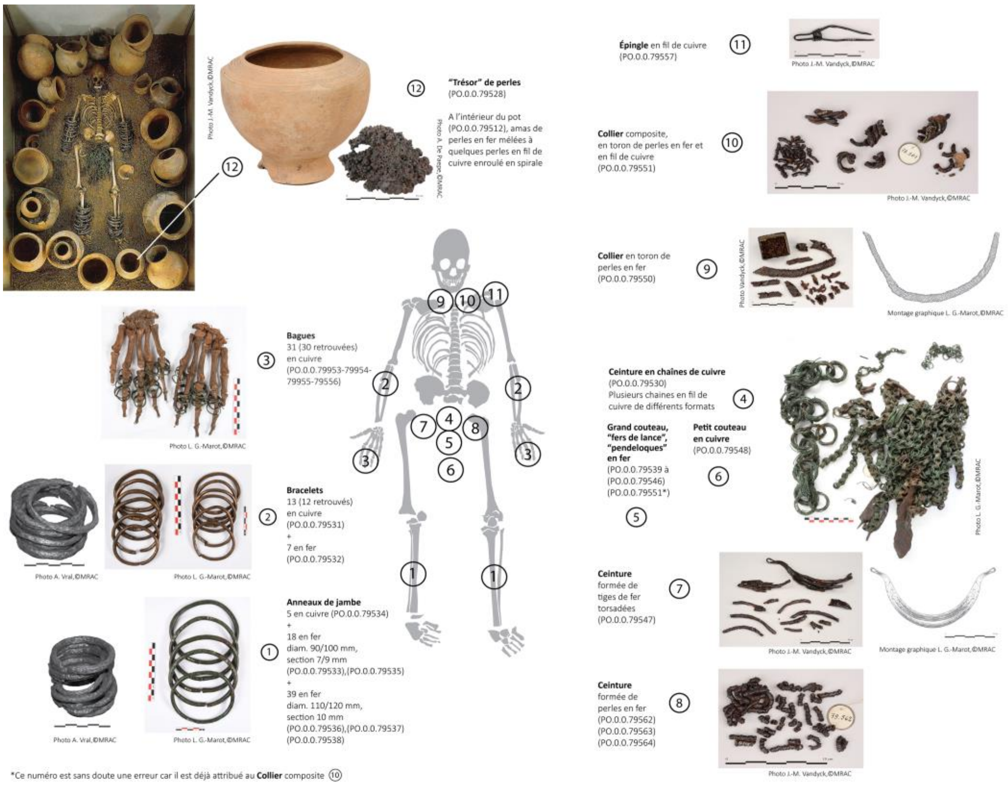
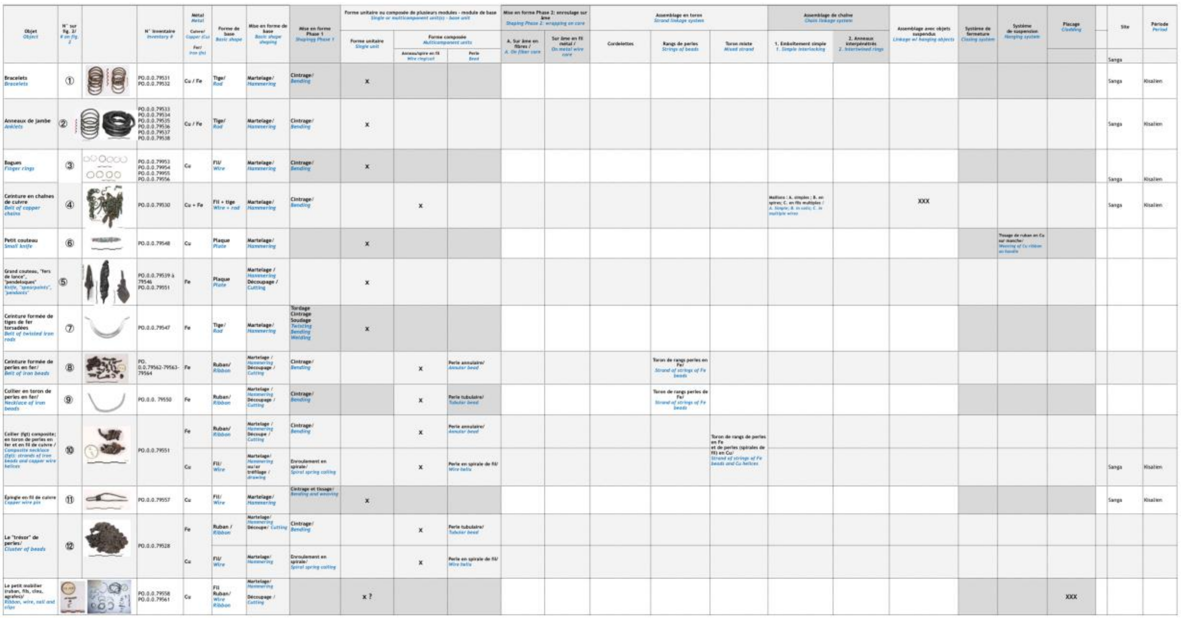


Figure 11: Hypothesized position of the metal objects of grave 53 of Sanga cemetery (graphic L. Garenne-Marot). Top left: postcard dated from the 1970s of the burial reconstructed and on display at the RMCA © Archives section Prehistory, Heritages, RMCA.



Objet Object	N° sur fig. 2/ # on fig. 2		N° Inventaire Inventory #	Métal Metal Cuivre/ Copper (Cu) Fer/ Iron (Fe)	Forme de base Basic shape	Mise en forme de base Basic shape shaping	Mise en forme Phase 1 Shaping Phase 1	Forme unitaire ou composée de plusieurs modules - module de base Single or multicomponent unit(s) - base unit			Mise en forme Phase 2: enroulage sur âme Shaping Phase 2: wrapping on core		Assemblage en toron Strand linkage system			Assemblage de chaîne Chain linkage system		Assemblage avec objets suspendus Linkage w/ hanging objects	Système de fermeture Closing system	Système de suspension Hanging system	Placage Cladding		Site	Période Period
								Forme unitaire Single unit	Forme composée Multicomponent units		A. Sur âme en fibres / A. On fiber core	Sur âme en fil métal / On metal wire core	Cordelettes	Rangs de perles Strings of beads	Toron mixte Mixed strand	1. Emboîtement simple 1. Simple interlocking	2. Anneaux interpénétrés 2. Intertwined rings							
									Anneau/spire en fil Wire ring/coil	Perle Bead													Sanga	
Bracelets Bracelets	①		PO.0.0.79531 PO.0.0.79532	Cu / Fe	Tige/ Rod	Martelage/ Hammering	Cintrage/ Bending	x															Sanga	Kisalien
Anneaux de jambe Anklets	②		PO.0.0.79533 PO.0.0.79534 PO.0.0.79535 PO.0.0.79536 PO.0.0.79537 PO.0.0.79538	Cu / Fe	Tige/ Rod	Martelage/ Hammering	Cintrage/ Bending	x															Sanga	Kisalien
Bagues Finger rings	③		PO.0.0.79953 PO.0.0.79954 PO.0.0.79955 PO.0.0.79956	Cu	Fil/ Wire	Martelage/ Hammering	Cintrage/ Bending	x															Sanga	Kisalien
Ceinture en chaînes de cuivre Belt of copper chains	④		PO.0.0.79530	Cu + Fe	Fil + tige Wire + rod	Martelage/ Hammering	Cintrage/ Bending		x							Maillons : A. simples ; B. en spires; C. en fils multiples / A. Simple; B. in coils; C. in multiple wires		XXX					Sanga	Kisalien
Petit couteau Small knife	⑥		PO.0.0.79548	Cu	Plaque Plate	Martelage/ Hammering		x												Tissage de ruban en Cu sur manche/ Weaving of Cu ribbon on handle				
Grand couteau, "fers de lance", "pendeloques" Knife, "spearpoints", "pendants"	⑤		PO.0.0.79539 à 79546 PO.0.0.79551	Fe	Plaque Plate	Martelage / Hammering Découpage / Cutting		x																
Ceinture formée de tiges de fer torsadées Belt of twisted iron rods	⑦		PO.0.0.79547	Fe	Tige/ Rod	Martelage/ Hammering	Tordage Cintrage Soudage Twisting Bending Welding	x																
Ceinture formée de perles en fer/ Belt of iron beads	⑧		PO. 0.0.79562-79563-79564	Fe	Ruban/ Ribbon	Martelage / Hammering Découpage / Cutting	Cintrage/ Bending		x	Perle annulaire/ Annular bead				Toron de rangs perles en Fe/ Strand of strings of Fe beads										
Collier en toron de perles en fer/ Necklace of iron beads	⑨		PO.0.0. 79550	Fe	Ruban/ Ribbon	Martelage / Hammering Découpage / Cutting	Cintrage/ Bending		x	Perle tubulaire/ Tubular bead				Toron de rangs perles de Fe/ Strand of strings of Fe beads										
Collier (fgt) composite; en toron de perles en fer et en fil de cuivre / Composite necklace (fgt): strands of iron beads and copper wire helices	⑩		PO.0.0.79551	Fe	Ruban/ Ribbon	Martelage / Hammering Découpe / Cutting	Cintrage/ Bending		x	Perle annulaire/ Annular bead					Toron de rangs de perles en Fe et de perles (spirales de fil) en Cu/ Strand of strings of Fe beads and Cu helices									
				Cu	Fil/ Wire	Martelage/ Hammering ou/or tréfilage / drawing	Enroulement en spirale/ Spiral spring coiling		x	Perle en spirale de fil/ Wire helix													Sanga	Kisalien
Épingle en fil de cuivre Copper wire pin	⑪		PO.0.0.79557	Cu	Fil/ Wire	Martelage/ Hammering	Cintrage et tissage/ Bending and weaving	x															Sanga	Kisalien
Le "trésor" de perles/ Cluster of beads	⑫		PO.0.0.79528	Fe	Ruban / Ribbon	Martelage/ Hammering Découpe/ Cutting	Cintrage/ Bending		x	Perle tubulaire/ Tubular bead														
				Cu	Fil/ Wire	Martelage/ Hammering	Enroulement en spirale/ Spiral spring coiling		x	Perle en spirale de fil/ Wire helix														
Le petit mobilier (ruban, fils, clou, agrafes)/ Ribbon, wire, nail and clips			PO.0.0.79558 PO.0.0.79561	Cu	Fil Ruban/ Wire Ribbon	Martelage/ Hammering Découpage / Cutting		x ?													XXX			

Figure 12: Recording of the shaping processes for the metal objects of grave 53 of Sanga. Graphic L. Garenne-Marot, © RMCA Tervuren

b) Antiquity of wire-drawing in central Africa

The question at stake was: is the thinnest copper wire of the archaeological jewellery of the RMCA collections dated from the (Classical) Kisalian time period (late 9th – 12th century AD) drawn wire? If it was so it would mean that the wire-drawing technique was present in South-Central Africa as early as the late 9th century AD. Results were of great interest for they had historical implications: the technique is said to have been imported in the Katanga (close to the Upemba region) by newcomers from the North-East of Lake Tanganyika in the 19th century.

- The investigation: the archaeological and ethnographic data.

The rich and diverse nature of the RMCA collections allowed to document at length the wire-drawing technique and any variants in the processes. In an attempt to unearth and unravel the "*sleeping beauties*" of the Museum, an integrated collation of all data related to the C.O. of these processes were done together with observation conducted on the end-products of these lines of processes.

- The investigation: laboratory work.

Terry Childs, Africanist archaeologist and archaeometallurgist, worked on the RMCA archaeological collections in late 1980s (Childs, 1991) analyzing copper and iron-based material from the Upemba Depression cemeteries, carrying about a 100 metallographic analyses (some of them on wire material). She came in the RMCA in October 2017 for a ten-day period. She brought along all the material related to this laboratory work: the mounted samples, the micrographs and all her notebooks (carefully filed), together with the remaining “non-utilized samples”. All previous work that could be further exploited for reconstructing past manufacture processes. This was the occasion to revive an ancient network in archaeometallurgy along the setting of new collaboration, within the RMCA and with the *Centre de Recherches et de Restauration des Musées de France (C2RMF)*, Paris. Stereoscopic examination and metallographic analyses conducted on the Upemba wire objects by Terry Childs in 1990-1991 were not conclusive and had to be sustained by new ones. The impression is that drawn wire was reserved for specific objects -spiraled wire cylinders- within rich tombs. These spiraled-wire cylinders recalled other ones –‘cordelettes’- prepared for the manufacture of bangles, the wire of which was proven to be drawn wire, that were collected at the end of the 19th century and are host in the RMCA ethnographic collections (Garenne-Marot & Child 2017).

Three samples were taken from these from ethnographic ‘cordelettes”. They were meant as attested drawn wire to be used as comparative material for SEM and metallographic investigation of the archaeological material. SEM were conducted at the RMCA in the Biology department (Didier van den Spiegel) to identify (and record) the presence of surface striations and other characteristics surface marks (with the archaeological objects having been submitted to a cleaning phase). Chemical analyses (ICP-MS; RMCA, Laurence Monin, Geology Dept.) were performed on the archaeological objects. Metallography was performed at the C2RMF (Paris, France) during four weeks parted between June 2018 and January 2019, following an agreement signed between the RMCA and the Centre de Recherches et de Restauration des Musées de France (C2RMF, Paris, France). This metallography project –four archaeological samples (taken from jewellery made from spiraled-wire cylinders); three ethnographic- was led by Benoît Mille, archaeometallurgist head of Group of Research ‘Objects’ at the C2RMF with strong assistance by Nathalie Gandolfo, Technician in the same Group ‘Objects’ at the C2RMF.

- The results.

Wire in late 9th century wire from the Upemba cemeteries was not drawn (Garenne-Marot, Childs, B. Mille and N. Nikis (in the last stages of writing)). The high degree of fragmentation of the wire of the archaeological *cordelettes* had been attributed to the soil conditions of the archaeological deposits. Binocular microscopic examination of the fragments from any of the supposed *cordelettes* showed diverse ends of the pieces and sound metal along the entire wire that questioned fragmentation due to burial taphonomy. Difference in the wire diameters was seen from one piece to another but such a difference was even seen within one fragment when the cross-cuts of two coils mounted side by side for metallographic examination allowed the comparison between the four sections of the same wire. Such a difference in diameters for a single wire could not have possibly occurred if the wire had been drawn. Microstructures revealed in two of the archaeological samples that the copper wire seems to have been made of two pieces hammer welded together.

This led us to conclude that there was no *cordelettes* of long wire in Classic Kisalian graves but that these elements of jewelry were made of numerous small spirally coiled wires either mounted as helix-beads on a string or wound around a fiber core. Drawing wire allowed the manufacture of almost unlimited lengths of wire from any ductile metal, while the manufacture of small, helix wire beads certainly did not require that the wire was drawn first.

These resultsrule out the assumption that wire-drawing has been practiced in the Upemba cemeteries in Classic Kisalian times. Interestingly after the Classic Kisalian period (late 9th-second half 12th century), there was but almost no occurrence of wire in the Upemba archaeological record. In the later periods (post 14th century) spirally coiled metal wound around a fibrous core is encountered mostly in iron material but in either cases, copper or iron, it is a ribbon which is thus coiled and wound.

The earliest date for wire drawing in East, south-central, and South Africa to date are the 16th century multiple-hole drawplates and the drawn wire recovered from the archaeological site of Ingombe Ilede (Zambia) (Fagan 1969). These are related to imported –the origin of the material remains to be determined- bronze trade wire.

Part 3: Strand of *cordelettes* and "drawn" wire

Manufacture of *cordelette* (*mutuga*), village of Nguba, Katanga, DRC, 1956.

Left: preparation of the supple fiber core
Right: rolling of the wire in tight coils on this core

HP.1984.25.69-28 and HP.1984.25.69-25, collection RMCA Tervuren; photo UMHK, 1957, RMCA Tervuren ©

Cordelette
Known in the ethnographic record

Produced up to the colonial period in Eastern Central Africa, East Africa and South Africa

This bundle comprises a hundred of *cordelettes* ready to be transformed into bangles

This bundle has been collected in Katanga at the end of the 19th cent.

EO.0.0.26754-2, RMAC collection, Tervuren

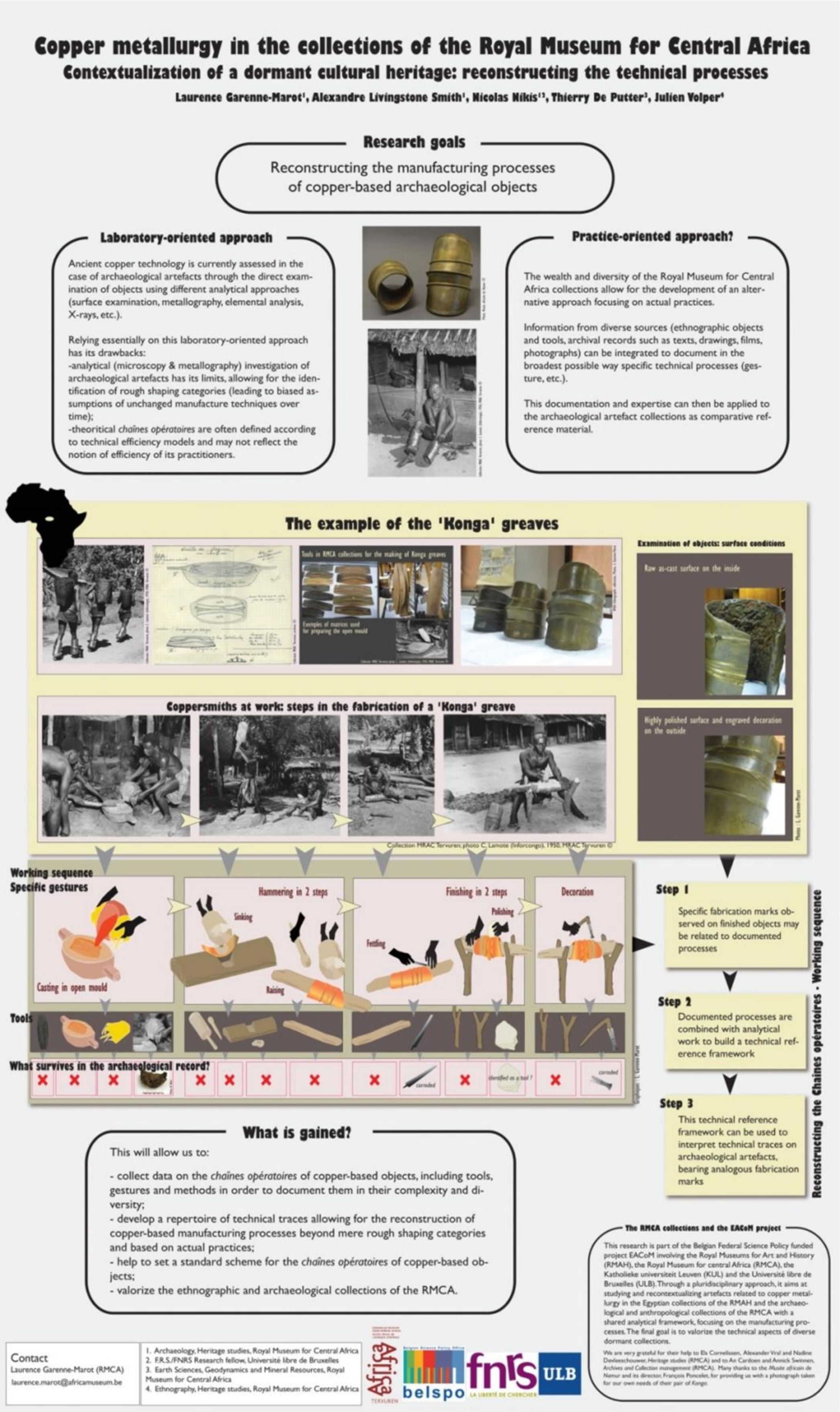
Figure 13: Slides of a PPT presentation. Communication presented at the African Archaeology Research Day, 24th November 2018, McDonald Institute for Archaeological Research at the University of Cambridge, England. Laboratory work to determine the presence of drawn wire in archaeological copper wire jewellery from the Upemba cemeteries, using ethnographic wire as reference material. Graphic L. Garenne-Marot, © RMCA Tervuren.

The seemingly late occurrence and scarcity of drawing tools in archaeological context has led us to a search for any clues about the antiquity of the technique by reviewing the ethnographic material. The work done in the RMCA ethnographic collections -inventory of the devices and tools; and all related film- and photographic material in relation to the wire drawing technique- helped to set this question within a larger issue of the origin and diffusion of the wire-drawing technique in East, South Central and South Africa. We insisted on the marked diversity in the wire-drawing processes and among the drawing devices that remains to be further explored, especially regarding the origin of each type. There is a clear-cut difference between two types of drawing tools - a single hole hemispherical die and a multiple-hole drawplate. It is the latter that has been recovered to date from archaeological contexts but there is no clue as to it being more ancient than the single-hole hemispherical one. Its cartographical distribution is mostly in the region east of Lake Victoria and it could be linked to an Arab-Indian-Persian influence and its inward diffusion via trade from the Indian Ocean coast.

The literature and ethnographic accounts suggest ties between wire drawing as it was practiced by the Yeke smiths in central Katanga, the arrival there of Yeke traders in the 19th century, and the relationships between the single-hole hemispherical drawing die used in the interlacustrine area between lakes Victoria, Kivu and Tanganyika (the Yeke home country) and the Katanga. Drawing wire allowed the manufacture of almost unlimited lengths of wire from any ductile metal. This technology could have been a later development tied to the wide diffusion of coiled and wound wire bangles and the development of trade in such items.

c) The Case of Konga Greaves in DRC

The same methodology was applied through a specific example: **the manufacture of the copper and copper alloys *Konga* greaves**. These large, heavy leglets (or greaves), parts of the dowry and meant as a specific-purpose currency but also worn as personal adornment by married women, were manufactured in pre-colonial times in the Equateur region (DRC) (Garenne-Marot, Nikis, De Putter & Livingstone Smith 2016; Fig. 14).



Copper metallurgy in the collections of the Royal Museum for Central Africa

Contextualization of a dormant cultural heritage: reconstructing the technical processes

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Research goals

Reconstructing the manufacturing processes of copper-based archaeological objects

Laboratory-oriented approach

Ancient copper technology is currently assessed in the case of archaeological artefacts through the direct examination of objects using different analytical approaches (surface examination, metallography, elemental analysis, X-rays, etc.).

Relying essentially on this laboratory-oriented approach has its drawbacks:
-analytical (microscopy & metallography) investigation of archaeological artefacts has its limits, allowing for the identification of rough shaping categories (leading to biased assumptions of unchanged manufacture techniques over time);
-theoritical *chaînes opératoires* are often defined according to technical efficiency models and may not reflect the notion of efficiency of its practitioners.

Practice-oriented approach?

The wealth and diversity of the Royal Museum for Central Africa collections allow for the development of an alternative approach focusing on actual practices.

Information from diverse sources (ethnographic objects and tools, archival records such as texts, drawings, films, photographs) can be integrated to document in the broadest possible way specific technical processes (gesture, etc.).

This documentation and expertise can then be applied to the archaeological artefact collections as comparative reference material.

The example of the 'Konga' greaves

Examination of objects: surface conditions

Coppersmiths at work: steps in the fabrication of a 'Konga' greave

Collection MRAC Tervuren; photo C. Lamote (Inforcongo), 1950, MRAC Tervuren ©

Step 1

Specific fabrication marks observed on finished objects may be related to documented processes

Step 2

Documented processes are combined with analytical work to build a technical reference framework

Step 3

This technical reference framework can be used to interpret technical traces on archaeological artefacts, bearing analogous fabrication marks

Reconstructing the Chaînes opératoires - Working sequence

What is gained?

This will allow us to:

- collect data on the *chaînes opératoires* of copper-based objects, including tools, gestures and methods in order to document them in their complexity and diversity;
- develop a repertoire of technical traces allowing for the reconstruction of copper-based manufacturing processes beyond mere rough shaping categories and based on actual practices;
- help to set a standard scheme for the *chaînes opératoires* of copper-based objects;
- valorize the ethnographic and archaeological collections of the RMCA.

The RMCA collections and the EACoM project

This research is part of the Belgian Federal Science Policy funded project EACoM involving the Royal Museums for Art and History (RMAH), the Royal Museum for central Africa (RMCA), the Katholieke universiteit Leuven (KUL) and the Université libre de Bruxelles (ULB). Through a pluridisciplinary approach, it aims at studying and recontextualizing artefacts related to copper metallurgy in the Egyptian collections of the RMAH and the archaeological and anthropological collections of the RMCA with a shared analytical framework, focusing on the manufacturing processes. The final goal is to valorize the technical aspects of diverse dormant collections.

We are very grateful for their help to Els Cornelissen, Alexander Vral and Nadine Devleeschouwer, *Heritage studies* (RMCA) and to An Cardoen and Annick Swinnen, *Archives and Collection management* (RMCA). Many thanks to the *Musée africain de Namur* and its director, François Poncelet, for providing us with a photograph taken for our own needs of their pair of Kongo.

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Figure 14: Poster on the manufacture of Konga Greaves presented at the Society for Africanist Archaeology, Toulouse 2016. Graphic L. Garenne-Marot, © RMCA Tervuren.

DISSEMINATION AND VALORISATION

Museum display and outreach

The work done contributed to improve the RMCA's internal databases and make them fit for Internet release. In the course of data collection in the ethnographic collections, a glossary was elaborated to precise (both in English and French) the adequate terms to designate both the object (tools: wooden patterns, wire drawing dies and vises) and the technique(s) related to this material. This was meant to correct inappropriate data (wrong definition or terminologies) encountered in the data base. Some modifications based on the objects' cards were done in *TMS* (*The Museum System*, an integrated database, used by the RMCA for ethnographic collections). We started to work on the system to adapt the TMS database to the recording of the archaeological copper and iron artefacts, with a special emphasis on the C.O. of forming techniques. The grave T.53 of the cemetery of Sanga, Upemba Depression (RDC) has been used as a case-study.

Some results of the project were integrated into the new RMCA permanent exhibition, opened since the 8/12/2018. Results of the works on the Upemba graves and on the copper cross-shaped ingots and the trade of copper have been valorised in the 'Long History' section (curator: ALS, P2 RMCA, with collaboration of LGM and NN, P4 ULB & TDP, P5 RMCA) and the results of the investigation around the wire-drawing Katanga post-and-lever process were also integrated in the 'Resources paradox' section (LGM, P2 RMCA)

Furthermore, copper object from Sanga have been digitized by Aurore Mathys, DIGIT-03 project, RMCA in collaboration with members of the project for the selection and the metadata (ALS & LGM P2 RMCA; NN P4 ULB) and publicly released on sketchfab plateform (https://skfb.ly/YEuz).

Strengthening of research data was done by archiving former research (Terry Childs' data of elemental analyses and metallographic expertise) and evaluating its potential for future research **and acting the development of other collaborative networks.** At the request of the *Institut des Sciences Humaines* of Mali, an expertise was conducted in 2017 on copper-based monetary items from a ca 13th cent. AD -at the time of the West African Mali empire- archaeological context. The expertise was done in collaboration with Laurence Monin (RMCA Earth Sciences Department; ICP-MS analyses), Arnaud Henrard (RMCA, Invertebrates, Biology Department; macro-photographs taken at different depths with a Leica MZ16 and combined in a one unique and sharp final image using the Leica Application Suite (LAS) software). A 3D image of one of these items was also done by Aurore Mathys (RMCA, 3D graphic designer). Other collaborative work was done with the CAST:ING Group international network, researchers from diverse fields with expertise on bronze sculpture initiated in 2016 by the J. Paul Getty Museum, Los Angeles, CA, USA and the C2RMF (*Centre de Recherches des Musées de France*, Paris). The CAST:ING project aims at promoting/fostering the sharing of skills, experience, and vocabularies and knowledge-making among this group of experts (metallurgists, archaeologists, art historians, conservators, curators, etc.) with the final objectives of writing *Guidelines to good practices for technical study of cast bronzes* and *Vocabulary/Thesaurus* and their online publication.

University lectures.

Results of the research on copper manufacturing processes and the RMAH and RMCA collections has been further integrated in several university lectures:

L. Garenne-Marot. Les terres cuites Nok. Expressions de cultures encore peu connues du Nigeria au 1er millénaire av. J.-C., lecture at *Art et archéologie : Afrique* (HAAR-B100, Prof Olivier Gosselain), Université libre de Bruxelles, 20/02//2017

L. Garenne-Marot. Arts du métal et cultures au Nigeria : Ife-Benin-Igbo Ukwu, lecture at *Art et archéologie : Afrique* (HAAR-B100, Prof Olivier Gosselain), Université libre de Bruxelles, 23/02/2017

N. Nikis, A Thousand Years of Copper Production, Trade, and Polities in Central Africa, lecture at *Prähistorisches Kolloquium* (Prof Hans-Peter Wotzka), Universität zu Köln, 8/07/2019.

N. Nikis & **A. Livingstone Smith**, Poterie, cuivre et politique : quand la culture matérielle et les chaines opératoires nous renseignent sur les anciens espaces politiques, lecture at *Approches croisées des cultures matérielles africaines* (HAAR-D400, Prof Olivier Gosselain), Université libre de Bruxelles, 9/11/2018.

N. Nikis. La métallurgie du cuivre en Afrique centrale, lecture at *Travaux dirigés : Archéologie de l'Afrique* (HAAR-B-260, Prof Pierre de Maret), Université libre de Bruxelles, 15/03/2017

N. Nikis. La métallurgie du cuivre en Afrique centrale, lecture at *Travaux dirigés : Archéologie de l'Afrique* (HAAR-B-260, Prof Pierre de Maret), Université libre de Bruxelles, 14/04/2016

F.W. Rademakers Reconstructing ancient metallurgy, Seminar at Vrije Universiteit Brussel, ArCPIG meeting, 27/10/2015

F.W. Rademakers Into the crucible! (But how, and why?), Seminar at KU Leuven, LARS meeting, 26/11/2015

F.W. Rademakers Copper for Pharaoh... and his people? Approaching early Egyptian metallurgy through excavation, analysis and experiment, Tuesday Seminar Series, University of Sheffield, Department of Archaeology, 7/02/2017

F.W. Rademakers Copper and its alloys in ancient Egypt, Aegyptologisches Seminar, Freie Universität Berlin, 14/05/2019

G. Verly. Bruxelles, Musée d'Art et d'Histoire de Bruxelles MRAH, 2019, premier et second quadrimestres, maître de stage, encadrement de 12 stagiaires pour l'étude d'artefacts égyptiens, au sein des collections égyptologiques des MRAH, liés à la production métallurgique en Egypte antique.

G. Verly, Bruxelles, Université Libre de Bruxelles, 2019, second quadrimestre, assistant invité au séminaire Préhistoire et Protohistoire HAARB-4200 MA1 et MA2 : Technicité des matériaux - Méthodologie - Archéologie expérimentale et archéométallurgie.

G. Verly, Bruxelles, Musée d'Art et d'Histoire de Bruxelles MRAH, 2018, premier et second quadrimestres, maître de stage, encadrement de neuf stagiaires pour l'étude d'artefacts égyptiens, au sein des collections égyptologiques des MRAH, liés à la production métallurgique en Egypte antique.

G. Verly, Bruxelles, Université Libre de Bruxelles, 2018, second quadrimestre, assistant invité au séminaire Préhistoire et Protohistoire HAARB-4200 MA1 et MA2 : Technicité des matériaux - Méthodologie - Archéologie expérimentale et archéométallurgie.

G. Verly, Bruxelles, Musée d'Art et d'Histoire de Bruxelles MRAH, 2017, premier et second quadrimestres, maître de stage, encadrement de sept stagiaires pour l'étude d'artefacts égyptiens, au sein des collections égyptologiques des MRAH, liés à la production métallurgique en Egypte antique.

G. Verly, Tel Aviv, Université de Tel Aviv, 2017, 31 janvier, conférencier, encadrement pour le projet « Mines restitutions at Timna » et conférence pour le MA2 et chercheurs de la CTV.

G. Verly, Bruxelles, Université Libre de Bruxelles, 2017, second quadrimestre, assistant invité au séminaire Préhistoire et Protohistoire HAARB-4200 MA1 et MA2 : Technicité des matériaux - Méthodologie - Archéologie expérimentale et archéométallurgie.

G. Verly, Bruxelles, Musée d'Art et d'Histoire de Bruxelles MRAH, 2016, second quadrimestre, maître de stage, encadrement de deux stagiaires pour l'étude d'artefacts égyptiens, au sein des collections égyptologiques des MRAH, liés à la production métallurgique en Egypte antique.

G. Verly, Bruxelles, Université Libre de Bruxelles, 2015, premier quadrimestre, assistant invité au séminaire Préhistoire et Protohistoire HAARB-4200 MA1 et MA2 : Technicité des matériaux - Méthodologie - Archéologie expérimentale et archéométallurgie.

Conference presentations

2015

Garenne-Marot L. (with the collaboration of Barbara Plakensteiner) “*African bronzes*”: current state of knowledge, issues, and inquiries pertaining to African bronze sculpture.Launch meeting of the *CAST:ING* Group project at the J. Paul Getty Museum, Los Angeles, CA, USA, 18th-19th October 2015. Presentations on “Summaries of the state of technical research on bronze sculpture”. **Oral presentation**.

Nikis, N., & Livingstone Smith, A. Copper and exchange networks in southern central Africa in 2nd millennium AD: Contribution of the material culture spatial analysis. Presented at the Journal of Southern African Studies 1st biennial conference. “Southern Africa beyond the west: Political, economic and cultural relationships with the BRICS countries and the global South”, Livingstone, Zambia, 11/08/2015. **Oral presentation**

Nikis, N., Verly, G., & Garenne-Marot, L. Un apport de l’ethnographie à l’archéologie expérimentale? Réflexion à partir de la production de cuivre au début du 20e s. Au Katanga (RDC). *International Conference ICA 2015: Non-Ferrous Metals Metallurgy and Experimental Archaeology*. Brussels, Belgium, 3rd-4th October 2015. **Oral presentation**

Rademakers, F.W., Making bronze for the pharaoh: a look into the Pi-Ramesse crucibles, Exhibition *Gegossene Götter – Metallhandwerk und Massen production im Alten Agypten*. Universität Bonn – Agyptisches Museum Bonn, Germany, 6th-8th March 2015, **Oral presentation**.

Rademakers, F.W., Rehren, Th. and Cholakova, A., Fragmented remains of Roman crucible metallurgy in Thrace. *Archaeometallurgy in Europe 2015, 4rd International Conference*. Madrid, Spain, 1st-3rd June 2015**. Oral presentation.**

Rademakers, F.W., Rehren, Th., Pernicka, E. and Pusch, E.B., Bronze production in Pi-Ramesse: crucible and metal evidence. *International Conference ICA 2015: Non-Ferrous Metals Metallurgy and Experimental Archaeology***.** Brussels, Belgium, 3rd-4th October 2015. **Oral presentation**.

Verly, G., Melting furnace R4 – ME, Exhibition *Gegossene Götter – Metallhandwerk und Massen production im Alten Agypten*. Universität Bonn – Agyptisches Museum Bonn, Germany, 6th-8th March 2015, **Oral presentation**.

Verly, G., Smelting furnace in Ayn Soukhna. *Archaeometallurgy in Europe 2015, 4rd International Conference*. Madrid, Spain, 1st-3rd June 2015**. Oral presentation.**

Verly, G., Presentation of the experimentations on the Osirian bronze statuettes, in the presentation of Johannes Auenmüller: Late Period Bronze Casting: The workshop artifacts from the Qubbet el-Hawa. *International Congress of Egyptologists, ICE XI*. Florence, Italy, 23rd-30th August 2015. **Oral presentation**.

Verly, G., Méthodologie de l'archéologie expérimentale, Fours de réduction et Four de fusion du Moyen Empire à Ayn Soukhna. *International Conference ICA 2015: Non-Ferrous Metals Metallurgy and Experimental Archaeology***.** Brussels, Belgium, 3rd-4th October 2015. **Oral presentations**.

Verly, G., Bronze Final III – Site de Presles Entonnoir du trou des Noutons : Technique de la cire retrouvée, études archéologiques et restitutions expérimentales. *International Conference ICA 2015: Non-Ferrous Metals Metallurgy and Experimental Archaeology* Brussels, Belgium, 3rd-4th October 2015. **Poster presentation**.

Verly, G., Fours de réduction du Moyen Empire à Ayn Soukhna. SAFEMM, Rencontre annuelle de la SAFEMM**.** Forges-les-Eaux, France, 30th October – 1st November 2015. **Oral presentation**.

2016

Garenne-Marot, L. & Mille B. Physical evidence of trans-Saharan trade: New analytical data for assessing the copper trade of the south-western Sahara during the medieval period" Presented at the Society of Africanist Archaeologists 23rd Biennial meeting, Toulouse, France, 26/06-02/07/2016. **Oral presentation**

Garenne-Marot, L., Nikis, N., De Putter, T., Livingstone Smith, A. Copper Metallurgy in the Collections of the Royal Museum for Central Africa: The Study and Technical

Contextualization of a Dormant Cultural Heritage. Presented at the Society of Africanist Archaeologists 23rd Biennial meeting, Toulouse, France, 26/06-02/07/2016. **Poster**

De Putter, T., & Nikis, N. The Mindouli (Republic of the Congo) mining district revisited (1): Geological context and preliminary results on the formation of complex, multiphase, Cu-Pb-Zn deposits. Presented at the 5th International Geologica Belgica 2016 Congress, Mons, 26-29/01/2016. **Poster**

Nikis, N., & De Putter, T. A geological context for ancient copper production in the Niari basin (Republic of Congo). Presented at the 5th International Geologica Belgica 2016 Congress, Mons, 26-29/01/2016 **Oral presentation**

Nikis, N. On Copper Production in Western Central Africa in the 2nd Millennium AD: New Insights from Recent Archaeological Fieldwork in the Niari Basin (Republic of the Congo). Presented at the Society of Africanist Archaeologists 23rd Biennial meeting, Toulouse, France, 26/06-02/07/2016 **Oral presentation**

Nikis, N. Cuivre, entités politiques et réseaux d'échange au sud de l'Afrique centrale durant le 2e millénaire AD. Presented at the Du passé au présent, et inversement. Hommage à Pierre de Maret, Université libre de Bruxelles, 02/10/2016. **Oral presentation**

Rademakers F.W., Verly G., Delvaux, L. and Degryse, P. , Egyptian bronzes at the RMAH. ISA 2016, the 41st International Symposium on Archaeometry. Kalamata, Greece, 15th-21st May 2016. **Oral and poster presentation.**

Rademakers F.W., Verly, G., Delvaux, L. & Degryse, P. Chemical and lead isotope characterisation of copper production in the 13th-19th century CE Niari Basin, Republic of Congo. Presented at the Society of Africanist Archaeologists 23rd Biennial Meeting, Toulouse, France, 26/06-02/07/2016. **Oral presentation.**

Verly, G. La métallurgie du cuivre en Egypte ancienne : premiers résultats du projet EACOM**.** Journée de contact FNRS Egyptologie in the RMAH. **Brussels**, Belgium, 23 April 2016. **Oral presentation**.

Verly, G. and Rademakers F.W., Experimental analysis of Ayn Soukhna copper smelting**.** *ISA 2016, the 41st International Symposium on Archaeometry*. Kalamata, Greece, 15th-21st May 2016. **Oral and poster presentation**.

Verly, G., Tools used in the production of copper chisels at Ayn Soukhna, Egypt – A holistic approach to the Middle Kingdom *chaîne opératoire*: archaeology and experiment**.** *The metalworker and his tools: symbolism, function and technology in the Bronze and Iron Ages*. Belfast, United Kingdom, 23rd-26th June 2016. **Oral and poster presentations**.

Verly, G., Réduction du minerai de cuivre au Moyen Empire (Egypte) : données archéologiques d'Ayn Soukhna, archéologie expérimentale et méthodologie. *L'expérimentation d'un séchoir de Gaule romaine. Orientations et perspectives de l'expérimentation en archéologie.* Paris, France, 6th October 2016. **Oral presentation**.

2017

Rademakers, F.W., Verly, G., Delvaux, L. & Degryse, P., Copper provenance in ancient Egypt: new insights from lead isotope data**,** *SAEMT (Science of Ancient Egyptian Materials and Technologies Conference),* Cairo**,** Egypt, November 2017. **Oral presentation**.

Verly, G., Qubbet el-Hawa casting moulds (Aswan, Egypt) : Experimental Archaeology of the Process of Antique Lost Wax Technique : données archéologiques de Qubbet el-Hawa, archéologie expérimentale, archéométrie et µCT-scans. *Experimental Archaeology Conference EAC10: 2017***.** Leiden, Netherlands, 19th-22nd April 2017. With support of the Fondation Roi Baudouin – Fonds Comhaire. **Oral and poster presentations**.

Verly, G., Delvaux, L., Degryse, P. & Rademakers, Integrating excavation, experiment and analysis into the study of ancient Egyptian metallurgy: the EACOM project approach**,** *EAA 2017 Building Bridges, the 23rd Annual Meeting of the European Association of Archaeologists 2017***.** Maastricht, Netherlands, 30th Augustus-3rd September 2017. **Oral presentation**.

Verly, G., Rademakers, F.W., Auenmüller, J., Delvaux, L. & Degryse, P., Smelting process in Ain Sukhna (Red sea coast) : experimental archaeology and archaeology**,** *SAEMT (Science of Ancient Egyptian Materials and Technologies Conference),* Cairo**,** Egypt, November 2017. **Oral presentation**.

Verly, G., Auenmüller, J., Delvaux, L. & Rademakers, F., Le mythe osirien au vu des données archéologiques et technologiques. Association Egyptologique Reine Elisabeth - RMAH, Brussels, Belgium, 19th November 2017. **Oral presentation**.

2018

Garenne-Marot, L. African ‘bronzes’, from a royal art to a more mundane one: reconstructing the forming techniques. Presented at Conference ‘Methods and techniques of archeometallurgy: recent research on bronze sculpture’, APSARA National Authority, Cambodia, 11/01/2018 (conference held within the framework of the CAST:ING Group project). **Oral presentation**

Garenne-Marot, L. Reconstructing the chaînes opératoires in the manufacture of archaeological metal objects: the Upemba Depression (DRC) jewellery. Presented at the African Archaeology Research Day, University of Cambridge, 24/11/2018. **Oral presentation**

Nikis, N., Rademakers, F., & De Putter, T. Technological shifts for cultural changes? Copper productions in Niari (Rep. Congo), 10th-19th century. Presented at the Society of Africanist Archaeologists (SAfA). 24th Biennial Meeting: Building Bridges to the African Past, Toronto, Canada, 18-21/06/ 2018. **Oral presentation**

Nikis, Nicolas, & Livingstone Smith, A.. Documenting the trade from the copper deposits in Central Africa. The case of the Niari Basin in the second millennium AD. Presented at the African Archaeology Research Day, University of Cambridge, 24/11/2018. **Oral presentation**

Rademakers, F.W., Verly, G., Delvaux, L. & Degryse, P Copper provenance in ancient Egypt: new data from Pre-Dynastic to Old Kingdom Egypt. Presented at the 42nd International Symposium on Archaeometry. **Oral presentation.**

Rademakers, F.W., Verly, G., Degryse, P., Boone, M. & Auenmüller, J. Late Period Egyptian casting technology: a new methodology integrating experiment and archaeometry. Presented at the 42nd International Symposium on Archaeometry**. Poster presentation**

Verly, G., Paris, France, 15 mars 2018 : **RIMs**, 6e séance Université Paris Nanterre, MAE - Rencontres Interdisciplinaires sur les Métaux. Présentation : L’organisation spatiale d’un atelier métallurgique au Moyen Empire (Egypte, AynSoukhna) : croisement des sources archéologique, expérimentale, ethnographique et iconographique.http://www.mae.u-paris10.fr/rencontres-interdisciplinaires-sur-les-metaux-rims/

Verly, G., Mérida, Mexico, 20-26 mai 2018 : **ISA 2018**, the 42nd International Symposium on Archaeometry. Présentations : Verly G., Rademakers F.W., Auenmüller J., Delvaux L., Degryse P. and Somaglino Cl., The *chaîne opératoire* of Middle Kingdom smelting batteries and the problem of fuel: excavation, experimental and analytical studies of ancient Egyptian metallurgy **and** Copper provenance in ancient Egypt: new data from Old to New Kingdom Egypt **and** Late Period Egyptian casting mould technology: experiment and archaeometry. http://isa2018.mx/program#scientific

2019

Debèque A., **Verly G., Rademakers F.W.,** Marchi S. and Bonnet Ch Les trois fours circulaires de Doukki Gel: archéologie expérimentale, étude technologique et archéométrie. Epoque méroïtique (300 AEC-300EC) Soudan. Poster pour International Conference ICA 2019 (ICA2), Paris – Melle, 25 septembre - 2 octobre 2019 : Contributions of Experimental Archaeology to Excavation and Material Studies. Second colloque international d'EACOM aux universités Lettres Sorbonne et MSH Paris-Saclay et à la plateforme expérimentale de Melle. **Poster presentation**.

Derenne, B., Verly, G. & Boone, M., Complementary between in situ studies and photogrammetry: Methodological feedback from a roman shipwreck in Caesarea**.** *ISPRS XLII 2019, Underwater 3D Recording and Modelling "A Tool for Modern Applications and CH Recording*". Limassol, Cyprus, 2nd–3rd May 2019. **Oral and poster presentations**.

Rademakers, F.W., Verly, G., Somaglino, Cl. & Degryse, P., Geochemical analysis of experimental Egyptian copper smelting. *AEIE V 2019 The 5th International Conference Archaeometallurgy in Europe*, Miskole, Hungary, 11th June 2019. **Oral presentation**.

Rademakers, F.W., Verly, G., Delvaux, L. & Degryse, P., From desert ores to Middle Kingdom copper: first chemical and lead isotope data from the AHM collection, Belgium**.** *ICAS-EMME 2 - 2nd International Congress on Archaeological Sciences in the Eastern Mediterranean and the Middle East,* Nicosia, Cyprus, 12th-14th November 2019. **Oral presentation**.

Verly, G. & Delvaux, L., Osirian rite and archeometallurgy. About a set of Late Period pottery molds**.** *Rethinking Osiris. CAMNES International Conference*. Florence, Italy, 26th-27th March 2019. **Oral presentation**.

Derenne, Br., Verly G., Nantet E. Limassol, Cyprus, 2–3 May 2019 : ISPRS XLII 2019, Underwater 3D Recording and Modelling "A Tool for Modern Applications and CH

Recording”. Présentations: Derenne B., Nantet E., Verly G. and Boone M., Complementary between in situ studies and photogrammetry: Methodological feedback from a roman shipwreck in Caesarea. https://www.int-arch-photogramm-remote-sens-spatial-inf-sci.net/XLII-2-W10/77/2019/

Verly, G., Rademakers, F. & Somaglino, Cl., L’apport de l’archéométrie aux fouilles et à l’étude des artefacts métallurgiques à Ayn Soukhna**.** *4^{e} Journée de rencontres en archéométrie de l’IFAO, Underwater 3D Recording and Modelling “A Tool for Modern Applications and CH Recording”*, Paris, France, 11th June 2019. **Oral presentation**.

Verly, G., Rademakers, F., Marchi, S. & Bonnet, Ch**.,** The bronze furnace of Kerma revisited a unique casting technology reconstructed through experiment, (re-)excavation and archaeometry. *AEIE V 2019 The 5th International Conference Archaeometallurgy in Europe*, Miskole, Hungary, 11th June 2019. **Oral and poster presentations**.

Verly, G., Rademakers, F. & Somaglino, Cl., Ayn Sukhna - melting and finishing part of the copper chaîne opératoire (Middle Kingdom - Egypt), Journée du Groupe de Contact FNRS « Arts et Techniques métallurgiques pré-industriels. Étude et conservation » (sous les auspices du FRS-FNRS). *Forging the “Chaîne Opératoire”*. **Oral presentation**.

Verly, G., Bruxelles, Belgique, 2 décembre 2019 : Université libre de Bruxelles - Centre de Recherches en Archéologie et Patrimoine, Journée du Groupe de Contact FNRS « Arts et Techniques métallurgiques pré-industriels. Étude et conservation » (sous les auspices du FRS-FNRS). Forging the “Chaîne Opératoire”. Présentation : Verly G., Rademakers Fr. and Somaglino Cl., Ayn Sukhna - melting and finishing part of the copper chaîne opératoire (Middle Kingdom - Egypt). https://crea.centresphisoc.ulb.be/fr/actualite/nouvelles/journee-du-groupe-de-contact-fnrs-arts-et-techniques-metallurgiques-pre

Dissemination publications (others).

Cornelissen, E. E., and **N. Nikis**. 2019. Een Archeologisch Perspectief Op Oude Handelsnetewerken in Centraal-Afrika. *Hermes. Tijdschrift van de Vlaamse Vereniging Voor Leraren Geschiedenis* 23 (1): 25–29.

Nikis, N., **and T. De Putter**. 2016. Het Koper van Niari, Een Eeuwenlang Gegeerde Natuurlijke Rijkdom. Geologische En Archeologische Studies van Koper-Lood-Zinkmijnen van Het Niaribekken (Republiek Congo). *Science Connection*, 2016.

Nikis, N., **and T. De Putter**. 2016. Le Cuivre Du Niari, Une Ressource Ancienne et Prisée. Études Géologique et Archéologique Des Mines de Cuivre-Plomb-Zinc Du Bassin Du Niari (République Du Congo). *Science Connection*, 2016.

Other dissemination activities

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Organisation of international symposia

Verly G., Rademakers Fr. W., **Clerbois S. and Nikis N.** 3rd-4th October 2015, RMAH : International Conference ICA 2015 : Non-Ferrous Metals Metallurgy and Experimental

Archaeology : http://eacom.be/wp/non-ferrous-metals-metallurgy-and-experimental-archaeology/ et http://www.kmkg-mrah.be/fr/non-ferrous-metals-metallurgy-and-experimental-archaeology

Verly G., Rademakers Fr. W., Auenmüller J. and Téreygeol Fl. 25th September – 2nd October 2019, Université Lettres Sorbonne, MSH Paris-Saclay and Melle experimental platform: International Conference ICA 2019 (ICA2) : Contributions of Experimental Archaeology to Excavation and Material Studies : https://metallurgy-ica.wixsite.com/ica2/

Participation in international symposia

Verly, G. 3-4 October 2015: International Conference ICA 2015: Non-Ferrous Metals Metallurgy and Experimental Archaeology

Organization of the first EACOM international symposium at the MRAHs: http://eacom.be/wp/non-ferrous-metals-metallurgy-and-experimental-archaeology/ and http://www.kmkg-mrah.be/fr/non-ferrous-metals-metallurgy-and-experimental-archaeology

May 6-7, 2017: creation of the metallurgical workshop and assembly of reduction furnaces

Purpose: to learn how to build mud walls and learn how to build a reduction frills battery section

Technique: Ayn Sukhna, Middle Kingdom (smelting furnaces)

Verly, G. 14-30 June 2018: creation of the metallurgical workshop to study lost wax techniques

Purpose: to test a dewaxing kiln according to archaeological data and to experiment with pottery protocols based on the technique of the archaeological evidence of Qubbet el-Hawa (Museum Bonn)

Technique: Qantir - KomTuman - Kerma, Middle Kingdom (dewaxing and melting furnaces) / Qubbet el-Hawa, Late Period (moulds QH 207)

Verly, G. 15 June - 6 July 2019: creation of the metallurgical workshop to study the production of metal plates

Purpose: to test a "de-waxing" kiln according to the archaeological data of Kerma.

Technique: Kerma, Classic Kerma (cross ovens)

Verly, G. 15 June - 8 July 2019: International Conference ICA 2019 (ICA2): Contributions of Experimental Archaeology to Excavation and Material Studies

Organization of the second EACOM international conference at the Sorbonne and MSH Paris-Saclay universities and at the Melle experimental platform: https://metallurgy-ica.wixsite.com/ica2/

Verly, G. 3-20 June 2019: creation of the metallurgical workshop to study lost wax techniques
Purpose: to test a dewaxing kiln according to archaeological data and to experiment with pottery protocols based on the technique of the archaeological evidence of Qubbet el-Hawa (Museum Bonn)
Technique: Qantir - KomTuman - Kerma, New Kingdom (dewaxing and melting furnaces) / Qubbet el-Hawa, Late Period (moulds QH 207)

Verly, G. 25 September - 2 October 2019: International Conference ICA 2019 (ICA2): Contributions of Experimental Archaeology to Excavation and Material Studies
Organization of the second EACOM international conference at the Sorbonne and MSH Paris-Saclay universities and at the Melle experimental platform: https://metallurgy-ica.wixsite.com/ica2/

Archaeological excavations
2020 **Verly, G.** Excavation of a metallurgical site dating from the Middle Kingdom, Ayn Soukhna-Egypte, Paris IV - Sorbonne : http://www.ifao.egnet.net/archeologie/ayn-soukhna/

2019 **Verly, G., Rademakers, F. W.** Excavation of a metallurgical site dating from the Middle Kingdom, Ayn Soukhna-Egypte, Paris IV - Sorbonne : http://www.ifao.egnet.net/archeologie/ayn-soukhna/

2018 **Verly, G., Rademakers, F. W.** Excavation of a metallurgical site dating from Middle Kerma, Kerma-Soudan, Mission archéologique suisse-franco-soudanaise : http://kerma-doukkigel.ch/

2018 **Verly, G.** Excavation of a metallurgical site dating from the Middle Kingdom, Ayn Soukhna-Egypte, Paris IV - Sorbonne : http://www.ifao.egnet.net/archeologie/ayn-soukhna/

2017 **Verly, G.** Excavation of a metallurgical site dating from the Middle Kingdom, Ayn Soukhna-Egypte, Paris IV - Sorbonne : http://www.ifao.egnet.net/archeologie/ayn-soukhna/

2017 **Verly, G., Rademakers F. W.** Excavation of a metallurgical site dating LBA-IA, Timna-Israël : http://archaeology.tau.ac.il/ben-yosef/CTV/

2017 **Verly, G.** Excavation of a metallurgical site dating from the Middle Ages, Castel-Minier, IRAMAT : http://castelminier.eu/Accueil&structure=site+structure&no_bl=y&page_ref_id=80/

2016 **Verly, G.** Excavation of a metallurgical site dating from the Middle Kingdom, Ayn Soukhna-Egypte, Paris IV - Sorbonne : http://www.ifao.egnet.net/archeologie/ayn-soukhna/

2016 **Verly, G.** Excavation of a metallurgical site dating LBA-IA, Timna-Israël : http://archaeology.tau.ac.il/ben-yosef/CTV/

2015 **Verly, G.** Excavation of a metallurgical site dating from the Middle Kingdom, Ayn Soukhna-Egypte, Paris IV - Sorbonne : http://www.ifao.egnet.net/archeologie/ayn-soukhna/

Collaborations

To consolidate the results and the international visibility of the project, cooperation agreements have been drafted and are in the course of being signed with each of the following:

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Regina-Pacis-Weg 7 - 53113 Bonn, Germany
Represented by Dr. des. Johannes Auenmüller (Projektmitarbeiter)

Archéosite et Musée d'Aubechies – Beloeil asbl
Rue de l'Abbaye, 1y - 7972 Aubechies

L’Institut de Recherche sur les ArchéoMATériaux IRAMAT – Melle, France

UGent : µCT scan and photogrammetry

AMeRS Association Mer Rouge-Sinaï

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ANNEXES

Annexe 1: Summary of forming techniques on copper-based objects

Copper (and its alloys) belongs to the category of “plastic solids” (A. Leroi-Gourhan) the common characteristic of which is to proceed from the “molten step to a solid one”, either by desiccation (cement), firing (clay), or cooling (wax and metal). The main characteristic of the material “copper” is that its forming can be done in either a “molten” or “solid”, state. This table of the production techniques of copper-based objects has thus been divided into two categories, “casting” and “fabrication”, depending on the state of the metal during the actual working: molten or “solid” state. The tools and techniques of the two categories overlap to some degree and ancient metalworking ateliers (and therefore) metalworkers may have been involved in the two or restricted to a specific set of techniques related to one category.

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Metal state	Designation	Technical gestures	Effects	Products / objects	African ethnographic and archaeological examples
“Molten”	Casting = Shaping of molten metal and moulding. Casting implies a wide range of metalworking activities: from the production of primary and secondary ingots to the casting of semi-finished preforms and finished objects. The casting can thus either be a single step process, the final shape of the object achieved with this single cast, or it can be a preliminary step in the manufacturing process - the cast, semi-finished object is then worked in its “solid” state. Moulds might be open or closed (bivalve or multiple-	Casting in an open mould The mould can be carved in stone or made of clay (crude or baked) or it can result from the impressing (and removal) of a form or matrix into wet sand or clay. Matrices are often made of carved wood but can be made of any hard material, such as previously cast form.		One can likewise cast some secondary ingots or shapes that will be exchanged as such. For more elaborate shapes, this casting in open mould is a preliminary stage = the cast form or semi-finished object is then worked in its solid state by shaping.	The cross-shaped ingots of the Katanga region and other regions of central Africa (13th-19th cent. AD) were cast in open-moulds, some of them made of terracotta. More recently, the discovery of a terracotta mould for the casting of coin blanks was made on the site of Essuk (ancient Tadmekka). Central Africa metalworkers in the early 20th c. cast semi-finished objects in sand using wood matrices; some of these matrices, now in the Royal Museum of central Africa (Tervuren, Belgium), bore very finely carved motives. The cast semi-finished objects obtained in open mould casting are then shaped using

piece) and can be reused if made of metal, stone or terracotta. Sand, sand/clay or clay moulds are destroyed in the “lost model” casting process, known more commonly as “lost wax” if the model is made of wax. .	Casting in a closed mould - Reusable moulds These bivalve or multiple- piece moulds can be made of metal, bronze (not found yet in sub-Saharan Africa), fine-grained stone, and terracotta. -“Lost” moulds Sand casting: finely powdered sand is packed into hinged wooden boxes to make a bivalve mould. The mould is destroyed when the cast object is removed.	The principle is to create a hollow space which will be filled by the metal. Sand casting in closed moulds: after two sides of a 3D shape are impressed in the sand (clay sand) contained in two hinged wooden boxes, the boxes are closed and the metal will be poured into the cavity that has been created.	The object is in a next-to-final stage. The stages to follow are those of joining (for pieces cast in parts) and surface treatment (metal finishing and decoration).	Stone moulds for the casting of medals were found in the 1970s on the site of Tegdaoust (ancient Awdaghust) in Mauritania. Sand casting in closed moulds is widely practiced in Africa nowadays. Determining the age of the technique is however a challenge, given the impermanence of sand moulds under archaeological conditions. Such moulds have a great resistance to heat, making them an excellent casting material, but they break down when exposed to water and wind.

		-Investment (lost-model casting) moulds Investment moulds are made by first modelling an object in wax (or other plastic material with a low melting point, such as latex or resin) and coating the model thickly in clay or a clay/organic mixture, known as the “investment »:The clay/wax assembly is then fired and the wax melted or burnt out to leave a cavity within a fired clay mould. Molten metal is then poured into the cavity, taking the exact shape of the previously present wax (hence the name lost-wax). Once the metal is solidified, the mould is broken to remove the casting. An investment-mould is like a multiple-piece mould without the constraint of having to assemble the parts.	The casts can be solid or hollow; in the case of the latter, the wax is modelled over a clay core and the metal will be poured in the space between the inner core and the outer clay investment; the core will be then covered by the metal in the finished casting. Variations in the chaîne opératoire: i.e.the joined mould and crucible method (called also the mould-cum-crucible or closed crucible method). Instead of separate crucible and mould, the two are enclosed within the same investment. Once the molten wax has run out, the fired dry mould is luted to the crucible containing the metal to be melt. The crucible part is heated and when the metal is molten, the assembly is turned upside down so that the molten metal flows into the cavity of the mould.	The object is in a next-to-final state like for objects cast from bivalve and multiple pieces mould and closed sand mould with the adding possibility of casting intricate forms.??? The stages to follow are also those of joining (for pieces cast in parts) and post-casting finishing.	An example are the beautiful casts of the ancient Nigerian cultures of Igbo Ukwu, Ife, Benin. One of the most impressive examples is the seated figure from Tada, attributed to the Ife culture (14th cent. AD), cast in pure copper, a metal not usually chosen for casting as it generates gases causing blowholes and unevenness in the surface of the casting. The process of the mould-cum-crucible (or closed crucible) technique (see Herbert, 1984:40-41 for complete description of process), and characteristic of sub-Saharan Africa, is still used in ateliers in Ivory Coast, Ghana (Akan-Baule casters), the Cameroon grasslands and in north-east Nigeria. It may have been in use as early as the 9th cent. AD at the site of Tegdaoust in Mauritania. Variations in the lost model casting process, including methods of forming the model, types of moulding and/or modelling materials, the replication or not of the model (direct or indirect casting), and methods of attaching the core to the mould and the crucible to the investment assembly, are often diagnostic of particular technological styles. Examination of these manufacturing techniques can be indicative of age and diffusion trends.

			In Africa, the“lost-wax” casting technique includes other materials besides wax for making the model: latex, gum, resin or any other material which can be modelled or carved (i.e.wood) and which can be melted and burned out at a low temperature.		A 1940 ethnographic citation (see Maquet 1965) mentions the the use among Lower Congo metallurgists of the trunk of a banana tree, being carved out and coated in clay investment. The clay/banana trunk assembly was fired leaving the imprint within the mould.
			Life-casting process. In this case, a real object or animal (often an insect) is used as a matrix or model, instead of other lost material.		The term “lost beetle” casting is also found: Akan craftsmen have delighted in casting small objects directly: beetles, crustaceans (crab claws), chicken feet and the like. These were used for producing brass gold weights.

“Solid” The metal would have been cast in a rough shape before beginning the “shaping”	Shaping or forming is the term applied to the fashioning of metal through plastic deformation, using stretching, shrinking, bending and cutting the metal. Techniques include raising, sinking, bending, punching, and drawing of the metal. “Wrought” metalwork describes the state of having been shaped by hammering and other mechanical means.	Hammering or Forging: the most common form of shaping. It is the controlled shaping of metal by the force of a hammer on an anvil or a stake. “Hammering” is the term more often applied to non-ferrous metals and “forging” to ferrous ones. Sinking and raising are two methods of forming metal objects either by hammering into a concave indented anvil or on a raised stake or stout pole.	Hammering/Forging can be done while the metal is hot or cold. Forging not only shapes the object, but also hardens it (important for edging tools). In order to restore the original softness and ductility of the metal so that the working can continue, heating (annealing) the work-hardened metal at intervals is necessary in order to allow the regeneration of the metal’s crystalline structure	Ingots and bars are transformed into wire.	There are many objects, which have been made by hammering. Among the archaeological items are the hammered spiralling snakes that acted as ferrules on Igbo Ukwu ceremonial staffs (Willet 1983:65).
				Masses can be hammered into plaque and into flat sheet.	Example of an elaborate sheet work: the Ibo Ogba anklets (Nigeria) “were beaten out of a solid brass bar… They not only sheathed the lower leg but projected out at right angles as much as six inches” (Herbert 1984: 16; see examples in Catalogue “Parure”, musée Barbier- Mueller: 62, n°65).
			Sinking, sometimes called doming or dishing, results from hammering sheet into a depression in an anvil or other hard form..	The two techniques are sometimes employed conjointly.	Other uses of sheet copper-based metal: the gold dust and shea butter boxes (forowa) of the Akan (Ghana); the beaten brass wares of the Nupe (Nigeria) and Fang (Gabon) and chamfrons bedecking and protecting Hausa and Nupe horses (northern Nigeria).
			Raising = hammering over a shaped stake or form with repeated courses of hammering and annealing.		
Example of a chaîne opératoire that involves two major steps, casting in an open mould and shaping by sinking and raising = the forming of konga (huge leggings or greaves worn by women until the 1950s on the left bank of the Congo). Photographs, taken by M.C. Lamotte in Congo in the 1930s (and published by E. Maquet ,1965: 13-14), record the making of these ornaments. The mould, a wooden container with handles, is filled with clay onto which a wood matrix -a large flat parallelepiped, which has a midrib and two secondary ribs that protrude- is impressed. The molten metal is poured into the mould where it is spread using a flat wood. The greave, flat, is then curved still hot, by hammering (sinking) it onto a depression carved out in a log, using an enormous wooden mallet. To perform this operation, the greave is hammered from the interior not directly on the metal but on a piece of wood of a crude oval shape resting on it. After, the final closing of the tubular form of the grease is done by working it from the outside on a branch, the approximate size of a leg. Then follows the finish: filing, polishing and engraving.					

		Wire making Drawing A rod of copper (or brass) is drawn through a succession of ever-smaller holes in a greased drawplate. The drawplates are either iron plates pierced with holes of decreasing diameters or a pierced tubular mass the hole of which is made narrower in the course of the process.	The drawing is forced by mechanical traction of a block or a bar (of several centimetres in diameter) through holes of more and more reduced diameter until the wire obtained is (sometimes) less than half a millimetre in diameter. Drawn wire is very even in thickness and sometimes shows irregularities in the surface caused by imperfections in the drawing plate holes.	Wire-drawing has been found in eastern, east-central, and southern Africa but is less well known in other parts of central and West Africa. In the case of wire drawing, it produces “veritable works of art” as a traveller to Katanga remarked, wire that was as fine as “the strings of a musical instrument in Europe” (Capello and Ivens 1888: 112). At Ingombe Ilede (Zambia), wire drawing implements (draw plates and tongs) where recovered in 15th cent. tombs together with copper wire bobbins (wire wound onto cores of folded wires) prepared for trade (Fagan et al. 1969; Phillipson and Fagan, 1969). Among the tools collection of the Royal Museum of Central Africa, examples of different systems of drawplates and tongs from the ethnographic record are found.	
		Hammering The wire is formed by hammering longitudinal strips of metal into wire forms.	Hammering produces wire of uneven thickness, often distinguished by remnants of the longitudinal seam lines which were formed during the overlapping of sheet metal edges.	Transformation of this wire: Twisting, braiding, etc.	The wire thus obtained could be wrapped around fibre or animal hair but could also be twisted and braided. Examples of jewels made by these diverse transformations of the wire products have been recovered in the tombs of the Upemba cemeteries as early as the 8th cent. AD. Ethnographic literature for Central,

					Eastern and Southern Africa is rich in examples of the use of wire in making jewels and ornaments and in decorating ceremonial weapons.
				Transformation of wire: Cutting the wire in order to make links for chain-making. Bending and soldering.	Chains were wrapped around ankles or arms as ornaments. Some were found in the Upemba graves. In the ethnographic record, G. Lindblom spoke in 1920s of the Akamba metalworkers who were famous over the whole of East Africa for their chain-making. The finest of the chains, used by the young men for leg ornaments, look, as Lindblom wrote, at a few metres' distance like painting on their legs.
	Shaping by material removal	Cutting Piercing Openwork	Many thin objects are cut out of sheet metal of varying thickness, including ribbons, discs, and plaque-like forms.	Ribbons are thus made. Cf. Decorative techniques	Several traditions of working copper material by cutting and piercing in Central Africa: examples in some ceremonial weapons Suka or Tshokwe
“Molten” and “solid” Joining techniques	Cold joining Most of these techniques can only be applied to metal sheet or thin-walled cast objects.	Joining of pieces of metal through mechanical interlocking of metal components.	Two metal sheet are assembled by overlapping the edge of one of the sheets with the edge of the other, often with hammering..		
		Stapling	Two metal sheets are assembled and held together with staples of different shapes.	The staples are segments of wire introduced into holes pierced in the pieces to be joined. The wires are bent over, folded or knotted in place.	

		Nailing	Nailing can be a joining technique - i.e. fastening with nails of an element of metal on another metal or on another material; it can also be a decorative technique. Cf. Decorative techniques.	In the case of the Kota reliquaries, copper or brass ribbons are fastened to the wooden core by small hand-formed nails. They are also inserted into openings in the wood to fasten them.	
		Riveting	Rivets: either cast into shape or formed by rolling up a short length of strip or sheet. The rivet is inserted into the sheets to be joined and hammered from both sides to form 'heads' of metal to secure it in place.	The joining is non reversible	
		Joining with locking pin	This is used in order to secure several pieces with a small straight metallic rod	The joining is reversible	Uses of locking pins in the case of elements with three loops used for

			which goes through holes previously made in the pieces to be joined.		connecting chains in horse harnesses from the site of Sintiu Bara (Senegal River, 12th cent.) (Thilmans et Ravisé 1980; Garenne-Marot 1996).
		Joining with a ring	A metal ring or ferrule can encase elements in materials susceptible to cracking and splitting (wood, ivory, bone).	The rings can have both a functional and decorative purpose	Examples of the javelins of Natamatao (Mali, 13th cent. AD); the iron heads of which were attached and maintained to the wooden shaft with a brass ring. The two metal colours thus associated created a decorative effect (Ouedraogo 2013).
	Hot joining	Hot joining without added material Fusing or welding using-welding	The pieces are joined by simultaneously fusing their edges.	Only iron is susceptible to give perfectly invisible fused edges thanks to its great capacities of welding when subjected to high temperatures. This is not the case with copper and alloys.. Metallographic analyses performed by T.S. Childs (1991) on archaeological objects from the Upemba cemeteries (Katanga, RDC) have shown that fusing –welding were systematically conducted on copper objects but with poor results.	

		Hot joining with added material Soldering. Soldering with brazing material (brazing)	Solder = the pieces are joined with an intermediate fusible alloy (added metal) the melting point of which is lower than that of the metals to be joined. A type of soldering that uses a brazing material made commonly of a copper/zinc alloy whose color can be suited to the copper alloy parts to be joined. The melting point of brazing material is higher than that of soft solders.	Examinations using magnification may reveal the use of soldering or brazing material on copper-based objects south of the Sahara. For the moment it is only on objects in gold and silver, in order to solder on the metal body threads, beads and cupula decors (filigree and granule techniques) that solders have been highlighted (examples of the jewels – among these a large round pectoral- of the tumuli of Rao, Senegal; 14th cent.; Joire 1955). In the case of a pendant from Sintiu Bara (Senegal river, 12th cent. AD), made of an interesting alloy of brass-silver, the tubular suspension loop is joined to the braid-shaped body by means of a solder (Thilmans et Ravisé 1980).	
What is seen in the Akan tradition tells a lot on manufacture choices in African metalworking traditions: objects of foreign design –such as the kuduo– were copied but instead of shaping them by soldering the motifs on the sheet metal body such as in the original manufacture, the kuduo were entirely (body and decoration) made using casting techniques (lost wax or latex). Other Akan objects such as brass pendants have a decoration imitating filigree but were also made by wax modelling and casting; the colour of the alloy together with the thinness of the decoration give to these objects the appearance of gold filigree jewels. Kapsiki cache-sex pendants from the ethnographic record of Northern Cameroon recall the twisted wire jewels of the Congo basin (See Herbert 1984a: 6, fig. 8) but were also done by wax modelling and casting.					
		Casting-on The concept = a molten metal can fuse with a metallic base of the same metal composition if the surface of the primary metal is perfectly cleaned and protected from oxidation, in an oxygen-free atmosphere.	It means pouring molten metal (of the same composition as the cast metal) to add on an additional element. These castings-on involve the whole lost-wax process: the linking parts being first modelled in wax in position, the whole enveloped in clay, and metal run in to replace the wax.	This method can be used to assemble a complex object but is also used to carry out a repair.	The “ropepot” of Igbo Ukwu (a bronze vessel on a stand encased in a network of bronze “ropework”) is one of the best examples of assemblage by casting-on (Shaw 1970). F. Willett (1983) reviewed the multi-stage process: the rope was passed over the pot from above and bent to fit. Next, the rim was attached to the body of the pot by casting-on more metal. The two parts of the base were joined in the same way, the lower loops of the ropework being added at the same time.

					The modelled décor of the 'flanged cylinders' of thin sheet from the site of Podor (Senegal, 12th cent. AD; Chavane 1985) was performed by the casting-on method.
“Solid” Surface treatments Finishing	On cast objects	Cleaning Deburring	In sculpture: cutting of all the sprues, vents and runners with chisels.		A good example of an examination of the “cleaning” of a sculpture is that of the Olokun head found in 1912 by Leo Frobenius. The observation was made at the Scientific department of the British Museum (Craddock et al.2013). It showed that the chiselling work was not conducted in a systematic way and certain casting flaws are still visible at the base of the crest.
		Chiselling	Removing surface flashes and casting flaws. Refers also to general further elaboration for fashioning details.		
		Repairing	Providing the means to repair gas porosity, shrinkage, and other defects that were incurred during casting, ie., a cold shut, in which the metal failed to flow properly into the mould.	There are several repair techniques. For small defects, set-in patches: round plugs and rectangular or other geometrically shaped patches with some bearing a rivet to maintain the plug or the patch in place. For large defects, castings-on (see above) could have been used.. The “seated figure of Tada” (see above) shows, according to F. Willett (1983) who closely examined it, “evidence of a score of repairs”. The fact that pure copper was used for the casting may explain all the defects in the cast figure. Still no explanation is given as to by which means these repairs were made. Repairing in African sculpture remains to be investigated.	
	On hammered objects	Planishing	A fine even hammering with a highly polished hammer	Create a smoother surface on hammered objects.	In the case of archaeological objects corrosion layers may conceal such surface treatments.

	On cast and hammered objects	Filing Polishing	To smooth the surface by abrasion. The polisher may be an abrasive stone (sandstone for ex.) or sand.		
		Burnishing	It is the plastic deformation of a surface due to the sliding contact with a hard and smooth surfaced objet: tooth, stone (agate or hematite)	It smears the texture of a rough surface and makes it smoother and shinier.	
	Colouring and patina	This can be done either by chemical treatment or by heat, or by a combination of the two. In the case of heat patina, copper is submitted to heat variations. The oxidation thus produced vary accordingly to the metal composition and the heating intensity: the colour of the copper can vary from orange red to brown to deep purple. As for the decoctions used for patinas, in workshops the recipes are always kept secret! Corrosion products mask largely old intentional patinas.	E. Herbert (1984) cites different cases of copper colouring: the Lemba metallurgists in the northern Transvaal who were able to give copper wire a fine yellow colour by using a decoction from a certain plant and then weave it with other wire that had been burnished a brilliant flame red; the so-called purple copper of Luanda that had a market as far away as Calabar in the eastern Niger Delta. Nowadays, the copper workers of Brazzaville use heat patina to add varied colours to the motifs on their repoussé copper plates.		

"solid" Decorative	By removing material The tools are files, chisels.	Cutting Piercing Punching	Removal of chunks of metal. The tools are sharp chisels.	Motifs are cut out right through the material (the sheet of metal) providing	Ethnographic examples: Songye and Suku ceremonial weapons displayed intricate openwork

techniques				openwork decoration	decorations. Tuareg wooden boxes and coffers were lavishly decorated with openwork decoration cut-out of copper, brass and iron sheets.
		Incising Stippling Engravingi	Linear motifs The tools are sharp, hard-cutting tools: chisels, gravers and burins.	The metal is cut and pushed, so that the tool removes a long, thin curl of metal. The shape of the incised line (often “v” shaped) is given by the end of the tool used.	The chiefs’ collars of the Teke (Middle Congo, region of the Stanley Pool) highly decorated with geometric motifs by means of a white-hot burin dipped in oil to heighten its resistance to heat.
	By material sinking By indirect striking with a mass	Tracing	Linear motifs The tools are chisels with a slight blunt edge that is pushed with a little hammer.	The result is a “linear impression”. The metal is displaced, turned again on the sides, and can afterwards be levelled with a hammer.	
		Stamping Punching Matting The work is done on the obverse side	Use of dies (the ends of them bear a motif) and punches (the end of them are shaped in a circle or half a circle, a triangle, etc.). In the case of stamping work, the work may be performed on a solid object or sheet metal. In stamping solid objects, dies and punches will have to be in a harder material than the surface to be hit: in iron for example for striking copper and copper-based objects.	There exist numerous examples of objects thus decorated by using dies and punches. Several ethnographic objects stored in the Royal Museum of Central Africa (Tervuren, Belgium) bore decorative work done by repoussé : Kusu stools; Salampasu masks, etc	

		Repoussé work Repoussé is used to work on the reverse of the metal (a sheet metal) to form a raised design on the front.	The working of the metal mainly by stretching using hammers and punches to create relief (usually on the frontside). The repoussé work is necessarily performed on a metal sheet, which is placed on a soft base in order not to crush the motifs in relief on the obverse. The main reliefs are then reworked on the obverse by chasing with chisels. When working on metal sheet, dies and punches might be in iron or stone but can also be in softer material: wood or bone. Chasing tools such as rounded chisels are also used.	
		Chasing	The working of the metal from the frontside (usually by sinking) to refine the design created by repoussé.	
		Repoussé-stamping Embossing	Hammering thin metal sheets over (repoussé work) or into (stamping) a form, mould or pattern to make the metal sheet conform to all outlines and details. Wooden models can also serve as a “core” on which the metal sheets are attached by means of nails and rivets.	Sheet metal was used to sheath wooden objects such as the Kota reliquary figures (Gabon); in the case of the Obamba ones, the entire wooden surface is covered by copper and brass sheet, some of it worked by repoussé. Nowadays Brazzaville craftsmen specialize in repoussé- stamping of copper sheets such as those that decorate the doors of the Cathedral in Brazzaville (Congo).

	By adding material	Nailing	A decorative device as well as a joining technique.	There are two types of decorative nails in statuary and other archaeological and ethnographic objects from Central Africa = the first are the so-called brass "upholsterer" nails, imported, of European manufacture and the second (copper or brass), conical in shape (recalling

				the shape of an anvil), are of native manufacture. The conical forged nails are found on a variety of objects (sculptures, seats, arms, etc.) in several Central African traditions (Kusu, Songye, Luba, etc.). An example from another region and from archaeological context: on an ornament identified as coming from the Inland Niger delta (Mali), but out of context, and which represents a male figure with raised arms, small copper nails together with iron elements, were inlaid to enhance the brass object leading to a rich polychromy (Barbier-Mueller collection; the object may date from the 13th -15th cent. AD).
	By inlaying material	Inlaying or inserting Craft of inlaying metal into another metal substrate	A piece of worked metal is inserted into the cut recesses of another and held mechanically in place.	The Songye and Kuba (southern Democratic Congo) were especially skilled in inlaying and chasing copper into iron, on both ceremonial weapons and sculpture. The iron blades have a circular opening in which a cut-out and open-worked copper piece has been hot incorporated. Two techniques are thus used = piercing and inlaying.
		Damascening A type of decorated metal surface created through the inlaying of one or more metals into one another	Notches carved at the surface of the metal are filled with elements in motifs cut out of sheets or threads. Matting is then performed to trap the added metal element.	In Benin sculptures (Nigeria), =iron inlays (eye pupils and vertical lines on the foreheads are seen on brass sculptures.

		Marquetry (also spelled as marqueterie) is the art and craft of applying pieces to a structure (often wood or ivory) to form decorative patterns, designs or pictures	The inlays or overlays are of metal but the substrate is non-metallic : wood, stone or ivory.	As in the case of wood marquetry , the substrate is carved or incised to form a depression to receive the metal inlay or overlay. Another method of joining is to insert the metal wire or strip into holes in the substrate, a type of pressure fit join.	The two Benin ivory masks (Nigeria) often associated with the Queen mother Idia, 16th cent. AD have also iron inlays. One of the eye pupils on one of the mask still bears its iron inlay. The foreheads were inlaid with a pair of metal strips (iron strips ?)

		Inlays of other materials into metal	The inlaid material is metal but other material than metal (such as niello, wax, bitumen) fill designs cut from the metal base.		Examples of the use of niello are known from Egyptian pharaonic times but it may be the absence of investigation which explain the lack of evidences from sub-Saharan Africa. Further research is necessary!

[i] Boucher Stéphanie, 1990, Surface Working, Chiseling, Inlays, Plating, Silvering, and Gilding, in *Small Bronze Sculpture from the Ancient World*: 161-178.

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Annexe 2. Analytical Methods and results of the geochemical analyses on copper metallurgical remains and objects from the Niari Basin area.

Published in

Rademakers, F.W., Nikis, N., De Putter, T. and Degryse, P.: Copper production and trade in the Niari Basin (Republic of Congo) from the 13th-19th century CE: chemical and lead isotope characterization, *Archaeometry*

Contents

Analytical Methods

Sample preparation for LI analysis by MC-ICP-MS (Multi-Collector Inductively-Coupled-Plasma Mass Spectrometry) was carried out at the Geochemical laboratory, KU Leuven. Ore and slag fragments were ground down to a fine powder in an agate mortar, from which homogenised samples were taken. Metal samples were either taken whole (small fragments) or cut using a clean rotary saw and weighed. The obtained samples (ca. 0.1g) were digested using HNO_3 (65%) and HCl (37%), with HF (48%) added to the ore samples to break down silicate fractions. The closed beakers were placed on a hotplate (110°C) over night, and then opened to evaporate the acids to dryness. If necessary, this digestion step was repeated. The remaining residues were dissolved in 1M HNO_3 for lead isolation (De Muynck *et al.*, 2008), carried out at a class 10 clean lab at Ghent University (Belgium). Pb concentrations of the solutions were determined using a Thermo Scientific Element XR sector field ICP-MS instrument (Ghent University), to calculate dilutions for MC-ICP-MS measurements. 30 µg/l Tl (NIST SRM 997) was added as an internal standard to both standards and samples for mass bias correction calculations (see, e.g., Ketterer *et al.*, 1991; Walder *et al.*, 1993). Pb isotope ratios were determined using a Thermo Scientific Neptune MC-ICP-MS (see De Muynck *et al*. (2008) for instrumental settings). To calculate the LI ratios, a blank correction was carried out and the measured intensities for $^{202}Hg^+$ were used to correct for Hg contribution to the apparent intensity for the $^{204}Pb^+$ isotope. The raw sample ratios thus obtained were corrected for Tl-based mass discrimination following Russel's Law[2], based on replicate measurements of the NIST SRM 981 common lead standard (commonly accepted ratio values from Galer and Abouchami (1998)). Errors and error correlation were calculated after Ludwig (1980): the 2□ uncertainties were better than 0.032% for the corrected ratios to ^{204}Pb, and better than 0.014% for ratios to ^{206}Pb. Measured LI ratios (Tables 1 and 2) are plotted with their 95% confidence intervals (2□).

Bulk chemical analysis of all ore and slag and five metal samples was carried out by ICP-OES (Inductively-Coupled-Plasma Optical Emission Spectroscopy) at the Geochemical laboratory, KU Leuven, using a Varian 720-ES instrument supplied with double-pass glass cyclonic spray chamber, concentric glass Sea-Spray nebulizer and "extended high solids" torch. An ionisation buffer (1% $CsNO_3$ in 4% HNO_3), intended to eliminate ionisation effects, was added. Concentrations of 21 elements were determined from a single analysis run. Calibration solutions were prepared from certified Plasma HIQU single element solutions (CHEM-LAB, Belgium). Ultra-pure water (>18 M□/cm^3) and analytical reagent grade acids were used. A NIST GBW-7411 soil standard and MBH CRM CR32-PB110 bronze standard were digested and analysed in the same run. Detection limits were calculated for all elements by repeated measurements of independent standard solutions.

Ores, slag and three metal samples were further analysed by ICP-MS (digested samples) in the Earth Sciences Department, RMCA. After cleaning with several acids (HAc 15% then HCl-HNO_3-H_2O 3:1:6), samples were digested using HCl-HNO_3-H_2O mixture with a 3:1:1

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[2] $R_{true} = R_{observed} * (m_1/m_2)^\wedge\beta$, where m_1 and m_2 are the masses of the isotopes of interest for the ratio R and ・ is the mass bias factor (see, e.g., Belshaw *et al.*, 1998).

ratio at 60°C until complete dissolution. The solutions were analysed for major and trace elements by ICP-MS (Quad X-Series2, Thermo). The external calibrations were performed with multi-element solutions and were assessed by analysing ERM-EB386 copper CRM standard prepared with the same protocol as the samples. Overall accuracy was better than 10%, but below 15% for Mg, Al, Mn and Bi. The precision was between 1 and 4% RSD.

All metal samples have been analysed by LA-ICP-MS at the Earth Sciences Department, RMCA. A New-Wave UP-193 FX fast excimer (193nm) laser coupled with a Thermo Scientific X-Series2 quadrupole ICP-MS was used. The laser was run at 30 Hz with 150 µm spot size during 60 sec ablation time. He gas at a flow rate of 0.65 l/min was flushed into the ablation cell and was mixed after the cell with Ar carrier gas at a flow rate of 0.76 l/min. The LA-ICP-MS operating conditions were optimized to have low oxides and double-charge levels. ^{65}Cu was used as internal standard for correcting instrumental drift and ablation rate. The NIST 610, NIST 612 and ERM-EB386 copper CRM standards were used as external standards and were measured frequently during the course of the analyses. The accuracy was better than 10% and the precision was below 10% RSD.
Note that the small spot size increases sensitivity to sample heterogeneities for LA-ICP-MS. In particular, insoluble lead globules dispersed throughout the metal can cause large variations.

Chemical data are reported in Tables 5, 6 and 7. All elemental concentrations are either reported in wt% (as %) or □g/g.

Overview of sample analysed

Objects and metallurgical remains

Type	Site	Area	Code	Period	Context
Cu fragment in slag	Kindangakanzi	Boko-Songho	KNA 14-1	15th-17th century CE	Excavation - Fill in of a copper smelting furnace
Malachite	Kindangakanzi	Boko-Songho	KNA 14-3	15th-17th century CE	Excavation (SVII -7cm) - Metallurgical remains layer
Slag	Kindangakanzi	Boko-Songho	KNA 14-2	15th-17th century CE	Excavation (SVII -7cm) - Metallurgical remains layer
Ingot fragment	Mouadabambi	Boko-Songho	MDB 14-1	19th century CE	Excavation (SIII -10cm) - Metallurgical remains layer
Malachite	Mouadabambi	Boko-Songho	MDB 14-3	19th century CE	Excavation (SIII -10cm) - Metallurgical remains layer
Slag (1)	Mouadabambi	Boko-Songho	MDB 14-2	19th century CE	Excavation (SIII -10cm) - Metallurgical remains layer
Slag (2)	Mouadabambi	Boko-Songho	MDB 14-2	19th century CE	Excavation (SIII -10cm) - Metallurgical remains layer
Barrette	Misenga	Mindouli	MSG70628-5	13th-14th century CE	M. Bequaert excavation, 1951
Ingot	Misenga	Mindouli	MSG70628-2	13th-14th century CE	M. Bequaert excavation, 1951
Bracelet	Misenga	Mindouli	MSG70620	13th-14th century CE	M. Bequaert excavation, 1951
Bar fragment	Kisaba	Mindouli	KIS 14-1	13th-14th century CE	Excavation (SIV -20cm) - Metallurgical remains layer
Malachite	Kisaba	Mindouli	KIS 14-4 (*)	13th-14th century CE	Excavation (SIV -20cm) - Metallurgical remains layer
Slag (aggregate)	Kisaba	Mindouli	KIS 14-2	13th-14th century CE	Excavation (SIV -10cm) - Metallurgical remains layer
Slag (1)	Kisaba	Mindouli	KIS 14-3	13th-14th century CE	Excavation (SIV -20cm) - Metallurgical remains layer
Slag (2)	Kisaba	Mindouli	KIS 14-3	13th-14th century CE	Excavation (SIV -20cm) - Metallurgical remains layer
Bar fragment	Makuti	Mindouli	MKU3 14-1	13th-14th century CE	Excavation (SVI -5cm) - Metallurgical remains layer
Barrette	Makuti	Mindouli	MKU 13-1	13th-14th century CE	Surface collection
Mineral refuse	Makuti	Mindouli	MKU3 14-2	13th-14th century CE	Excavation (SVI -25cm) - Metallurgical remains layer
Tuyere	Makuti	Mindouli	MKU3 14-3	13th-14th century CE	Excavation (SVI -25cm) - Metallurgical remains layer
Barrette	Nkabi	Mindouli	GPSNN 294-1	13th-14th century CE	Surface collection
Ingot	Nkabi	Mindouli	GPSNN 294-2	13th-14th century CE	Surface collection
Cu prill	Kitchounga	Mindouli	KTG 14-3	19th century CE	Excavation (SI -30cm) - Metallurgical remains layer
Cu (+ Slag?)	Kitchounga	Mindouli	KTG 14-1	19th century CE	Excavation (SI -30cm) - Metallurgical remains layer
Slag	Kitchounga	Mindouli	KTG 14-2	19th century CE	Excavation (SI -30cm) - Metallurgical remains layer
Copper extracted from slag	Ntominsie	Mindouli	NTM15-1	16th-18th century CE	Excavation (SI) - Metallurgical remains layer
Slag	Ntominsie	Mindouli	NTM15-1	16th-18th century CE	Excavation (SI) - Metallurgical remains layer
Lead slag	Mfouati "Songa-Melka"	Mfouati	GPSNN-290-1	19th-20th century CE	Surface collection
Ingot fragment	Ubangi	Ubangi	RGM583-1	19th century CE	Collected by Coquilhat in late 19th c. in Mbandaka region
Ingot fragment	Ubangi	Ubangi	RGM583-2	19th century CE	Collected by Coquilhat in late 19th c. in Mbandaka region

Geological samples

Type	Site	Area	Code	Description
Malachite	Grande Mine	Boko-Songho	GPSNN 271	Laminar/botryoidal malachite coating in iron-rich laterite host-rock
Malachite	Grande Mine	Boko-Songho	GPSNN 271-3	Laminar/botryoidal malachite coating in iron-rich laterite host-rock
Malachite	Malembe	Boko-Songho	GPSNN272	Botroyoidal malachite + azurite
Malachite	Malembe	Boko-Songho	Malembe-1	Botroyoidal malachite in weathered argillaceous host-rock
Malachite	Djenguele	Boko-Songho	Djen Geol	Malachite and galena in highly weathered host-rock
Malachite	Mpassa Mine	Mindouli	Mpassa-1	Malachite and azurite in highly weathered porous host-rock
Malachite	Mpassa Mine	Mindouli	Mpassa-2	Malachite and azurite in highly weathered porous host-rock
Malachite	Mpassa Mine	Mindouli	MPA-3	Malachite and azurite in highly weathered porous host-rock
Malachite	Ntola	Mindouli	Ntola	Laminar/botryoidal malachite coating in weathered host-rock
Malachite	Ntola	Mindouli	NTO-3	Laminar/botryoidal malachite coating in weathered host-rock
Malachite	Gotala	Mindouli	GPSNN 259	Thin malachite coating in highly weathered host-rock
Malachite	Gotala	Mindouli	GPSNN 259-6	Malachite coating and later cuprite, dioptase
Malachite	Moutele "faille"	Mindouli	GPSNN 263	Malachite coating (+ chrysocolla, dioptase) in weathered argillaceous host-rock
Malachite	Moutele	Mindouli	MOU-2	Botryoidal malachite in weathered porous host-rock
Malachite	Travers-Banc	Mindouli	TRB-01	Malachite and chrysocolla with quartz vein infilling
Malachite	Mfouati	Mfouati	GPSNN 284	Thin malachite coating in highly weathered, porous limestone host-rock
Malachite	Malouende	Mfouati	MAL-1	Laminar/botryoidal malachite in weathered argillaceous host-rock
Malachite	Mfouati/Hapilo	Mfouati	GPSNN 289	Malachite coating in silicified skeletal host-rock
Galena	Djenguele	Boko-Songho	BSD-01	Galena spots in weathered host-rock
Galena	Ntola	Mindouli	NTO-02	Galena cement in brecciated host-rock
Galena	Mpassa	Mindouli	MPA-01	Galena spots within massive sulfides
Galena	Hapilo	Mfouati	HAP-01	Galena spots within massive sulfides

Lead isotope data

Tables

Table 1: LI ratios for geological samples

Lab code	Type	Site	Area	Lab no		$^{206}Pb/^{204}Pb$	$^{207}Pb/^{204}Pb$	$^{208}Pb/^{204}Pb$	$^{207}Pb/^{206}Pb$	$^{208}Pb/^{206}Pb$	$^{208}Pb/^{207}Pb$
GPSNN 271	Malachite	Grande Mine	Boko-Songho	M12	Average	**18.866**	**15.703**	**38.947**	**0.832**	**2.064**	**2.480**
					☐	*0.005*	*0.005*	*0.013*	*0.000*	*0.000*	*0.000*
GPSNN 271-3	Malachite	Grande Mine	Boko-Songho	FA-40	Average	**18.223**	**15.674**	**38.192**	**0.860**	**2.096**	**2.437**
					☐	*0.002*	*0.002*	*0.005*	*0.000*	*0.000*	*0.000*
GPSNN 272	Malachite	Malembe	Boko-Songho	M22	Average	**18.110**	**15.667**	**38.138**	**0.865**	**2.106**	**2.434**
					☐	*0.003*	*0.003*	*0.008*	*0.000*	*0.000*	*0.000*
Malembe-1	Malachite	Malembe	Boko-Songho	FA-39	Average	**18.113**	**15.670**	**38.158**	**0.865**	**2.107**	**2.435**
					☐	*0.002*	*0.003*	*0.007*	*0.000*	*0.000*	*0.000*
Djen Geol	Malachite	Djenguele	Boko-Songho	M14	Average	**17.993**	**15.663**	**38.129**	**0.870**	**2.119**	**2.434**
					☐	*0.004*	*0.003*	*0.009*	*0.000*	*0.000*	*0.000*
BSD-01	Galena	Djenguele	Boko-Songho	M7	Average	**18.001**	**15.660**	**38.087**	**0.870**	**2.116**	**2.432**
					☐	*0.005*	*0.005*	*0.013*	*0.000*	*0.000*	*0.000*
Mpassa-1	Malachite	Mpassa Mine	Mindouli	M15	Average	**17.886**	**15.655**	**38.003**	**0.875**	**2.125**	**2.428**
					☐	*0.005*	*0.005*	*0.013*	*0.000*	*0.000*	*0.000*
Mpassa-2	Malachite	Mpassa Mine	Mindouli	M16	Average	**17.888**	**15.656**	**38.005**	**0.875**	**2.125**	**2.428**
					☐	*0.004*	*0.003*	*0.009*	*0.000*	*0.000*	*0.000*
MPA-3	Malachite	Mpassa Mine	Mindouli	FA-43	Average	**17.896**	**15.657**	**38.000**	**0.875**	**2.123**	**2.427**
					☐	*0.004*	*0.003*	*0.010*	*0.000*	*0.000*	*0.000*
Ntola	Malachite	Ntola	Mindouli	M17	Average	**17.872**	**15.653**	**38.015**	**0.876**	**2.127**	**2.429**
					☐	*0.003*	*0.003*	*0.008*	*0.000*	*0.000*	*0.000*
NTO-3	Malachite	Ntola	Mindouli	FA-41	Average	**17.876**	**15.655**	**38.022**	**0.876**	**2.127**	**2.429**
					☐	*0.002*	*0.002*	*0.005*	*0.000*	*0.000*	*0.000*

Lab code	***Type***	***Site***	***Area***	***Lab no***		***^{206}Pb/^{204}Pb***	***^{207}Pb/^{204}Pb***	***^{208}Pb/^{204}Pb***	***^{207}Pb/^{206}Pb***	***^{208}Pb/^{206}Pb***	***^{208}Pb/^{207}Pb***
GPSNN 259	Malachite	Lagotala	Mindouli	M20	Average	**17.919**	**15.666**	**38.107**	**0.874**	**2.127**	**2.432**
					□	*0.005*	*0.004*	*0.012*	*0.000*	*0.000*	*0.000*
GPSNN 259-6	Malachite	Lagotala	Mindouli	FA-44	Average	**17.942**	**15.664**	**38.103**	**0.873**	**2.124**	**2.432**
					□	*0.002*	*0.003*	*0.007*	*0.000*	*0.000*	*0.000*
GPSNN 263	Malachite	Moutele "faille"	Mindouli	M21	Average	**17.863**	**15.645**	**37.991**	**0.876**	**2.127**	**2.428**
					□	*0.004*	*0.004*	*0.011*	*0.000*	*0.000*	*0.000*
MOU-2	Malachite	Moutele	Mindouli	FA-42	Average	**17.985**	**15.661**	**38.136**	**0.871**	**2.120**	**2.435**
					□	*0.007*	*0.006*	*0.014*	*0.000*	*0.000*	*0.000*
TRB-01	Malachite	Travers-Banc	Mindouli	FA-45	Average	**17.925**	**15.670**	**38.090**	**0.874**	**2.125**	**2.431**
					□	*0.002*	*0.002*	*0.006*	*0.000*	*0.000*	*0.000*
NTO-02	Galena	Ntola	Mindouli	M6	Average	**17.903**	**15.665**	**38.087**	**0.875**	**2.127**	**2.431**
					□	*0.007*	*0.006*	*0.016*	*0.000*	*0.000*	*0.000*
MPA-01	Galena	Mpassa	Mindouli	M8	Average	**17.884**	**15.650**	**37.991**	**0.875**	**2.124**	**2.427**
					□	*0.007*	*0.007*	*0.017*	*0.000*	*0.000*	*0.000*
GPSNN 284	Malachite	Mfouati	Mfouati	M23	Average	**18.392**	**15.688**	**38.060**	**0.853**	**2.069**	**2.426**
					□	*0.004*	*0.004*	*0.010*	*0.000*	*0.000*	*0.000*
MAL-1	Malachite	Malouende	Mfouati	FA-38	Average	**18.609**	**15.701**	**38.059**	**0.844**	**2.045**	**2.424**
					□	*0.003*	*0.002*	*0.006*	*0.000*	*0.000*	*0.000*
GPSNN 289	Malachite	Mfouati/Hapilo	Mfouati	M24	Average	**17.856**	**15.651**	**37.987**	**0.877**	**2.127**	**2.427**
					□	*0.008*	*0.008*	*0.018*	*0.000*	*0.000*	*0.000*
HAP-01	Galena	Hapilo	Mfouati	FA-47	Average	**17.834**	**15.650**	**37.978**	**0.877**	**2.129**	**2.427**
					□	*0.004*	*0.004*	*0.010*	*0.000*	*0.000*	*0.000*

Table 2: LI ratios for archaeological samples

Lab code	*Type*	*Site*	*Area*	*Lab no*		$^{206}Pb/^{204}Pb$	$^{207}Pb/^{204}Pb$	$^{208}Pb/^{204}Pb$	$^{207}Pb/^{206}Pb$	$^{208}Pb/^{206}Pb$	$^{208}Pb/^{207}Pb$
KNA14-1	Cu fragment in slag	Kindangakanzi	Boko-Songho	FA--50	Average	**17.983**	**15.662**	**38.112**	**0.871**	**2.119**	**2.433**
					□	*0.004*	*0.004*	*0.010*	*0.000*	*0.000*	*0.000*
KNA 14-3	Malachite	Kindangakanzi	Boko-Songho	FA-37	Average	**18.022**	**15.663**	**38.110**	**0.869**	**2.115**	**2.433**
					□	*0.003*	*0.002*	*0.007*	*0.000*	*0.000*	*0.000*
KNA 14-2	Slag	Kindangakanzi	Boko-Songho	FJ6	Average	**17.996**	**15.662**	**38.113**	**0.870**	**2.118**	**2.433**
					□	*0.006*	*0.005*	*0.014*	*0.000*	*0.000*	*0.000*
MDB14-1	Ingot fragment	Mouadabambi	Boko-Songho	M3	Average	**17.860**	**15.654**	**38.001**	**0.876**	**2.128**	**2.428**
					□	*0.004*	*0.004*	*0.010*	*0.000*	*0.000*	*0.000*
MDB 14-3	Malachite	Mouadabambi	Boko-Songho	FA-36	Average	**17.947**	**15.658**	**38.084**	**0.872**	**2.122**	**2.432**
					□	*0.003*	*0.002*	*0.006*	*0.000*	*0.000*	*0.000*
MDB 14-2	Slag (1)	Mouadabambi	Boko-Songho	FA-48	Average	**17.852**	**15.651**	**37.989**	**0.877**	**2.128**	**2.427**
					□	*0.003*	*0.003*	*0.007*	*0.000*	*0.000*	*0.000*
MDB 14-2	Slag (2)	Mouadabambi	Boko-Songho	FA-59	Average	**17.851**	**15.650**	**37.983**	**0.877**	**2.128**	**2.427**
					□	*0.002*	*0.002*	*0.006*	*0.000*	*0.000*	*0.000*
MSG70628-5	Barrette	Misenga	Mindouli	FA-29	Average	**17.886**	**15.660**	**38.044**	**0.876**	**2.127**	**2.429**
					□	*0.003*	*0.003*	*0.009*	*0.000*	*0.000*	*0.000*
MSG70628-2	Ingot	Misenga	Mindouli	FA-30	Average	**17.976**	**15.674**	**38.118**	**0.872**	**2.120**	**2.432**
					□	*0.003*	*0.003*	*0.009*	*0.000*	*0.000*	*0.000*
MSG70620	Bracelet	Misenga	Mindouli	FA-31	Average	**18.263**	**15.659**	**38.338**	**0.857**	**2.099**	**2.448**
					□	*0.003*	*0.003*	*0.008*	*0.000*	*0.000*	*0.000*
KIS14-1	Bar fragment	Kisaba	Mindouli	M4	Average	**17.951**	**15.675**	**38.135**	**0.873**	**2.124**	**2.433**
					□	*0.004*	*0.003*	*0.009*	*0.000*	*0.000*	*0.000*
KIS 14-2	Slag (aggregate)	Kisaba	Mindouli	FJ5	Average	**17.953**	**15.665**	**38.094**	**0.873**	**2.122**	**2.432**
					□	*0.002*	*0.002*	*0.007*	*0.000*	*0.000*	*0.000*

Lab code	***Type***	***Site***	***Area***	***Lab no***		***^{206}Pb/^{204}Pb***	***^{207}Pb/^{204}Pb***	***^{208}Pb/^{204}Pb***	***^{207}Pb/^{206}Pb***	***^{208}Pb/^{206}Pb***	***^{208}Pb/^{207}Pb***
KIS 14-3	Slag (1)	Kisaba	Mindouli	FA-49	Average	**17.985**	**15.672**	**38.108**	**0.871**	**2.119**	**2.432**
					□	*0.003*	*0.003*	*0.009*	*0.000*	*0.000*	*0.000*
KIS 14-3	Slag (2)	Kisaba	Mindouli	FA-60	Average	**17.995**	**15.667**	**38.093**	**0.871**	**2.117**	**2.431**
					□	*0.002*	*0.003*	*0.007*	*0.000*	*0.000*	*0.000*
MKU-314-1	Bar fragment	Makuti	Mindouli	M5	Average	**17.949**	**15.673**	**38.134**	**0.873**	**2.125**	**2.433**
					□	*0.005*	*0.004*	*0.011*	*0.000*	*0.000*	*0.000*
MKU 13-1	Barrette	Makuti	Mindouli	FA-28	Average	**17.928**	**15.676**	**38.137**	**0.874**	**2.127**	**2.433**
					□	*0.002*	*0.002*	*0.008*	*0.000*	*0.000*	*0.000*
MKU-314-2	Mineral refuse	Makuti	Mindouli	M13	Average	**18.061**	**15.669**	**38.097**	**0.868**	**2.109**	**2.431**
					□	*0.004*	*0.004*	*0.011*	*0.000*	*0.000*	*0.000*
MKU 314-3	Tuyère	Makuti	Mindouli	FJ1	Average	**18.016**	**15.668**	**38.118**	**0.870**	**2.116**	**2.433**
					□	*0.002*	*0.002*	*0.008*	*0.000*	*0.000*	*0.000*
GPSNN 294-1	Barrette	Nkabi	Mindouli	FA-32	Average	**17.900**	**15.663**	**38.087**	**0.875**	**2.128**	**2.432**
					□	*0.003*	*0.003*	*0.008*	*0.000*	*0.000*	*0.000*
GPSNN 294-2	Ingot	Nkabi	Mindouli	FA-33	Average	**17.995**	**15.679**	**38.137**	**0.871**	**2.119**	**2.432**
					□	*0.002*	*0.002*	*0.007*	*0.000*	*0.000*	*0.000*
KTG 14-3	Cu prill	Kitchounga	Mindouli	FA-27	Average	**17.908**	**15.670**	**38.113**	**0.875**	**2.128**	**2.432**
					□	*0.003*	*0.003*	*0.008*	*0.000*	*0.000*	*0.000*
KTG 14-1	Cu (+ Slag?)	Kitchounga	Mindouli	FD27	Average	**17.913**	**15.665**	**38.087**	**0.874**	**2.126**	**2.431**
					□	*0.004*	*0.004*	*0.011*	*0.000*	*0.000*	*0.000*
KTG 14-2	Slag	Kitchounga	Mindouli	FJ2	Average	**17.933**	**15.667**	**38.092**	**0.874**	**2.124**	**2.431**
					□	*0.002*	*0.002*	*0.007*	*0.000*	*0.000*	*0.000*
NTM15-1	Copper extracted from slag	Ntominsier	Mindouli	FA-35	Average	**17.978**	**15.670**	**38.093**	**0.872**	**2.119**	**2.431**
					□	*0.002*	*0.003*	*0.007*	*0.000*	*0.000*	*0.000*
NTM15-1	Slag	Ntominsier	Mindouli	FA-34	Average	**17.972**	**15.666**	**38.083**	**0.872**	**2.119**	**2.431**
					□	*0.003*	*0.004*	*0.011*	*0.000*	*0.000*	*0.000*

Lab code	***Type***	***Site***	***Area***	***Lab no***		***^{206}Pb/^{204}Pb***	***^{207}Pb/^{204}Pb***	***^{208}Pb/^{204}Pb***	***^{207}Pb/^{206}Pb***	***^{208}Pb/^{206}Pb***	***^{208}Pb/^{207}Pb***
GPSNN-290-1	Lead slag	Mfouati "Songa-Melka"	Mfouati	M11	Average	**17.845**	**15.649**	**37.981**	**0.877**	**2.128**	**2.427**
					□	*0.005*	*0.004*	*0.012*	*0.000*	*0.000*	*0.000*
RGM583-1	Ingot fragment	Ubangi	Ubangi	M18	Average	**17.854**	**15.653**	**38.002**	**0.877**	**2.128**	**2.428**
					□	*0.003*	*0.003*	*0.008*	*0.000*	*0.000*	*0.000*
RGM583-2	Ingot fragment	Ubangi	Ubangi	M19	Average	**17.863**	**15.653**	**38.007**	**0.876**	**2.128**	**2.428**
					□	*0.004*	*0.003*	*0.009*	*0.000*	*0.000*	*0.000*

Plots

The first plots (Figures 3-6) show comparisons to published LI ratios for Central African copper ores: data from Haest *et al*., 2009, 2010; Kamona *et al*., 1999; Key *et al*., 2001; Molofsky *et al*., 2014; Richards *et al*., 1988; Walraven and Chabu, 1994. Ore data published by Willet and Sayre (2006) is omitted here, as their ore samples from Morocco, Tunisia, Algeria and Namibia are too few (and sometimes from unclear geological context) to be representative of deposits there.

Figures 7-10 present plots at different scales of the Mindouli metals and slags.

Figures 11-14 present plots at different scales of the Boko Songho, Ubangi and Mfouati metals and slags.

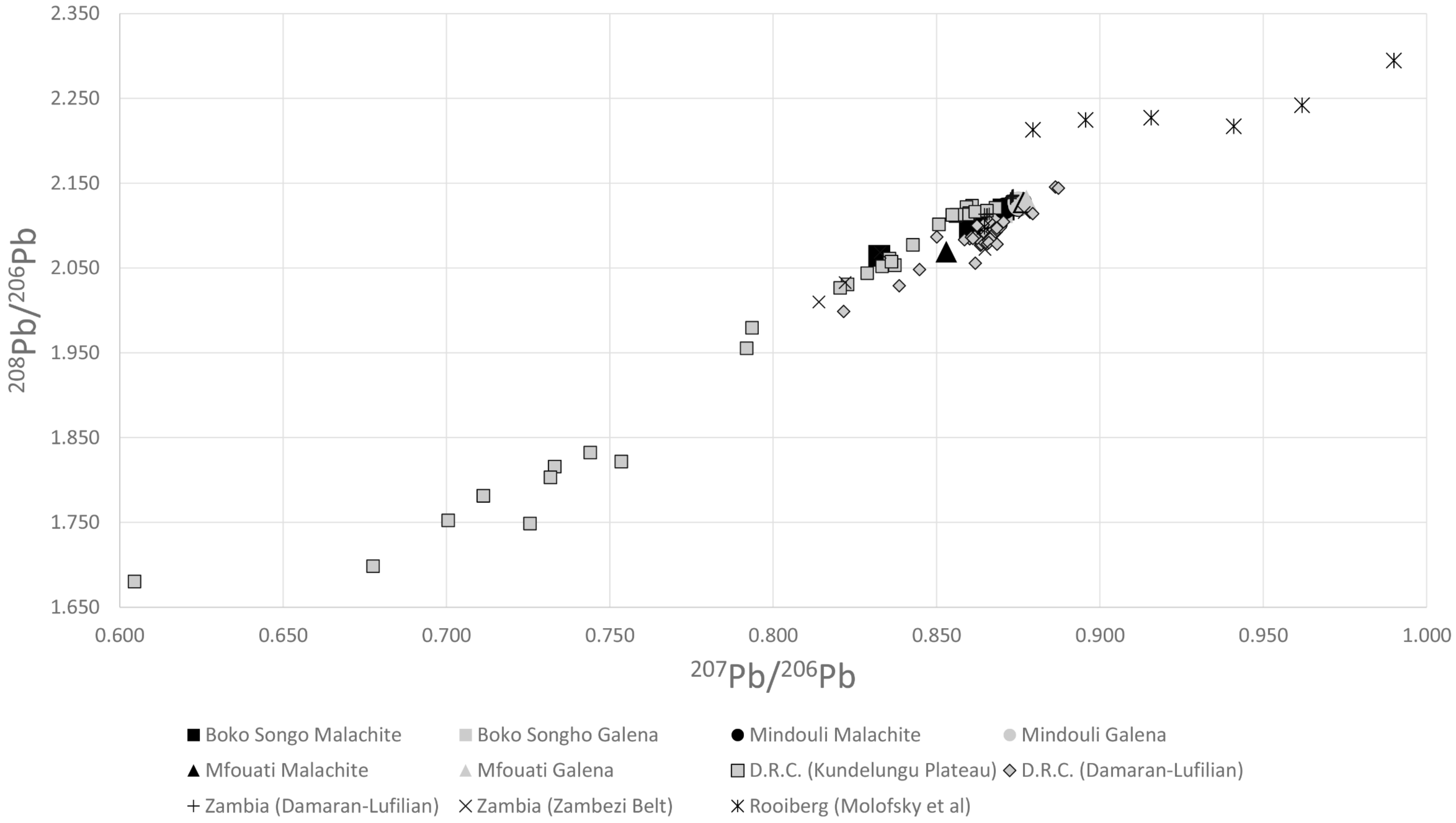

Figure 1: LI ratios for Niari Basin ores compared to available regional copper ore data (1)

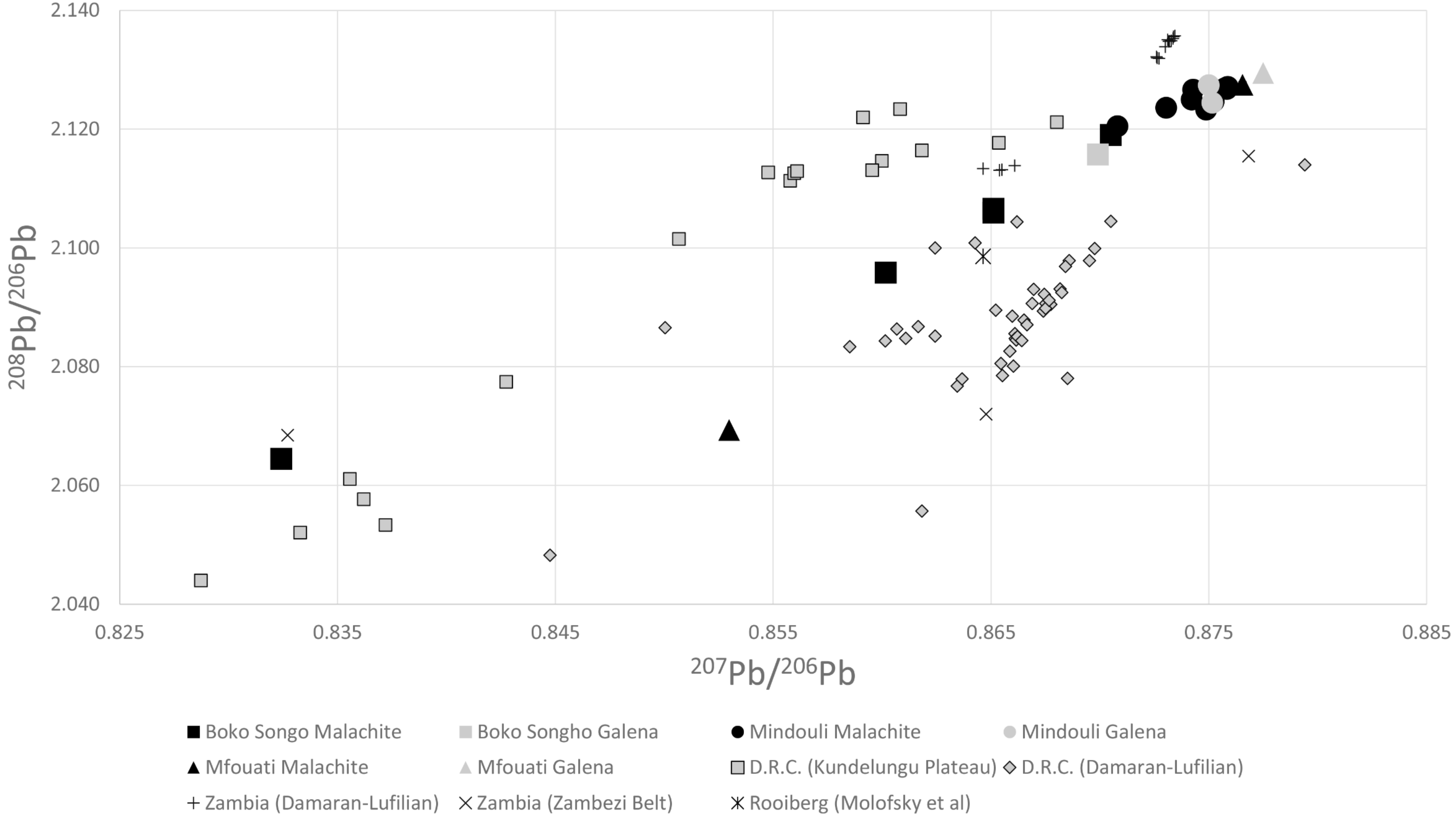

Figure 2: (Close-up of Figure 3) LI ratios for Niari Basin ores compared to available regional copper ore data (2)

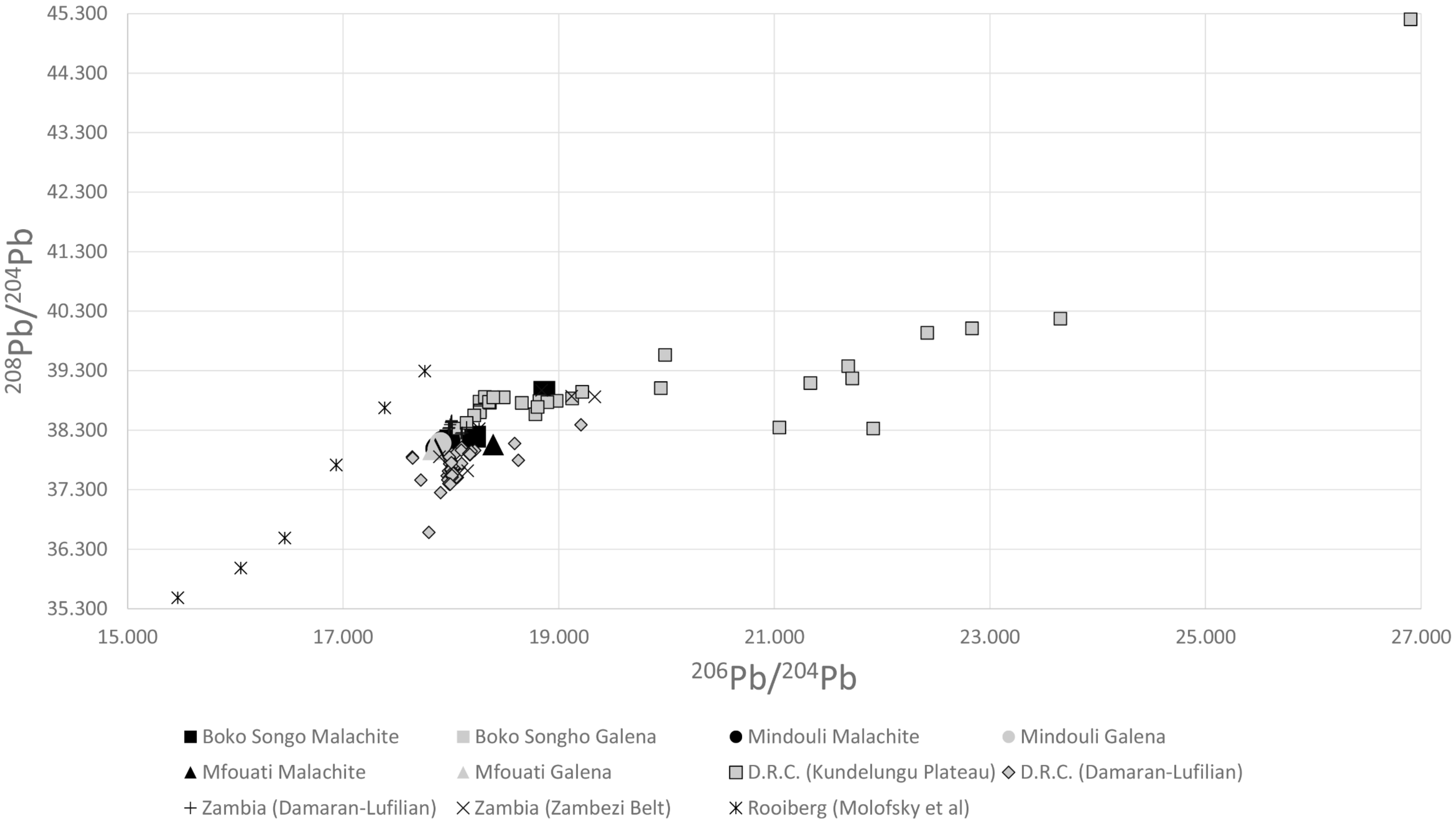

Figure 3: LI ratios for Niari Basin ores compared to available regional copper ore data (3)

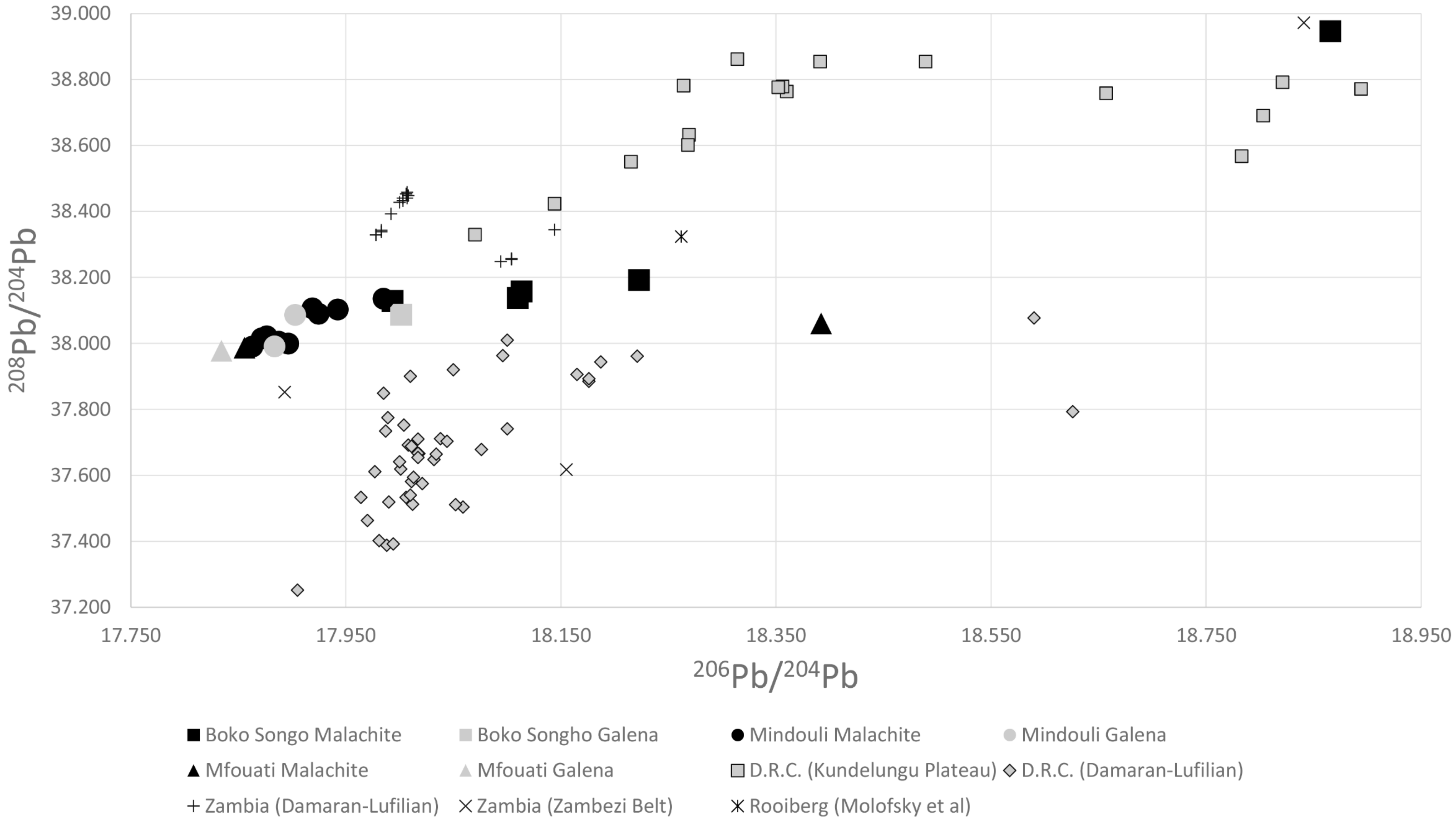

Figure 4: (Close-up of Figure 5) LI ratios for Niari Basin ores compared to available regional copper ore data (4)

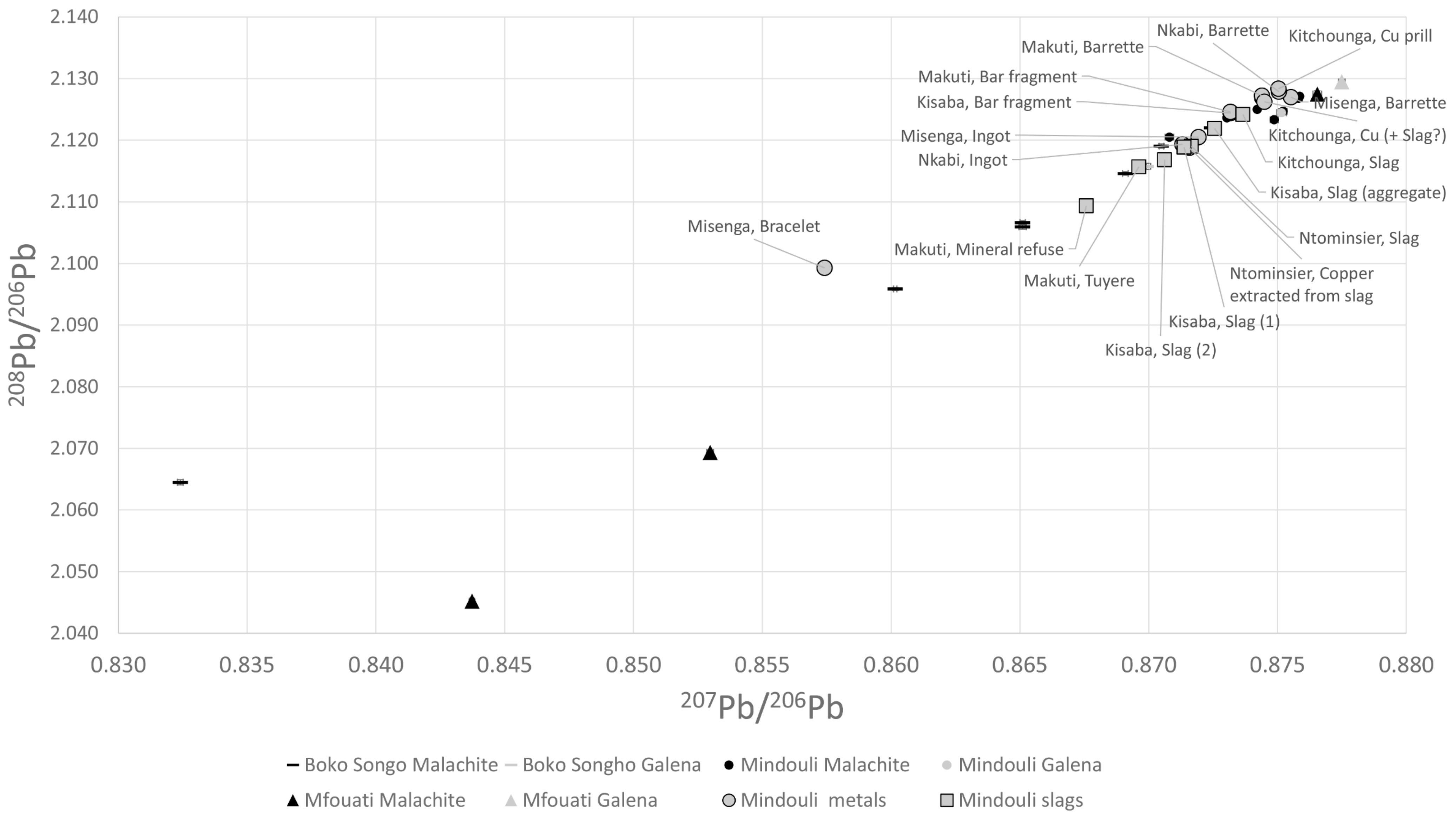

Figure 5: LI ratios for Mindouli metals and slags (1)

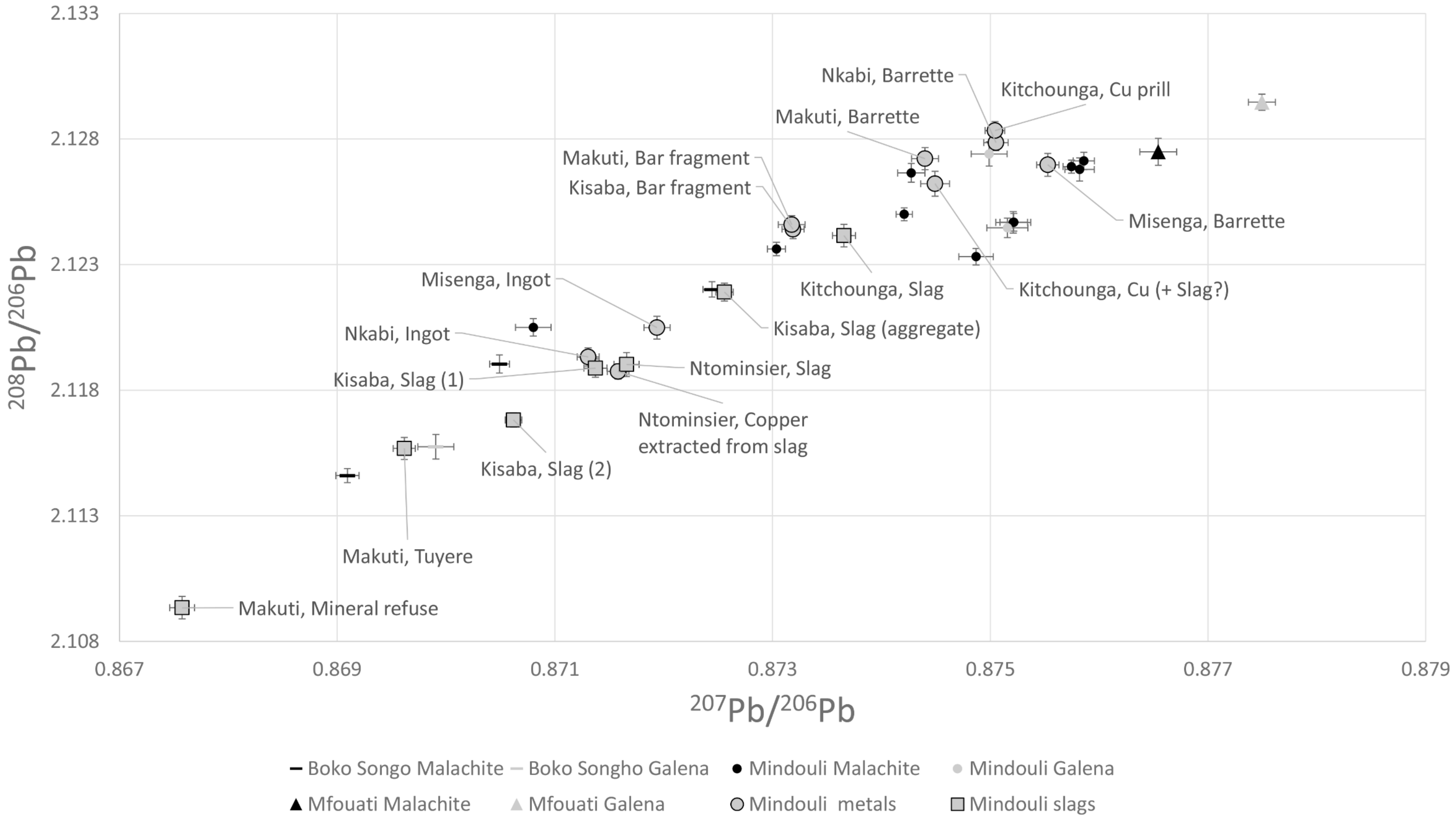

Figure 6: (Close-up of Figure 8) LI ratios for Mindouli metals and slags (2)

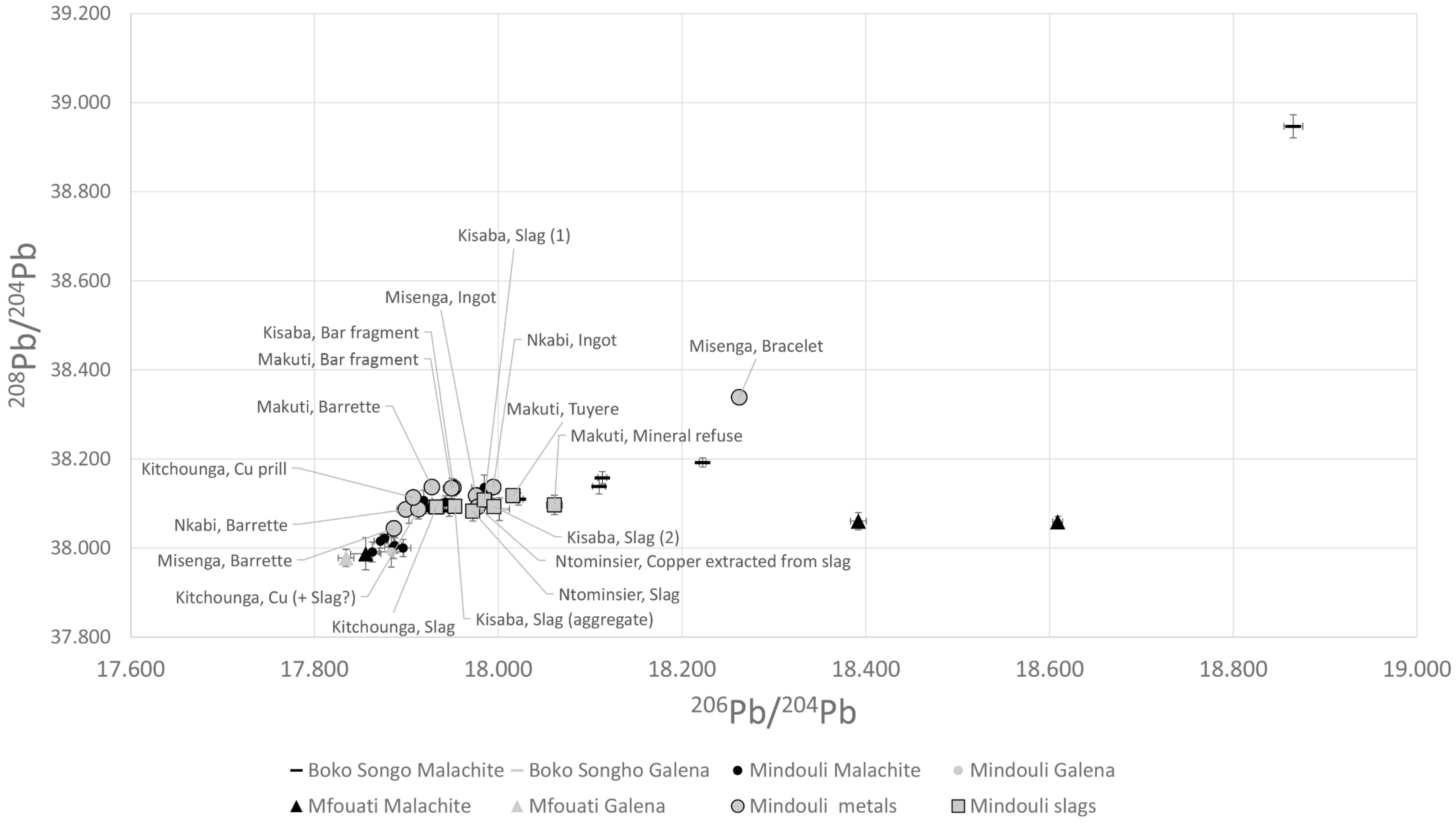

Figure 7: LI ratios for Mindouli metals and slags (3)

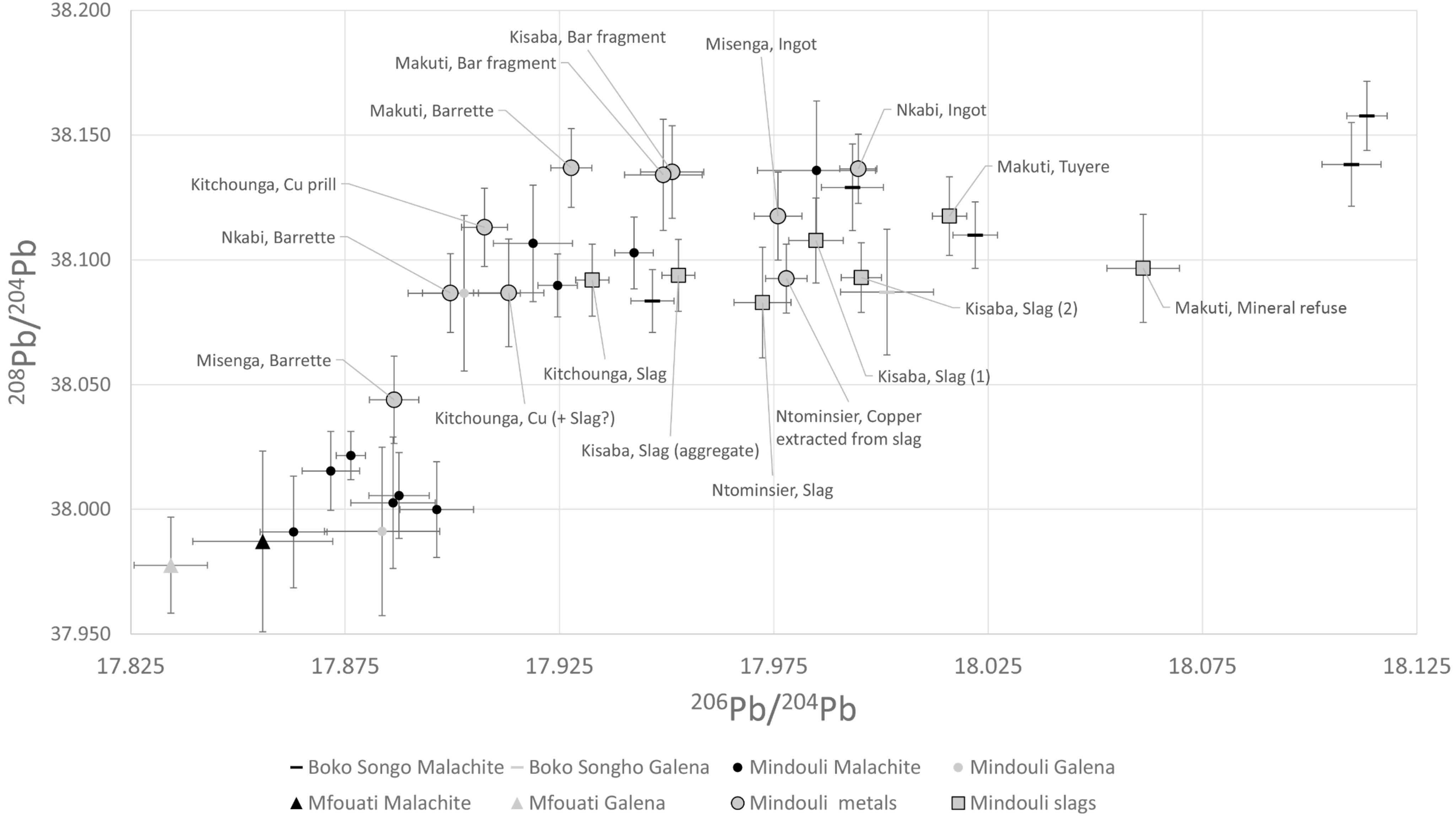

Figure 8: (Close-up of Figure 9) LI ratios for Mindouli metals and slags (4)

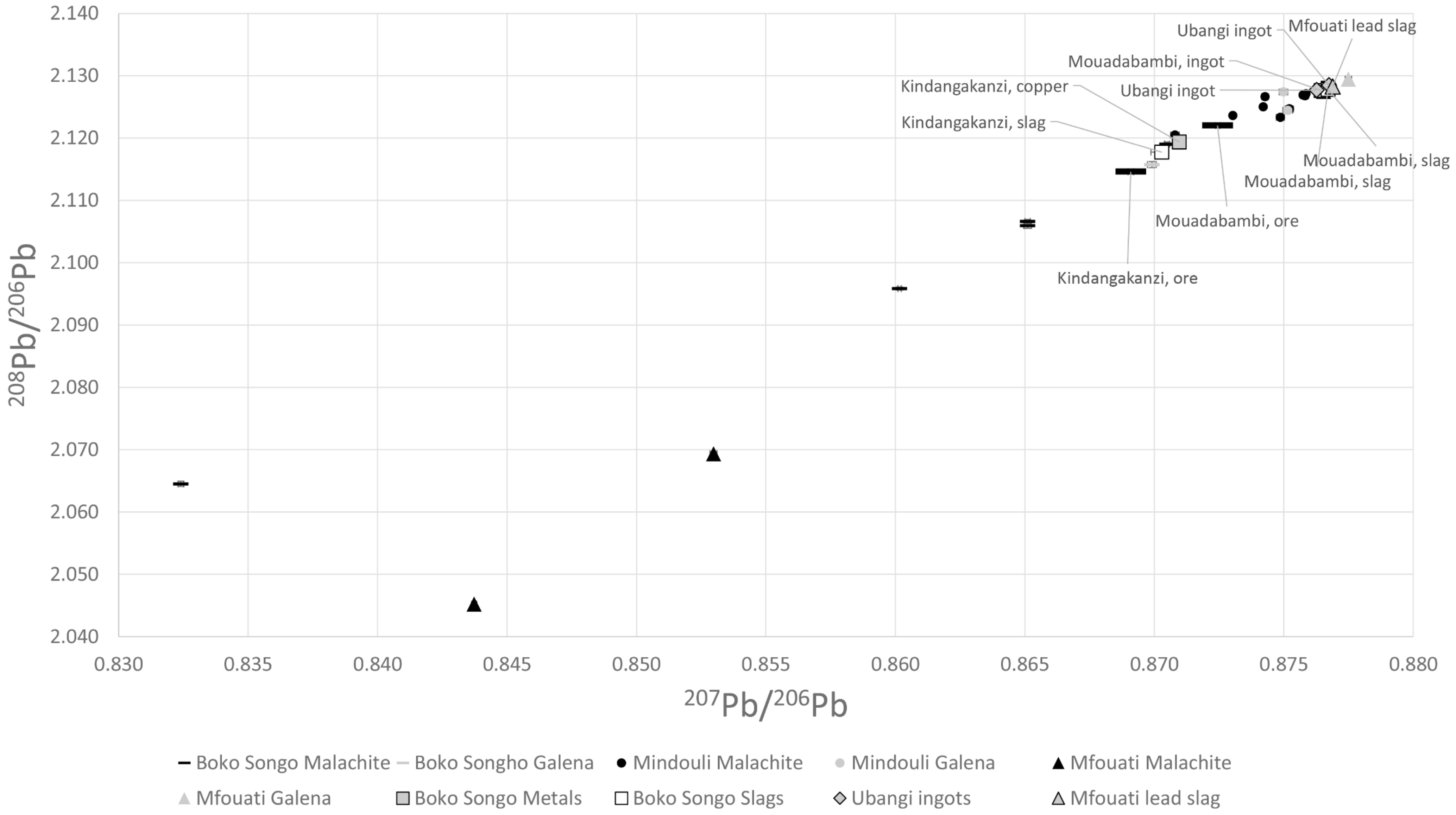

Figure 9: LI ratios for Boko Songho, Ubangi and Mfouati metals and slags (1)

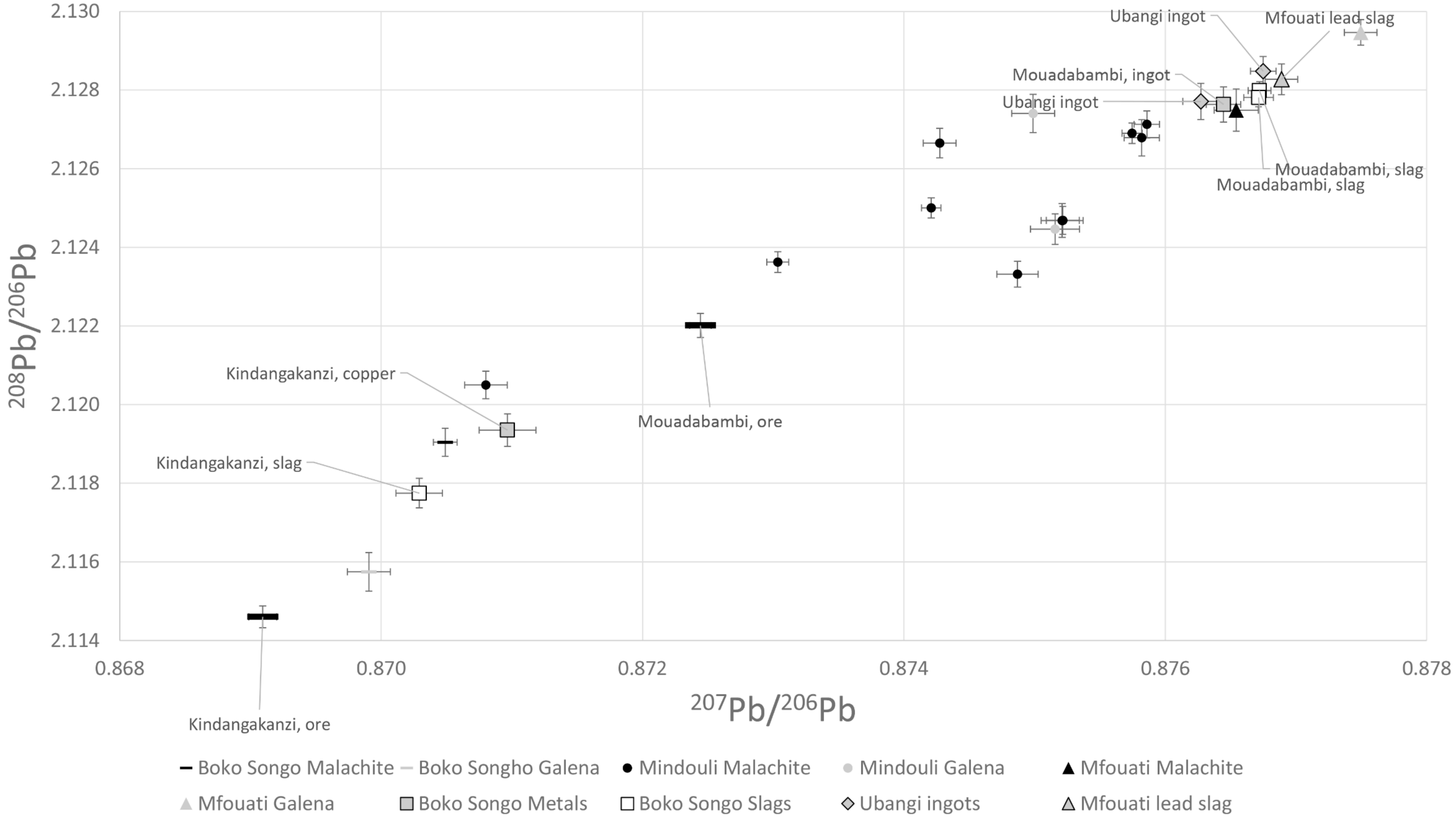

Figure 10: (Close-up of Figure 11) LI ratios for Boko Songho, Ubangi and Mfouati metals and slags (2)

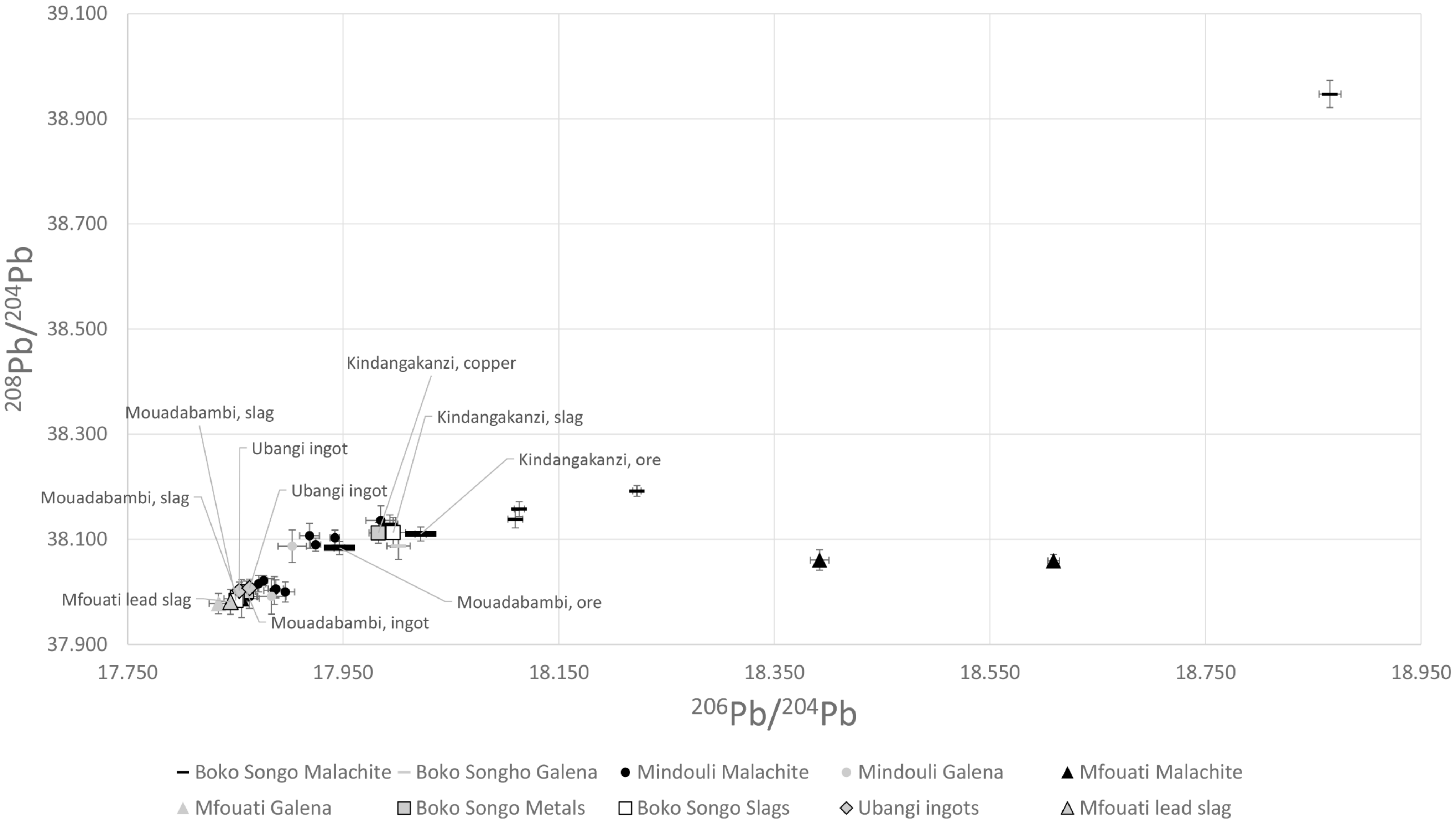

Figure 11: LI ratios for Boko Songho, Ubangi and Mfouati metals and slags (3)

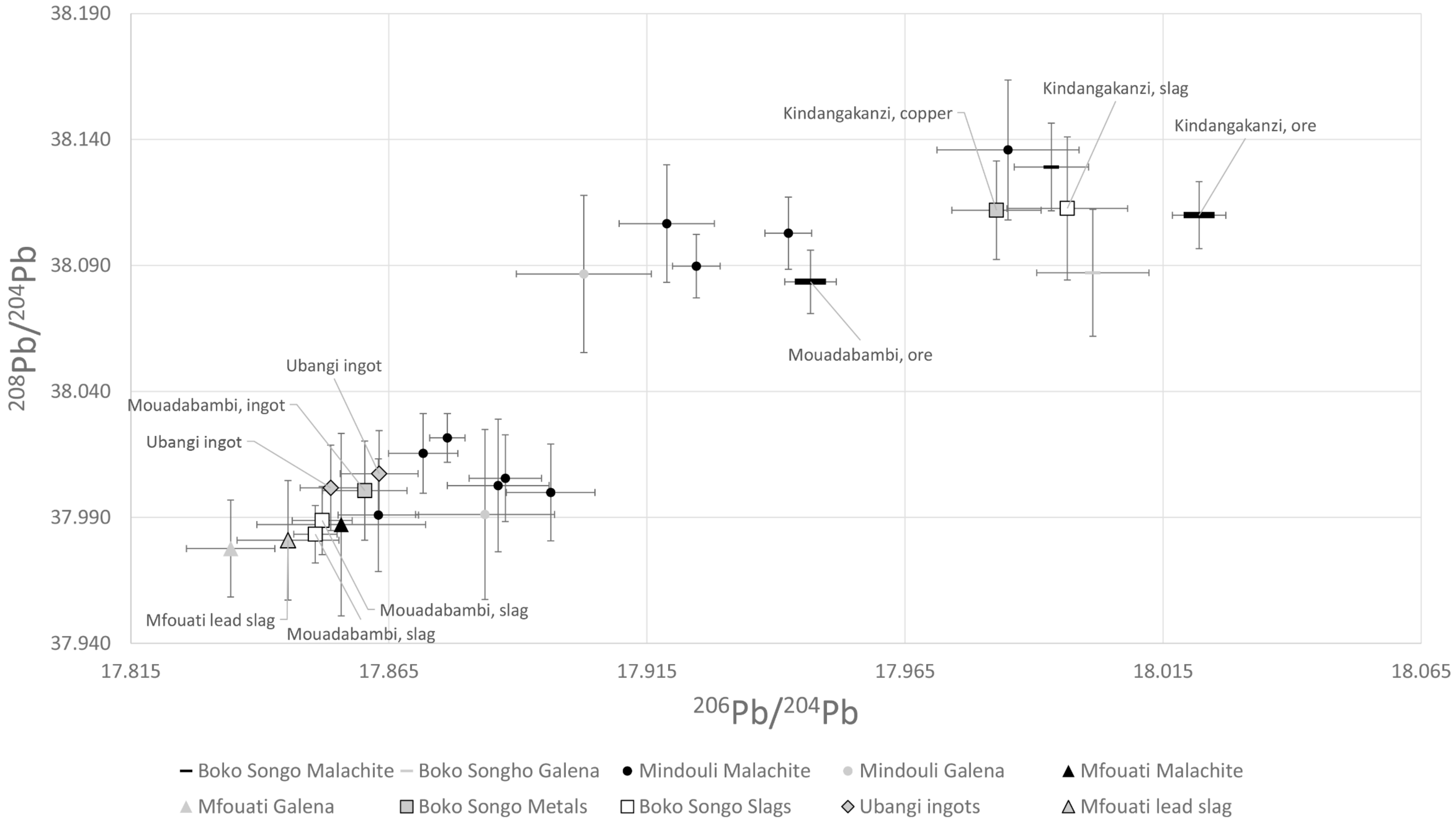

Figure 12: (Close-up of Figure 13) LI ratios for Boko Songho, Ubangi and Mfouati metals and slags (4)

Age Calculations

Calculated using Albarède *et al*. (2012) toolbox.

Table 3: LI ratios and calculated ages for geological samples

Lab code	***Type***	***Area***	***Lab no***		***^{206}Pb/^{204}Pb***	***^{207}Pb/^{204}Pb***	***^{208}Pb/^{204}Pb***	***^{207}Pb/^{206}Pb***	***^{208}Pb/^{206}Pb***	***^{208}Pb/^{207}Pb***	***T (Ma)***	***□ (U/Pb)***	***□ (Th/U)***	***f=0***
GPSNN 271	Malachite	Boko-Songho	M12	Average	**18.866**	**15.703**	**38.947**	**0.832**	**2.064**	**2.480**	**60**	**9.92**	**3.91**	**3.92E-08**
				□	*0.005*	*0.005*	*0.013*	*0.000*	*0.000*	*0.000*				
GPSNN 271-3	Malachite	Boko-Songho	FA-40	Average	**18.223**	**15.674**	**38.192**	**0.860**	**2.096**	**2.437**	**476**	**9.95**	**3.93**	**-1E-06**
				□	*0.002*	*0.002*	*0.005*	*0.000*	*0.000*	*0.000*				
GPSNN 272	Malachite	Boko-Songho	M22	Average	**18.110**	**15.667**	**38.138**	**0.865**	**2.106**	**2.434**	**544**	**9.96**	**3.97**	**-2.1E-08**
				□	*0.003*	*0.003*	*0.008*	*0.000*	*0.000*	*0.000*				
Malembe-1	Malachite	Boko-Songho	FA-39	Average	**18.113**	**15.670**	**38.158**	**0.865**	**2.107**	**2.435**	**546**	**9.96**	**3.98**	**-1.2E-07**
				□	*0.002*	*0.003*	*0.007*	*0.000*	*0.000*	*0.000*				
Djen Geol	Malachite	Boko-Songho	M14	Average	**17.993**	**15.663**	**38.129**	**0.870**	**2.119**	**2.434**	**621**	**9.97**	**4.05**	**-4.2E-06**
				□	*0.004*	*0.003*	*0.009*	*0.000*	*0.000*	*0.000*				
BSD-01	Galena	Boko-Songho	M7	Average	**18.001**	**15.660**	**38.087**	**0.870**	**2.116**	**2.432**	**610**	**9.96**	**4.02**	**4.06E-06**
				□	*0.005*	*0.005*	*0.013*	*0.000*	*0.000*	*0.000*				
Mpassa-1	Malachite	Mindouli	M15	Average	**17.886**	**15.655**	**38.003**	**0.875**	**2.125**	**2.428**	**684**	**9.98**	**4.05**	**-5.8E-07**
				□	*0.005*	*0.005*	*0.013*	*0.000*	*0.000*	*0.000*				
Mpassa-2	Malachite	Mindouli	M16	Average	**17.888**	**15.656**	**38.005**	**0.875**	**2.125**	**2.428**	**685**	**9.98**	**4.05**	**-8.7E-08**
				□	*0.004*	*0.003*	*0.009*	*0.000*	*0.000*	*0.000*				
MPA-3	Malachite	Mindouli	FA-43	Average	**17.896**	**15.657**	**38.000**	**0.875**	**2.123**	**2.427**	**681**	**9.98**	**4.04**	**-2.2E-07**
				□	*0.004*	*0.003*	*0.010*	*0.000*	*0.000*	*0.000*				
Ntola	Malachite	Mindouli	M17	Average	**17.872**	**15.653**	**38.015**	**0.876**	**2.127**	**2.429**	**692**	**9.97**	**4.07**	**8.46E-07**
				□	*0.003*	*0.003*	*0.008*	*0.000*	*0.000*	*0.000*				
NTO-3	Malachite	Mindouli	FA-41	Average	**17.876**	**15.655**	**38.022**	**0.876**	**2.127**	**2.429**	**692**	**9.98**	**4.07**	**-2.8E-08**
				□	*0.002*	*0.002*	*0.005*	*0.000*	*0.000*	*0.000*				

Lab code	***Type***	***Area***	***Lab no***		***^{206}Pb/^{204}Pb***	***^{207}Pb/^{204}Pb***	***^{208}Pb/^{204}Pb***	***^{207}Pb/^{206}Pb***	***^{208}Pb/^{206}Pb***	***^{208}Pb/^{207}Pb***	***T (Ma)***	***□ (U/Pb)***	***□ (Th/U)***	***f=0***
GPSNN 259	Malachite	Mindouli	M20	Average	**17.919**	**15.666**	**38.107**	**0.874**	**2.127**	**2.432**	**680**	**10.01**	**4.09**	**1.36E-06**
				□	*0.005*	*0.004*	*0.012*	*0.000*	*0.000*	*0.000*				
GPSNN 259-6	Malachite	Mindouli	FA-44	Average	**17.942**	**15.664**	**38.103**	**0.873**	**2.124**	**2.432**	**660**	**10.00**	**4.07**	**4.78E-06**
				□	*0.002*	*0.003*	*0.007*	*0.000*	*0.000*	*0.000*				
GPSNN 263	Malachite	Mindouli	M21	Average	**17.863**	**15.645**	**37.991**	**0.876**	**2.127**	**2.428**	**685**	**9.94**	**4.06**	**8.31E-06**
				□	*0.004*	*0.004*	*0.011*	*0.000*	*0.000*	*0.000*				
MOU-2	Malachite	Mindouli	FA-42	Average	**17.985**	**15.661**	**38.136**	**0.871**	**2.120**	**2.435**	**624**	**9.97**	**4.06**	**7.6E-07**
				□	*0.007*	*0.006*	*0.014*	*0.000*	*0.000*	*0.000*				
TRB-01	Malachite	Mindouli	FA-45	Average	**17.925**	**15.670**	**38.090**	**0.874**	**2.125**	**2.431**	**682**	**10.03**	**4.08**	**-7.3E-07**
				□	*0.002*	*0.002*	*0.006*	*0.000*	*0.000*	*0.000*				
NTO-02	Galena	Mindouli	M6	Average	**17.903**	**15.665**	**38.087**	**0.875**	**2.127**	**2.431**	**689**	**10.01**	**4.09**	**-2.5E-08**
				□	*0.007*	*0.006*	*0.016*	*0.000*	*0.000*	*0.000*				
MPA-01	Galena	Mindouli	M8	Average	**17.884**	**15.650**	**37.991**	**0.875**	**2.124**	**2.427**	**679**	**9.96**	**4.04**	**7.03E-07**
				□	*0.007*	*0.007*	*0.017*	*0.000*	*0.000*	*0.000*				
GPSNN 284	Malachite	Mfouati	M23	Average	**18.392**	**15.688**	**38.060**	**0.853**	**2.069**	**2.426**	**379**	**9.96**	**3.75**	**6.51E-06**
				□	*0.004*	*0.004*	*0.010*	*0.000*	*0.000*	*0.000*				
MAL-1	Malachite	Mfouati	FA-38	Average	**18.609**	**15.701**	**38.059**	**0.844**	**2.045**	**2.424**	**244**	**9.96**	**3.62**	**2.95E-07**
				□	*0.003*	*0.002*	*0.006*	*0.000*	*0.000*	*0.000*				
GPSNN 289	Malachite	Mfouati	M24	Average	**17.856**	**15.651**	**37.987**	**0.877**	**2.127**	**2.427**	**701**	**9.97**	**4.06**	**-2.6E-06**
				□	*0.008*	*0.008*	*0.018*	*0.000*	*0.000*	*0.000*				
HAP-01	Galena	Mfouati	FA-47	Average	**17.834**	**15.650**	**37.978**	**0.877**	**2.129**	**2.427**	**713**	**9.97**	**4.07**	**-2.2E-08**
				□	*0.004*	*0.004*	*0.010*	*0.000*	*0.000*	*0.000*				

Table 4: LI ratios and calculated ages for archaeological samples

Lab code	***Type***	***Area***	***Lab no***		***^{206}Pb/^{204}Pb***	***^{207}Pb/^{204}Pb***	***^{208}Pb/^{204}Pb***	***^{207}Pb/^{206}Pb***	***^{208}Pb/^{206}Pb***	***^{208}Pb/^{207}Pb***	***T (Ma)***	***□ (U/Pb)***	***□ (Th/U)***	***f=0***
KNA14-1	Cu fragment in slag	Boko-Songho	FA--50	Average	**17.983**	**15.662**	**38.112**	**0.871**	**2.119**	**2.433**	**628**	**9.98**	**4.05**	**2.7E-07**
				□	*0.004*	*0.004*	*0.010*	*0.000*	*0.000*	*0.000*				
KNA 14-3	Malachite	Boko-Songho	FA-37	Average	**18.022**	**15.663**	**38.110**	**0.869**	**2.115**	**2.433**	**601**	**9.97**	**4.02**	**2.49E-07**
				□	*0.003*	*0.002*	*0.007*	*0.000*	*0.000*	*0.000*				
KNA 14-2	Slag	Boko-Songho	FJ6	Average	**17.996**	**15.662**	**38.113**	**0.870**	**2.118**	**2.433**	**618**	**9.97**	**4.04**	**8E-06**
				□	*0.006*	*0.005*	*0.014*	*0.000*	*0.000*	*0.000*				
MDB14-1	Ingot fragment	Boko-Songho	M3	Average	**17.860**	**15.654**	**38.001**	**0.876**	**2.128**	**2.428**	**701**	**9.98**	**4.07**	**5.11E-06**
				□	*0.004*	*0.004*	*0.010*	*0.000*	*0.000*	*0.000*				
MDB 14-3	Malachite	Boko-Songho	FA-36	Average	**17.947**	**15.658**	**38.084**	**0.872**	**2.122**	**2.432**	**646**	**9.97**	**4.05**	**-3E-07**
				□	*0.003*	*0.002*	*0.006*	*0.000*	*0.000*	*0.000*				
MDB 14-2	Slag (1)	Boko-Songho	FA-48	Average	**17.852**	**15.651**	**37.989**	**0.877**	**2.128**	**2.427**	**703**	**9.97**	**4.07**	**6.24E-07**
				□	*0.003*	*0.003*	*0.007*	*0.000*	*0.000*	*0.000*				
MDB 14-2	Slag (2)	Boko-Songho	FA-59	Average	**17.851**	**15.650**	**37.983**	**0.877**	**2.128**	**2.427**	**702**	**9.97**	**4.06**	**-1.6E-07**
				□	*0.002*	*0.002*	*0.006*	*0.000*	*0.000*	*0.000*				
MSG70628-5	Barrette	Mindouli	FA-29	Average	**17.886**	**15.660**	**38.044**	**0.876**	**2.127**	**2.429**	**693**	**10.00**	**4.08**	**-2E-07**
				□	*0.003*	*0.003*	*0.009*	*0.000*	*0.000*	*0.000*				
MSG70628-2	Ingot	Mindouli	FA-30	Average	**17.976**	**15.674**	**38.118**	**0.872**	**2.120**	**2.432**	**653**	**10.03**	**4.06**	**-1E-06**
				□	*0.003*	*0.003*	*0.009*	*0.000*	*0.000*	*0.000*				
MSG70620	Bracelet	Mindouli	FA-31	Average	**18.263**	**15.659**	**38.338**	**0.857**	**2.099**	**2.448**	**419**	**9.88**	**3.98**	**5.98E-07**
				□	*0.003*	*0.003*	*0.008*	*0.000*	*0.000*	*0.000*				
KIS14-1	Bar fragment	Mindouli	M4	Average	**17.951**	**15.675**	**38.135**	**0.873**	**2.124**	**2.433**	**672**	**10.04**	**4.09**	**-1.9E-08**
				□	*0.004*	*0.003*	*0.009*	*0.000*	*0.000*	*0.000*				
KIS 14-2	Slag (aggregate)	Mindouli	FJ5	Average	**17.953**	**15.665**	**38.094**	**0.873**	**2.122**	**2.432**	**653**	**9.99**	**4.06**	**4.13E-07**
				□	*0.002*	*0.002*	*0.007*	*0.000*	*0.000*	*0.000*				

Lab code	*Type*	*Area*	*Lab no*		$^{206}Pb/^{204}Pb$	$^{207}Pb/^{204}Pb$	$^{208}Pb/^{204}Pb$	$^{207}Pb/^{206}Pb$	$^{208}Pb/^{206}Pb$	$^{208}Pb/^{207}Pb$	*T (Ma)*	*□ (U/Pb)*	*□ (Th/U)*	*f=0*
KIS 14-3	Slag (1)	Mindouli	FA-49	Average	**17.985**	**15.672**	**38.108**	**0.871**	**2.119**	**2.432**	**642**	**10.01**	**4.05**	**9.59E-07**
				□	*0.003*	*0.003*	*0.009*	*0.000*	*0.000*	*0.000*				
KIS 14-3	Slag (2)	Mindouli	FA-60	Average	**17.995**	**15.667**	**38.093**	**0.871**	**2.117**	**2.431**	**627**	**9.99**	**4.03**	**2.46E-06**
				□	*0.002*	*0.003*	*0.007*	*0.000*	*0.000*	*0.000*				
MKU-314-1	Bar fragment	Mindouli	M5	Average	**17.949**	**15.673**	**38.134**	**0.873**	**2.125**	**2.433**	**670**	**10.03**	**4.09**	**-1.8E-07**
				□	*0.005*	*0.004*	*0.011*	*0.000*	*0.000*	*0.000*				
MKU 13-1	Barrette	Mindouli	FA-28	Average	**17.928**	**15.676**	**38.137**	**0.874**	**2.127**	**2.433**	**691**	**10.05**	**4.10**	**5.63E-06**
				□	*0.002*	*0.002*	*0.008*	*0.000*	*0.000*	*0.000*				
MKU-314-2	Mineral refuse	Mindouli	M13	Average	**18.061**	**15.669**	**38.097**	**0.868**	**2.109**	**2.431**	**583**	**9.98**	**3.98**	**2.59E-07**
				□	*0.004*	*0.004*	*0.011*	*0.000*	*0.000*	*0.000*				
MKU 314-3	Tuyère	Mindouli	FJ1	Average	**18.016**	**15.668**	**38.118**	**0.870**	**2.116**	**2.433**	**613**	**9.99**	**4.03**	**1.12E-06**
				□	*0.002*	*0.002*	*0.008*	*0.000*	*0.000*	*0.000*				
GPSNN 294-1	Barrette	Mindouli	FA-32	Average	**17.900**	**15.663**	**38.087**	**0.875**	**2.128**	**2.432**	**689**	**10.01**	**4.09**	**-2E-07**
				□	*0.003*	*0.003*	*0.008*	*0.000*	*0.000*	*0.000*				
GPSNN 294-2	Ingot	Mindouli	FA-33	Average	**17.995**	**15.679**	**38.137**	**0.871**	**2.119**	**2.432**	**648**	**10.04**	**4.06**	**1.99E-07**
				□	*0.002*	*0.002*	*0.007*	*0.000*	*0.000*	*0.000*				
KTG 14-3	Cu prill	Mindouli	FA-27	Average	**17.908**	**15.670**	**38.113**	**0.875**	**2.128**	**2.432**	**695**	**10.03**	**4.10**	**7.06E-06**
				□	*0.003*	*0.003*	*0.008*	*0.000*	*0.000*	*0.000*				
KTG 14-1	Cu (+ Slag?)	Mindouli	FD27	Average	**17.913**	**15.665**	**38.087**	**0.874**	**2.126**	**2.431**	**682**	**10.01**	**4.08**	**-1.2E-07**
				□	*0.004*	*0.004*	*0.011*	*0.000*	*0.000*	*0.000*				
KTG 14-2	Slag	Mindouli	FJ2	Average	**17.933**	**15.667**	**38.092**	**0.874**	**2.124**	**2.431**	**672**	**10.01**	**4.07**	**-7.2E-07**
				□	*0.002*	*0.002*	*0.007*	*0.000*	*0.000*	*0.000*				
NTM15-1	Copper extracted from slag	Mindouli	FA-35	Average	**17.978**	**15.670**	**38.093**	**0.872**	**2.119**	**2.431**	**645**	**10.01**	**4.04**	**-1.9E-07**
				□	*0.002*	*0.003*	*0.007*	*0.000*	*0.000*	*0.000*				
NTM15-1	Slag	Mindouli	FA-34	Average	**17.972**	**15.666**	**38.083**	**0.872**	**2.119**	**2.431**	**641**	**9.99**	**4.04**	**5E-07**
				□	*0.003*	*0.004*	*0.011*	*0.000*	*0.000*	*0.000*				

Lab code	***Type***	***Area***	***Lab no***		***^{206}Pb/^{204}Pb***	***^{207}Pb/^{204}Pb***	***^{208}Pb/^{204}Pb***	***^{207}Pb/^{206}Pb***	***^{208}Pb/^{206}Pb***	***^{208}Pb/^{207}Pb***	***T (Ma)***	***□ (U/Pb)***	***□ (Th/U)***	***f=0***
GPSNN-290-1	Lead slag	Mfouati	M11	Average	**17.845**	**15.649**	**37.981**	**0.877**	**2.128**	**2.427**	**703**	**9.96**	**4.06**	**4.94E-07**
				□	*0.005*	*0.004*	*0.012*	*0.000*	*0.000*	*0.000*				
RGM583-1	Ingot fragment	Ubangi	M18	Average	**17.854**	**15.653**	**38.002**	**0.877**	**2.128**	**2.428**	**705**	**9.98**	**4.07**	**-2.1E-07**
				□	*0.003*	*0.003*	*0.008*	*0.000*	*0.000*	*0.000*				
RGM583-2	Ingot fragment	Ubangi	M19	Average	**17.863**	**15.653**	**38.007**	**0.876**	**2.128**	**2.428**	**698**	**9.98**	**4.07**	**8.39E-08**
				□	*0.004*	*0.003*	*0.009*	*0.000*	*0.000*	*0.000*				

The Isoplot calculation (cfr. main text) for the entire dataset (Figure 15) does not provide a good fit (nor does the software allow good fitting of sub-sets of the data). For better modeling of mineralisation using Pb isotopic constraints (similar to that done by Haest *et al*., 2010, for example) analysis of sulphides and other minerals from associated mineralisation is preferable.

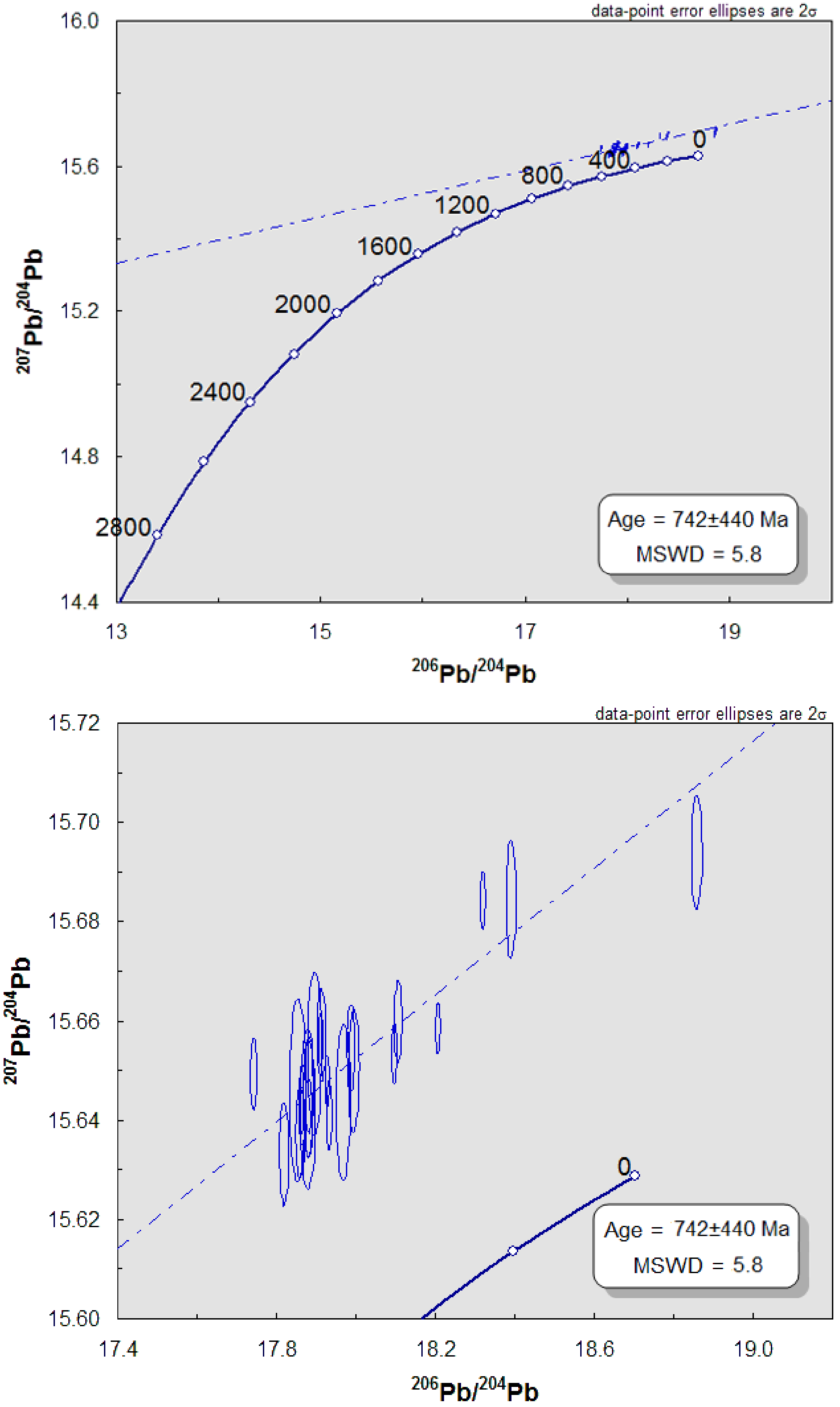

Figure 13: Alternative geological age calculation using Isoplot software for all data

Chemical data

As the acid digestion process destroys and volatilises (most) carbonates and silicates – which are bulk constituents of the ores and slags – ICP-OES analytical totals are typically well below 100%. ICP-OES analysis of metals results in totals between ca. 80-100%, where losses in recovery are mostly related to the presence of (minor) corrosion in the majority of our samples. For the (LA-)ICP-MS analyses, copper content was not measured and no analytical totals can be calculated – the aim of using this method was to quantify trace element contents only.

Table 5: Chemical composition of geological samples[3] (for each sample, the first line shows ICP-OES results, the second line ICP-MS results)

Code	Type	Site	Area	Lab no	Cu %	Ag µg/g	As µg/g	Au µg/g	Ba µg/g	Bi µg/g	Co µg/g	Cr µg/g	Fe µg/g	Mg µg/g	Mn µg/g	Ni µg/g	P µg/g	Pb µg/g	S µg/g	Sb µg/g	Se µg/g	Sn µg/g	Te µg/g	Ti µg/g	Zn µg/g
GPSNN 271	Malachite	Grande Mine	Boko-Songho	M12	56.4	<10	235	<8	<1	<30	<10	<5	320	<50	40	<25	835	<25	<16	<8	<150	<50	<16	<4	115
					56.8				1		2	<0.5				25		7							215
GPSNN 271-3	Malachite	Grande Mine	Boko-Songho	FA-40	47.6	13	250	<8	7	<8	35	11	5500	315	1060	<2	1110	180	<16	<8	<8	20	<16	40	630
					54.1		230		11		50	3				8		175		1		1		50	590
GPSNN 272	Malachite	Malembe	Boko-Songho	M22	53.9	<10	950	<30	<8	<150	35	<5	230	6000	275	<10	1230	35	<250	<8	<150	<50	<16	<15	195
					62.1				4		45	<0.5				3		30							320
Malembe-1	Malachite	Malembe	Boko-Songho	FA-39	47.8	5	150	11	2	<8	30	4	275	7200	135	11	1025	190	<16	<8	<8	<8	<16	15	940
					57.8		125		4		40	<0.5				13		210		<0.1		<0.1		20	900
Djen Geol	Malachite	Djenguele	Boko-Songho	M14	29.7	700	180	<8	<30	205	12	<5	4700	275	50	<10	1160	326000	6200	115	<150	<50	<16	<4	12600
					27.0				25		20	2				6		330000							11700
BSD-01	Galena	Djenguele	Boko-Songho	M7	0.3	245	330	9	8	185	<1	5	3000	75	5	<2	485	670000	99000	1000	<8	<8	<16	<4	13500
					0.2	12.0	350				1	<0.5				<0.5		730000		1200		2		8	12700
Mpassa-1	Malachite	Mpassa Mine	Mindouli	M15	32.3	590	1060	<8	60	<8	<1	6	4800	3100	760	<2	170	1500	8000	<8	45	<8	40	<4	33400
					37.4		1050		75		16	1				3		2100		4		0		8	39400
Mpassa-2	Malachite	Mpassa Mine	Mindouli	M16	45.5	775	360	<8	45	<8	2	6	5600	240	230	<2	330	1300	43000	<8	100	<8	35	30	3200
					51.5		380		60		6	2				<0.5		3400		14		<0.1		35	3300
MPA-3	Malachite	Mpassa Mine	Mindouli	FA-43	51.3	3170	700	<8	1200	225	<10	<1	5300	335	30	<10	365	2200	44000	<8	<150	<50	<16	<15	3900
					49.0				1300		4	<0.5				<0.5		1400							3800
Ntola	Malachite	Ntola	Mindouli	M17	48.3	10	3250	9	<1	120	<1	5	1050	1300	140	<2	805	200	<16	<8	<8	14	75	60	630
					53.2		3150		1		6	1				3		200		2		<0.1		70	730

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[3] For MOU-3 (Mindouli), a different malachite fragment from the same locality was sampled for chemical and LI analysis.

Code	*Type*	*Site*	*Area*	*Lab no*	*Cu* %	*Ag* µg/g	*As* µg/g	*Au* µg/g	*Ba* µg/g	*Bi* µg/g	*Co* µg/g	*Cr* µg/g	*Fe* µg/g	*Mg* µg/g	*Mn* µg/g	*Ni* µg/g	*P* µg/g	*Pb* µg/g	*S* µg/g	*Sb* µg/g	*Se* µg/g	*Sn* µg/g	*Te* µg/g	*Ti* µg/g	*Zn* µg/g
NTO-3	Malachite	Ntola	Mindouli	FA-41	55.1	<10	6100	<8	<1	<30	<10	<1	670	115	320	<10	1155	200	<16	<8	<50	<50	<16	<4	545
					52.9				1		7	<0.5				2		190							620
GPSNN 259	Malachite	Lagotala	Mindouli	M20	49.0	680	20	<8	10	85	<1	6	13000	440	800	<2	235	870	120000	25	<8	14	75	20	130
					55.1		40		13		1	1				1		1100		17		1		25	370
GPSNN 259-6	Malachite	Lagotala	Mindouli	FA-44	29.1	<30	<30	<30	<8	285	<10	<1	<50	90	780	<25	810	80	<16	<8	<150	<8	<16	<4	245
					53.8				18		1	25				26		190							595
MOU-3	Malachite	Moutele	Mindouli	FA-42	45.6	<10	60	<8	<1	<100	<10	<1	5	140	60	<10	550	93000	305	<8	<50	<50	<16	<4	1100
					45.7				1		16	<0.5				2		86000							1000
TRB-01	Malachite	Travers-Banc	Mindouli	FA-45	32.9	10	15	25	9	<8	<1	50	1000	2300	2045	<2	225	1700	125	<8	<8	<8	<16	20	3600
					31.9		30		13		5	45.0				4		4500		1		1		30	3400
NTO-02	Galena	Ntola	Mindouli	M6	0.4	45	<8	<8	<1	<8	<1	2	1600	13000	690	<2	<8	600000	120000	55	<8	<8	<16	7	240
					0.2		25		1		7	<0.5				<0.5		700000		90		<0.1		6	270
MPA-01	Galena	Mpassa	Mindouli	M8	0.4	35	40	<8	1	<8	1	3	1000	35	50	<2	70	510000	160000	40	<8	<8	<16	<4	140000
					0.3		65		2		12	<0.5				<0.5		560000		11		1		1	160000
GPSNN 284	Malachite	Mfouati	Mfouati	M23	44.6	<10	135	<30	<1	<100	<10	<1	160	28000	45	<10	2670	<25	<250	<8	<8	<50	<50	<50	110
					40.8				2		<0.2	<0.5				9		9							205
MAL-1	Malachite	Malouende	Mfouati	FA-38	37.0	6	70	10	9	<8	<1	9	1300	9800	65	15	1800	70	<16	<8	<8	<8	70	270	735
					40.6		25		13		1	4				14		35		0		0		280	750
GPSNN 289	Malachite	Mfouati/Hapilo	Mfouati	M24	36.0	<30	490	<8	<8	225	<10	<5	7700	22000	555	<10	500	28000	<250	<8	<50	<50	105	430	91000
					30.5				10		1	3				6		26000							94000
HAP-01	Galena	Hapilo	Mfouati	FA-47	0.1	85	1900	<8	<1	<8	2	3	160000	150	360	<2	180	120000	320000	100	<8	15	25	10	210000
					0.0		1840		1		<0.2	1				<0.5		130000		125		2		10	250000

Table 6: Chemical composition of archaeological samples[4] *(for ores and slags, the first line shows ICP-OES results, the second line ICP-MS results. For metals, the first line shows ICP-OES results, the second line LA-ICP-MS results*[5]*, and the third line ICP-MS results (MDB14-1, KIS14-1 and RGM 583-1 only))*

Code	***Type***	***Site***	***Area***	***Lab no***	***Cu*** *%*	***Ag*** *µg/g*	***As*** *µg/g*	***Au*** *µg/g*	***Ba*** *µg/g*	***Bi*** *µg/g*	***Co*** *µg/g*	***Cr*** *µg/g*	***Fe*** *µg/g*	***Mg*** *µg/g*	***Mn*** *µg/g*	***Ni*** *µg/g*	***P*** *µg/g*	***Pb*** *µg/g*	***S*** *µg/g*	***Sb*** *µg/g*	***Se*** *µg/g*	***Sn*** *µg/g*	***Te*** *µg/g*	***Ti*** *µg/g*	***Zn*** *µg/g*
KNA 14-4	Malachite	Kindanga-kanzi	Boko-Songho	FA-37	24.2	<10	140	<30	<1	235	<10	<1	570	305	120	<10	810	1200	<250	<8	150	<50	<16	30	4200
					44.9				5		17	<0.5				4		1000							7500
KNA 14-2	Slag	Kindanga-kanzi	Boko-Songho	FJ6	16.7	<50	630	<30	240	<40	55	16	30000	10300	1600	<25	2600	26000	<250	<8	<150	<50	<16	1700	2700
					15.0				260		60	16				13		25000							2700
MDB 14-1	Ingot fragment	Mouada-bambi	Boko-Songho	M3	69.1	240	6700	20	2	<8	<1	<1	305	55	6	100	145	270000	<16	80	<8	25	<16	25	17
						860	32000		20	2	3	5	2700		120	100		420000		430		2		575	50
					100.1		*10500*		*0*	*1*	*1*	*<0.05*	*60*		*<0.02*	*165*		*65000*		*160*		*1*		*25*	*5*
MDB 14-4	Malachite	Mouada-bambi	Boko-Songho	FA-36	54.2	<10	240	<8	<8	<40	<10	<5	1200	1400	250	<10	1100	465	<250	<8	<150	<8	<16	110	355
					53.4				6		4	<0.5				5		475							450
MDB 14-2	Slag (1)	Mouada-bambi	Boko-Songho	FA-48	3.2	16	1400	25	95	<8	100	20	160000	9500	2200	50	1900	300000	915	<8	50	25	40	925	2800
					3.4		1400		125		120	15				55		320000		17		4		1000	2700
MSG 70628-5	Barrette	Misenga	Mindouli	FA-29																					
						4400	390			0	0		20		6	6		775		16		4			3
MSG 70628-2	Ingot	Misenga	Mindouli	FA-30																					
						7200	90		0	4	0		450		155	6		405		9		4		15	30
MSG 70620	Bracelet	Misenga	Mindouli	FA-31																					
						2800	225		0	0	4		50		12	5		135		9		4			7

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[4] For KNA14-4 and MDB 14-4, a different malachite fragment from the same locality was sampled for chemical and LI analysis. KTG 14-3 bis does not correspond to the same fragment analysed for LI ratios (FD-27), but is a tiny copper-slag fragment. KNA14-1 and KTG14-1 were not analysed for chemical composition. From Ntominsier, FA-34-A has been analysed for LI ratios and ICP-OES, while FA-34-B is another slag fragment analysed for ICP-OES and MS. FA-35-A has been analysed for LI ratios and ICP-OES, while FA-35-B is other prill analysed by LA-ICP-MS.

[5] Average values are reported in Table 6, which exclude measurements of pure lead inclusions. Complete raw LA-ICP-MS data are reported below.

Code	***Type***	***Site***	***Area***	***Lab no***	***Cu*** %	***Ag*** *μg/g*	***As*** *μg/g*	***Au*** *μg/g*	***Ba*** *μg/g*	***Bi*** *μg/g*	***Co*** *μg/g*	***Cr*** *μg/g*	***Fe*** *μg/g*	***Mg*** *μg/g*	***Mn*** *μg/g*	***Ni*** *μg/g*	***P*** *μg/g*	***Pb*** *μg/g*	***S*** *μg/g*	***Sb*** *μg/g*	***Se*** *μg/g*	***Sn*** *μg/g*	***Te*** *μg/g*	***Ti*** *μg/g*	***Zn*** *μg/g*
KIS 14-1	Bar fragment	Kisaba	Mindouli	M4																					
						1600	170		0	0	0		30		2	3		735		3		0			11
					100.9		*240*		*0*	*<0.001*	*0*	*<0.05*	*40*		*3*	*4*		*365*		*8*		*0*		*1*	*10*
KIS 14-4	Malachite	Kisaba	Mindouli		44.5	360	500	<8	630	<50	150	25	4200	20000	590	<25	360	3400	370	<8	<50	<8	<16	55	495
					47.6				670		155	25				13		2300							565
KIS 14-2	Slag aggregate	Kisaba	Mindouli	FJ5	12.0	240	55	<8	1900	200	<10	55	35000	51000	1300	<25	2300	1400	<250	<8	<50	<8	<16	2400	590
					5.1				1700		20	50				20		755							185
KIS 14-3	Slag (1)	Kisaba	Mindouli	FA-49	29.3	350	205	20	9600	<8	17	20	11000	24000	2700	11	5900	2300	3000	<8	70	<8	<16	935	260
					31.7		200		13100		35	14				13		3500		3		1		1000	320
MKU 314-1	Bar fragment	Makuti	Mindouli	M5																					
						3060	465		0	0	0		50		12	4		460		2		0			5
MKU 13-1	Barrette	Makuti	Mindouli	FA-28																					
						7200	1100			1	0		19		1	6		210		12		4			10
MKU 314-2	Mineral refuse	Makuti	Mindouli	M13	14.3	1200	<8	<8	9	<8	2	9	2500	600	7	<2	445	445	18000	<8	<8	<8	<16	25	30
					15.7		14		13		1	4				1		505		1		<0.1		30	65
MKU 314-3	Tuyère	Makuti	Mindouli	FJ1	2.6	<50	<8	<30	815	220	<10	20	17000	27000	1500	<25	650	350	<250	<8	<50	<8	<16	1600	630
					12.1				2100		16	60				25		385							1200
GPSNN 294-1	Barrette	Nkabi	Mindouli	FA-32																					
						4050	235			0	0		40		2	6		460		11		4			10
GPSNN 294-2	Ingot	Nkabi	Mindouli	FA-33																					
						2200	140		0	0	0		195		5	5		345		9		4		17	9
KTG 14-3	Cu prill	Kitchounga	Mindouli	FA-27	90.2	30	915	<16	<1	<24	1	<1	930	<8	6	11	420	27000	12000	30	<8	<16	100	11	310
KTG 14-2	Slag	Kitchounga	Mindouli	FJ2	5.2	110	95	<50	410	260	<10	18	160000	11000	5000	<10	3700	4800	1400	<8	<50	<8	<16	850	3400
					5.3				430		18	17				6		3700							3300
KTG 14-3 bis	Slag	Kitchounga	Mindouli	-																					
						280	1100		4900	13	185	190	470000		51000	110		24000		30		6		4100	14000

Code	***Type***	***Site***	***Area***	***Lab no***	***Cu*** *%*	***Ag*** *µg/g*	***As*** *µg/g*	***Au*** *µg/g*	***Ba*** *µg/g*	***Bi*** *µg/g*	***Co*** *µg/g*	***Cr*** *µg/g*	***Fe*** *µg/g*	***Mg*** *µg/g*	***Mn*** *µg/g*	***Ni*** *µg/g*	***P*** *µg/g*	***Pb*** *µg/g*	***S*** *µg/g*	***Sb*** *µg/g*	***Se*** *µg/g*	***Sn*** *µg/g*	***Te*** *µg/g*	***Ti*** *µg/g*	***Zn*** *µg/g*
NTM 15-1	Copper extracted from slag	Ntominsier	Mindouli	FA-35-A	99.9	40	175	2	1	12	0	0	60	25	50	3	25	15	25	4	5	2	140	6	3
				FA-35-B		190	150		17	0	0		550		935	5		70		8		4		135	30
NTM 15-1	Slag	Ntominsier	Mindouli	FA-34-A	84.0	7	1500	Mn interf	1020	40	25	100	52000	32000	43000	65	18000	2800	430	19	40	35	145	6400	1600
				FA-34-B	20.2	40	460	120	240	<8	<1	30	13000	8900	10000	20	5100	920	1700	<8	25	<8	<16	1500	445
				FA-34-B	22.3		360		360		5	25				18		1500		8		2		1600	465
GPSNN 290-1	Lead slag	Mfouati Songa-Melka	Mfouati	M11	0.1	20	900	50	15	<8	<1	10	33000	1800	4800	<2	495	410000	630	<8	<8	16	<16	100	905
					0.0		970		20		4	4				1		460000		15		1		120	890
RGM 583-1	Ingot fragment	Ubangi	Ubangi	M18	78.8	50	860	<8	<1	25	<1	1	75	40	5	40	155	140000	240	45	16	<8	60	5	17
						570	2700		1	1	0	2	200		170	45		150000		95		1		50	50
					97.2		*620*		*<0.02*	*<0.001*	*0*	*<0.05*	*2*		*0*	*65*		*34000*		*25*		*0*		*2*	*7*
RGM 583-2	Ingot fragment	Ubangi	Ubangi	M19	87.0	55	15000	<8	<1	<8	1	<1	30	13	1	45	<8	56000	260	95	10	9	40	<4	13
						1060	29000		1	20	1		150		4	35		190000		150		4		16	30

Ingot fragments for which multiple samples were taken for ICP-OES analysis:

M3-A was taken from the side of the ingot fragment, M3-B from closer to the fragment centre. For the Ubangi sample, two different loose fragments were analysed (fragmented ingot, original position of fragments unclear). The main difference between the samples is the sample weight, chosen to investigate analytical bias introduced by sample size. No major distinctions are noted.

Code	*Type*	*Site*	*Area*	*Lab no*	*Weight (g)*	***Cu*** *%*	***Ag*** *µg/g*	***As*** *µg/g*	***Au*** *µg/g*	***Ba*** *µg/g*	***Bi*** *µg/g*	***Co*** *µg/g*	***Cr*** *µg/g*	***Fe*** *µg/g*	***Mg*** *µg/g*	***Mn*** *µg/g*	***Ni*** *µg/g*	***P*** *µg/g*	***Pb*** *µg/g*	***S*** *µg/g*	***Sb*** *µg/g*	***Se*** *µg/g*	***Sn*** *µg/g*	***Te*** *µg/g*	***Ti*** *µg/g*	***Zn*** *µg/g*
MDB 14-1	Ingot fragment	Mouadabambi	Boko-Songho	M3-A	0.0133	69.1	240	6700	20	2	<8	<1	<1	305	55	6	100	145	270000	<16	80	<8	25	<16	25	17
				M3-B	0.0717	59.4	100	6300	<8	4	<8	<1	<1	420	50	8	85	40	280000	310	70	<8	<8	25	30	10
RGM 583-1	Ingot fragment	Ubangi	Ubangi	M18-A	0.0325	77.0	165	545	<8	3	<8	<1	2	435	190	150	50	370	150000	180	40	35	110	35	50	45
				M18-B	0.1016	78.8	50	860	<8	<1	25	<1	1	75	40	5	40	155	140000	240	45	16	<8	60	5	17

Table 7: Raw LA-ICP-MS results for metal samples (in ppm)

Code		*Ti*	*V*	*Cr*	*Mn*	*Fe*	*Co*	*Ni*	*Zn*	*Ga*	*Ge*	*As*	*Nb*	*Mo*	*Ag*	*Cd*	*In*	*Sn*	*Sb*	*Ba*	*La*	*Ce*	*Sm*	*W*	*Pb*	*Bi*
	MDB14S3-p1	<10	<0.05	<0.5	<0.5	8	0.47	109	6.6	<0.5	<0.5	5757	<0.02	<0.05	580	0.5	<0.05	0.30	37	<0.01	<0.01	<0.01	<0.05	<0.05	41319	1.09
	MDB14S3-p2	<10	<0.05	<0.5	<0.5	15	0.46	119	9.6	<0.5	<0.5	8839	<0.02	<0.05	1031	0.8	<0.05	0.42	54	<0.01	<0.01	<0.01	<0.05	<0.05	83249	1.31
	MDB14S3-i1	484	8.8	4.7	246	4560	6.49	63	119	1.4	0.7	108797	1.70	0.36	106	26	0.39	7.4	1677	34	3.0	4.6	0.46	0.24	1022472	0.27
	MDB14S3-z2p3	<10	<0.05	<0.5	<0.5	17	0.64	123	8.8	<0.5	<0.5	13430	<0.02	<0.05	1406	1.3	<0.05	0.46	63	0.08	<0.01	<0.01	<0.05	<0.05	284226	2.70
	MDB14S3-z2i2ext	<10	<0.05	<0.5	<0.5	<1	<0.01	27	<0.2	<0.5	<0.5	1441	<0.02	<0.05	3028	1.1	<0.05	<0.05	46	<0.01	<0.01	<0.01	<0.05	<0.05	1186377	6.29
	MDB14S3-i3ext	<10	0.21	<0.5	1.7	98	0.60	124	8.9	<0.5	<0.5	12320	0.07	<0.05	994	1.6	<0.05	0.61	115	0.45	0.05	0.09	<0.05	<0.05	287299	2.06
	MDB14S3-z3p4	<10	<0.05	<0.5	<0.5	<1	0.50	119	9.0	<0.5	<0.5	14370	<0.02	<0.05	920	1.4	<0.05	0.28	88	<0.01	<0.01	<0.01	<0.05	<0.05	108284	1.51
	MDB14S3-z4p5	<10	<0.05	<0.5	<0.5	<1	0.43	122	10.5	<0.5	<0.5	16440	<0.02	<0.05	564	1.3	<0.05	0.50	75	<0.01	<0.01	<0.01	<0.05	<0.05	41934	0.88
	MDB14S3-long z1p1	<10	<0.05	<0.5	<0.5	3	0.69	119	13.1	<0.5	<0.5	14900	<0.02	<0.05	449	1.4	<0.05	0.55	67	<0.01	<0.01	<0.01	<0.05	<0.05	28224	0.63
	MDB14S3-long z1i1	928	13.6	6.9	146	9586	14.76	60	216	2.4	1.7	123305	3.9	0.85	37	47	0.63	10.2	2181	51	5.4	8.0	0.66	0.24	1092059	0.11
	MDB14S3-long z2i2	317	6.1	3.3	89	7649	12.70	49	178	1.4	1.5	58152	1.23	0.51	73	26	0.31	4.1	1106	26	2.5	3.7	0.40	0.08	1001167	0.23
	MDB14S3-long z2p2	<10	<0.05	<0.5	<0.5	<1	0.50	123	7.7	<0.5	<0.5	14570	<0.02	<0.05	909	0.9	<0.05	0.39	54	<0.01	<0.01	<0.01	<0.05	<0.05	177245	1.73
	MDB14S3-long z3p3	<10	<0.05	<0.5	<0.5	<1	0.52	124	7.2	<0.5	<0.5	12760	<0.02	<0.05	1073	0.7	<0.05	0.34	50	<0.01	<0.01	<0.01	<0.05	<0.05	121918	1.91
MDB14-1 Lab no: M3	**Average**	**576**	**7**	**5**	**121**	**2742**	**3**	**99**	**50**	**2**	**1**	**31160**	**2**	**1**	**859**	**9**	**0**	**2**	**432**	**22**	**3**	**4**	**1**	**0**	**421213**	**2**
	RGM583-z1p1	<10	0.06	<0.5	2.6	21	0.19	48	8.7	<0.5	<0.5	955	<0.02	<0.05	483	0.3	<0.05	0.19	34	0.16	<0.01	0.02	<0.05	<0.05	10867	0.47
	RGM583-z1i1	<10	0.06	2.4	2.8	20	0.18	45	9.5	<0.5	<0.5	878	<0.02	<0.05	600	0.3	<0.05	0.19	34	0.14	<0.01	0.03	<0.05	<0.05	5495	0.36
	RGM583-z1ext i2	25	0.60	<0.5	36	202	0.37	34	39	<0.5	<0.5	5316	0.08	<0.05	916	2.3	<0.05	0.81	201	1.50	0.15	0.35	<0.05	<0.05	514476	1.63
	RGM583-z1ext p2	<10	<0.05	<0.5	1.5	10	0.20	47	15.9	<0.5	<0.5	1708	<0.02	<0.05	494	0.4	<0.05	0.16	39	0.07	<0.01	0.02	<0.05	<0.05	15750	0.45
	RGM583-z2 p3	<10	0.17	<0.5	14.4	76	0.37	55	32	<0.5	<0.5	3509	0.03	<0.05	450	0.7	<0.05	0.41	123	0.58	0.04	0.09	<0.05	<0.05	70596	0.46
	RGM583-z3 i3	18	0.98	2.9	940	342	0.89	30	208	<0.5	<0.5	2067	0.06	0.16	651	1.1	<0.05	3.1	73	0.91	0.12	0.23	<0.05	0.42	432505	1.25
	RGM583-z4 p4	43	0.88	1.0	100	232	0.35	52	26	<0.5	<0.5	2142	0.15	0.13	596	0.5	<0.05	0.24	62	1.83	0.05	0.15	<0.05	<0.05	36233	0.66
	RGM583-z5ext i4	56	1.33	1.5	155	341	0.44	53	33	<0.5	<0.5	1665	0.19	0.24	447	0.6	<0.05	0.27	56	2.7	0.09	0.33	<0.05	<0.05	32176	0.29
	RGM583-z5ext p5	97	1.83	1.7	285	577	0.75	59	58	<0.5	<0.5	6068	0.33	0.33	449	1.3	<0.05	0.79	255	4.5	0.15	0.66	<0.05	<0.05	218383	0.49
RGM583-1 Lab no: M18	**Average**	**48**	**1**	**2**	**171**	**202**	**0**	**47**	**48**			**2701**	**0**	**0**	**565**	**1**	**0**	**1**	**97**	**1**	**0**	**0**		**0**	**148498**	**1**

Code		*Ti*	*V*	*Cr*	*Mn*	*Fe*	*Co*	*Ni*	*Zn*	*Ga*	*Ge*	*As*	*Nb*	*Mo*	*Ag*	*Cd*	*In*	*Sn*	*Sb*	*Ba*	*La*	*Ce*	*Sm*	*W*	*Pb*	*Bi*
	KIS14 s4-1	<10	<0.05	<0.5	2.7	36	0.19	2.2	7.1	<0.5	<0.5	82	<0.02	<0.05	1509	<0.1	<0.05	0.28	1.8	0.04	<0.01	<0.01	<0.05	<0.05	611	0.35
	KIS14 s4-1 ablation 2	<10	<0.05	<0.5	2.2	22	0.23	3.6	10.3	<0.5	<0.5	167	<0.02	<0.05	1435	<0.1	<0.05	0.24	2.5	<0.01	<0.01	<0.01	<0.05	<0.05	480	0.26
	KIS14 s4-2	<10	0.39	<0.5	0.9	23	0.20	2.2	11.5	<0.5	<0.5	204	<0.02	0.11	1678	<0.1	<0.05	0.31	2.9	0.59	<0.01	<0.01	<0.05	<0.05	699	0.37
	KIS14 s4-2 ablation 2	<10	<0.05	<0.5	2.5	35	0.25	3.1	14.8	<0.5	<0.5	215	<0.02	<0.05	1672	<0.1	<0.05	0.27	2.9	0.05	<0.01	<0.01	<0.05	<0.05	1146	0.41
KIS14-1 Lab no: M4	**Average**				**2**	**29**	**0**	**3**	**11**			**167**		**0**	**1574**			**0**	**3**	**0**					**734**	**0**
	MKU14 s6-1	<10	0.23	<0.5	11.4	100	0.21	4.7	5.0	<0.5	<0.5	457	<0.02	<0.05	2748	<0.1	<0.05	0.29	2.4	0.11	<0.01	0.03	<0.05	<0.05	655	0.18
	MKU14 s6-1 ablation 2	<10	0.09	<0.5	1.9	39	0.14	4.5	3.4	<0.5	<0.5	458	<0.02	0.11	3006	<0.1	<0.05	0.25	2.2	0.04	<0.01	<0.01	<0.05	<0.05	690	0.22
	MKU14 s6-2	<10	0.12	<0.5	15.2	41	0.18	4.5	7.2	<0.5	<0.5	426	<0.02	0.12	3254	<0.1	<0.05	0.26	2.4	0.05	<0.01	<0.01	<0.05	<0.05	70	<0.05
	MKU14 s6-2 ablation 2	<10	<0.05	<0.5	22	48	0.29	4.2	6.7	<0.5	<0.5	494	<0.02	0.09	3170	<0.1	<0.05	0.20	2.3	<0.01	<0.01	<0.01	<0.05	<0.05	167	<0.05
	MKU14 s6-3	<10	<0.05	<0.5	8.8	28	0.18	4.3	4.8	<0.5	<0.5	465	<0.02	0.06	3057	<0.1	<0.05	0.18	2.5	0.02	<0.01	<0.01	<0.05	<0.05	493	0.11
	MKU14 s6-3 ablation 2	<10	<0.05	<0.5	11.0	27	0.15	4.1	5.2	<0.5	<0.5	494	<0.02	<0.05	3104	<0.1	<0.05	0.18	2.3	<0.01	<0.01	<0.01	<0.05	<0.05	678	0.17
MKU-314-1 Lab no: M5	**Average**		**0**		**12**	**47**	**0**	**4**	**5**			**466**		**0**	**3057**			**0**	**2**	**0**					**459**	**0**
	NTM15-2 s1	235	27	<0.5	5110	2914	1.72	9.0	93	24	2.3	95	0.76	0.88	103	0.2	<0.05	4.4	8.1	94	3.2	4.8	0.58	0.34	309	0.06
	NTM15-2 s1-2	37	3.0	<0.5	441	274	0.34	5.0	16.7	2.4	<0.5	57	0.13	<0.05	100	<0.1	<0.05	4.0	7.0	9.3	0.41	0.53	0.17	0.07	34	<0.05
	NTM15-2 s2	<10	<0.05	<0.5	2.3	10	0.14	3.3	0.3	<0.5	<0.5	109	<0.02	0.99	326	<0.1	<0.05	3.9	7.7	0.03	<0.01	<0.01	<0.05	<0.05	<0.5	<0.05
	NTM15-2 s2-2	<10	<0.05	<0.5	2.5	10	0.13	4.5	<0.2	<0.5	<0.5	206	<0.02	0.27	225	<0.1	<0.05	4.0	7.4	0.04	<0.01	<0.01	<0.05	<0.05	1.8	<0.05
	NTM15-2 s3	<10	0.67	<0.5	47	94	0.17	5.0	4.4	<0.5	<0.5	206	0.06	0.44	185	<0.1	<0.05	4.0	7.9	1.35	0.08	0.17	<0.05	<0.05	5.7	<0.05
	NTM15-2 s3-2	<10	<0.05	<0.5	2.1	12	0.14	4.7	<0.2	<0.5	<0.5	229	<0.02	<0.05	180	<0.1	<0.05	3.9	7.4	0.03	<0.01	<0.01	<0.05	<0.05	0.8	<0.05
NTM15-1 Lab no: FA35	**Average**	**136**	**10**		**934**	**552**	**0**	**5**	**29**	**13**	**2**	**150**	**0**	**1**	**187**	**0**		**4**	**8**	**17**	**1**	**2**	**0**	**0**	**70**	**0**
	MSG70628-5 s1	<10	<0.05	<0.5	12.6	27	0.33	5.5	3.4	<0.5	<0.5	416	<0.02	<0.05	4508	<0.1	<0.05	4.1	17.0	<0.01	<0.01	<0.01	<0.05	<0.05	1182	0.40
	MSG70628-5 s2	<10	<0.05	<0.5	1.3	16	0.26	5.4	1.7	<0.5	<0.5	383	<0.02	<0.05	4468	<0.1	<0.05	4.0	14.9	<0.01	<0.01	<0.01	<0.05	<0.05	366	0.22
	MSG70628-5 s3	<10	<0.05	<0.5	5.3	24	0.35	5.7	2.8	<0.5	<0.5	370	<0.02	<0.05	4271	<0.1	<0.05	4.0	14.8	<0.01	<0.01	<0.01	<0.05	<0.05	771	0.29
MSG70628-5 Lab no: FA-29	**Average**				**6**	**22**	**0**	**6**	**3**			**390**			**4416**			**4**	**16**						**773**	**0**

Code		***Ti***	***V***	***Cr***	***Mn***	***Fe***	***Co***	***Ni***	***Zn***	***Ga***	***Ge***	***As***	***Nb***	***Mo***	***Ag***	***Cd***	***In***	***Sn***	***Sb***	***Ba***	***La***	***Ce***	***Sm***	***W***	***Pb***	***Bi***
	MSG70620-1 s1	<10	<0.05	<0.5	2.0	19	2.9	7.3	3.5	<0.5	<0.5	225	<0.02	<0.05	2321	<0.1	<0.05	3.9	8.6	<0.01	<0.01	<0.01	<0.05	<0.05	48	0.31
	MSG70620-1 s2	<10	<0.05	<0.5	30	103	5.2	5.2	10.6	<0.5	<0.5	229	<0.02	<0.05	2918	0.3	<0.05	4.3	9.2	0.04	<0.01	<0.01	<0.05	<0.05	206	0.51
	MSG70620-1 s3	<10	<0.05	<0.5	3.4	28	2.8	3.9	7.3	<0.5	0.7	227	<0.02	<0.05	3023	<0.1	<0.05	4.1	8.7	<0.01	<0.01	<0.01	<0.05	<0.05	151	0.40
MSG70620 Lab no: FA-31	**Average**				**12**	**50**	**4**	**5**	**7**		**1**	**227**			**2754**	**0**		**4**	**9**	**0**					**135**	**0**
	KTG14-3 s1	4054	937	205	53379	489437	190	102	14317	62	85	717	13.0	277	251	7.6	0.73	6.6	29	4972	137	222	30	24	24355	10.6
	KTG14-3 s1-2	3307	705	151	42466	391881	147	120	11394	45	68	523	9.8	205	131	6.0	0.67	4.5	22	4022	111	175	25	18.3	19861	4.1
	KTG14-3 s2	5155	1004	210	56658	507680	197	114	14557	64	87	1918	15.4	300	530	11.6	0.71	7.3	36	5599	173	243	38	24	26221	8.3
	KTG14-3 s3	3857	910	192	53185	481944	196	102	15128	59	92	1407	12.2	289	198	11.9	0.80	6.5	38	4900	131	216	29	22	26854	29
KTG 14-3 Lab no: FA-27	**Average**	**4093**	**889**	**189**	**51422**	**467736**	**183**	**109**	**13849**	**57**	**83**	**1141**	**13**	**268**	**277**	**9**	**1**	**6**	**31**	**4873**	**138**	**214**	**31**	**22**	**24323**	**13**
	GPSNN294-1 s1	<10	<0.05	<0.5	2.8	39	0.32	5.5	11.6	<0.5	<0.5	315	<0.02	<0.05	5228	<0.1	<0.05	4.1	12.1	<0.01	<0.01	<0.01	<0.05	<0.05	595	0.13
	GPSNN294-1 s2	<10	<0.05	<0.5	2.1	46	0.36	5.4	10.9	<0.5	<0.5	232	<0.02	<0.05	4164	<0.1	<0.05	4.0	10.4	<0.01	0.08	<0.01	<0.05	<0.05	499	0.11
	GPSNN294-1 s3	<10	<0.05	<0.5	1.4	42	0.48	7.5	7.7	<0.5	<0.5	152	<0.02	<0.05	2764	<0.1	<0.05	4.0	9.1	<0.01	<0.01	<0.01	<0.05	<0.05	279	0.06
GPSNN 294-1 Lab no: FA-32	**Average**				**2**	**42**	**0**	**6**	**10**			**233**			**4052**			**4**	**11**		**0**				**458**	**0**
	MKU13-1 s1	<10	<0.05	<0.5	<0.5	17	0.15	5.3	8.7	<0.5	<0.5	1203	<0.02	<0.05	6987	<0.1	<0.05	4.0	11.8	<0.01	<0.01	<0.01	<0.05	<0.05	181	0.57
	MKU13-1 s2	<10	<0.05	<0.5	1.0	20	0.17	6.0	11.8	<0.5	<0.5	1213	<0.02	<0.05	8100	<0.1	<0.05	4.0	13.1	<0.01	<0.01	<0.01	<0.05	<0.05	258	0.71
	MKU13-1 s3	<10	<0.05	<0.5	0.8	18	0.17	8.0	8.3	<0.5	<0.5	1121	<0.02	<0.05	6603	<0.1	<0.05	3.9	11.8	<0.01	<0.01	<0.01	<0.05	<0.05	194	0.51
MKU 13-1 Lab no: FA-28	**Average**				**1**	**19**	**0**	**6**	**10**			**1179**			**7230**			**4**	**12**						**211**	**1**

Code		*Ti*	*V*	*Cr*	*Mn*	*Fe*	*Co*	*Ni*	*Zn*	*Ga*	*Ge*	*As*	*Nb*	*Mo*	*Ag*	*Cd*	*In*	*Sn*	*Sb*	*Ba*	*La*	*Ce*	*Sm*	*W*	*Pb*	*Bi*
	RG583-2 s1	15	0.43	<0.5	4.7	150	2.5	23	68	<0.5	<0.5	60123	0.07	0.16	494	2.5	<0.05	8.5	459	1.42	0.17	0.27	<0.05	0.15	574521	0.36
	RG583-2 s1-2	<10	0.07	<0.5	2.2	61	1.59	50	48	<0.5	<0.5	50080	<0.02	<0.05	852	2.4	<0.05	4.7	306	0.39	0.10	0.11	0.08	<0.05	308000	164
	RG583-2 s2	26	1.64	<0.5	8.5	136	0.99	37	76	<0.5	<0.5	25460	0.05	<0.05	1183	0.6	<0.05	7.0	110	0.60	0.02	0.05	0.07	<0.05	232100	0.76
	RG583-2 s2-2	<10	0.06	<0.5	1.0	491	1.12	40	28	<0.5	<0.5	26560	<0.02	<0.05	1157	<0.1	<0.05	4.1	111	0.02	<0.01	0.35	<0.05	<0.05	137900	41
	RG583-2 s3	<10	0.40	<0.5	3.1	374	0.70	41	25	<0.5	<0.5	27260	<0.02	<0.05	1009	0.5	<0.05	4.4	104	0.92	0.23	0.58	<0.05	<0.05	109700	0.43
	RG583-2 s3-2	<10	<0.05	<0.5	<0.5	26	0.77	43	18.9	<0.5	<0.5	26310	0.03	<0.05	838	0.4	<0.05	4.1	110	0.14	<0.01	<0.01	<0.05	<0.05	61930	0.19
	RG583-2 s4	<10	0.19	<0.5	4.0	58	0.40	12.4	5.7	<0.5	<0.5	7200	0.04	<0.05	624	0.3	<0.05	1.0	49	0.71	0.08	0.12	<0.05	<0.05	131399	0.14
	RG583-2 s4-2	12	0.34	<0.5	5.5	79	0.60	18.8	15.9	<0.5	<0.5	22325	0.03	<0.05	499	0.5	<0.05	1.5	98	1.47	0.21	0.28	<0.05	<0.05	223382	0.14
	RG583-2 s5	11	0.36	<0.5	4.3	97	0.62	43	20	<0.5	<0.5	30210	0.04	<0.05	2534	0.8	<0.05	4.3	127	0.82	0.09	0.12	<0.05	<0.05	109400	0.23
	RG583-2 s5-2	<10	<0.05	<0.5	<0.5	15	0.64	46	3.1	<0.5	<0.5	10080	<0.02	<0.05	1427	<0.1	<0.05	3.9	44	0.05	<0.01	<0.01	<0.05	<0.05	31700	3.5
RGM583-2 Lab no: M19	**Average**	**16**	**0**		**4**	**149**	**1**	**35**	**31**			**28561**	**0**	**0**	**1062**	**1**		**4**	**152**	**1**	**0**	**0**	**0**	**0**	**192003**	**21**
	MSG70628-2 s1	18	0.07	<0.5	392	1016	0.44	6.1	38	2.8	<0.5	74	0.06	0.26	8767	0.2	<0.05	4.3	8.9	0.19	<0.01	<0.01	0.13	<0.05	712	6.4
	MSG70628-2 s2	<10	<0.05	<0.5	39	161	0.28	5.9	24	<0.5	<0.5	75	<0.02	<0.05	6575	<0.1	<0.05	4.2	8.2	<0.01	<0.01	0.08	<0.05	<0.05	222	2.0
	MSG70628-2 s3	12	0.18	<0.5	28	170	0.38	7.1	30	<0.5	<0.5	126	<0.02	<0.05	6349	<0.1	<0.05	4.1	9.4	<0.01	0.10	0.06	0.08	<0.05	280	2.6
MSG70628-2 Lab no: FA-30	**Average**	**15**	**0**		**153**	**449**	**0**	**6**	**31**	**3**		**92**	**0**	**0**	**7230**	**0**		**4**	**9**	**0**	**0**	**0**	**0**		**405**	**4**
	GPSNN294-2 s1	18	0.18	<0.5	8.2	131	0.24	4.3	8.8	<0.5	<0.5	131	<0.02	<0.05	3052	<0.1	<0.05	4.2	9.7	0.43	0.17	0.36	<0.05	<0.05	591	0.65
	GPSNN294-2 s2	17	0.37	<0.5	3.7	238	0.27	5.8	8.0	<0.5	<0.5	125	0.13	0.08	1790	0.3	<0.05	4.0	8.5	0.25	0.14	0.13	<0.05	<0.05	149	0.26
	GPSNN294-2 s3	15	0.67	<0.5	4.3	211	0.25	5.3	9.2	<0.5	<0.5	164	<0.02	<0.05	1763	<0.1	<0.05	4.1	9.1	0.40	0.14	0.08	<0.05	<0.05	291	0.33
GPSNN 294-2 Lab no: FA-33	**Average**	**17**	**0**		**5**	**194**	**0**	**5**	**9**			**140**	**0**	**0**	**2202**	**0**		**4**	**9**	**0**	**0**	**0**			**344**	**0**

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Annexe 3. Analytical Methods and results of the geochemical analyses on copper ingots from Katanga, South-East DRC

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Contents

Analytical methods

Sample preparation for LI analysis by MC-ICP-MS (Multi-Collector Inductively-Coupled-Plasma Mass Spectrometry) was carried out at the Geochemical laboratory, KU Leuven. A small surface area of the selected artefacts was mechanically cleaned (Dremel rotary tool (Bosch®), steel brush) prior to sampling with a clean TiN-coated steel 1mm drill bit to obtain core material. For the smallest HH *croisettes*, a sample was obtained by cutting a fragment using a clean rotary saw. Prior to dissolution, the samples were weighed. The samples (ca. 0.1g) were digested using *aqua regia*: HNO_3 (65%) and HCl (37%). After adding the acids, the beakers were closed and placed on a hotplate (110°C) over night, and then opened to evaporate the acids to dryness. The remaining residues were dissolved in 1M HNO_3, from which one aliquot was retained for elemental analysis by ICP-OES, one aliquot for elemental analysis by Q-ICP-MS and one aliquot for lead isotope analysis by MC-ICP-MS.

Bulk chemical analysis of all metal samples was carried out by ICP-OES (Inductively-Coupled-Plasma Optical Emission Spectroscopy) at the Geochemical laboratory, KU Leuven, using a Varian 720-ES instrument supplied with double-pass glass cyclonic spray chamber, concentric glass Sea-Spray nebulizer and “extended high solids” torch. An ionisation buffer (1% $CsNO_3$ in 4% HNO_3), intended to eliminate ionisation effects, was added. Concentrations of 21 elements were determined from a single analysis run. Calibration solutions were prepared from certified Plasma HIQU single element solutions (CHEM-LAB, Belgium). Ultra-pure water (>18 M□/cm^3) and analytical reagent grade acids were used. A NIST GBW-7411 soil standard and MBH CRM CR32-PB110 bronze standard were digested and analysed in the same run. Detection limits were calculated for all elements by repeated measurements of independent standard solutions.

All metal samples were further analysed by ICP-MS (Quad X-Series2, Thermo) for major and trace elements at the Earth Sciences Department, RMCA. The external calibrations were performed with multi-element solutions and were assessed by analysing ERM-EB386 copper CRM standard prepared with the same protocol as the samples. Overall accuracy was better than 10%, but below 15% for Mg, Al, Mn and Bi. The precision was between 1 and 4% RSD.

Lead isolation (procedure detailed by De Muynck *et al*., 2008) was carried out at a class 10 clean lab at Ghent University (Belgium). Pb concentrations of the solutions were determined using a Thermo Scientific Element XR sector field ICP-MS instrument (Ghent University), to calculate dilutions for MC-ICP-MS measurements. 30 µg/l Tl (NIST SRM 997) was added as an internal standard to both standards and samples for mass bias correction calculations (see, e.g., Ketterer *et al*. 1991, Walder *et al*. 1993). Pb isotope ratios were determined using a Thermo Scientific Neptune MC-ICP-MS (see De Muynck *et al*. (2008) for instrumental settings). To calculate the LI ratios, a blank correction was carried out and the measured intensities for $^{202}Hg^+$ were used to correct for Hg contribution to the apparent intensity for the $^{204}Pb^+$ isotope. The raw sample ratios thus obtained were corrected for Tl-based mass discrimination following Russel’s Law[6], based on replicate measurements of the NIST SRM 981 common lead standard (commonly accepted ratio values from Galer and Abouchami (1998)). Errors and error correlation were calculated after Ludwig (1980): the 2□ uncertainties were better than ca. 0.01% for ratios to ^{206}Pb and ca. 0.01-0.03% – up to 0.078% for extremely low lead samples – for ratios to ^{204}Pb.

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[6] $R_{true} = R_{observed} * (m_1/m_2)^\wedge\beta$, where m_1 and m_2 are the masses of the isotopes of interest for the ratio R and ・ is the mass bias factor (see, e.g., Belshaw *et al*., 1998).

Due to a dilution error for the first batch of analysed samples (too low Tl NIST concentrations in FE-11 to FE-29), the ratios for this first batch were calculated using a standard bracketing method, rather than a Tl-based mass discrimination correction following Russel’s Law (cfr. Rademakers *et al*. 2018). For the second batch of samples (K-1 through K-26), the relative difference between the mass-bias corrected values and the standard bracketing corrected values has been verified to be smaller than the relative measurement error. This signals good agreement between both calculation methods and allows the integration of both datasets.

Overview of samples analysed

Table 1: Overview of ingots analysed

Type	Site	Unit	Cultural/Chronological Attribution	Absolute dating associated	Museum ID	Lab number	Excavation Ref.
Ingot	Luano river	Survey	(pre-8e s.)	-	Luano_25_7_81_1	FE-11	-
HIH (large)	Kikulu	Burial 20	Kabambian A	795 +/- 65 (Hv-7505); 530 +/- 50 (Hv-8269)	KUL_T20_U1	FE-12	de Maret 1992: 78
HIH (large)	Kikulu	Burial 15	Kabambian A	665 +/- 30 bp (Poz-108342)	KUL_T15_U1_S	FE-13	de Maret 1992: 73
HIH (medium)	Kikulu	Burial 10	Kabambian A	-	KUL_T10_U2_L_1	FE-14	de Maret 1992: 66
HIH (small)	Kikulu	Burial 10	Kabambian A	-	KUL_T10_U2_S_1	FE-15	de Maret 1992: 66
HIH (medium)	Sanga	Burial 11	Kabambian A	-	Sanga_79293	FE-16	Nenquin 1963
HX	Sanga	Trench (occupation layer?)	(Transition to Kabambian/Kabambian A)	-	Sanga_79647_4pack_bottom	FE-17	Nenquin 1963
HX	Sanga	Trench (occupation layer?)	(Transition to Kabambian/Kabambian A)	-	Sanga_79647_4pack_middle	FE-18	Nenquin 1963
HX	Sanga	Trench (occupation layer?)	(Transition to Kabambian/Kabambian A)	-	Sanga_79647_4pack_top	FE-19	Nenquin 1963
HX	Sanga	Trench (occupation layer?)	(Transition to Kabambian/Kabambian A)	-	Sanga_79647_2pack_bottom_1	FE-20	Nenquin 1963
HX	Sanga	Trench (occupation layer?)	(Transition to Kabambian/Kabambian A)	-	Sanga_79647_2pack_top_1	FE-21	Nenquin 1963
HX	Sanga	Burial 32	Transition to Kabambian A	-	Sanga_79579_L_1	FE-22	Nenquin 1963
HX	Sanga	Burial 32	Transition to Kabambian A	-	Sanga_79579_M_1	FE-23	Nenquin 1963
HX	Sanga	Burial 32	Transition to Kabambian A	-	Sanga_79579_S_1	FE-24	Nenquin 1963
HH (-HX)	Sanga	Burial 6	Kabambian A	-	Sanga_79573_A_1	FE-25	Nenquin 1963
HX (-HH)	Sanga	Burial 6	Kabambian A	-	Sanga_79573_L_1	FE-26	Nenquin 1963
HH (large)	Sanga	Burial 4	Kabambian A	-	Sanga_79265_A_2	FE-27	Nenquin 1963
HH (-HX)	Sanga	Burial 4	Kabambian A	-	Sanga_79265_F_1	FE-28	Nenquin 1963
HH (large)	Sanga	Burial 4	Kabambian A	-	Sanga_79265_G_1	FE-29	Nenquin 1963
HH (large)	Sanga	Burial 4	Kabambian A	-	Sanga_79265_H_1	K-1	Nenquin 1963
HH (large)	Sanga	Burial 4	Kabambian A	-	Sanga_79265_I_1	K-2	Nenquin 1963
HH (large)	Sanga	Burial 55	(Transition to) Kabambian A?	-	Sanga_79568_A_bottom_3	K-3	Nenquin 1963
HH (large)	Sanga	Burial 55	(Transition to) Kabambian A?	-	Sanga_79568_A_top_1	K-4	Nenquin 1963
HH (large)	Sanga	Burial 55	(Transition to) Kabambian A?	-	Sanga_79568_B_bottom_3	K-5	Nenquin 1963
HH (large)	Sanga	Burial 55	(Transition to) Kabambian A?	-	Sanga_79568_B_top_2	K-6	Nenquin 1963
HH (large)	Kamilamba	Burial 5	Transition to Kabambian A	470 +/- 120 bp (Hv-7501)	KMI_T5_U1_1	K-7	de Maret 1992: 66
HH (small)	Sanga	Burial 42	Kabambian B	-	Sanga_79589_B_1	K-8	Nenquin 1963
HH (medium)	Sanga	Burial 42	Kabambian B	-	Sanga_79589_C_1	K-9	Nenquin 1963
HH (small)	Sanga	Burial 42	Kabambian B	-	Sanga_79590_A_1	K-10	Nenquin 1963

HH (small)	Sanga	Burial 42	Kabambian B	-	Sanga_79590_E_1	K-11	Nenquin 1963
HH (small)	Katongo	Burial 4	Kabambian B	430 +/- 160 bp (Hv-6618)	KTG_T4_U2F_A_1	K-12	de Maret 1985: 211
HH (small)	Katongo	Burial 4	Kabambian B	430 +/- 160 bp (Hv-6618)	KTG_T4_U2F_B_1	K-13	de Maret 1985: 211
HH (small)	Katongo	Burial 4	Kabambian B	430 +/- 160 bp (Hv-6618)	KTG_T4_U2F_E_1	K-14	de Maret 1985: 211
HH (small)	Katongo	Burial 4	Kabambian B	430 +/- 160 bp (Hv-6618)	KTG_T4_U2F_F_1	K-15	de Maret 1985: 211
HH (small)	Katongo	Burial 4	Kabambian B	430 +/- 160 bp (Hv-6618)	KTG_T4_U2F_G_1	K-16	de Maret 1985: 211
HIH	Bolela	Survey	(ca. 9th-14th c.)	-	47763_1	K-17	-
HIH	Bolela	Survey	(ca. 9th-14th c.)	-	47764_1	K-18	-
HH (medium)	Katongo	Burial 4	Kabambian B	430 +/- 160 bp (Hv-6618)	KTG_74_T4_U1_1	K-19	de Maret 1985: 211
HH (small)	Malemba-Nkulu	Burial 8ter	Kabambian B	-	MAK_T8ter_U1_1	K-20	de Maret 1992: 103
HH (small)	Malemba-Nkulu	Burial 13	Kabambian B	375 +/- 40 (Hv-8495)	MAK_T13_U1_1	K-21	de Maret 1992: 113
HH (small)	Malemba-Nkulu	Burial 35	Kabambian B	860 +/- 55 (Hv-8497)* ; 955 +/- 30 (Poz-108343)*	MAK_T35_U1_A_1	K-22	de Maret 1992: 143
HH (small)	Malemba-Nkulu	Burial 35	Kabambian B	860 +/- 55 (Hv-8497)* ; 955 +/- 30 (Poz-108343)*	MAK_T35_U1_B_1	K-23	de Maret 1992: 143
HH (very small)	Katongo	Burial 8	Kabambian B	250 +/- 85 (Hv-6621)	KTG_74_T8_U1_1	K-24	de Maret 1985: 221
HH (very small)	Moero	Survey	(ca. 16th-18th c.)	-	Moero_47725_A	K-25	-
HH (very small)	Moero	Survey	(ca. 16th-18th c.)	-	Moero_47725_B	K-26	-

Legend () = hypotheses through comparison or relative chronology

* radiocarbon dates discarded

Croisettes sizes: Large > 3 cm > Medium > 2 > Small > 1 > Very small.

Lead isotope data

Tables

Table 2: Calculated LI ratios of analysed ingots

Site	206Pb/204Pb	207Pb/204Pb	208Pb/204Pb	207Pb/206Pb	208Pb/206Pb	208Pb/207Pb	σ 206Pb/204Pb	σ 207Pb/204Pb	σ 208Pb/204Pb	σ 207Pb/206Pb	σ 208Pb/206Pb	σ 208Pb/207Pb
Luano river	22.324	15.940	38.189	0.714	1.711	2.396	0.005	0.004	0.012	0.000	0.000	0.000
Kikulu	18.400	15.687	38.452	0.853	2.090	2.451	0.005	0.004	0.012	0.000	0.000	0.000
Kikulu	19.517	15.779	38.594	0.808	1.977	2.446	0.007	0.007	0.018	0.000	0.000	0.000
Kikulu	21.465	15.851	38.823	0.738	1.809	2.449	0.011	0.009	0.022	0.000	0.000	0.000
Kikulu	18.616	15.662	38.335	0.841	2.059	2.448	0.004	0.004	0.010	0.000	0.000	0.000
Sanga	21.060	15.840	38.773	0.752	1.841	2.448	0.014	0.010	0.025	0.000	0.000	0.000
Sanga	18.406	15.682	38.054	0.852	2.068	2.427	0.004	0.003	0.008	0.000	0.000	0.000
Sanga	18.984	15.702	38.414	0.827	2.023	2.446	0.013	0.010	0.025	0.000	0.000	0.000
Sanga	19.424	15.758	38.551	0.811	1.985	2.446	0.007	0.007	0.016	0.000	0.000	0.000
Sanga	18.168	15.648	38.125	0.861	2.098	2.437	0.006	0.005	0.012	0.000	0.000	0.000
Sanga	18.817	15.703	38.498	0.835	2.046	2.452	0.011	0.009	0.025	0.000	0.000	0.000
Sanga	18.929	15.705	38.363	0.830	2.027	2.443	0.006	0.006	0.013	0.000	0.000	0.000
Sanga	18.979	15.733	38.392	0.829	2.023	2.440	0.006	0.005	0.012	0.000	0.000	0.000
Sanga	18.870	15.735	38.508	0.834	2.041	2.447	0.006	0.005	0.013	0.000	0.000	0.000
Sanga	18.545	15.630	38.324	0.843	2.067	2.452	0.002	0.002	0.005	0.000	0.000	0.000
Sanga	19.408	15.764	38.542	0.812	1.986	2.445	0.005	0.005	0.011	0.000	0.000	0.000
Sanga	18.814	15.713	38.566	0.835	2.050	2.454	0.015	0.012	0.029	0.000	0.000	0.000
Sanga	30.988	16.442	39.460	0.531	1.273	2.400	0.020	0.011	0.027	0.000	0.000	0.000
Sanga	18.068	15.630	37.644	0.865	2.084	2.408	0.005	0.005	0.013	0.000	0.000	0.000
Sanga	18.070	15.637	37.646	0.865	2.083	2.408	0.002	0.002	0.006	0.000	0.000	0.000
Sanga	18.314	15.674	38.198	0.856	2.086	2.437	0.002	0.002	0.006	0.000	0.000	0.000
Sanga	19.242	15.754	38.361	0.819	1.994	2.435	0.002	0.002	0.005	0.000	0.000	0.000
Katongo	20.521	15.826	38.438	0.771	1.873	2.429	0.002	0.002	0.005	0.000	0.000	0.000
Katongo	19.760	15.786	39.377	0.799	1.993	2.494	0.005	0.004	0.012	0.000	0.000	0.000
Katongo	20.811	15.852	38.542	0.762	1.852	2.431	0.003	0.002	0.006	0.000	0.000	0.000
Katongo	22.041	15.902	39.887	0.722	1.810	2.508	0.005	0.004	0.010	0.000	0.000	0.000
Katongo	20.194	15.804	39.594	0.783	1.961	2.505	0.005	0.004	0.010	0.000	0.000	0.000
Bolela	18.878	15.689	38.850	0.831	2.058	2.476	0.002	0.002	0.006	0.000	0.000	0.000
Bolela	18.539	15.655	38.261	0.844	2.064	2.444	0.004	0.003	0.007	0.000	0.000	0.000
Katongo	20.327	15.770	38.766	0.776	1.907	2.458	0.002	0.002	0.005	0.000	0.000	0.000
Sanga	18.725	15.712	38.255	0.839	2.043	2.435	0.003	0.003	0.006	0.000	0.000	0.000

Malemba-Nkulu	20.919	15.860	38.711	0.758	1.851	2.441	0.002	0.002	0.005	0.000	0.000	0.000
Malemba-Nkulu	20.658	15.809	39.196	0.765	1.897	2.479	0.005	0.004	0.011	0.000	0.000	0.000
Malemba-Nkulu	20.663	15.844	38.580	0.767	1.867	2.435	0.002	0.002	0.005	0.000	0.000	0.000
Malemba-Nkulu	18.402	15.673	37.815	0.852	2.055	2.413	0.002	0.002	0.004	0.000	0.000	0.000
Katongo	20.816	15.834	39.269	0.761	1.887	2.480	0.004	0.003	0.009	0.000	0.000	0.000
Moero	21.295	15.873	39.417	0.745	1.851	2.483	0.005	0.004	0.011	0.000	0.000	0.000
Moero	19.699	15.783	39.347	0.801	1.998	2.493	0.003	0.003	0.008	0.000	0.000	0.000
Sanga	18.741	15.699	38.282	0.838	2.043	2.438	0.004	0.003	0.009	0.000	0.000	0.000
Sanga	18.180	15.678	38.150	0.862	2.099	2.433	0.003	0.002	0.007	0.000	0.000	0.000
Sanga	18.211	15.647	37.730	0.859	2.072	2.411	0.002	0.002	0.006	0.000	0.000	0.000
Sanga	18.305	15.655	37.860	0.855	2.068	2.418	0.004	0.004	0.010	0.000	0.000	0.000
Kamilamba	18.066	15.637	37.646	0.866	2.084	2.407	0.002	0.003	0.007	0.000	0.000	0.000
Sanga	21.494	15.888	39.315	0.739	1.829	2.475	0.013	0.010	0.023	0.000	0.000	0.000
Sanga	20.498	15.827	38.470	0.772	1.877	2.431	0.003	0.002	0.006	0.000	0.000	0.000

Plots

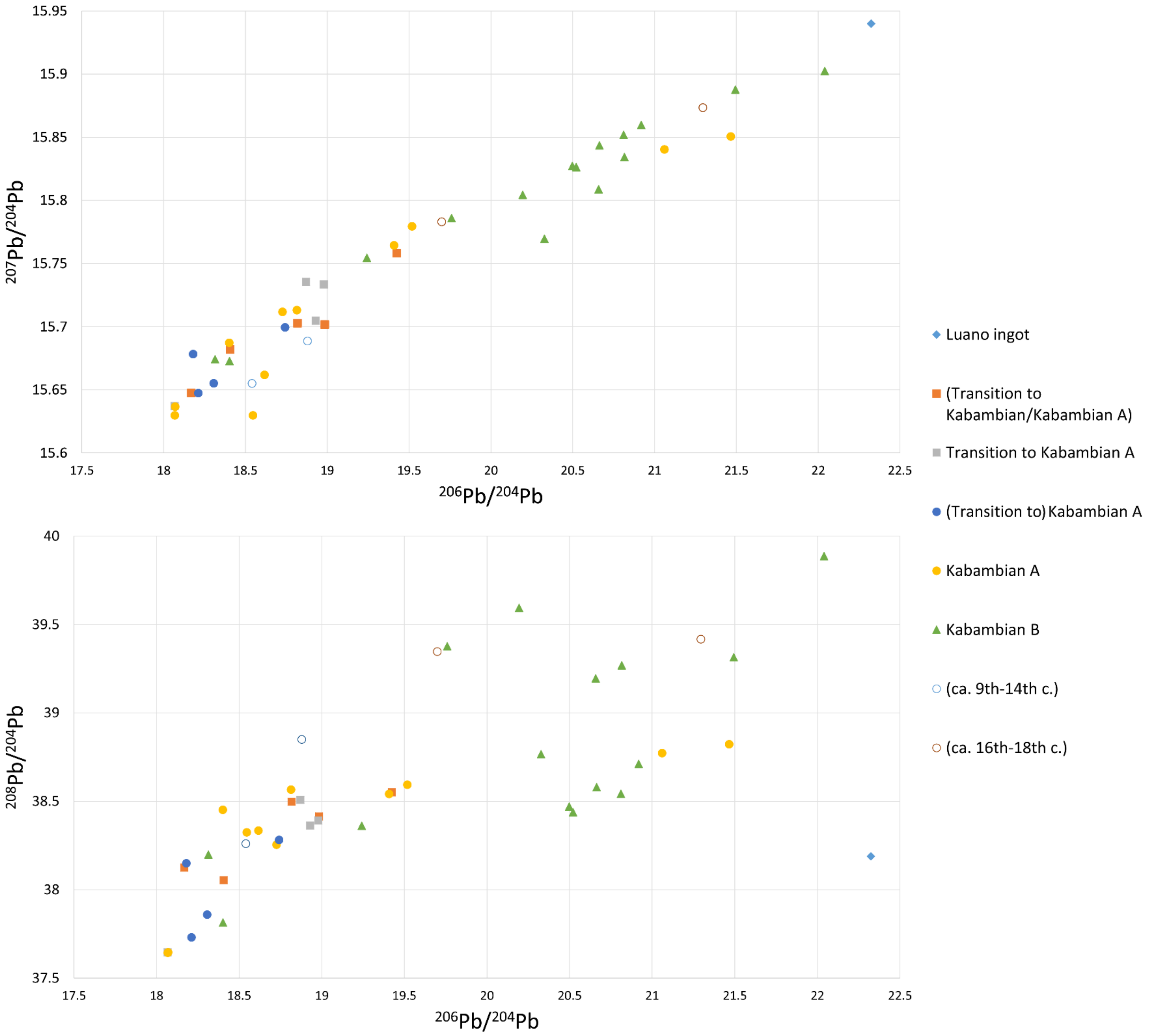

Figure 14: Croisette LI ratios, grouped by chronological period

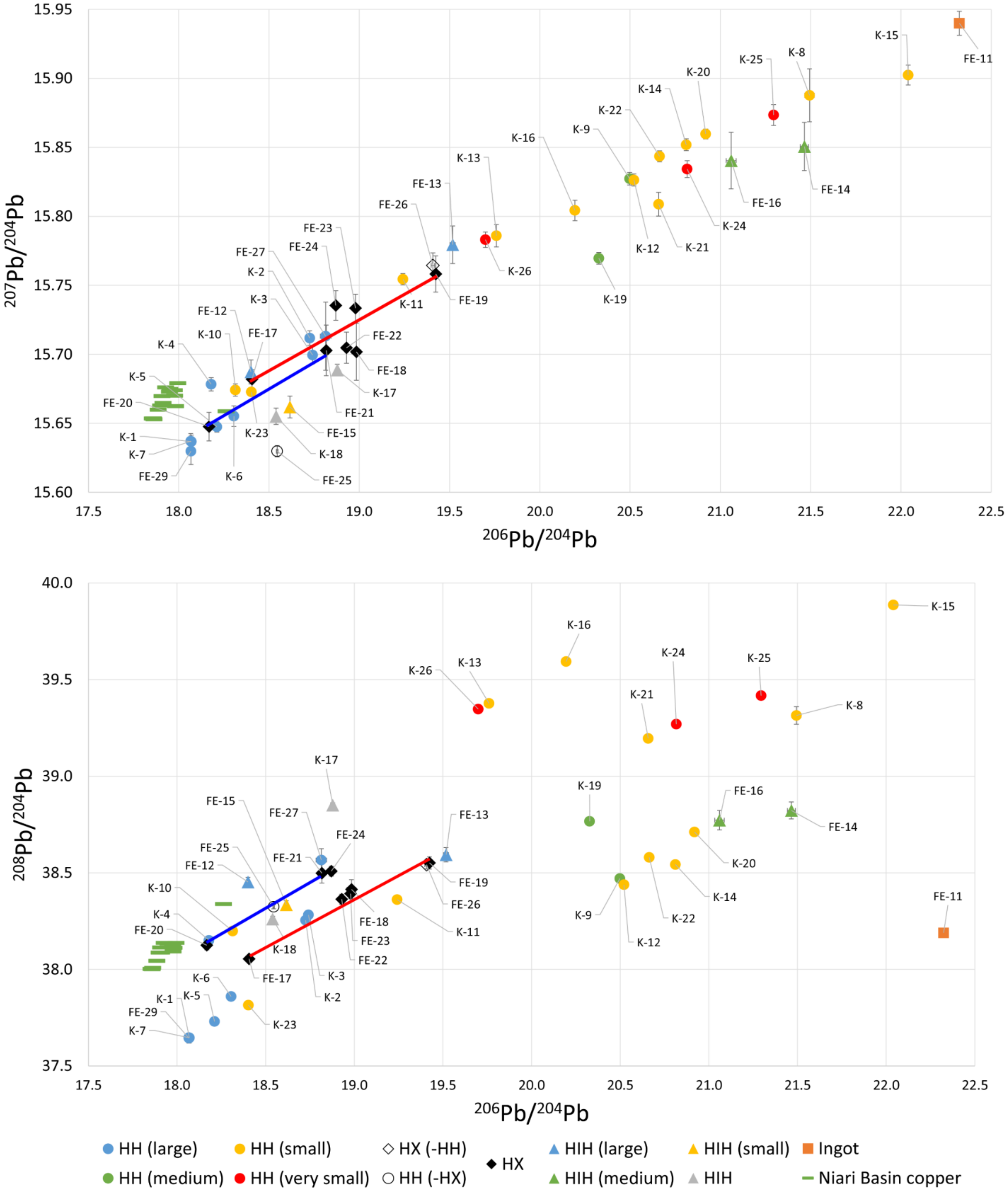

Figure 2: Croisette LI ratios. Lines indicate relation between croisettes from bundle in PO 0.0.79647 (red: four in bundle, blue: two in bundle)

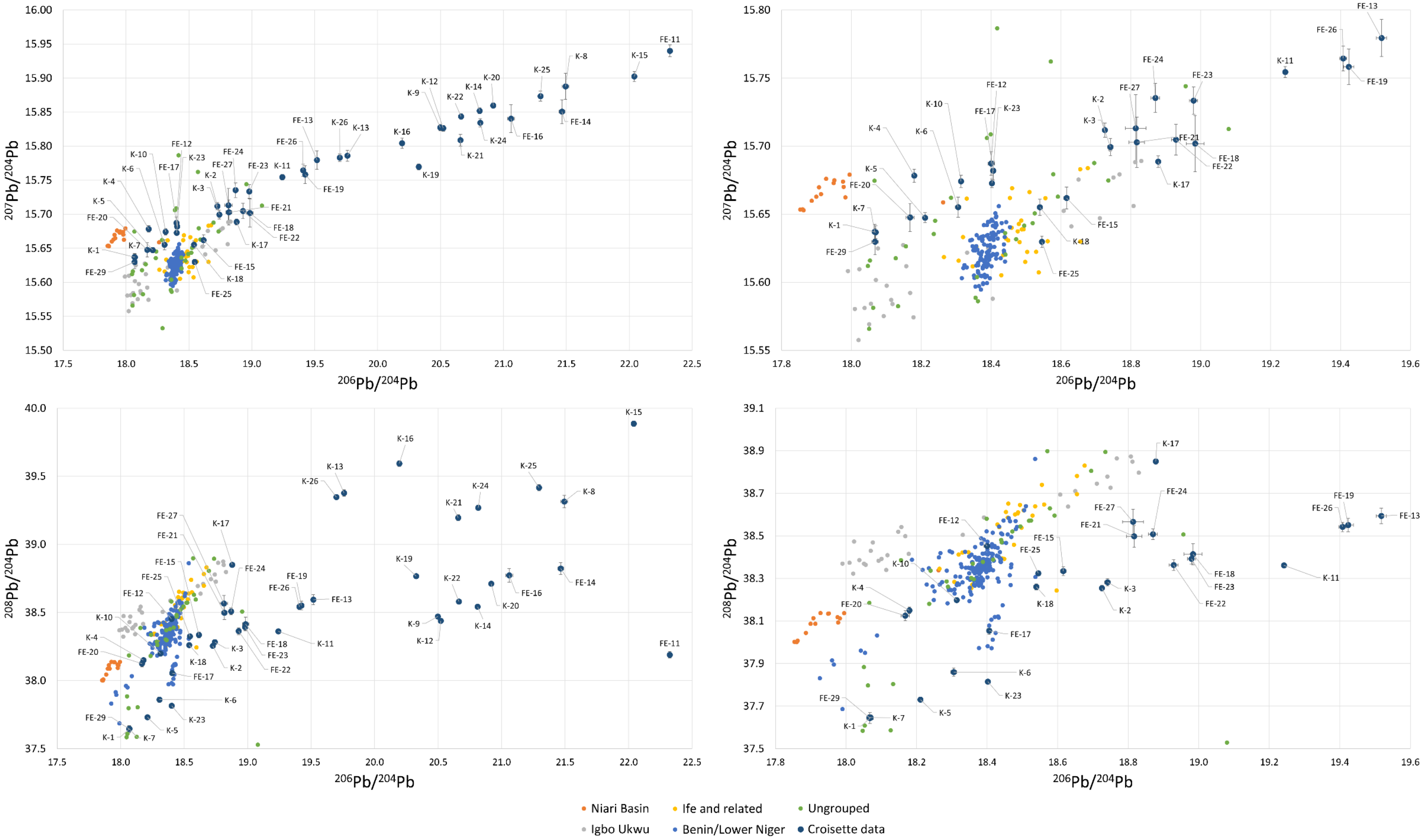

Figure 15: Croisette LI ratios compared to LI ratios of West-African copper alloys presented by Willet and Sayre (2006)

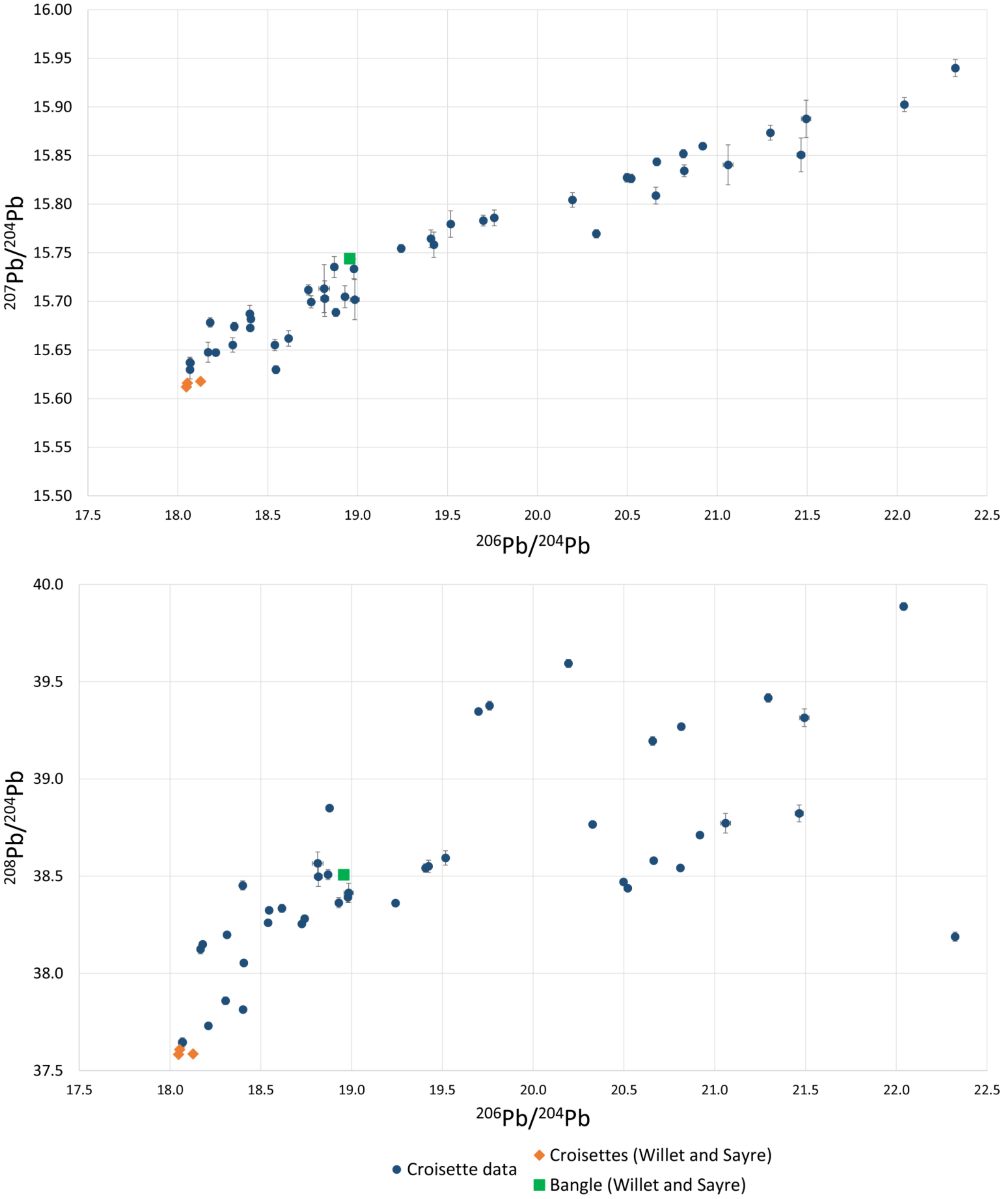

Figure 16: Croisette LI ratios compared to three croisettes and bangle published by Willet and Sayre (2006)

descriptive histograms and bi-plots of the elemental data

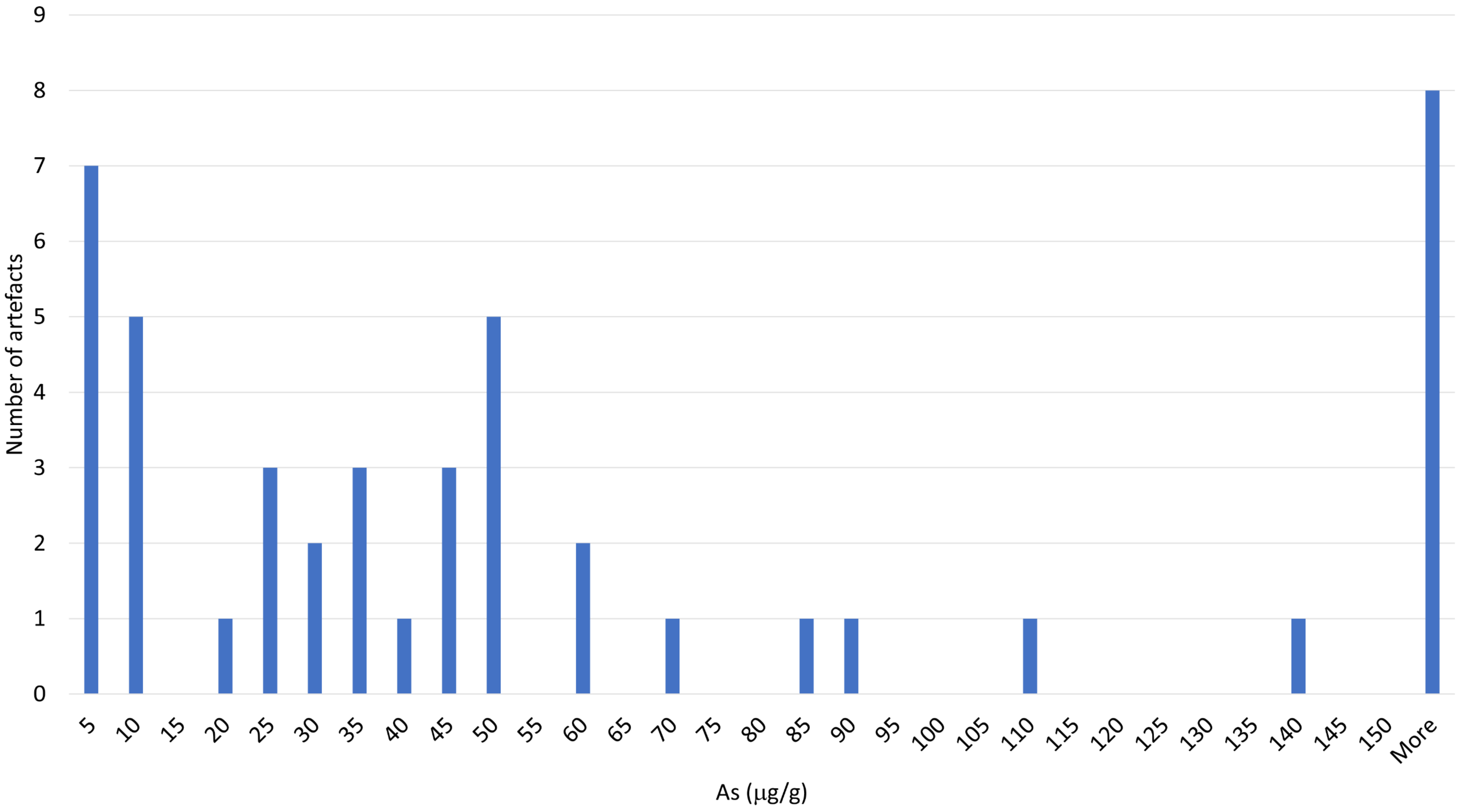

Figure 5: Presence of As in samples analysed

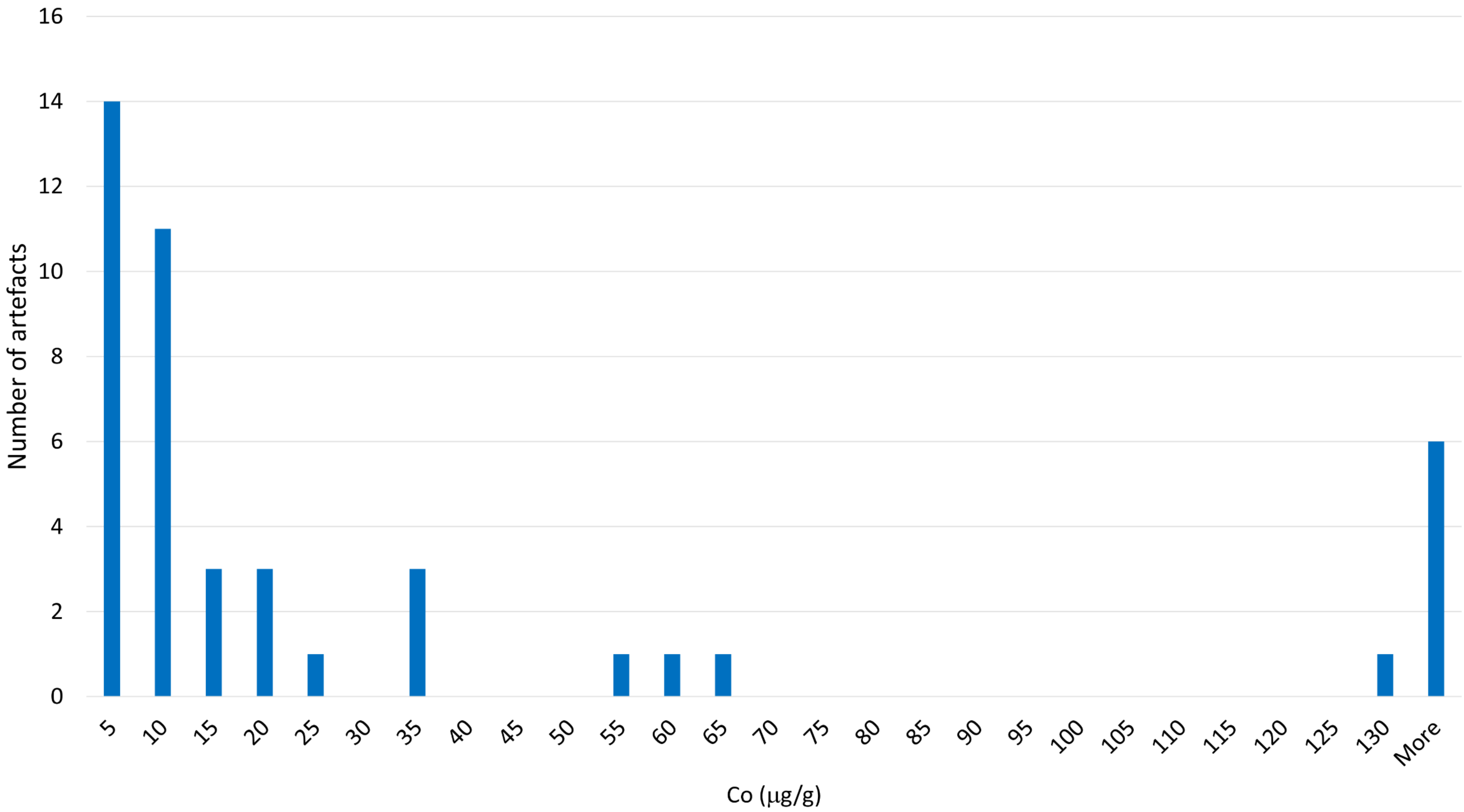

Figure 6: Presence of Co in samples analysed

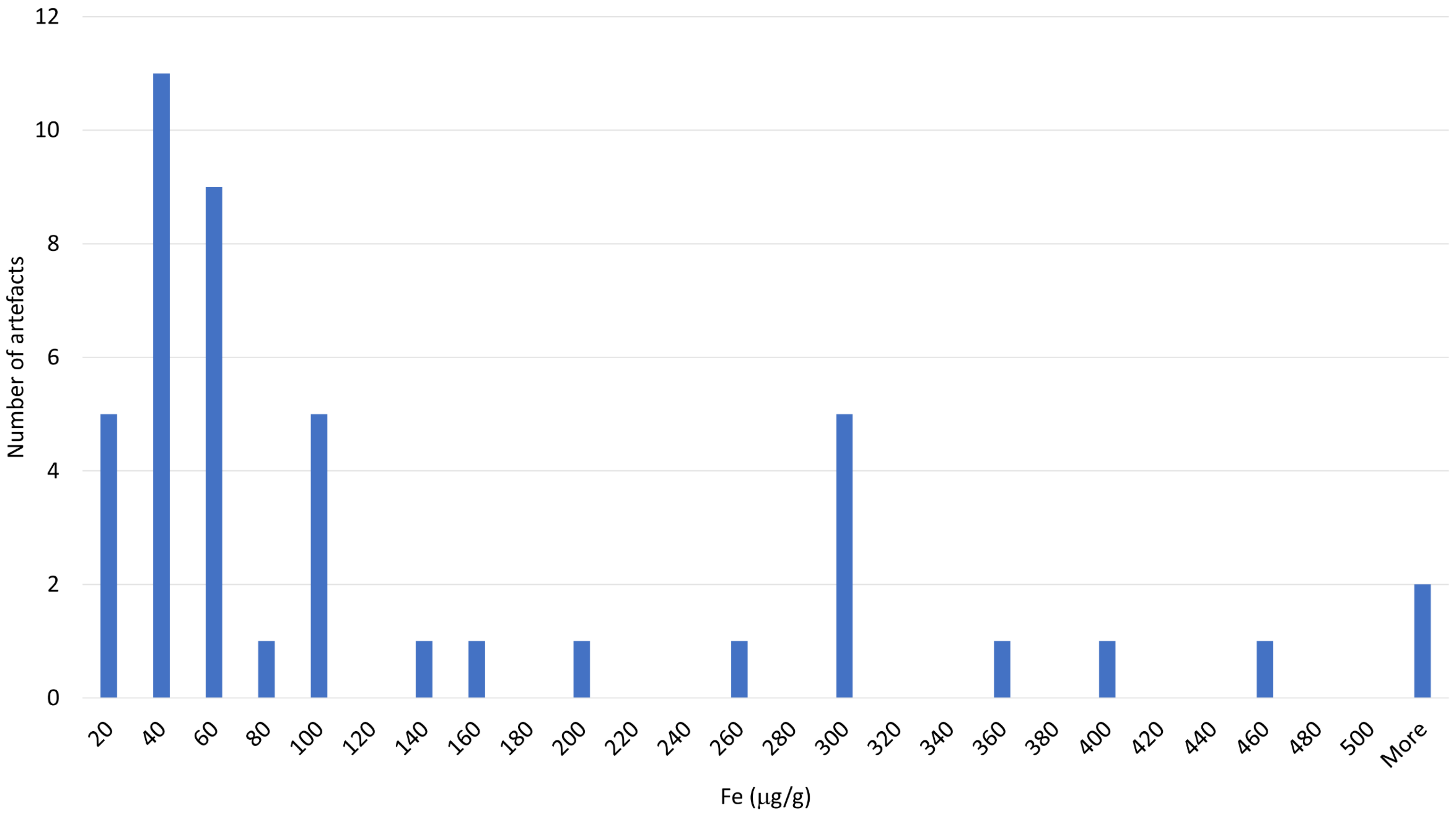

Figure 7: Presence of Fe in samples analysed

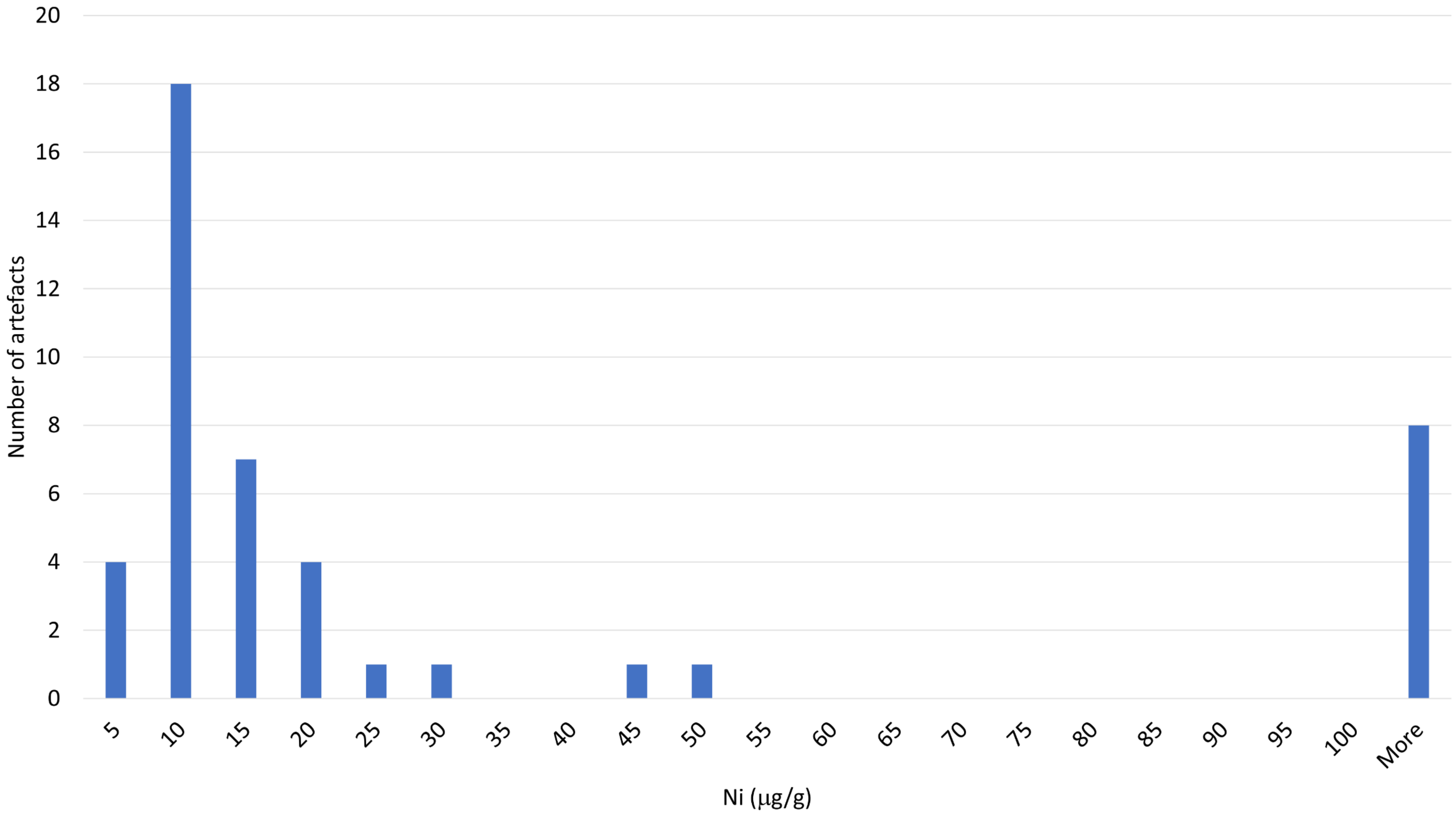

Figure 8: Presence of Ni in samples analysed

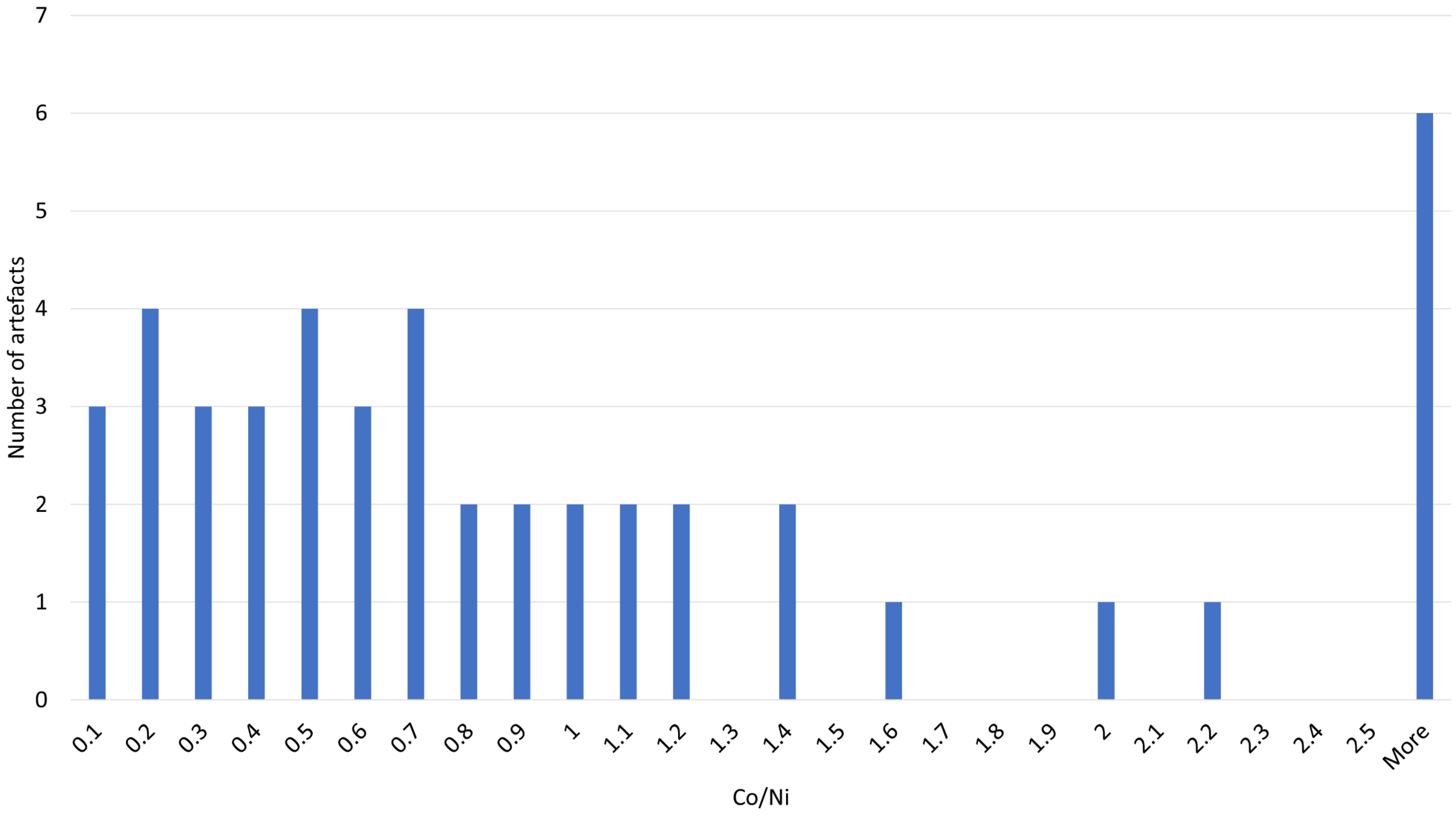

Figure 9: Ratio Co/Ni in samples analysed

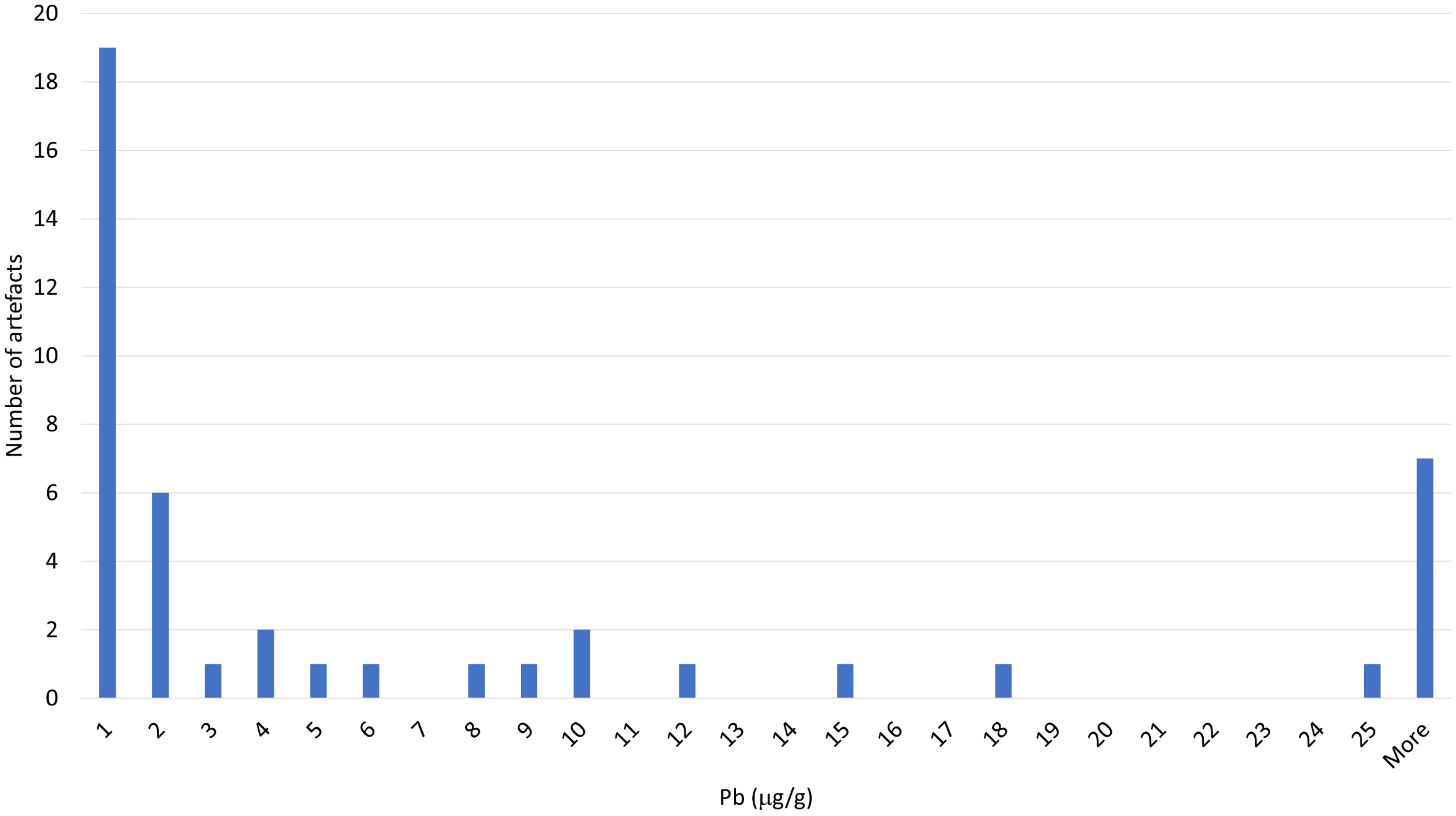

Figure 10: Presence of Pb in samples analysed

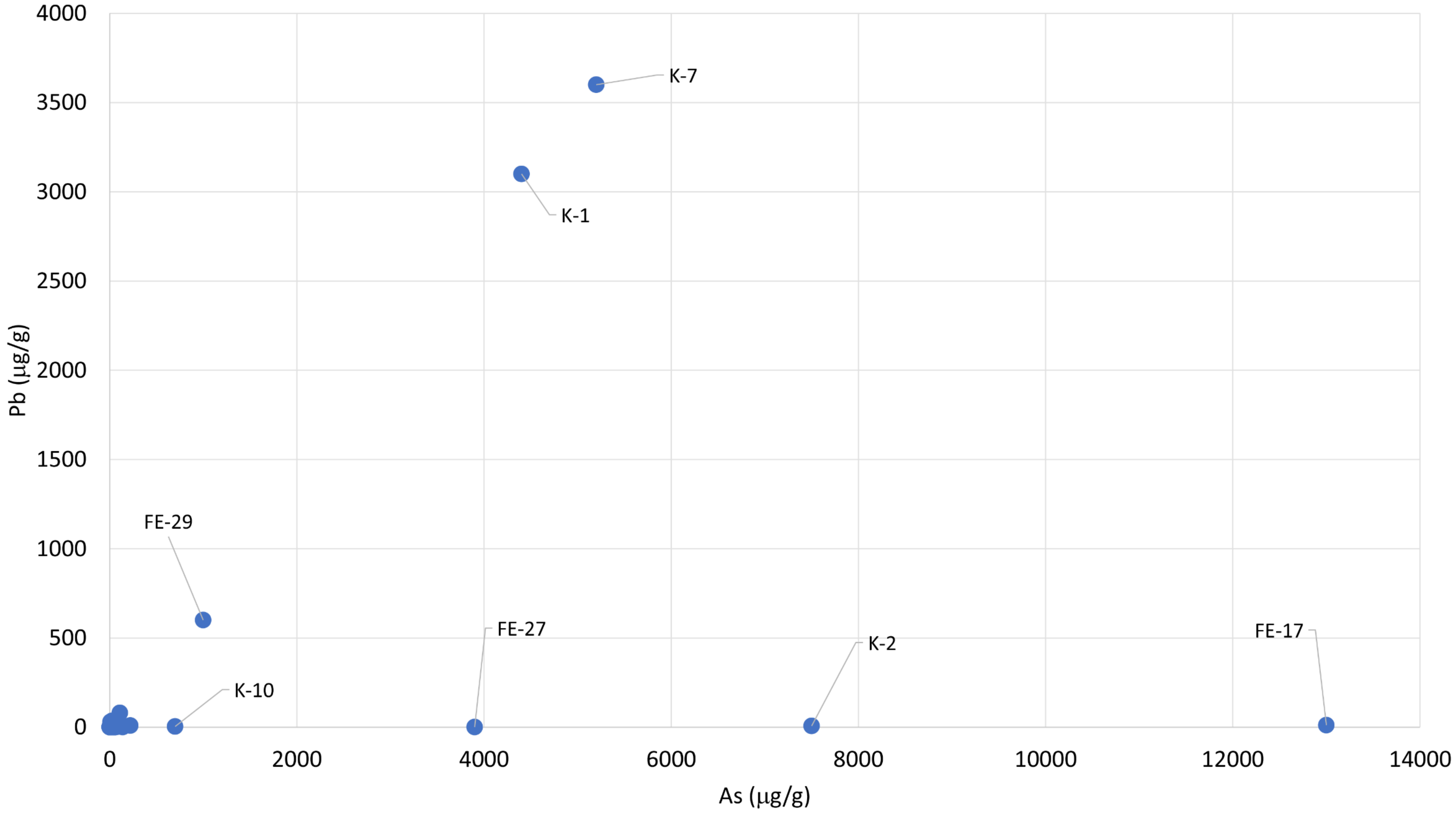

Figure 11: By-plot of Pb vs As in samples analysed

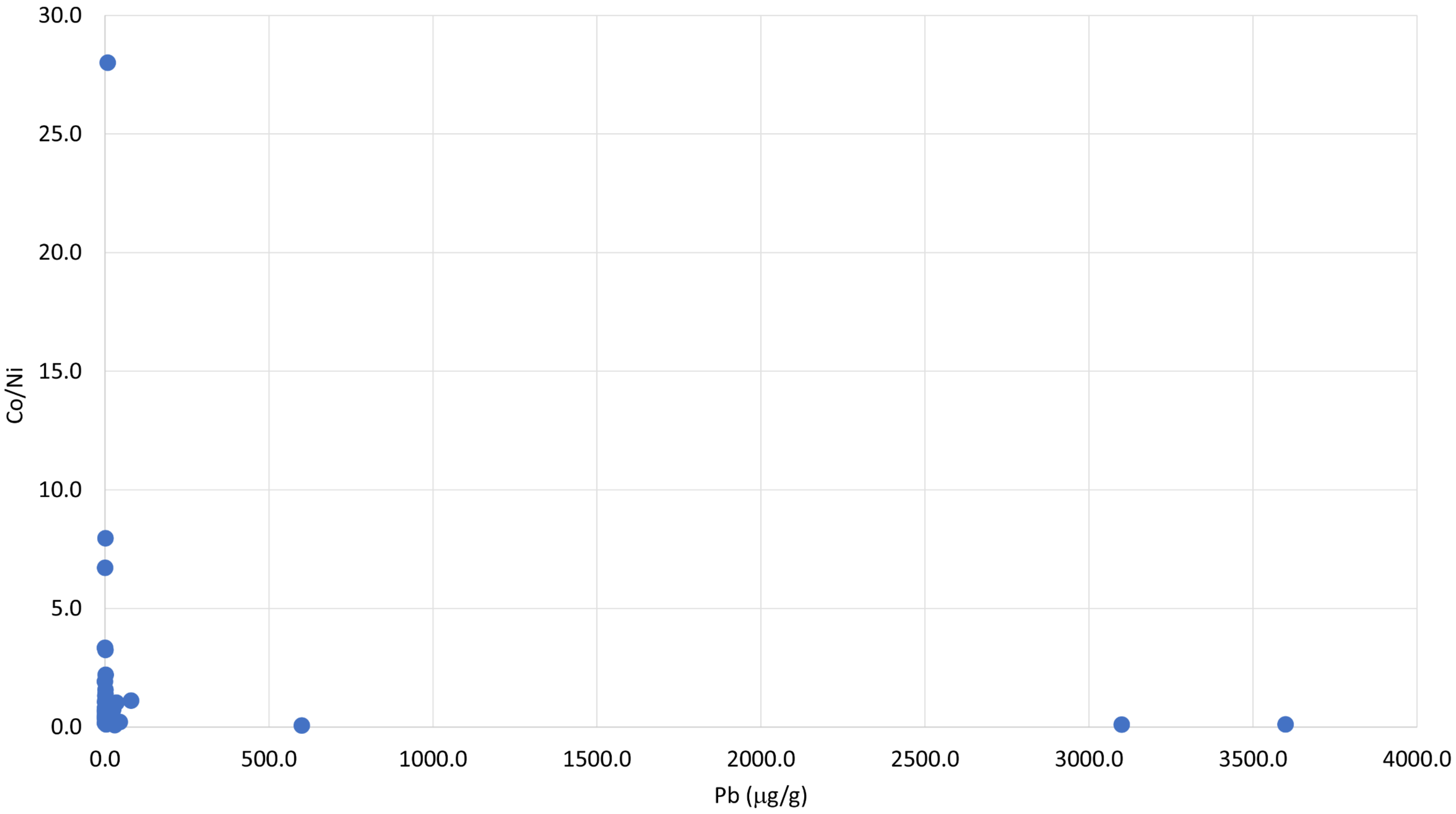

Figure 12: By-plot of Pb vs Co/Ni in samples analysed

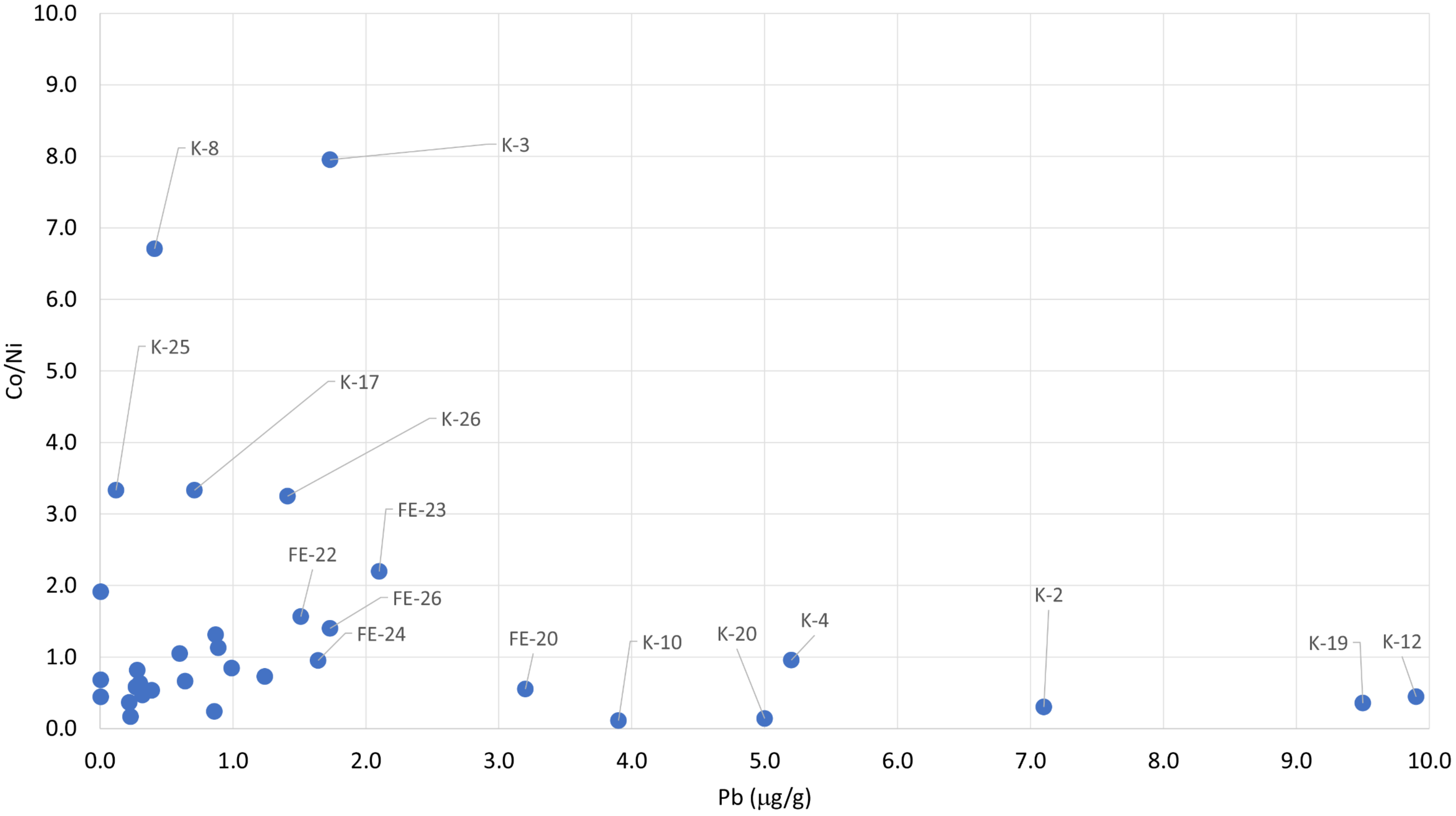

Figure 13: details of fig. 12, By-plot of Pb vs Co/Ni in samples analysed

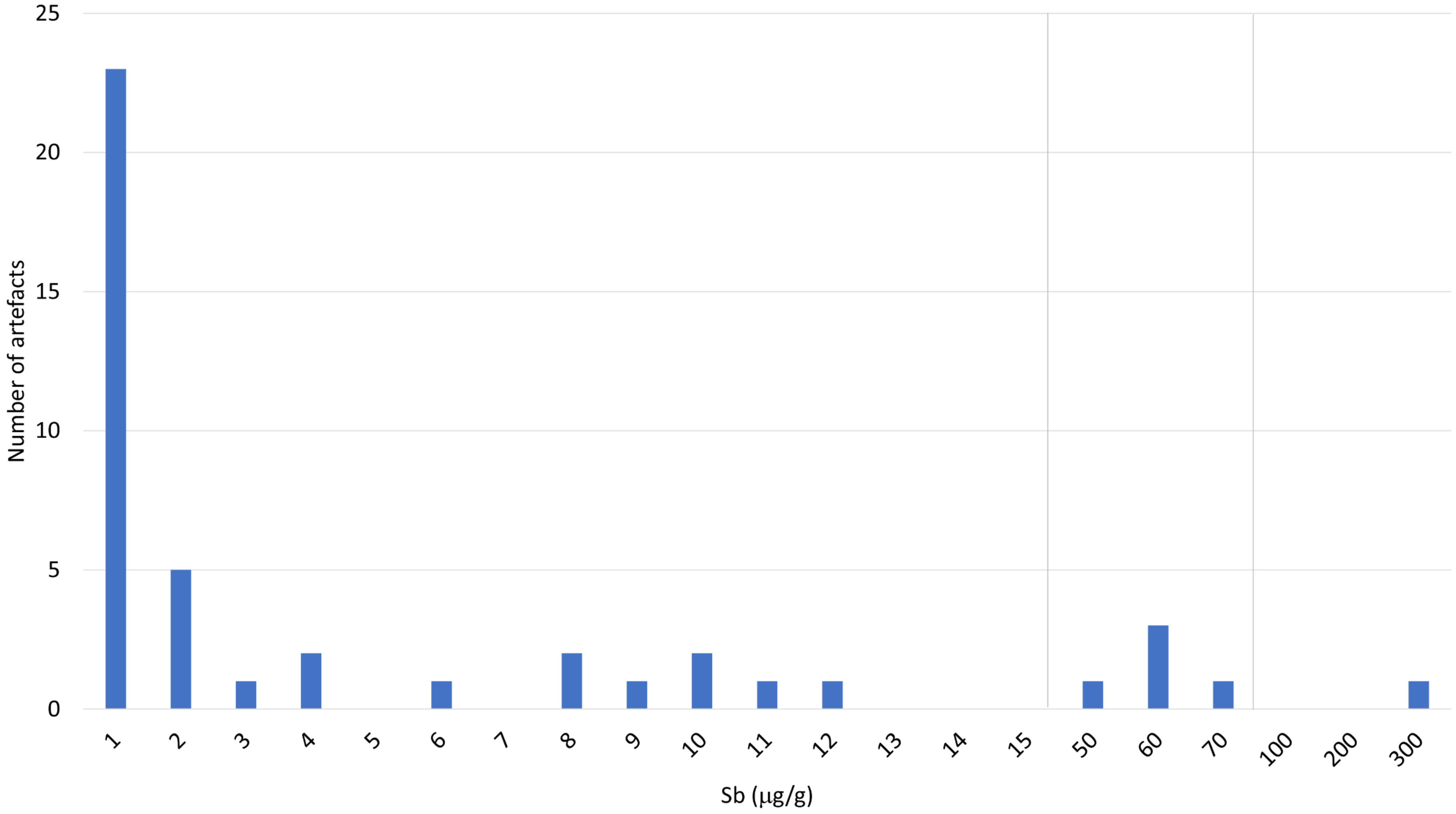

Figure 14: Presence of Sb in samples analysed

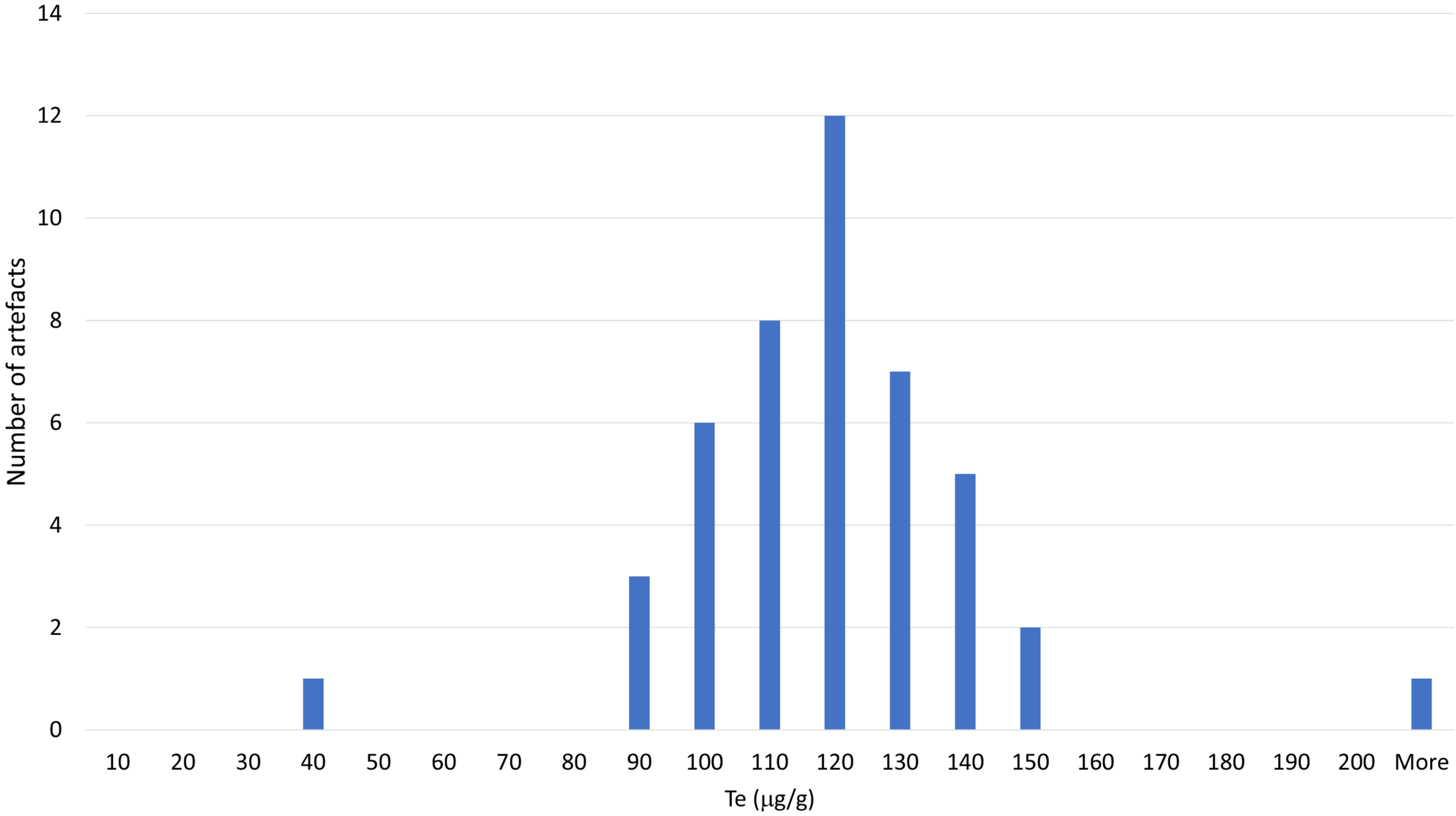

Figure 15: Presence of Te in samples analysed

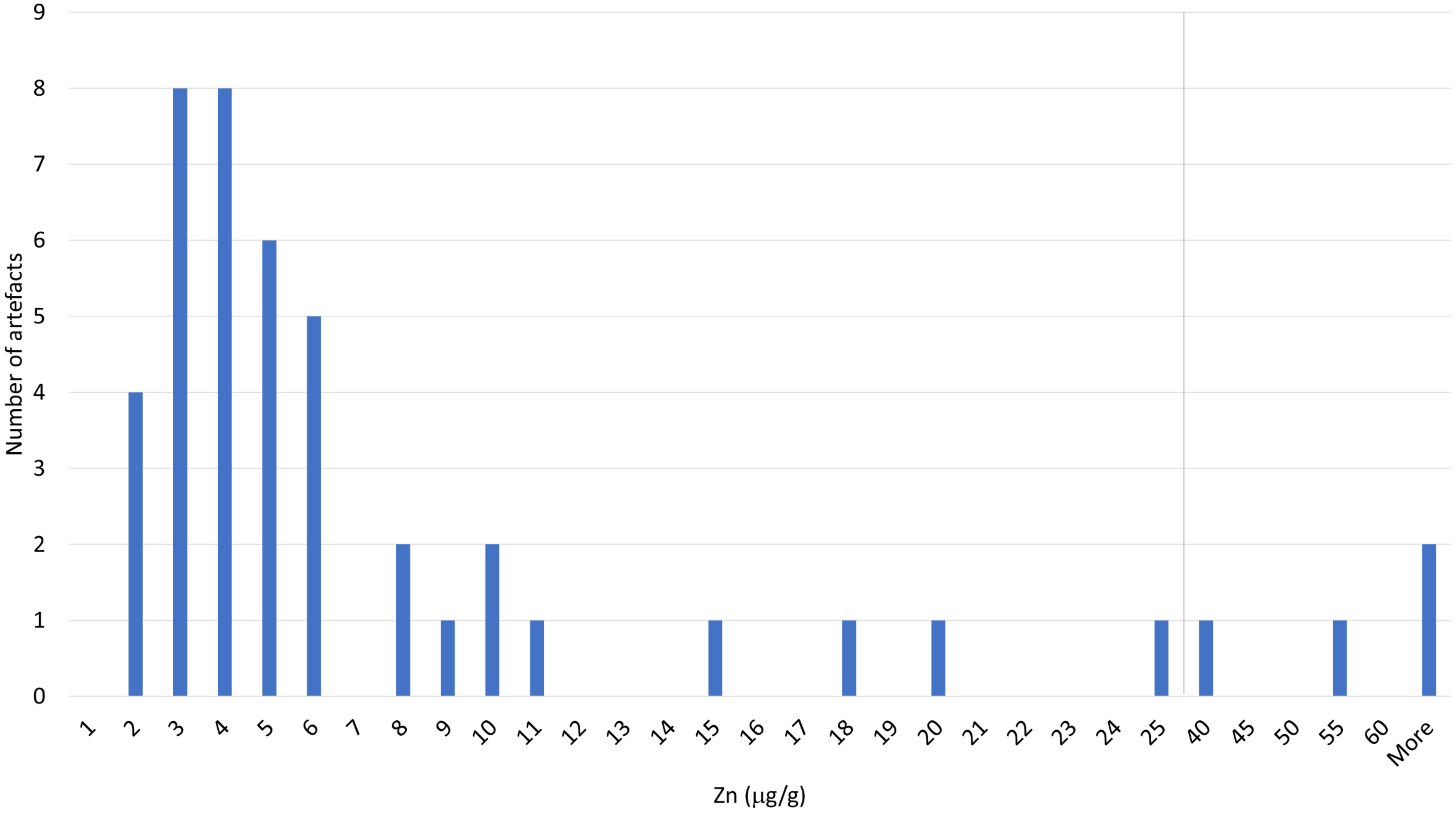

Figure 16: Presence of Zn in samples analysed

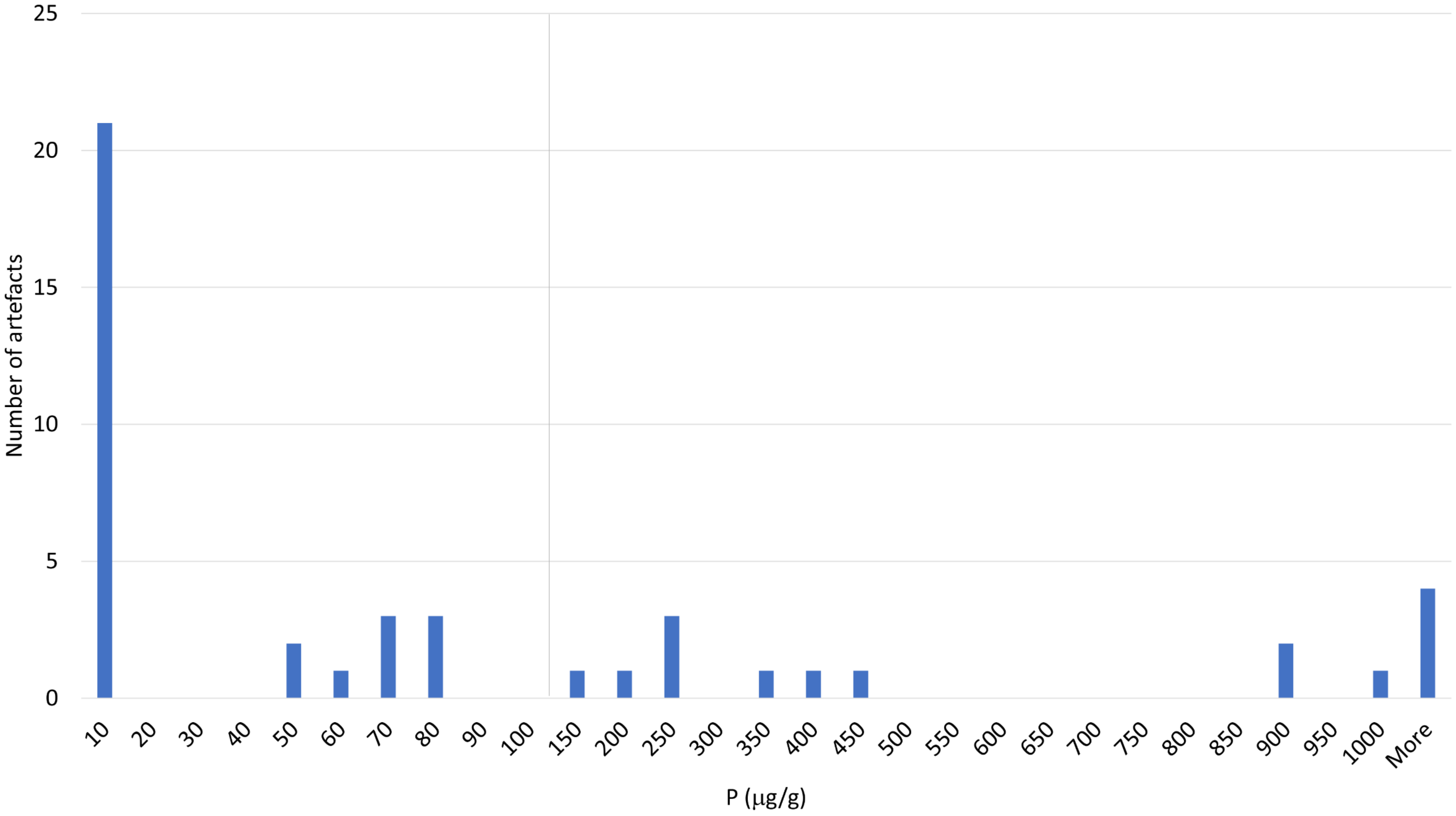

Figure 17: Presence of P in samples analysed

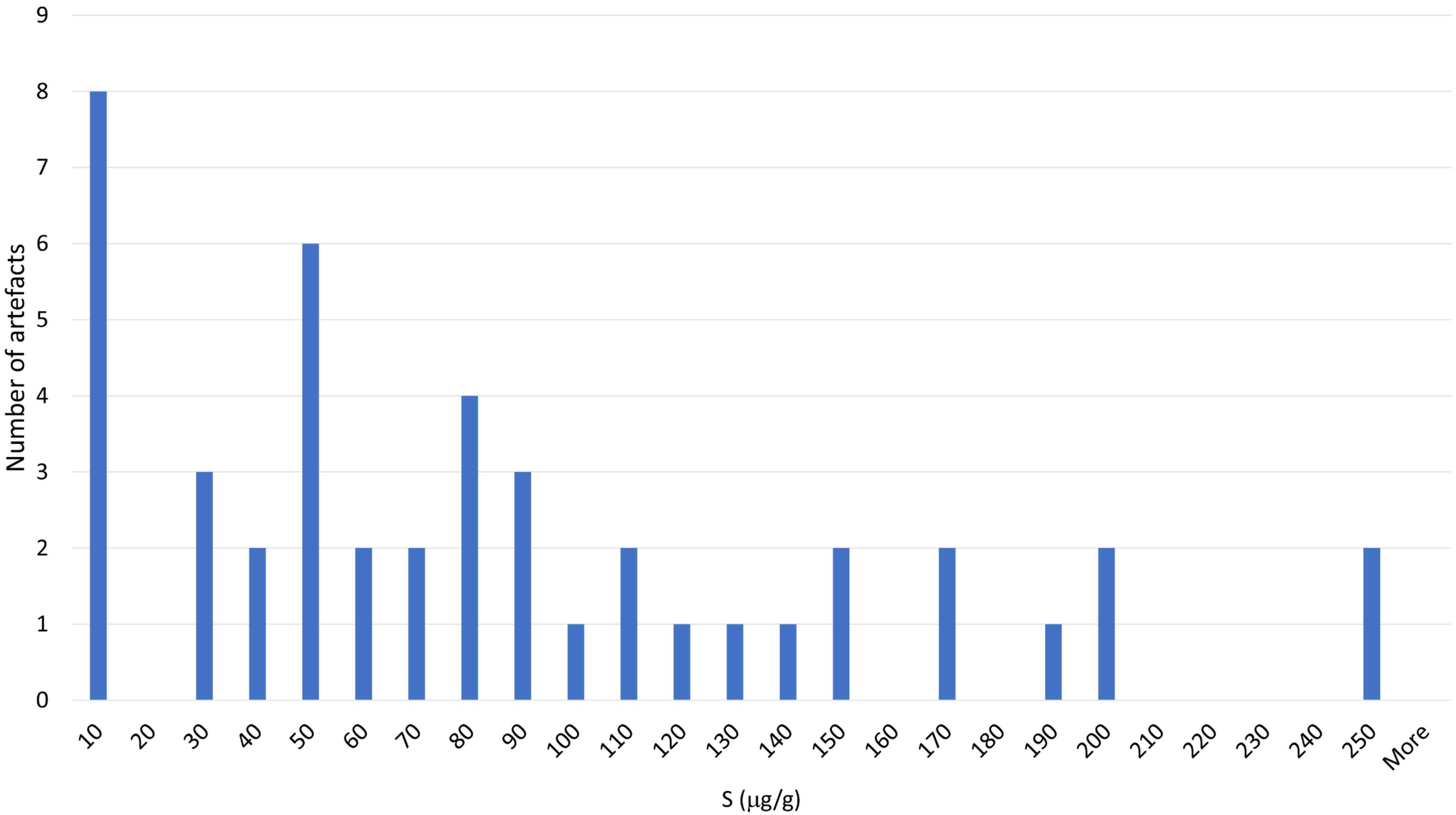

Figure 18: Presence of S in samples analysed

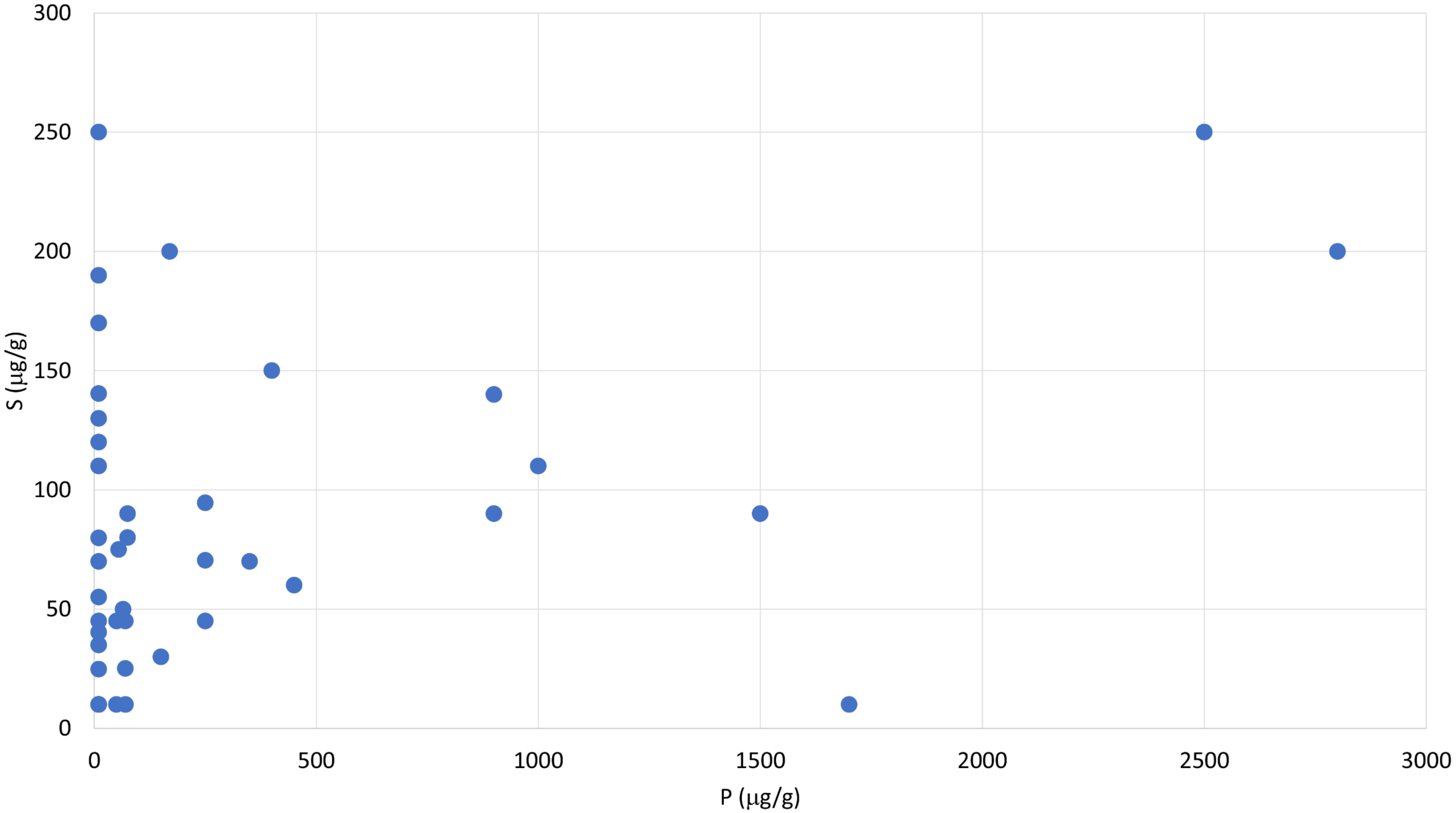

Figure 19: By-plot of S vs P in samples analysed

Chemical data

Tables

Table 3: Elemental composition of analysed ingots. Top rows: ICP-OES results, bottom rows: ICP-MS results ("<x" refers to values below element-specific detection limit "x" – zero values (ICP-MS) refer to concentrations below 0.1 µg/g – empty cells: W, Th, U were not measured by ICP-OES; Au, P, S, Se and Te were not measured by ICP-MS).

Type - Site	**Lab number**	**Sample weight (g)**	**Cu**	**Ag**	**As**	**Au**	**Ba**	**Bi**	**Co**	**Cr**	**Fe**	**Mg**	**Mn**	**Ni**	**P**	**Pb**	**S**	**Sb**	**Se**	**Sn**	**Te**	**Ti**	**Zn**	**W**	**Th**	**U**
			%	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g	µg/g
Lingot - Luano river	FE-11	0.0463	96.1	<5	70	<5	<5	<35	690	3	260	45	2	25	<50	<15	125	<10	<30	<20	145	18	<10			
				1.8	25		5.3	5	715	3	250	40	2	23		8.7		2		1		17	3.4	2.5	0.06	0.04
HIH (grande) - Kikulu	FE-12	0.0688	96.5	<5	55	<5	<5	<35	10	<3	30	<10	<3	11	<50	<15	70	<10	<30	<20	120	<10	<10			
				0	30		1	0	10.8	1	30	<1	0	7.8		0.9		0		0		1	2.5	1.1	0	0
HIH (grande) - Kikulu	FE-13	0.0760	97.5	<5	30	0	<5	<35	11	<3	40	<10	1	12	75	<15	90	<10	<30	<20	145	<10	<10			
				0.1	18		1.7	0	12	1	35	<1	0	9.5		0.9		0		0		1	2.7	1	0	0.01
HIH (moyenne) - Kikulu	FE-14	0.0641	97.5	<5	50	0	<5	<35	3	<3	35	<10	<3	7	<50	<15	40	<10	<30	<20	145	<10	<10			
				0	35		0.3	0	3.1	0	35	<1	0	4.9		0.3		0		0		0	3.6	0.6	0	0
HIH (petite) - Kikulu	FE-15	0.0486	97.0	<5	60	0	<5	<35	<5	<3	50	<10	<3	10	170	<15	210	<10	<30	<20	135	<10	<10			
				0	40		2.7	1	4.3	1	45	<1	0	4.1		0.6		0		1		3	3.6	1.6	0	0
HIH (moyenne) - Sanga	FE-16	0.0469	97.4	<5	60	1	<5	<35	<5	<3	<25	<10	<3	13	<50	<15	70	<10	<30	<20	140	<10	<10			
				0	40		0.2	0	1.2	1	20	<1	0	7.2		0.2		0		0		4	2.5	1.1	0	0
HX - Sanga	FE-17	0.044	94.5	15	13000	0	<5	<35	<5	<3	105	<10	<3	<5	<50	<15	170	65	<30	<20	95	4	9			
FE-17	FE-17	FE-17		8	12000		1.2	5	0.7	2	100	<1	0	2.7		11		60		1		7	9.5	1.9	0	0.03
HX - Sanga	FE-18	0.0365	96.4	<5	135	<5	<5	<35	4	2	90	<10	1	7	<50	<15	80	<10	<30	<20	115	<10	<10			
FE-18	FE-18	FE-18		1.4	85		0.9	0	3.1	2	85	3	1	5.8		0.4		1		0		1	4.9	2.1	0	0
HX - Sanga	FE-19	0.0576	96.6	<5	55	<5	<5	<35	4	2	70	<10	<3	11	75	<15	80	<10	<30	<20	130	<10	<10			
FE-19	FE-19	FE-19		0.1	25		3	0	4.9	2	60	<1	0	7.4		0.6		0		0		3	3.4	2.4	0	0.02
HX - Sanga	FE-20	0.0450	96.5	<5	85	0	<5	<35	7	<3	<25	<10	<3	15	<50	<15	<50	<10	<30	<20	115	<10	<10			
FE-20	FE-20	FE-20		0	50		1.6	0	6.8	1	20	<1	0	12.3		3.2		0		0		0	5.9	0.8	0	0.02
HX - Sanga	FE-21	0.0230	97.4	<5	<10	2	<5	<35	4	<3	<25	<10	<3	14	70	<15	70	<10	<30	<20	120	<10	9			
FE-21	FE-21	FE-21		0.1	9		1.2	0	5.1	1	20	<1	0	10.9		0.3		0		4		0	8.7	0.7	0	0.02
HX - Sanga	FE-22	0.0509	92.6	<5	40	3	40	<35	8	<3	430	55	6	12	1500	<15	95	<10	<30	<20	115	14	<10			
FE-22	FE-22	FE-22		0.1	20		36	0	9.7	1	410	55	6	6.2		1.5		0		0		5	5.7	0.9	0.13	0.11
HX - Sanga	FE-23	0.0453	94.4	<5	<10	0	11	<35	19	<3	330	40	4	15	890	<15	90	<10	<30	<20	125	6	<10			

FE-23	FE-23	FE-23		0	5		9.4	0	19.8	1	310	45	3	9.1		2.1		0		0		5	4.8	0.9	0.08	0.1
HX - Sanga	FE-24	0.0616	94.9	<5	<10	1	40	<35	9	<3	320	45	5	14	1700	<15	65	<10	<30	<20	120	<70	7			
FE-24	FE-24	FE-24		0	9		39	0	9.8	1	310	45	4	10.3		1.6		0		1		60	5.6	0.6	0.11	0.11
HH (-HX) - Sanga	FE-25	0.0823	95.7	<5	25	0	10	<35	9	<3	50	<10	1	11	270	40	70	<10	<30	<20	110	2	<10			
FE-25	FE-25	FE-25		0.1	15		9.3	0	9.3	1	50	<1	1	9.1		35		10		2		3	3.4	0.8	0	0.05
HH (-HX) - Sanga	FE-26	0.0679	95.8	<5	45	1	9	<35	10	<3	105	15	1	10	340	<15	70	<10	<30	<20	140	<10	<10			
FE-26	FE-26	FE-26		0.5	35		7.9	0	10.5	0	100	10	1	7.5		1.7		0		0		1	4.3	0.3	0.02	0.03
HH (grande) - Sanga	FE-27	0.0491	96.8	<5	3900	<5	<5	<35	6	<3	<25	<10	<3	18	<50	<15	120	70	45	<20	95	<10	<10			
FE-27	FE-27	FE-27		13	3500		0	6	5.7	0	20	<1	0	15.6		0.2		60		0		0	7.4	0.4	0	0
HH (-HX) - Sanga	FE-28	0.0610	96.2	<5	45	<5	<5	<35	4	<3	<25	<10	<3	9	<50	<15	60	<10	<30	<20	135	<10	<10			
FE-28	FE-28	FE-28		0.4	12		0.1	0	4.4	0	20	<1	0	5.4		0.3		0		0		0	3.3	0.2	0	0
HH (grande) - Sanga	FE-29	0.0388	96.4	15	980	1	<5	<35	<5	<3	40	<10	<3	14	<50	650	190	<10	<30	<20	125	<10	240			
FE-29	FE-29	FE-29		13	820		1.2	4	0.9	0	30	<1	0	10.6		610		10		17		0	230	0.5	0	0
HH (grande) - Sanga	K-1	0.0572	97	150	4400	<5	<5	<35	17	<3	<25	<10	<3	170	<50	2800	170	60	<30	<20	110	<10	45			
K-1	K-1	K-1		385	4000		0.5	3	18.5	1	30	3	0	190		3100		55		0		1	41	0.6	0	0.01
HH (grande) - Sanga	K-2	0.0439	96	210	7500	<5	<5	<35	<10	<3	<25	10	<3	<10	<50	<10	110	290	<30	<20	<60	<10	21			
K-2	K-2	K-2		465	7000		0.6	9	4.4	0	85	10	1	14.6		7.1		300		0		3	18	0.5	0	0
HH (grande) - Sanga	K-3	0.0532	93	<2	35	<5	180	<35	35	<3	280	80	4	<10	2800	<10	210	<10	<30	<20	110	25	<10			
K-3	K-3	K-3		1.1	19		190	7	37	1	290	85	4	4.4		1.7		5		0		25	9.7	0.5	0.05	0.88
HH (grande) - Sanga	K-4	0.0345	87	<2	25	<5	160	<35	<10	<3	260	90	3	<10	2500	13	250	<10	<30	<20	125	30	<10			
K-4	K-4	K-4		0.8	25		175	1	6.8	1	280	95	2	7.1		5.2		1		0		25	7.9	0.6	0.1	1
HH (grande) - Sanga	K-5	0.0722	94	25	40	<5	70	<35	30	<3	300	55	3	45	880	20	135	<10	<30	<20	95	30	12			
K-5	K-5	K-5		30	50		73	16	33	1	330	65	3	50		15		2		0		20	10.2	0.3	0.09	0.13
HH (grande) - Sanga	K-6	0.0556	95	<2	45	<5	50	<35	<10	<3	190	45	<3	<10	960	<10	105	<10	<30	<20	90	20	<10			
K-6	K-6	K-6		0.3	45		50	0	3.6	0	200	50	2	5.3		0		0		0		20	5.6	0.3	0.06	0.12
HH (grande) - Kamilamba	K-7	0.0602	95	800	5200	<5	<2	<35	<10	<3	<60	<10	<3	14	<50	3300	260	60	<30	65	85	<10	55			
K-7	K-7	K-7		330	4700		0.3	1	1.1	0	55	6	0	10.2		3600		50		45		1	53	0.4	0	0
HH (grande) - Sanga	K-8	0.1761	98	3	35	<5	2	<35	50	<3	<60	<10	<3	<10	50	<10	<20	<10	<30	<20	110	<10	<10			
K-8	K-8	K-8		1.6	25		1.6	11	55	0	35	7	0	8.2		0.4		0		0		1	2.6	0	0.01	0.01
HH (grande) - Sanga	K-9	0.0652	95	150	45	<5	16	<35	350	<3	125	17	<3	450	400	30	150	14	<30	<20	120	17	25			
K-9	K-9	K-9		180	45		15.9	5	370	1	125	15	2	490		25		10		4		18	22	0.4	0.03	0.1
HH -Sanga	K-10	0.0778	93	140	690	<5	4	<35	<10	<3	420	20	3	20	55	13	75	15	<30	60	105	110	115			
K-10	K-10	K-10		180	680		4.1	1	2.2	0	450	20	3	22		3.9		8		2		105	125	0	1.19	0.36
HH -Sanga	K-11	0.1935	99	95	85	<5	3	<35	<10	<3	<60	14	<3	36	70	60	45	85	45	<20	105	<10	<10			
K-11	K-11	K-11		195	70		2.1	7	6.3	0	40	12	2	31		45		60		0		2	4.5	0	0.01	0.01
HH (petite) - Katongo	K-12	0.1101	97	105	30	<5	5	<35	160	<3	<60	<10	<3	410	450	14	60	<10	<30	<20	115	<10	<10			
K-12	K-12	K-12		120	25		4.8	10	180	0	25	7	0	450		9.9		8		0		0	1.5	0	0	0.03
HH (petite) - Katongo	K-13	0.1509	99	4	<10	<5	5	<35	<10	<3	60	<10	<3	<10	270	<10	45	<10	<30	<20	140	<10	<10			
K-13	K-13	K-13		2.8	3		5.5	2	3.4	0	60	10	1	7.7		0		0		0		2	2.5	0	0.01	0.06
HH (petite) - Katongo	K-14	0.1139	98	45	105	<5	2	<35	870	<3	300	11	<3	890	65	85	50	20	<30	200	130	7	<10			
K-14	K-14	K-14		47	100		0.9	6	960	0	320	19	1	950		80		4		9		6	2.5	0	0.01	0.01

HH (petite) - Katongo	K-15	0.061	97	<2	<10	<5	<2	<35	<10	<3	80	11	<3	<10	150	<10	30	<10	<30	<20	135	<10	<10			
K-15	K-15	K-15		0.3	0		1.5	0	2.2	0	85	14	1	9.2		0.9		0		0		2	5.3	0	0.01	0.01
HH (petite) - Katongo	K-16	0.0371	95	5	<10	<5	2	<35	<10	<3	55	7	<3	14	70	<10	25	<10	<30	<20	125	<10	<10			
K-16	K-16	K-16		0.6	1		1	0	7.2	0	50	15	0	12.4		0.3		0		0		4	2	0	0.01	0
HIH - Bolela	K-17	0.0847	96	<2	25	<5	5	<35	145	<3	2800	<10	4	45	<50	<10	35	<10	<30	<20	120	30	20			
K-17	K-17	K-17		0.8	35		5	3	165	2	3100	3	5	46		0.7		0		0		20	23	1.9	0.01	0.01
HIH - Bolela	K-18	0.0681	95	<2	19	<5	<2	<35	<10	<3	960	<10	<3	<10	<50	<10	25	<10	<30	<20	105	<10	15			
K-18	K-18	K-18		0.3	25		0.7	0	8.2	1	1100	10	0	9.7		1.0		0		0		1	14.5	0.8	0	0.01
HH (moyen) - Katongo	K-19	0.0913	95	35	220	<5	<2	<35	240	<3	<60	<10	<3	690	<50	17	140	<10	<30	<20	105	<10	<10			
K-19	K-19	K-19		40	210		0.1	30	270	0	30	2	0	750		9.5		9		0		1	3.7	0	0	0
HH (petite) - Malemba-Nkulu	K-20	0.0824	95	125	<10	<5	<2	<35	20	<3	60	8	<3	160	<50	14	45	10	<30	<20	130	<10	<10			
K-20	K-20	K-20		165	3		0.2	4	25	0	60	10	1	180		5.0		3		0		1	4.6	0	0	0
HH (petite) - Malemba-Nkulu	K-21	0.1881	99	10	<10	<5	<2	<35	28	<3	<60	10	<3	17	<50	<10	55	<10	<30	<20	85	<10	<10			
K-21	K-21	K-21		11	4		0.4	18	33	0	30	12	2	18.3		0		2		0		1	1.7	0	0.01	0.01
HH (petite) - Malemba-Nkulu	K-22	0.0755	96	135	30	<5	<2	<35	120	<3	<60	<10	<3	290	<50	25	40	<10	<30	<20	85	<10	<10			
K-22	K-22	K-22		155	28		0.1	5	129	0	45	4	1	310		18		4		1		1	3.5	0	0	0.01
HH (petite) - Malemba-Nkulu	K-23	0.1078	98	100	<10	<5	<2	<35	55	<3	<60	<10	<3	710	<50	25	<20	<10	<30	<20	90	<10	<10			
K-23	K-23	K-23		120	9		<0.02	6	61	0	30	5	1	780		30		11		0		0	2.7	0	0	0
HH (très petite) - Katongo	K-24	0.0750	96	<2	<10	<5	1	<35	<10	<3	<60	6	<3	<10	230	12	95	13	<30	<20	135	<10	<10			
K-24	K-24	K-24		0.2	3		1.2	0	5.9	0	45	12	1	8.1		1.2		2		0		6	4.3	0	0.02	0.01
HH (très petite) - Moero	K-25	0.1166	97	<2	<10	<5	1	<35	16	<3	<60	14	<3	<10	<50	<10	35	<10	<30	<20	120	<10	<10			
K-25	K-25	K-25		0.2	2		1.2	0	18.7	0	35	19	1	6		0.1		0		0		3	1.6	0	0.01	0.01
HH (très petite) - Moero	K-26	0.0928	95	<2	<10	<5	1	<35	60	<3	70	17	<3	19	50	<10	45	<10	<30	<20	105	<10	<10			
K-26	K-26	K-26		0.20	5		1.2	0	67	0	75	20	2	19.8		1.4		0		0		5	3	0	0.02	0.02

Boxplots

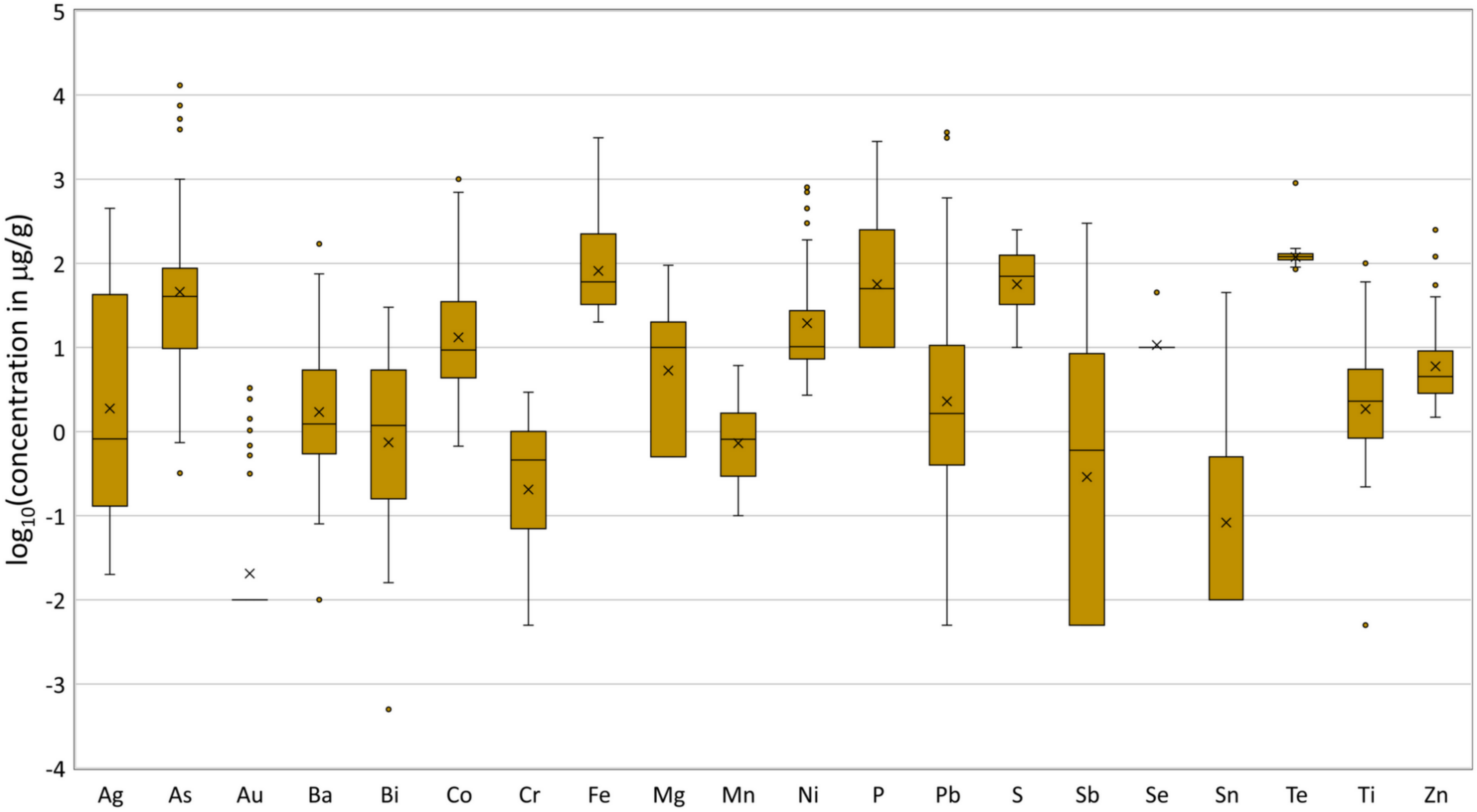

Figure 20: Boxplot showing distribution of element concentrations (in µg/g) for the entire analysed assemblage.

Raw Q-ICP-MS data

Table 4 & 5: full raw Q-ICP-MS data for the 45 sampled artefacts.

(□g/g)	Mg	Al	Sc	Ti	Cr	Mn	Fe	Co	Ni	Zn	Ga	Ge	As	Rb	Sr	Y	Zr	Nb	Mo	Ag	Cd	Sn	Sb
FE-11	40	115	0.04	17.2	2.5	2.0	249	716	23	3.4	0.06	<0.01	27	0.048	<0.02	0.076	1.78	0.090	1.75	1.81	0.024	0.51	1.64
FE-12	<1	<1	<0.02	0.95	0.82	0.17	28	10.8	7.8	2.5	<0.01	<0.01	30	<0.001	<0.02	<0.001	0.04	0.009	0.68	0.023	0.005	0.43	<0.01
FE-13	<1	<1	<0.02	1.12	0.99	0.43	37	12.0	9.5	2.7	<0.01	<0.01	18.1	<0.001	<0.02	<0.001	<0.01	0.013	0.63	0.135	0.004	0.43	<0.01
FE-14	<1	<1	<0.02	0.38	0.43	0.18	33	3.1	4.9	3.6	<0.01	<0.01	37	<0.001	<0.02	<0.001	<0.01	0.002	0.35	0.022	0.012	0.04	<0.01
FE-15	<1	<1	0.05	2.6	1.25	0.45	45	4.3	4.1	3.6	<0.01	<0.01	38	<0.001	<0.02	<0.001	<0.01	0.016	0.96	0.023	0.007	0.51	<0.01
FE-16	<1	<1	<0.02	4.2	0.92	0.12	22	1.19	7.2	2.5	<0.01	<0.01	38	<0.001	<0.02	<0.001	<0.01	0.010	0.72	0.020	0.007	0.24	<0.01
FE-17	<1	<1	0.04	7.1	1.60	0.46	102	0.67	2.7	9.5	<0.01	0.03	12055	<0.001	<0.02	<0.001	<0.01	0.029	1.29	8.0	0.015	0.62	60
FE-18	3	<1	0.04	0.70	1.74	0.65	85	3.1	5.8	4.9	<0.01	<0.01	85	<0.001	<0.02	<0.001	<0.01	0.026	2.1	1.42	0.018	0.07	0.60
FE-19	<1	<1	<0.02	2.7	1.92	0.32	62	4.9	7.4	3.4	<0.01	<0.01	26	<0.001	<0.02	<0.001	0.04	0.058	1.85	0.089	0.025	0.31	<0.01
FE-20	<1	<1	<0.02	0.28	0.54	0.29	21	6.8	12.3	5.9	<0.01	<0.01	48	<0.001	<0.02	<0.001	<0.01	0.014	0.63	0.043	0.028	<0.02	<0.01
FE-21	<1	<1	0.03	0.27	0.56	0.28	22	5.1	10.9	8.7	<0.01	<0.01	9.0	<0.001	<0.02	<0.001	<0.01	0.021	1.65	0.125	0.049	3.8	<0.01
FE-22	53	602	0.20	4.9	1.43	6.1	407	9.7	6.2	5.7	0.09	<0.01	22	0.91	2.3	0.30	0.03	0.035	0.81	0.063	0.044	0.30	<0.01
FE-23	45	371	0.12	5.2	1.13	3.2	307	19.8	9.1	4.8	0.08	<0.01	5.3	0.60	<0.02	0.21	<0.01	0.028	0.68	0.028	0.014	<0.02	<0.01
FE-24	46	828	0.12	62	1.02	4.4	305	9.8	10.3	5.6	0.13	0.03	9.4	1.99	3.7	0.31	1.84	0.26	0.52	0.034	0.005	0.49	<0.01
FE-25	<1	<1	<0.02	3.2	0.57	0.81	50	9.3	9.1	3.4	<0.01	<0.01	15.2	0.025	<0.02	0.004	0.06	0.017	0.56	0.105	0.010	2.1	9.8
FE-26	10	111	<0.02	1.36	0.49	0.90	99	10.5	7.5	4.3	0.04	<0.01	34	0.27	<0.02	0.052	<0.01	0.009	0.19	0.51	<0.001	0.21	<0.01
FE-27	<1	<1	<0.02	<0.05	0.29	0.12	21	5.7	15.6	7.4	<0.01	0.07	3475	<0.001	<0.02	<0.001	<0.01	0.007	0.26	13.1	0.003	0.20	59
FE-28	<1	<1	<0.02	<0.05	0.15	0.12	19	4.4	5.4	3.3	<0.01	<0.01	12.0	<0.001	<0.02	<0.001	<0.01	0.003	0.21	0.39	0.008	<0.02	<0.01
FE-29	<1	<1	<0.02	<0.05	0.37	0.25	32	0.89	10.6	228	<0.01	0.04	823	<0.001	<0.02	<0.001	<0.01	0.004	0.45	12.9	0.75	17.1	10.3
K-1	3	<1	<0.02	0.93	0.51	0.26	29	18.5	187	41	0.03	0.10	3978	0.084	<0.02	<0.001	<0.01	0.011	0.40	386	0.66	<0.02	53
K-2	10	<1	<0.02	3.1	0.44	1.20	87	4.4	14.6	17.6	0.07	0.11	6950	0.055	<0.02	<0.001	<0.01	0.018	0.28	466	0.007	<0.02	302
K-3	84	586	0.09	24	1.12	3.9	291	37	4.4	9.7	0.12	0.04	19.1	1.06	17.0	0.23	0.17	0.087	0.48	1.10	0.192	<0.02	5.1
K-4	94	709	<0.02	23	1.07	2.3	279	6.8	7.1	7.9	0.24	0.03	24	1.77	15.5	0.22	0.46	0.097	0.54	0.82	0.21	<0.02	1.32
K-5	64	707	0.05	19.3	0.83	3.0	333	33	50	10.2	0.20	0.04	48	1.19	5.6	0.26	0.74	0.080	0.23	30	<0.001	<0.02	1.54
K-6	51	437	0.04	20	0.42	1.91	202	3.6	5.3	5.6	0.10	<0.01	46	0.83	4.6	0.141	0.44	0.081	0.30	0.26	<0.001	<0.02	<0.01
K-7	6	<1	<0.02	0.76	0.43	0.24	57	1.12	10.2	53	0.30	3.3	4673	0.024	<0.02	<0.001	<0.01	0.008	0.36	328	0.048	44	51
K-8	7	<1	<0.02	1.29	<0.05	0.10	35	55	8.2	2.6	0.02	<0.01	27	0.041	<0.02	<0.001	<0.01	0.003	<0.01	1.60	<0.001	<0.02	0.47
K-9	15	42	<0.02	17.6	0.61	1.51	127	372	488	22	0.07	0.06	43	0.128	1.64	0.034	0.25	0.044	0.37	179	0.51	4.2	9.9
K-10	20	150	0.64	103	0.46	2.8	452	2.2	22	124	0.09	0.03	678	0.086	8.4	0.82	11.3	0.058	0.05	181	0.035	1.47	7.9
K-11	12	18	<0.02	1.64	<0.05	1.67	40	6.3	31	4.5	<0.01	0.13	72	0.071	0.37	0.016	0.23	0.008	<0.01	194	<0.001	<0.02	59
K-12	7	<1	<0.02	0.45	<0.05	0.44	23	180	455	1.48	<0.01	0.05	25	0.030	0.62	<0.001	0.03	0.003	<0.01	120	0.060	<0.02	7.5
K-13	10	15	<0.02	1.64	0.08	0.60	61	3.4	7.7	2.5	<0.01	<0.01	3.2	0.085	0.89	0.008	<0.01	0.012	<0.01	2.8	<0.001	<0.02	0.34
K-14	19	56	<0.02	5.5	0.07	0.86	312	957	948	2.5	0.82	0.22	102	0.111	0.15	0.042	0.12	0.003	<0.01	47	<0.001	8.7	3.7
K-15	14	29	<0.02	2.3	<0.05	1.07	84	2.2	9.2	5.3	<0.01	<0.01	0.32	0.080	0.13	0.018	0.04	0.009	<0.01	0.32	0.038	<0.02	<0.01
K-16	15	3	<0.02	4.0	0.07	0.41	49	7.2	12.4	2.0	<0.01	<0.01	0.74	0.095	<0.02	0.009	0.03	0.009	<0.01	0.58	0.009	<0.02	0.10
K-17	3	91	<0.02	20	1.83	4.7	3103	165	46	23	0.09	0.04	33	0.066	<0.02	0.030	0.06	0.025	1.51	0.79	0.057	<0.02	<0.01

K-18	10	35	<0.02	1.21	0.71	0.32	1098	8.2	9.7	14.5	<0.01	<0.01	25	0.056	<0.02	0.023	<0.01	0.007	0.57	0.33	0.012	<0.02	<0.01
K-19	2	<1	<0.02	0.92	0.06	0.30	31	266	745	3.7	<0.01	0.09	211	0.023	<0.02	<0.001	<0.01	0.004	<0.01	40	0.046	0.32	9.0
K-20	10	<1	<0.02	0.54	<0.05	0.95	61	25	178	4.6	0.03	0.03	3.2	0.078	<0.02	<0.001	<0.01	<0.001	<0.01	163	<0.001	<0.02	2.9
K-21	12	7	<0.02	0.96	<0.05	1.59	31	33	18.3	1.72	0.03	0.09	4.3	0.107	<0.02	0.005	<0.01	0.005	0.03	11.2	0.022	<0.02	1.85
K-22	4	<1	<0.02	0.92	<0.05	0.64	46	129	311	3.5	0.03	0.04	28	0.057	<0.02	<0.001	<0.01	0.004	0.09	154	0.091	0.57	3.5
K-23	5	<1	<0.02	0.22	<0.05	0.98	30	61	776	2.7	0.04	0.03	9	0.008	<0.02	<0.001	0.04	0.003	<0.01	118	<0.001	<0.02	11.3
K-24	12	72	<0.02	5.5	<0.05	1.05	45	5.9	8.1	4.3	0.06	<0.01	2.8	0.171	0.11	0.021	0.07	0.019	<0.01	0.22	0.006	<0.02	1.93
K-25	19	46	<0.02	3.2	0.07	1.06	33	18.7	6.0	1.57	0.06	<0.01	2.1	0.087	0.51	0.021	0.07	0.021	<0.01	0.22	<0.001	<0.02	<0.01

(µg/g)	**Cs**	**Ba**	**La**	**Ce**	**Pr**	**Nd**	**Sm**	**Eu**	**Gd**	**Dy**	**Ho**	**Er**	**Yb**	**Lu**	**Hf**	**Ta**	**W**	**Pb**	**Bi**	**Th**	**U**
FE-11	<0.002	5.3	0.066	0.083	0.031	0.114	0.007	0.003	0.021	0.014	0.003	0.007	0.010	0.003	0.031	<0.002	2.5	8.7	4.8	0.055	0.038
FE-12	<0.002	1.00	<0.001	<0.001	<0.001	<0.001	<0.005	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.002	1.07	0.87	0.184	<0.001	<0.001
FE-13	<0.002	1.68	<0.001	<0.001	<0.001	<0.001	<0.005	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.002	1.00	0.89	0.171	<0.001	0.011
FE-14	<0.002	0.34	<0.001	<0.001	<0.001	<0.001	<0.005	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.002	0.59	0.30	0.21	<0.001	<0.001
FE-15	<0.002	2.7	<0.001	<0.001	<0.001	<0.001	<0.005	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.002	1.55	0.60	0.80	<0.001	0.003
FE-16	<0.002	0.22	<0.001	<0.001	<0.001	<0.001	<0.005	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.002	1.09	0.23	0.167	<0.001	<0.001
FE-17	<0.002	1.23	<0.001	<0.001	<0.001	<0.001	<0.005	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.002	1.92	11.3	5.0	<0.001	0.033
FE-18	<0.002	0.85	<0.001	<0.001	<0.001	<0.001	<0.005	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.002	2.1	0.39	0.26	<0.001	0.004
FE-19	<0.002	3.0	<0.001	<0.001	<0.001	<0.001	<0.005	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.002	2.4	0.64	0.047	<0.001	0.022
FE-20	<0.002	1.63	<0.001	<0.001	<0.001	<0.001	<0.005	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.002	0.78	3.2	0.121	<0.001	0.018
FE-21	0.021	1.23	<0.001	<0.001	<0.001	<0.001	<0.005	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.002	0.67	0.32	<0.001	<0.001	0.021
FE-22	0.019	36	0.73	1.22	0.170	0.68	0.088	0.022	0.091	0.066	0.009	0.022	0.022	0.003	<0.001	<0.002	0.93	1.51	0.031	0.132	0.110
FE-23	<0.002	9.4	0.45	0.78	0.106	0.44	0.060	0.014	0.067	0.042	0.007	0.014	0.007	0.004	<0.001	<0.002	0.86	2.1	0.028	0.078	0.095
FE-24	0.029	39	0.51	0.89	0.119	0.47	0.083	0.018	0.086	0.057	0.013	0.031	0.029	0.003	0.036	<0.002	0.63	1.64	0.016	0.106	0.106
FE-25	<0.002	9.3	<0.001	<0.001	<0.001	0.006	<0.005	<0.001	0.004	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.002	0.78	36	0.089	<0.001	0.047
FE-26	<0.002	7.9	0.118	0.196	0.028	0.113	0.012	<0.001	0.016	0.014	<0.001	0.005	<0.001	<0.001	<0.001	<0.002	0.27	1.73	0.021	0.024	0.031
FE-27	<0.002	<0.02	<0.001	<0.001	<0.001	<0.001	<0.005	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.002	0.37	0.22	5.6	<0.001	<0.001
FE-28	<0.002	0.10	<0.001	<0.001	<0.001	<0.001	<0.005	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.002	0.21	0.28	0.150	<0.001	<0.001
FE-29	<0.002	1.21	<0.001	<0.001	<0.001	<0.001	<0.005	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.002	0.53	614	4.4	<0.001	<0.001
K-1	0.011	0.49	0.006	<0.001	<0.001	0.008	0.020	<0.001	0.003	0.003	<0.001	0.003	<0.001	<0.001	0.017	0.014	0.56	3051	3.3	0.003	0.008
K-2	<0.002	0.60	0.036	0.007	0.004	0.015	0.011	<0.001	0.004	0.004	<0.001	<0.001	<0.001	<0.001	0.015	0.022	0.45	7.1	8.5	0.004	<0.001
K-3	0.012	188	0.39	0.75	0.093	0.37	0.072	0.018	0.060	0.045	0.009	0.024	0.018	0.003	0.012	<0.002	0.50	1.73	7.1	0.048	0.88
K-4	0.005	174	0.43	0.80	0.102	0.36	0.083	0.023	0.070	0.046	0.009	0.023	0.019	0.005	0.023	0.014	0.57	5.2	1.19	0.097	1.00
K-5	0.013	73	0.40	0.73	0.095	0.39	0.069	0.018	0.060	0.058	0.009	0.029	0.027	0.004	0.022	0.007	0.33	14.7	15.6	0.089	0.129
K-6	<0.002	50	0.27	0.51	0.066	0.26	0.052	0.012	0.040	0.026	0.006	0.017	0.014	0.003	0.017	0.012	0.25	<0.01	0.135	0.063	0.121
K-7	<0.002	0.26	0.005	<0.001	<0.001	0.008	<0.005	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	0.003	<0.002	0.40	3585	1.18	0.003	<0.001
K-8	<0.002	1.61	0.009	0.017	<0.001	0.009	<0.005	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	0.003	0.012	0.007	0.41	11.0	0.005	0.010
K-9	<0.002	15.9	0.074	0.150	0.017	0.056	0.015	0.002	0.010	0.010	<0.001	0.007	0.005	<0.001	0.012	0.007	0.41	26	4.5	0.034	0.103
K-10	<0.002	4.1	1.41	3.21	0.29	1.00	0.193	0.035	0.169	0.175	0.033	0.113	0.107	0.016	0.34	<0.002	0.039	3.9	1.26	1.19	0.36
K-11	<0.002	2.1	0.022	0.034	0.003	0.020	0.008	<0.001	<0.001	0.005	<0.001	0.003	0.003	<0.001	0.006	<0.002	<0.002	47	6.9	0.009	0.012
K-12	<0.002	4.8	<0.001	0.003	<0.001	0.005	<0.005	<0.001	0.003	0.003	<0.001	<0.001	<0.001	<0.001	0.005	0.011	<0.002	9.9	9.6	<0.001	0.033
K-13	<0.002	5.5	0.018	0.028	0.004	0.014	<0.005	<0.001	0.004	<0.001	<0.001	<0.001	<0.001	<0.001	0.004	0.012	0.006	<0.01	2.0	0.006	0.060
K-14	<0.002	0.86	0.190	0.28	0.053	0.169	0.032	0.005	0.018	0.011	0.003	0.005	0.005	<0.001	0.005	<0.002	<0.002	80	6.1	0.011	0.005
K-15	<0.002	1.46	0.038	0.056	0.012	0.038	<0.005	0.003	0.006	0.006	<0.001	0.003	0.003	<0.001	0.003	0.012	<0.002	0.86	0.37	0.009	0.006

K-16	<0.002	0.96	0.043	0.047	0.009	0.030	0.009	<0.001	0.004	0.004	<0.001	0.004	<0.001	<0.001	0.004	0.022	<0.002	0.27	0.20	0.009	0.004
K-17	<0.002	5.0	0.043	0.100	0.011	0.042	0.011	<0.001	0.011	0.013	<0.001	0.006	0.004	<0.001	0.004	0.011	1.86	0.71	2.6	0.009	0.008
K-18	<0.002	0.74	0.035	0.075	0.007	0.023	0.007	<0.001	0.007	0.007	<0.001	0.005	<0.001	<0.001	<0.001	0.014	0.75	0.99	0.160	0.002	0.009
K-19	<0.002	0.11	<0.001	<0.001	<0.001	0.005	<0.005	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	0.011	0.009	9.5	29	<0.001	<0.001
K-20	<0.002	0.20	<0.001	<0.001	<0.001	0.006	<0.005	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	0.014	0.004	5.0	3.9	<0.001	0.004
K-21	<0.002	0.36	0.013	0.033	0.003	0.019	<0.005	<0.001	<0.001	0.006	<0.001	0.003	<0.001	<0.001	<0.001	0.006	<0.002	<0.01	17.6	0.005	0.014
K-22	<0.002	0.08	<0.001	<0.001	<0.001	0.011	<0.005	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.002	<0.002	17.8	5.3	<0.001	0.011
K-23	<0.002	<0.02	<0.001	<0.001	<0.001	0.006	<0.005	<0.001	<0.001	0.003	<0.001	<0.001	<0.001	<0.001	0.017	0.022	<0.002	28	5.5	0.003	<0.001
K-24	<0.002	1.19	0.036	0.070	0.009	0.032	<0.005	<0.001	0.006	0.004	<0.001	0.004	<0.001	<0.001	0.004	0.004	<0.002	1.24	0.181	0.015	0.006
K-25	<0.002	1.20	0.041	0.080	0.013	0.041	<0.005	<0.001	0.005	0.008	0.003	0.005	0.003	<0.001	0.005	0.018	<0.002	0.12	0.61	0.013	0.013

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