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# Data pretreatment and multivariate analyses for ochre sourcing: Application to Leopard Cave (Erongo, Namibia)

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## Abstract

Usually referred to as 'ochre' or 'pigment' in archaeological contexts, ferruginous rocks were commonly exploited during the Later Stone Age in southern Africa. While ochre could lead to crucial inferences about socio-cultural behaviours of past populations, the provenance and the procurement strategies of this material in LSA contexts, as well as its association to rock art remain largely understudied. In the present study, seventeen ochre sources from five geological zones in north-central Namibia and 41 archaeological ochre pieces discovered in the stratigraphic sequence of the Later Stone Age site of Leopard Cave, Erongo Mountains - Namibia, were analysed by ICP-OES and ICP-MS/MS. Geochemical data coupled to data pre-treatment considerations and multivariate statistical analyses demonstrate that archaeological ochres were both collected locally and regionally from more distant sources. Beyond shedding new light on ochre provenance for rock art in north-central Namibia during the Later Stone Age, our data provide new insight into the mobility of past populations and the interactions existing between distinct rock art areas in north-central Namibia.

## Keywords

Multivariate statistics; Sourcing; Ochre; ICP-OES; ICP-MS/MS; Later Stone Age; Rock art; Leopard Cave; Namibia

## 1. Introduction

Most archaeologists refer to ferruginous rocks and iron oxides that present a strong tinting strength and are commonly found in prehistoric archaeology as “ochre”. This material has long been used by humankind for a wider variety of functions including sun protection (Rifkin et al., 2015a, 2015b), mosquito repellent (Rifkin, 2015), hide tanning (Rifkin, 2011), medicine (Audouin and Plisson, 1982) or hafting loading agents (Wadley, 2005; Zipkin et al., 2014; Rots et al., 2017) as well as symbolic and social expression (Dart, 1975; McBrearty & Brooks, 2000; Hovers et al., 2003; Henshilwood et al., 2011; Dayet et al., 2017). This wide array of possible uses makes the analysis of this resource challenging but important within the archaeological material culture record.

Identifying sources of archaeological ochres, specific outcrops or more broadly all outcrops from the same geological formation is essential for understanding prehistoric population mobility, exchange networks and interaction with the environment. In doing so, it allows us to make crucial inferences about human evolution and socio-cultural behaviours (Watts, 2002, 2009; Wadley, 2005; Wadley et al., 2009; Salomon, 2009; Soriano et al., 2009; Hodgskiss, 2012, 2014; Salomon et al., 2012, 2014, 2015; Bernatchez, 2013; Dayet et al., 2013; Pradeau et al., 2014; Rosso et al., 2016, 2017; Hodgskiss and Wadley, 2017; Anderson et al., 2018; Brooks et al., 2018; MacDonald et al., 2018; Wolf et al., 2018). Here, we follow Andrefsky (1998) definition of provenance, as the geological origin of a rock, and we focus on sourcing the archaeological ochres discovered at the Later Stone Age (LSA) site of Leopard Cave (Erongo, Namibia).

To our knowledge, only three archaeometry studies focused on analyses of ochre discovered in southern African LSA contexts (Bernatchez, 2008; Hughes & Solomon, 2000; Steele et al., 2016), contrasting with the extensive number of sites where LSA ochres were recovered. During their preliminary work on LSA ochres from four rock art sites in KwaZulu-Natal (South Africa), Hughes & Solomon (2000) pointed to the lack of knowledge about this mineral resource. Their preliminary multi-analytical work demonstrated that the ochres they studied were highly diverse and might match rock painting materials. Following the work of Erlandson and colleagues (1999), Hughes and Solomon considered rare earth elements to be the key to differentiate ochre sources, and they performed provenance studies on these raw materials. But they could not go further due to the diversity of sites they studied both chrono- logically (between 1290 and 9180 years BP) and geographically (all part of KwaZulu-Natal).

In Namibia, archaeologists recovered ochres from numerous archaeological sites within rock-art-rich areas such as Twyfelfontein, the Rhino Desert, the Brandberg and the Erongo Mountains (Sandelowsky and Viereck, 1969; Wendt, 1972; Kinahan, 1990, 2010, 2018; Richter, 1991; Breunig, 2003; Vogelsang and Eichhorn, 2011; Veldman, 2015; Breunig et al., 2018; Mauran et al., 2020a). Existing literature focuses mostly on exploitation and technical functions of ochre *stricto sensu* in hunter-gatherers societies (Rifkin, 2011, 2012, 2015; Rifkin et al., 2015a, b).

The scarcity of publications focusing on Namibian archaeological ochre found at various rock art sites is rather surprising considering the abundance of sites that have delivered such material and the extensive literature related to rock art sites distribution, function, diversity and relative chronology (Breuil, 1955; Breuil et al., 1960; Scherz, 1970; Vallverdu et al., 1979; Richter, 1988, 1995, 2002; Pager and Breunig, 1989; Kinahan, 1990, 2010; Pager, 1993, 1995, 1998, 2001, 2005, 2006; Hollmann and Steyn, 2003; Lenssen-Erz, 2004; Richter and Vogelsang, 2008; Börner, 2013; Holl, 2017; Nankela, 2017; Breunig et al., 2018; Breunig, 2019). Studying provenances of ochres found at rock art sites could thus shed new lights on the temporal pattern of their exploitation, possibly linked to territorial changes and spatial use and reuse (Smith et al., 2004).

The recent excavations carried out since 2014 at the painted shelter, Leopard Cave, led to the discovery of numerous ochre blocks, fragments and residues on artefacts attesting to the intensive use of ochre (Pleurdeau et al., 2012; Mauran et al., 2020a). Beyond the scope of this article, we aim to identify the "evolutive chain" as defined by Fernandes et al. (2008) for siliceous materials, that is, the history of ochre from their primary geological formation to its exploitation, use, burial and discovery, thence including all the "chaînes opératoires" of ochre processing at Leopard Cave.

To identify potential ochre sources around the site, casual interviews were conducted with local actors including small scale miners, archaeologists and farm owners. During these interviews we asked where we could be find "ochre" that looks like the archaeological pieces we were showing them. As all these actors knew about the large outcrop of the Verbrande Berg, it led us to consider other potential non-local ochre procurement zones located more than 150 km from Leopard Cave. This hypothesis has led us to develop and study a reference collection of geological ochre from north-central Namibia.

From a geological perspective, North-central Namibia corresponds to the Damara Belt that incorporates different tectonostratigraphic zones directed from south-west to north-east: the north-Central Zone, south- Central Zone, North Zone and South-Kaoko Zone (Corner, 1983).

Within these zones, one can find some post-Karoo intrusions that have geochemical signatures different from each other: the Erongo complex, the Kalkfeld complex and the Brandberg formations. Our field survey led us to investigate and collect samples within five zones of interest: Brandberg, Kalkfeld, northern Central Zone (nCZ), North Zone (NZ) and southern Central Zone (sCZ) (Fig. 1). These areas were examined to investigate the intra-regional and the inter-regional ochre compositional variations. For the scope of the present paper, we consider the outcrops within the sCZ and nCZ zones as "local" sources (between 5 and 50 km from Leopard Cave) and the ones from the Brandberg, Kalkfeld complex and the NZ zones as "regional" (between 60 and 180 km from Leopard Cave).

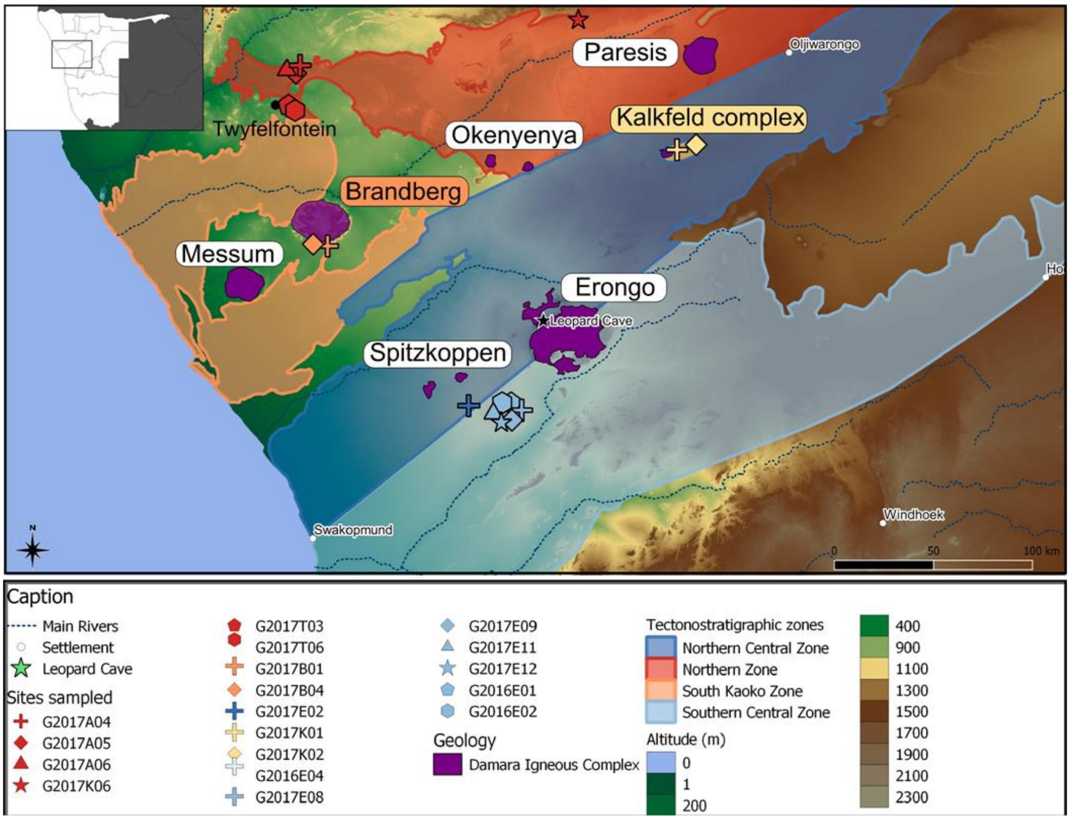

Fig. 1. Map of Namibian ochre acquisition locations for geological and archaeological samples analyzed in this study.

Mineral resource sourcing mostly relies on geochemical data and relative abundances of chemical elements and the process implies data treatments and the use of various numerical or statistical analyses (Zipkin et al., 2020). Moreover, identifying ochres' provenance and to a lesser extent their evolutive chains, requires analyzing geological fragments, archaeological fragments and residues on artefacts and rock shelter walls. Therefore, provenance studies of ochre fragments and the pigment used in rock art require the establishment of multi-technical analytical strategies aimed at avoiding the destruction of archaeological samples while making it possible to establish a clear distinction between the potential sources sampled (Mauran, 2019).

Although ochre has long been reported in archaeological contexts and rock art pigments have been analyzed since the end of the 19th century (Moissan, 1902, 1903), ochre geochemical fingerprinting only developed during the last two decades thanks to analytical developments (Chalmin & Huntley, 2017). Two approaches can be distinguished within ochre provenance studies, both involving the analyses of archaeological material and geological references: 1) the ana- lyses of elemental composition and more precisely trace elements (David et al., 1993; Green and Watling, 2007; Popelka-Filcoff et al., 2007, 2008; Eiselt et al., 2011, 2019; Beck et al., 2012; Montalto et al., 2012; Mathis et al., 2014; Scadding et al., 2015; Zipkin et al., 2017, 2020; Lebon et al., 2018; MacDonald et al., 2011, 2013, 2018; Velliky et al., 2019; Pierce et al., 2020); and 2) the analyses of geological, mineralogical and geochemical information (Iriarte et al., 2009; Salomon et al., 2012, 2014; Kingery-Schwartz et al., 2013; Pradeau et al., 2016; Dayet et al., 2016; Cavallo et al., 2017; Lebon et al., 2019).

The first approach rests solely on trace elements and it is the more widespread in ochre provenance studies. It relies on four dominating techniques: X-Ray Fluorescence spectrometry (XRF), Neutron Activation Analysis (NAA), Inductive Coupled Plasma – Mass Spectrometry (ICP- MS) and Proton-induced X-ray Emission (PIXE). These techniques pre-sent distinct characteristics in regards to accessibility, invasiveness, the quantity of material analyzed, the number of elements that can be quantified, sensitivity and accuracy.

As advocated by Zipkin and colleagues (2020), when selecting a compositional analysis for ochre sourcing, one has to consider the destructiveness of the method, the elements to be quantified and the sample size. Due to the specificities of the four aforementioned tech- niques and the analytical specificities of the analyzed material, it ap- pears that combining destructive analyses for characterizing the geological samples and non-destructive analyses for the archaeological ones would be ideal. But comparing the data obtained would then be tedious because the experimental conditions would be considerably different. Therefore, it is necessary to choose the best compromise to answer the research question about archaeological ochres.

The present study seeks to develop, for the first time in Namibia, a reference dataset for multiple previously uncharacterized ochre sources and to assign archaeological artefacts to one or several specific sources at the regional scale. Therefore, the most appropriate analytical technique should 1) allow the quantification of numerous elements to identify the composition of the ochre samples from the distinct zones; 2) be of good sensitivity and accuracy to identify the distinct chemical signatures; 3) be of minimal invasiveness to preserve the archaeological samples; 4) be easily accessible; and 5) allow the present study to serve as a preliminary step to later deploy the chemical analyses on red residues and paintings for future studies of "chaînes opératoires" of ochre processing.

Here, we present the results of a study to distinguish north-central Namibian ochre sources using Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES), triple quadrupole Inductively Coupled Plasma Mass Spectrometry (ICP-MS/MS) and multivariate statistical analyses. Considering all the constraints and the different characteristics of the analytical techniques, a micro-destructive ICP-MS approach coupled to ICP-OES analyses appeared to be best suited for our study.

This paper aims to 1) document the variety of geological ochres collected in various zones in north-central Namibia; 2) present the geochemical data from these reference collection measured by ICP-OES and ICP-MS/MS; 3) demonstrate the efficiency of our micro-sample ICP- OES and ICP-MS/MS protocol for ochre chemical fingerprinting; 4) investigate the Provenance Postulate with regard to the different geological sources sampled (Weigand et al., 1977) at an inter-regional scale, thus providing us with a baseline to source archaeological ochre and rock art pigments; and 5) to source some of the LSA ochres discovered at Leopard Cave and discuss the implications of these raw material provenances for past populations behaviours.

## 2. Materials and methods

### 2.1. *Archaeological and geological material*

#### 2.1.1. *Archaeological material*

Leopard Cave is an LSA painted rock shelter located within a Cretaceous granitic spur in the Omaruru region of the northwestern part of Erongo Mountains (Fig. 1). The site is located about 1.7 km south to the archaeological site of Fackelträger excavated by Wendt in the 1960s (Wendt, 1972; Richter, 1991). The excavations conducted yearly since 2014 have yielded ochre fragments, stone tools used for grinding and crushing and red-tinted ornaments in archaeological layers dated be- tween 5700 and 2300 BP below the painted wall (Pleurdeau et al., 2012; Mauran et al., 2020a).

Archaeological ochre blocks and fragments studied here were recovered from the excavations conducted between 2014 and 2018. They were sampled within a set of 366 pieces, which could be divided into two main macroscopic groups: 1) 171 black metallic iron oxides, including both massive hematite and some massive magnetite, these last ones were not studied in the present study, and 2) red ferruginous rocks (Fig. 2.A) including 65 ferruginous sandstones, 59 banded ironstones, 26 ferruginous mudstones, and 45 ferruginous altered rocks. Since we did not observe any -specific organisation in their spatial distribution or potential treatment, we decided to gather them into a single group of ferruginous rocks. They were all physically examined before any ana- lyses with a binocular microscope (LEICA) and described according to the descriptions methods presented in Mauran and colleagues (2020c). Descriptions of the archaeological samples are presented in the online dataset Mauran and colleagues (2020a).

In the present study, 41 archaeological ochre pieces were analysed with elemental techniques (ICP-OES and ICP-MS/MS). Before their analyses, the samples were systematically documented, before and after cleaning with deionized water for 3 min, through visual observations and photography.

#### 2.1.2. *Geological material*

To characterize the ochre sources, we conducted surveys in north- western central Namibia in summer 2015, 2016 and 2017. We first documented the potential ochre sources in this region thanks to existing geological literature (Blümel et al., 1979; Roesener & Schreuder, 1992; Schreiber et al., 2000; Schreiber & Becker, 2008), observations of satellite photographs, and interviews with local communities including archaeologists, farm owners, and small scale miners who have empirical knowledge of semi-precious mineral ore deposits of central Namibia.

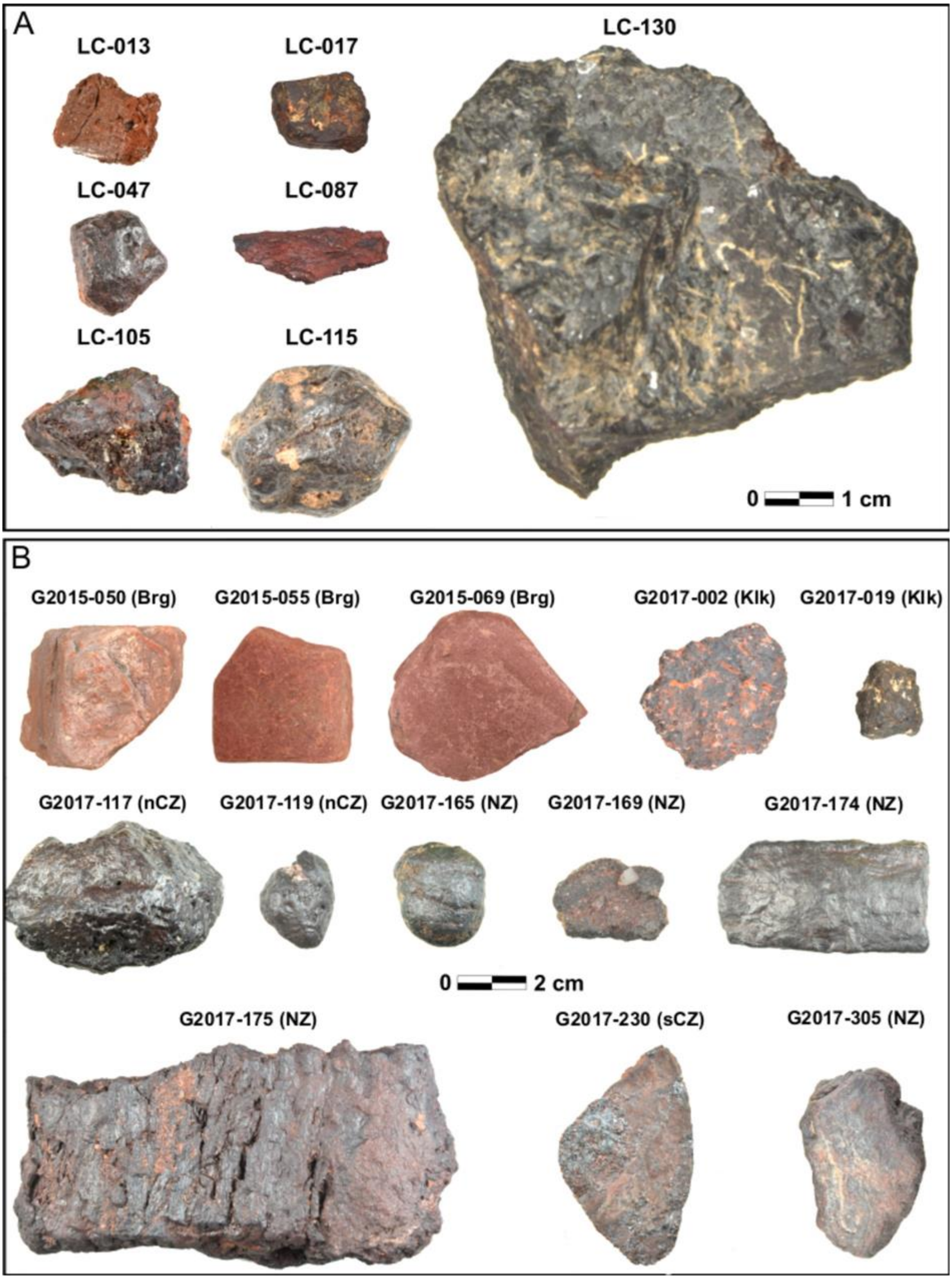

Fig. 2. Diversity of ochre samples analysed. A: Selected analyzed samples from Leopard Cave; B: Selected samples from geological outcrops of all investigated provenance zones.

From their information, we conducted physical surveys in the region located between Burnt Mountain and Twyfelfontein site (a rock art cluster located 160 km NW of the Erongo Mountains) and Leopard Cave. Fig. 1 displays a map with the locations of outcrops, presented in Appendix A. According to these interviews, the most important ochre source within central Namibia is the Burnt Mountain site in the immediate vicinity of Twyfelfontein.

The amount sampled at each outcrop depended partly on the extent and diversity of the ochre materials we found within each source. Additionally, sampling was more important at primary outcrops where the material was clearly in place in relation to the substrate but less intensive at secondary outcrops where the material could have originally come from distinct primary outcrops. Multiple samples were collected from each outcrop to gauge intra-source heterogeneity; in particular, we collected samples where we observed macroscopic differences in morphology, texture, apparent structure and density.

A total of 279 samples (12 kg) were collected from 17 outcrops within the five areas previously mentioned (Table 1). Each outcrop was documented photographically and its dimensions, diversity and con- centration of ochre, accessibility and geographic location were recorded with GPS equipment GPSMAP 64sx (Garmin). Due to geography, land partition, wildlife and limited time, we could not always reach primary ochre formations.

Before any analyses, all the geological samples collected were observed macroscopically and microscopically and described in the same way as the archaeological ochre fragments. They comprised both black metallic iron oxides and red ferruginous rocks (Fig. 2.B). Descriptions of the geological samples are also presented in the online dataset Mauran and colleagues (2020b).

These first observations and descriptions allowed to sub-sample from the various outcrops thereby reducing the number of samples for analysis to 94. For each outcrop, great care was taken to select samples with the most diverse structure, porosity and mineral inclusions, thus maximizing the intra-source variability of the samples.

Table 1 List of geological sources and samples studied (details sample are given in Mauran et al. (2020b)).

| *Site code* | *Zone* | *Number of samples analyzed (Number of* | *GPS data* | *Site nature* | *Geological formation* |
|---|---|---|---|---|---|
| **G2016-E04** | Erongo | 2 (2) | 15,51° E ;<br>21,55° S | Primary | Erongo granite (inclusion) |
| **G2017-E02** | nCZ | 10 (15) | 15,21° E ;<br>21,95° S | Sub-primary | Leucogranite (fault) |
| **G2016-E01** | sCZ | 6 (18) | 15,37° E ;<br>21,97° S | Sub-primary | Ozambada granite(fault) |
| **G2016-E02** | | 3 (11) | 15,35° E ;<br>21,96° S | Sub-primary | Leucogranite (fault) |
| **G2017-E08** | | 7 (14) | 15,37° E ;<br>21,97° S | Sub-primary | Ozambada (fault) |
| **G2017-E09** | | 5 (6) | 15,37° E ;<br>21,97° S | Sub-primary | Ozambada (fault) |
| **G2017-E11** | | 7 (13) | 15,36° E ;<br>21,97° S | Secondary | Leucogranite(fault) |

| | | | | | |
|---|---|---|---|---|---|
| **G2017-E12** | | 5 (8) | 15,36° E ; 21,97° S | Secondary | Leucogranite (fault) |
| **G2017-K01** | Kalkfeld | 3 (15) | 16,16° E ; 20,80° S | Sub-primary | Kalkfeld igneous complex |
| **G2017-K02** | | 5 (16) | 16,24° E ; 20,78° S | Sub-primary | Kalkfeld igneous complex |
| **G2017-B01** | Brandberg | 9 (38) | 14,58° E ; 21,25° S | Secondary | Brandberg igneous complex |
| **G2017-B04** | | 3 (10) | 14,51° E ; 21,22° S | Secondary | Brandberg igneous complex |
| **G2017-T03** | NZ | 3 (43) | 14,41° E ; 20,62° S | Primary | Karoo formations |
| **G2017-T06** | | 6 (9) | 14,42° E ; 20,62° S | Primary | Karoo formations (vein) |
| **G2017-A04** | | 5 (12) | 14,45° E ; 20,42° S | Sub-primary | Karoo formations (vein) |
| **G2017-A05** | | 5 (20) | 14,45° E ; 20,42° S | Sub-primary | Karoo formations (vein) |
| **G2017-A06** | | 8 (15) | 14,45° E ; 20,42° S | Sub-primary | Karoo formations (vein) |
| **G2017-K06** | | 3 (14) | 16,71° E ; 20,21° S | Secondary | Swakop formations (vein) |

## 2.2. *Analytical methods*

Elemental characterization of each sample was performed using so- lution Inductively Coupled Plasma Optical Emission Spectroscopy (ICP- OES) and triple quadrupole Inductively Coupled Plasma Mass Spectrometry (ICP-MS/MS). A triple quadrupole is a tandem mass spectrometer comprising of two transmission quadrupole mass spectrometers in series, with a collision cell interbedded. Both mass spectrometers (Q1 and Q2) are tuned (M/Z) together or independently, depending on isobaric interferences of the target element. Isobaric interferences of element y on target element x are mainly due to the oxide conditions (Mx/Z=(My + 16)/Z) and the doubled charged ion (Mx+/Z= My++/2Z) but are also due to the isotopes of other elements. Collision/reaction cells could use collision or reacting gases (e.g. He, H2, O2, NH3) to eliminate the isobaric interferences. In this work, we have used the mode MS/MS tandem with no gas.

Each sample/block was prepared separately. For each block analyzed, we sampled 1 g of the sample and homogenized it using an agate mortar and pestle. Out of the homogenized powder, two aliquots of 50 mg were sampled: one for the ICP-OES analysis and one for the ICP-MS/MS analysis. For both kind of analyses, we then digested the powder using acid attack (HCl, HF, HNO3) with Polypropylene DigiTUBEs inserted inside DigiPREP (SCP Sciences) hot blocks. The solution was diluted with 50 mL of boric acid (H3BO3) following the protocols of the ALIPP6 Platform (Sorbonne Université, ISTeP), where the ICP-OES and ICP-MS/MS were performed (Mauran, 2019). Supplementary dilution with MilliQ HNO3 2% v/v avoids saturating the detector. Blank, international and internal standards were digested each time. It is worth noting that our analyses involved 100 mg of ochre (50 mg for ICP-OES and 50 mg for ICP-MS/MS), half the amount used in previous ochre studies involving those techniques (Iriarte et al, 2009; Dayet et al., 2016; Roman et al., 2015; Moyo et al., 2016).

ICP-OES analyses were carried out using a 5100 SVDV Agilent ICP- OES and this quantified the following 21 chemical elements: Si, Al, Mg, Na, K, Ti, Fe, Mn, Ca, P, Sr, Ba, Sc, V, Cr, Zr, S, W, Cu, Zn, and Co. For each element, two to four wavelengths were measured over an exposure time of 3 times 20 s. The RSD of each analysis is < 5%.

ICP-MS/MS analyses were carried out using a 8800 Agilent ICP-MS/ MS and this quantifies the following 37 isotopes: $^{7}$Li, $^{45}$Sc, $^{51}$V - $^{53}$V, $^{52}$Cr, $^{59}$Co, $^{60}$Ni, $^{63}$Cu, $^{66}$Zn, $^{71}$Ga, $^{75}$As, $^{85}$Rb, $^{88}$Sr, $^{89}$Y, $^{90}$Zr, $^{93}$Nb, $^{95}$Mo, $^{133}$Cs, $^{137}$Ba, $^{139}$La, $^{140}$Ce, $^{141}$Pr, $^{146}$Nd, $^{147}$Sm, $^{153}$Eu, $^{157}$Gd, $^{159}$Tb, $^{163}$Dy, $^{165}$Ho, $^{166}$Er, $^{169}$Tm, $^{172}$Yb, $^{175}$Lu, $^{178}$Hf, $^{181}$Ta, $^{206}$Pb - $^{207}$Pb - $^{208}$Pb, $^{232}$Th, and $^{238}$U. The oxide (156/140) and double charged (70/ 140) are < 1.1% and < 3% respectively. The RSD of each analysis is < 3%.

For both ICP-OES and ICP-MS/MS, each measurement was per- formed in triplicates and the results were later averaged. Calibration of the quantification was performed thanks to international standards (AThO, AGV-2, BHVO-2, BIR-1, GSN, RGM-1) and internal laboratory references (Lip, Co5, M77-12, LGM). After every 10 measurements, we carried out MilliQ HNO3 2% v/v Blank and 3 standards (BHVO-2, AGV and BIR-1) measurements to detect and correct the eventual drifts of signal. The other standards were analyzed like blind samples to control the accuracy of results. International standards for iron oxides from Geological Survey of Canada were used to check the high matrix effect (Fer 1, 2, 3 and 4) with 50, 22.5, 29.4 and 22.7 wt% of Fe2O3 respectively (Pain and Ollier, 1992).

### *2.3. Statistical analyses*

Compositional analyses first rely on bivariate plots of geochemical data acquired from a unique analytical technique to inspect the dataset, detect the possible outliers and sometimes to distinguish different sources (Baxter, 2008). When relying on a small number of elements, compositional analyses sometimes fail to distinguish different ochre sources or might not be robust enough on their own for an ochre sourcing study (Zipkin et al., 2020). This is why sourcing issues also require the implementation of multivariate statistics. Several issues may bias the statistical analyses: 1) non-normality of the distribution of the measurements for each variable can bias the statistical test (Baxter et al., 2005; Leroy, 2010; Leroy et al., 2012), 2) the specific treatment of rounded zero usually dealt by substitution of the rounded zero value with a constant positive value smaller than the Limit Of Detection (LOD), 3) the difference of orders of magnitude lead the analyses to be overwhelmed by the variable presenting the highest order of magnitude (Baxter et al., 2005; Leroy, 2010; Leroy et al., 2012), and 4) the intrinsic relative nature of elemental datasets make it dependent on the representation used (Aitchison, 1999; Baxter et al., 2005). Multivariate statistical tests such as the unsupervised principal component analysis (PCA) and the supervised linear discriminant analysis (LDA) are sometimes considered robust to counter deviations from normality and rounded zeros value resulting from concentration below LOD (Zipkin et al., 2017, 2020).

In our study, only elements that could be measured reliably by ICP- OES or ICP-MS/MS in a majority of the samples (*i.e*. not below LOD) were used for the multivariate analysis. Some elements such as Ga, Na, Nb or S, were present below detection limits in more than 2% (N = 2) of samples analyzed and were consequently removed from the data set for subsequent statistical analyses.

Then we split our data sets into two groups: 1) the data measured on the geological samples, referred to as "geological dataset", and 2) the data measured on the archaeological samples, referred to here as "archaeological data set". We first considered the geological data sets to distinguish the sundry source zones. On this data set, we performed all steps described below to build a sourcing model. As for the archaeo- logical data set, we performed its projection on the sourcing model. Initially, the data set included two additional samples from locality G2017-E02 that were outliers, corresponding to quartz covered with an iron-rich weathering body on the exterior. As our analyses were per- formed on both the exterior body and the quartz, we decided not to include them in the present study.

All statistical analyses were performed with the R software version 3.6.2 and the “ggplot2”, “MVN”, “MASS”, “ade4”, “corrplot”, “plyr” packages (R Core Team, 2019; Chessel et al., 2004; Wickham, 2009, 2011; Ripley et al., 2013; Korkmaz et al., 2014; Wei et al., 2017). The R script used to produce the results is available as Supporting Information (Appendix F).

### 2.3.1. *Data correlation*

Since it has been established that reducing the number of variables could improve discrimination between the chemical signature of out- crops (Baxter & Freestone, 2006), we first tried to limit the number of variables by using the Fe-oxide signature approach for ochre compositional analyses: restricting us to the elements associated with iron (Fe).

On the raw data set, we performed a Spearman’s correlation test coupled with the “corrplot” function of the R “corrplot” package to determine which elements were associated with Fe. No element was significantly

positively correlated to Fe, due to the high diversity of the ferruginous materials considered, and this highly restricted our ability to exploit the usual statistical treatments for distinguishing ochre potential sources (Popelka-Filcoff et al., 2007, 2008). When considering each outcrop individually, we could not reduce the number of variables because there was at least one site where the elements correlated to iron.

### 2.3.2. *Data treatment*

For compositional analyses, data normalization is necessary to pro- vide representation-invariant datasets, taking into consideration the matter size variation. For materials other than ochre, this normalization is commonly performed using the centered-log-ratio transformation relying on the geometric mean of the elements measured (Aitchison, 1999; Baxter et al., 2005; Leroy, 2010; Leroy et al., 2012). To our knowledge, it has not yet been used on ochre.

Since the concentration of iron in ferruginous artefacts can vary significantly, using a Fe-ratio can assist in preventing the natural variation in Fe from influencing statistical analysis of trace elements, as it is the case with our archaeological samples according to our preliminary observations (Mauran et al., 2020a). This led researchers to normalize element concentration to iron and limit the analyses to the elements correlated to iron (Popelka-Filcoff et al., 2007, 2008). Recent studies have demonstrated that this method is not always the optimal one and proved it is necessary to consider multiple approaches to ochre compositional data when the variation of sources is unknown (Zipkin et al., 2017; Pierce et al., 2020).

In our dataset, Eu and Th exhibited one rounded zero, due to measurements below LOD. We performed, therefore, a basic substitution, replacing these rounded zeros with a positive value representing 10% of the lowest value measured on the other samples.

Considering all the previous studies, we transformed our geological dataset according to three distinct methods: 1) log transformation of the raw data, producing dataset 1 “log dataset”, 2) the iron normalized log transformation, leading to dataset 2 “log-Fe dataset”, and 3) the centred log-ratio transformation performed on the unmodified variables provided dataset 3 “ log-ratio dataset” and this transformation is common in statistical compositional analyses.

### 2.3.3. *Multivariate normality*

We tested the multivariate normality of our raw data with the Mardia's multivariate skewness and kurtosis test, Royston's multivariate Shapiro–Wilk test and the Henzee Zirkler empirical characteristic function test using the MVN statistical package for the R programming environment (Mecklin & Mundfrom, 2005; Korkmaz et al., 2014; Cain et al., 2017). All three MVN tests found that our data sets are non- multivariate normal. As LDA has been considered as robust to violations of data set multivariate normality, we pursued our analysis (Blanca et al., 2013; Enserro et al., 2019). The use of logarithm scaling decreases the existing bias due to the difference of scale of concentration between the distinct elements under consideration (Baxter, 1994; Baxter & Freestone, 2006; MacDonald et al., 2018). For ochre sourcing studies as ours, such a transformation is essential, given the difference of magnitude between the major, minor and trace elements. Moreover, the log transformation allows the reduction of the multivariate non-normality of the measures distribution and increases the robustness of the statis- tical performance (Buxeda i Garrigos, 2018).

### 2.3.4. *Multivariate statistics*

After these treatments we used Principal Components Analysis (PCA) and Linear Discriminant Analysis (LDA), to investigate the chemical signatures of the samples from the distinct outcrops. Both methods are discussed in details for ochre studies in Zipkin and colleagues (2017), and for iron slags in Leroy (2010) and Disser (2015). There is a more in- depth overview in Baxter (1994). In the present study, PCA was per- formed to observe the multivariate variations among all samples considered, while LDA was used to build the sourcing model. As dis- cussed by Harbottle (1976), LDA is sensitive to sample size: the number of observations considered in the analysis needs to be at least three times higher than the number of variables used to perform the LDA. This is why it appeared necessary to reduce the number of variables taken into account in our analyses from 48 to fewer than 20.

Reduction of the number of variables was performed in an empirical recursive way, thanks to backwards stepwise LDA. Similarly to what has been done recently by Pierce and colleagues (2020), elements were removed according to their explanation of variation. Elements removed were the ones presenting the fewest variations. We determined when the source groups were clearly distinguished at the level that satisfies the

Provenance Postulate by visualizing each group's 95% confidence ellipse on the two first axes of projection. We ensured that no ellipses overlapped and that few or no samples plotted within an incorrect group ellipse.

### 2.3.5. *Cross-validation*

To evaluate the performance of LDA on the different datasets considered, we also used cross-validations. They allowed the attribution of a score of differentiation between the sundry zones considered, using the confusion function published by Maindonald and Braun (2010). The confusion matrix is calculated from predictions of class membership that are derived from leave-one-out cross-validation and comparison of the actual given and predicted group assignments.

### 2.3.6. *Archaeological samples projection*

Once the sourcing model was built and selected from the different ones constructed from the sundry data treatments, we performed the data transformation on the archaeological dataset. We performed a PCA on both geological and archaeological data to ensure that they presented comparable chemical signatures and compositional variation. The ten samples that were not entirely comparable to our geological dataset were relegated to "unknown" provenance. The 31 others, we thence projected them on the sourcing model using predictive LDA and calculating the resemblance scores for the different zones considered (Table 2).

Table 2 LDA calculated scores of resemblance for the archaeological samples with the distinct provenance zones for the two sourcing models. Only the archaeological samples (31/41) presenting a chemical composition comparable to the geological ones are presented here. In bold score of resemblance higher than 75%.

| Arcaheological samples | 5 zones model | | | | | 4 zones model | | | | Provenance |
|---|---|---|---|---|---|---|---|---|---|---|
| | Brandberg | Kalkfeld | nCZ | NZ | sCZ | Brandberg | nCZ | NZ | sCZ | |
| LC-013 | **100** | 0 | 0 | 0 | 0 | **100** | 0 | 0 | 0 | Brandberg |
| LC-030 | 57 | 0 | 0 | 43 | 0 | 13 | 0 | 87 | 0 | |
| LC-038 | 0 | 0 | 5 | 5 | **89** | 0 | 2 | 1 | **98** | sCZ |
| LC-047 | 0 | 0 | 1 | 88 | 10 | 0 | 0 | 19 | 80 | |
| LC-056 | 0 | 0 | 1 | 8 | **92** | 0 | 0 | 0 | **100** | sCZ |
| LC-095 | 0 | 0 | 1 | 1 | **98** | 0 | 0 | 0 | **100** | sCZ |
| LC-096 | 0 | 0 | 0 | 89 | 10 | 0 | 0 | 39 | 61 | |
| LC-105 | 0 | 0 | 7 | 3 | **90** | 0 | 4 | 2 | **94** | sCZ |
| LC-106 | 0 | 0 | 48 | 3 | 50 | 0 | 64 | 5 | 31 | Central Zone |
| LC-110 | 0 | 0 | 0 | **96** | 4 | 0 | 0 | **98** | 2 | NZ |
| LC-114 | 0 | 0 | 0 | **95** | 5 | 0 | 0 | **96** | 4 | NZ |
| LC-115 | 0 | 0 | 18 | 69 | 13 | 0 | 61 | 38 | 0 | |
| LC-116 | 0 | 0 | 1 | 0 | **99** | 0 | 0 | 0 | **100** | sCZ |
| LC-133 | 60 | 0 | 0 | 4 | 36 | 45 | 0 | 5 | 50 | |
| LC-146 | 78 | 0 | 0 | 22 | 0 | 45 | 0 | 55 | 0 | |
| LC-184 | 0 | 0 | 1 | 2 | **97** | 0 | 0 | 1 | **99** | sCZ |
| LC-187 | 0 | 0 | 0 | **100** | 0 | 0 | 0 | **100** | 0 | NZ |
| LC-189 | 0 | 0 | 1 | **99** | 0 | 0 | 1 | **99** | 0 | NZ |
| LC-216 | 56 | 0 | 0 | 44 | 0 | 26 | 0 | 74 | 0 | |
| LC-225 | 0 | 0 | 68 | 21 | 12 | 0 | **84** | 15 | 0 | nCZ |
| LC-248 | **100** | 0 | 0 | 0 | 0 | **100** | 0 | 0 | 0 | Brandberg |
| LC-255 | **100** | 0 | 0 | 0 | 0 | **100** | 0 | 0 | 0 | Brandberg |
| LC-268 | **96** | 0 | 0 | 4 | 0 | **92** | 0 | 8 | 0 | Brandberg |
| LC-269 | 0 | 0 | 2 | 4 | **94** | 0 | 1 | 2 | **98** | sCZ |
| LC-281 | **100** | 0 | 0 | 0 | 0 | **100** | 0 | 0 | 0 | Brandberg |
| LC-283 | **100** | 0 | 0 | 0 | 0 | **100** | 0 | 0 | 0 | Brandberg |
| LC-296 | 0 | 0 | 1 | 17 | **82** | 0 | 0 | 3 | **96** | sCZ |
| LC-341 | 0 | 0 | 1 | 42 | 56 | 1 | 0 | 37 | 61 | |
| LC-354 | **97** | 0 | 0 | 3 | 0 | **78** | 0 | 22 | 0 | Brandberg |
| LC-396 | **100** | 0 | 0 | 0 | 0 | **100** | 0 | 0 | 0 | Brandberg |
| LC-402 | 0 | 0 | 0 | **100** | 0 | 0 | 0 | **100** | 0 | NZ |

## 3. Results

### 3.1. *ICP-OES and ICP-MS/MS results*

In total, 94 geological ochre samples were analyzed by ICP-OES and ICP-MS/MS. Elemental concentration data are provided in Mauran and colleagues (2020c).

Concentrations of the nine elements quantified both by ICP-OES and ICP-MS/MS (V, Cr, Co, Cu, Zn, Sr, Zr, Ba, and Sc) were compared to ensure correlation between the two techniques and attainment of a single unified dataset. These correlations are presented in supplementary data (Appendix Table C). A Spearman test was performed for each element to test the correlation of the ICP-OES and ICP-MS/MS results. Since the p-value for all nine elements was less than 2.2e-16 and the correlation coefficient was around 0.9, we considered the two techniques to be complementary. The ICP-OES analyses allow the quantification of major, minor and few trace elements, while ICP-MS/MS ones provide quantification of a large range of trace elements. To avoid having duplicate data of the nine elements mentioned above, we arbitrarily decided to use the ICP-MS/MS values for these elements for subsequent analyses.

#### 3.1.1. *Geological samples*

Fe, Al and Si are the main components of our geological samples (Fig. 3 and Mauran et al., 2020c). Fe2O3 weight percentage varies from 20 wt% in an indurated shale sample coming from the

Brandberg (Fig. 3. A) to 95 wt% in iron oxide samples from different zones considered in this study. Ferruginous sandstones (G2017A04, G2017A06) and breccia (G2017K01) present an intermediary total iron oxide weight (around 65 wt%). However, except for the Brandberg ochre, the intra-source variations are substantial as illustrated in Fig. 3.B and 3.C for North and southern Central zones, making it difficult to use major and minor minerals to discriminate the different zones. The use of bivariate plots considering all measured elements could not further help to discriminate the zones.

When screening the results it appears that Rare Earth Elements occurred in significantly higher concentrations in ochre from the Kalkfeld complex than in iron oxide samples from the other regions. This led us to focus the distinction between the four following zones: Brandberg igneous complex, northern Central Zone, southern Central Zone, and the North Zone.

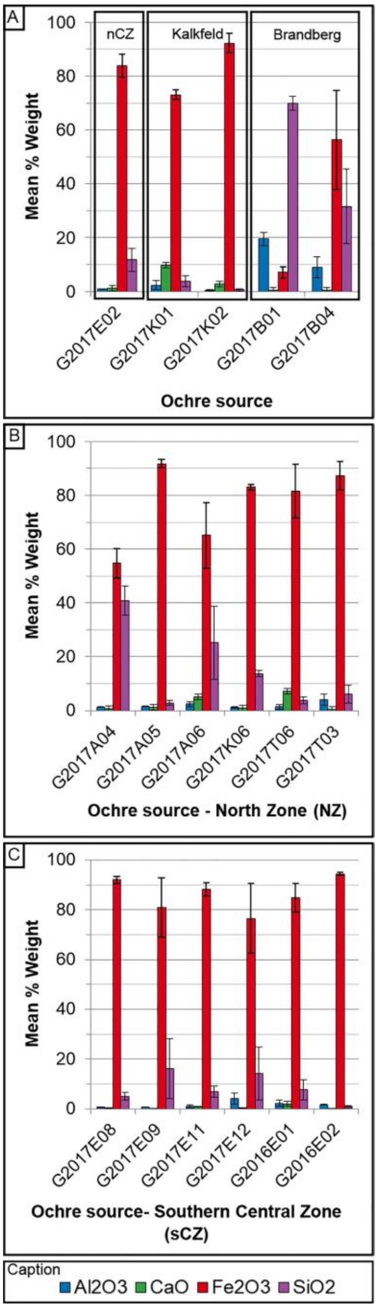

Fig. 3. Mean major element concentrations for sources as % weight oxides, as measured by ICP-OES and ICP-MS/MS. Error bars indicate the range for each element based on individual ICP-OES and ICP-MS/MS measures for each source; A: northern Central Zone (nCZ), Kalkfeld complex, Brandberg,; B: North Zone; C: southern Central Zone.

#### 3.1.2. *Archaeological samples*

The composition of the analysed archaeological samples is similar for major elements to that of the geological samples (Mauran et al., 2020b). Iron percentages vary as they do for geological samples. Therefore, the archaeological samples present a chemical signature that is coherent with the geological samples considered, allowing us to attempt sourcing them with our modern collection.

### 3.2. *Differentiation of the potential source zones*

We first investigated the possibility of identifying the ochres with REE but these elements appeared to be insufficient to differentiate effectively the different areas considered here (Appendix D). Following data exploration, we carried out multivariate statistical techniques of principal component analysis (PCA) and linear discriminant analyses LDA). Our procedure to determine the variables take into consideration the fifteen elements (Al, As, Ce, Cs, Eu, Fe, Hf, Mg, Sm, Sr, Th, U, V, Zn and Zr) to compare the three different transformations presented above and their ability to discriminate between the distinct zones. To achieve this we compared both the PCA biplot of the two first components and the LDA biplot of the two discriminant functions for each dataset: 1) “log dataset”, 2) “log-Fe-dataset”, and 3) “log-ratio-dataset”. Comparing the bivariate plots of the two first PCA components and LDA functions as well as the cross-validation scores for each dataset, it appeared that the “log-dataset” was the most effective approach to differentiate the various zones considered in our study. It was the only model providing a bivariate plot of the two first LDA functions with clear distinction of the zones and neat clustering of data points and a cross-validation score of 93% (the other transformations also presented a cross-validation accu- racy score of 93% but presented some overlaps on the bivariate plot of the two first LDA functions). We pursued our analyses with the “log dataset” and built the sourcing model with this dataset.

In the biplot of the two first component of the PCA performed on all samples including the ones of the Erongo member, principal component 1 explains 29.8% of the data set variance, while principal component 2 explains 19.9%. Principal component 1 allows us to distinguish the local zones (northern Central Zone and southern Central Zone) from the Kalkfeld complex and the Brandberg samples, while principal component 2 mainly enables us to differentiate the Kalkfeld complex from the Brandberg zone (Fig. 4.A and Appendix Tables E.1, E.2). However, this representation did not allow us to distinguish the different zones completely, confirming the large intra-zone variation and the proximity of the chemical signatures of the raw material from North Zone, northern Central Zone and southern Central Zone. It is worth noting here that remaining principal components did not enable a better distinction of the different zones.

In the bivariate plot of the LDA performed on the fives zones considered in the study (Fig. 4.B), discriminant function 1 (44.0% of the between-class variance) clearly distinguishes the Kalkfeld complex from the Brandberg Zone, while discriminant function 2 (30.1% of the same between-class variance) differentiates the remaining zones. In this bivariate plot, 90% confidence ellipses clearly distinguish the zones from one another, making it a reliable model for ochre provenance. This is confirmed by the cross-validation performed with 93% of the samples correctly attributed to their zone of origin.

We performed a second LDA considering only the four following zones: Brandberg, northern Central Zone, southern Central Zone and North Zone (Fig. 4.C and 6.D and Appendix Tables E.1, E.2). Both multivariate statistical techniques lead to similar results to those from all the five zones. Although the PCA tends to better differentiate the North Zone from the southern Central Zone and the northern Central Zone, confidence ellipses between the distinct zones still overlap (Fig. 4.C). As for the LDA, it provided a better distinction between the zones considered with a clear differentiation of the 90% and 95% confidence ellipses, while as expected it explained higher variance of the model (Fig. 4.D).

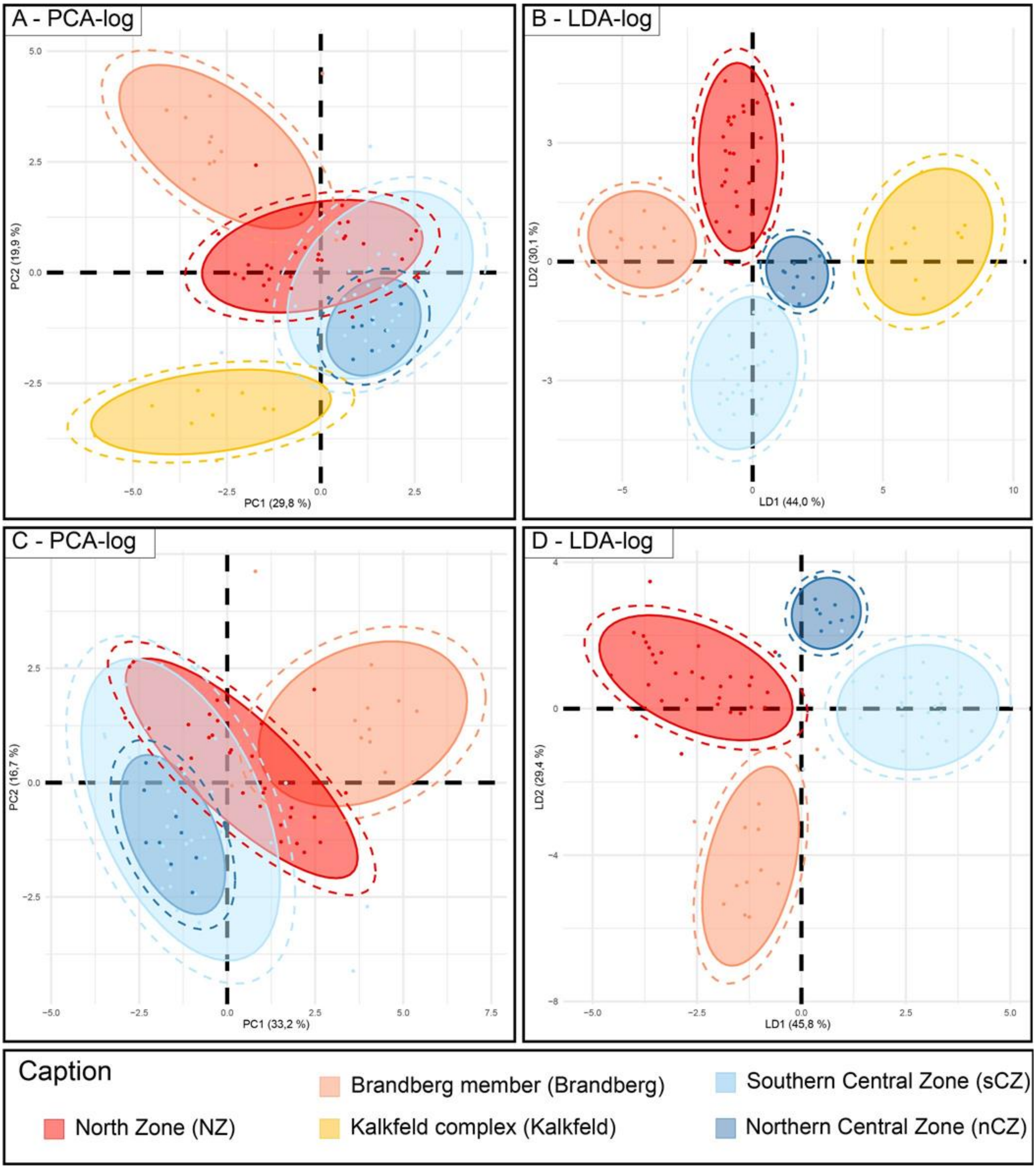

Fig. 4. Bivariate plots of Principal Component Analysis and Linear Discriminant Analysis constructed with geological samples for the 15 following elements: Al, As, Ce, Cs, Eu, Fe, Hf, Mg, Sm, Sr, Th, U, V, Zn and Zr. All samples are grouped by provenance zone with the 90% (plain) and 95% (dash) confidence ellipses shown for each group. A and B: PCA and LDA considering the five zones of provenance. C and D: PCA and LDA limiting the analysis to four zones (Brandberg, southern Central Zone, northern Central Zone and North Zone). PCA normed scores presented in Table B.3 and LDA variable coefficients in Table B.4.

### 3.3. *Provenances of archaeological samples*

We decided then to build the sourcing model of the archaeological ochres using the four previous LDA and PCA bivariate plots. First, we added the archaeological samples to the PCA with the five zones to ensure that their chemical signature was comparable to one of the geological samples considered here. In this projection, any samples that plotted outside of the 95% confidence ellipses for the source zones were considered to be of unknown provenance. This was necessary because subsequent modeling with predictive LDA would provide a resemblance score for these samples, though these scores would not be coherent because the samples do not have a high chemical signature resemblance. Ten of the 41 archaeological samples analyzed correspond to such cases: LC-008, LC-029, LC-039, LC-045, LC-087, LC-111, LC-125, LC-130, LC- 253 and LC-298 (Fig. 5). They most probably indicate that LSA populations who occupied Leopard Cave exploited ochre sources other than the ones we sampled.

For the 31 other samples that plot at least within one of the confidence ellipse of a zone considered here, we used the first LDA from five zones to associate each archaeological sample to one of these zones and the second LDA from the less differentiated four zones to confirm the proposed association. In each case, we calculated the resemblance score between the archaeological sample and the geological data of the considered zones. Table 2 documents all the scores obtained and the proposed origin attribution for samples presenting a score of resemblance higher than 75%. Other samples (N = 6) were considered of unknown provenance. Sample LC-106 presented a resemblance score of 50% with sCZ and 48% with nCZ (Table 2). Although we could not determine its provenance with certainty, we considered it to be of local origin from the Central Zone. As only one sample could be attributed to nCZ with certainty, we included it in the sCZ in Fig. 6, which sums up our results of the LSA Leopard Cave ochre provenance. One sample was attributed to the northern Central Zone (< 50 km from LC), nine samples were attributed to the southern Central Zone (< 50 km from LC) considered to be of local origin, eight to the Brandberg area (100 km from LC) and five to the North Zone (150 km from LC). The samples from the last two areas are both considered to be from regional provenances.

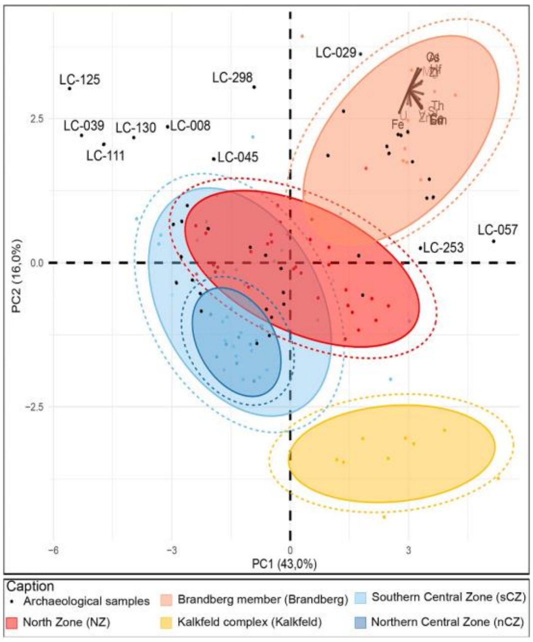

Fig. 5. Bivariate plot of PCA with archaeological and geological samples for the 15 following elements (Al, As, Ce, Cs, Eu, Fe, Hf, Mg, Sm, Sr, Th, U, V, Zn and Zr). Archaeological samples falling outside of confidence ellipse are considered of unknown provenance.

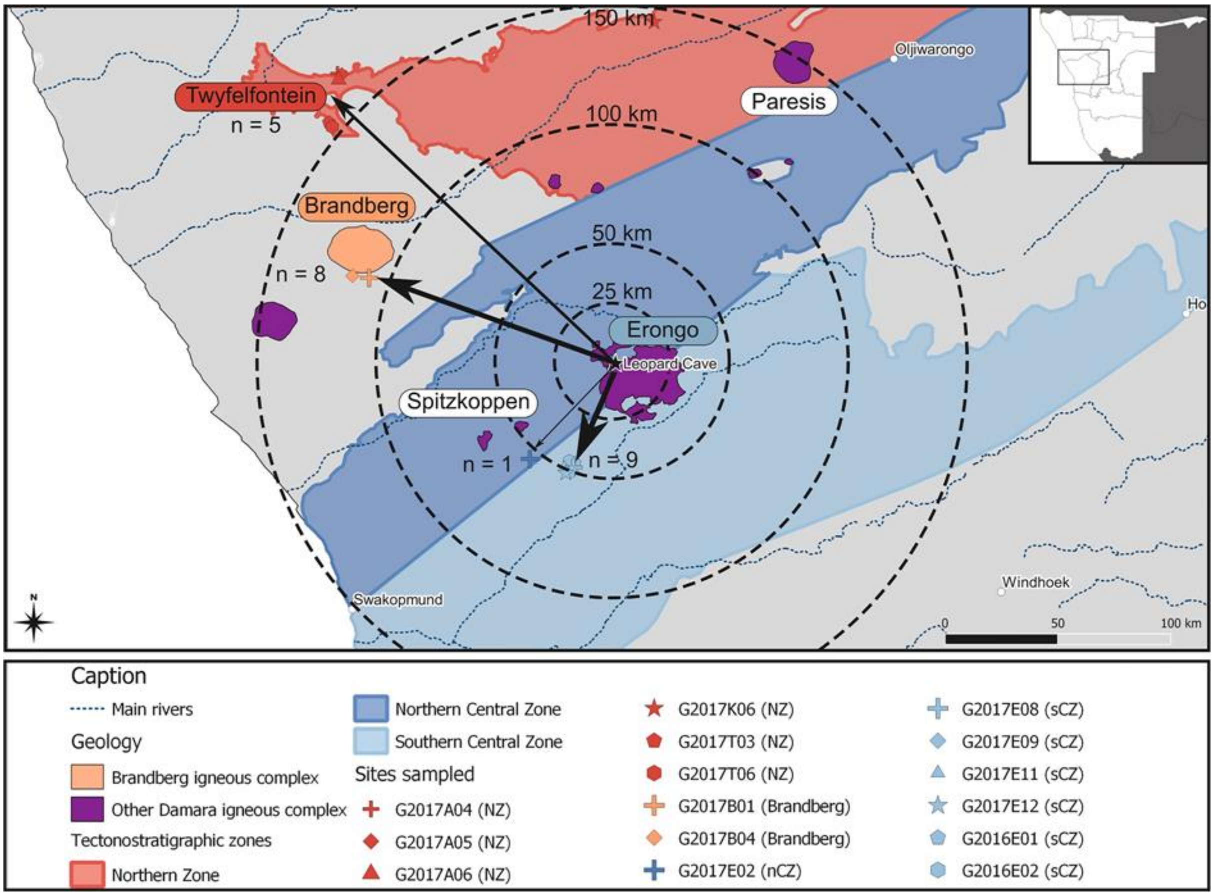

Fig. 6. North-central Namibia map presenting the main provenance zones where the archaeological ochre pieces studied come from.

# 4. Discussion

## 4.1. *Ochre analyses*

ICP-MS/MS and ICP-OES analyses were previously used successfully to study ochre sourcing (Iriarte et al, 2009; Dayet et al., 2016; Roman et al., 2015; Moyo et al., 2016). Measurements performed in these previous studies were carried out on at least 200 mg of samples. The method used in this study was based on analyzing 100 mg of samples (50 mg for ICP-OES and 50 mg for ICP-MS/MS), reducing the size of the samples considerably, thus destroying less of the archaeological samples. While reducing the mass of the samples, this method also allowed us to discriminate ochre originating from distinct tectonostratigraphic zones in north Central Namibia thanks to multivariate statistical analyses.

## 4.2. *Data treatment and differentiation of the ochre zones*

An important issue that should be tackled in future lies in the treatment of compositional data collected from ochre analyses. Our enquiry into the provenance of ochre fragments discovered at Leopard Cave, suggests that the simple log method was the most effective data treatment to properly distinguish the different sources areas considered. Although some previous studies proved the Fe-log-ratio to be efficient for distinguishing ochre sources (Popelka-Filcoff et al., 2007, 2008; Kiehn et al., 2007; Eiselt et al., 2011; Dayet et al., 2016; Zipkin et al., 2020), our results echo other recent studies for which the log-Fe ratio data treatment was not the most effective means to discriminate the sources. Poor results from previous studies were due to the high variability of the assemblages with numerous Fe-oxide sources leading to arbitrary iron-correlated elements (Zipkin et al., 2017; MacDonald et al., 2018; Pierce et al., 2020). We propose that such results call for more investigations of ochre data treatments, so as to define a robust method that can be applied to a large ochre assemblage of varied geological origin.

### 4.3. *Archaeological ochre provenance*

Out of the 41 archaeological ochres analyzed, we could attribute 23 of them to a probable provenance zone. The other 18 unprovenanced ochre fragments highlight the incomplete geological ochre sampling in north-central Namibia. Sampling should be extended in future. The ochre samples of unknown provenance highlight the large diversity of the ochre fragments exploited at Leopard Cave. Among these 18 samples, ten presented chemical signatures close to those of the central zones, suggesting them to be of local provenance, coherent with opportunistic procurement strategies highlighted for the lithic raw material of Leopard Cave (Pleurdeau et al., 2012). The ochre fragments of unknown provenance could either come from unsampled zones or unsampled outcrops within the zones studied here. Moreover, their diversity could be either imputed to the diversity of materials found at an unsampled outcrop or to the exploitation of numerous distinct outcrops. Among the ochre fragments that could be related to one of the provenance zones, our survey and geochemical analyses suggest that the ochres found at Leopard Cave come from both local (within a few km of the site) and regional (about 150 km from the site) ochre sources (Table 2 and Fig. 6). The interviews with local communities all point to long-distance ochre exploitation from the North Zone, these results are important. They demonstrate the existence of local exploitation of ochre outcrops, complemented by collecting of ochre from long distances but still within the regional context. Although the ochre fragments found at Leopard Cave could be the result of multiple occupations from a unique group or of distinct ones, the ochre provenance diversity interrogates about the ochre exploitation strategy of these populations and their ochre usages.

### 4.4. *Ochre exploitation strategy and population mobility*

According to ethnographic studies, mobility can be distinguished in two ways: 1) the foraging radius which defines a daily exploitable area around residential camp and 2) the logistical radius for resources more than a day's walk away (Binford, 1982, 1983; Kelly, 1983, 2013). To cope with diminishing resources over time, hunter-gatherer populations move camps beyond the foraging radius of the previous residential area (Binford, 1982).

Hunter-gatherer populations in arid environments in southern Africa have a foraging radius of less than 30 km. According to Kelly (1983), the hunter-gatherers G/wi had a foraging radius of 25 km while the Dobe ! Kung had one of 24 km. The G/wi, who lived in the Kalahari desert, historically covered a yearly total distance of around 275 km, while the Dobe !Kung covered a much-reduced area of 150 km. The areas around the Erongo and Brandberg Mountains at the ridge of the Namib desert presented a similar environment to the ones where the Dobe !Kung lived. Here, the strategic configuration between two iconic mountains meant that the area might have served as a corridor between the dry and barren Namib and Savanna grassland for migratory bands of hunter-gatherers and large game during dry seasons. With this in mind, the presence of non-local ochre material found at 150 km from Leopard Cave site is significant.

The presence of ochre fragments of diverse provenances with small- scale groups could indicate expedient opportunistic exploitations of numerous outcrops rather than a long-term focus on specific ochre outcrops (Couraud, 1988). For example, members of a failed hunting expedition may decide to acquire seeds or even nonfood resources discovered during the expedition, such as ochre (Binford, 1979; Lee, 1979) and this might result in opportunistic exploitation of diverse ochre outcrops. Thus, the diversity of ochre found at Leopard Cave could be either the result of some logistical expeditions to the Brandberg (8 LC archaeological ochre fragments were attributed to the Brandberg zone) and areas near Twyfelfontein (5 archaeological ochre fragments attributed to the North Zone) or the consequence of local ochre collection that was transported as populations migrated over time. This diversity of ochre could also be the result of numerous occasional occupations of Leopard Cave during which specific outcrops were exploited. Since there is no specific trend that defines the provenance of the ochre pieces and their stratigraphic distribution, the latter hypothesis seems less likely, leaving the possibility for numerous occupations with the exploitation of separate outcrops.

The ochre diversity might also point to a high rate of circulation of ochre fragments between hunter-gatherer camps (Steele et al., 2016). These high circulations might have involved networks over long distances all over southern Africa as mentioned by Ouzman, (2003), involving ochre outcrops of distinct regions of southern Africa. These networks could have involved gift-giving practices similar to the hxaro exchange, defined by Wiessner (1982, 2002) as long-term, semiformal relations of mutual assistance of delayed reciprocity as paid out in a wide range of currencies. The ochre fragments might, in this case, have born a specific value - symbolic or magical, such as lightning protection as mentioned by How (1962) - or for the activities in which they were involved (Dayet et al., 2016). Other scenarios could emerge from the seasonal grouping and later disbanding of hunter-gatherer populations over the region of north-central Namibia. Rock art specialists considered the extensive accumulation of rock art sites in the Erongo Mountains and the Brandberg to be the result of seasonal grouping of LSA hunter- gatherers who inhabited north-central Namibia at the time (Lenssen- Erz, 1994, 2004, 2008; Richter, 2002). Archaeological evidence for seasonal occupation of the Erongo (Wadley, 1984) supports the suggestion. The presence of strong aquifers associated with these Granite Mountains meant that water was likely readily accessible through shallow hand dug wells supplemented by other underground seepages during dry season. This therefore explain the majority of rock art sites observed in these regions. The scarcity of studies carried out on archaeological ochre fragments from various LSA sequences in southern Africa limited our study. Nonetheless, the presence of archaeological ochre fragments attributed to the Brandberg or the North Zone close to the Twyfelfontein rock art cluster implies a high level of human mobility associated to specific adaptations to extreme aridity and environmental uncertainty in north Central Namibia. This highlights potential interactions between the north-central Namibian key rock art areas.

### 4.5. *Interactions between the various central Namibian rock art clusters*

Rock art research carried out at the various rock art clusters of north- central Namibia (Erongo Mountains, Brandberg, Sptizkoppe, Twyfelfontein and Rhino Desert) along with various excavations carried out in these areas introduced some interesting data that can be discussed regarding seasonal mobility of LSA hunter-gatherers (Richter, 1984, 2002; Wadley, 1984; Kinahan, 1990; Lenssen-Erz, 1994, 2004, 2008;

Breunig, 2003, 2019; Richter and Vogelsang, 2008; Nankela, 2017; Breunig et al., 2018). Within these distinct regions, the Brandberg and Erongo massifs have been considered as a favourable environment that could have provided resources to hunters-gatherers populations all year long (Richter, 2002; Lenssen-Erz, 2008; Nankela, 2017) until recently due to their strategic configurations, while the Spitzkoppe, the Rhino Desert and Twyfelfontein area could have only been occupied seasonally due to the lower and restricted underground water sources (Kinahan, 1990; Richter, 2002; Breunig, 2019).

Given the extensive number of sites, the climatic conditions within the region and the ethnographic information about hunter-gatherers in southern Africa, researchers consider seasonal mobility to be important during the LSA with aggregation and dispersal of distinct groups depending on resource availability (Richter, 1991; Lenssen-Erz, 2008). Such practices would have led groups living in the area of the distinct rock art cluster to interact with each other.

According to Richter (2002), distinct groups must have occupied the Brandberg and the Erongo massifs. The author considered that these populations respectively visited Twyfelfontein and Spitzkoppe areas (Richter, 1984, 1991). Our results show rather a complex situation in which human groups living in the Erongo massif seem to have collected ochre from the Brandberg and Twyfelfontein areas. These results interrogate past relationships between the populations who occupied the different massifs. In light of the work of Breunig (2019), questions arise regarding the nature of these relations, but unfortunately, limited data prohibit investigation of these relations. However, numerous sites within these rock art clusters have yielded ochre fragments (Table 3), studying them would help to refine and strengthen our results.

Table 3 List of LSA Namibian rock art sites that have delivered ochre fragments

| Site name | Latitude | Longitude | Reference |
|---|---|---|---|
| **Aar 1** | -24,77 | 16,48 | Vogelsang, 1998 |
| **Affenfelsen Shelter** | -20,58 | 14,38 | Vogelsang, 1998 |
| **Amis 10** | -21,12 | 14,31 | Breunig, 2003 |
| **Apollo 11 Cave** | -27,75 | 17,10 | Wendt, 1972 |
| **Bremen 1** | -25,77 | 17,15 | Wendt, 1972 |
| **Etemba 14** | -21,45 | 15,65 | Richter, 1991 |
| **Etemba 2** | -21,46 | 15,65 | Richter, 1991 |
| **Fackeltrager Shelter** | -21,56 | 15,55 | Richter, 1991 |
| **Falls Rock Shelter** | -21,18 | 14,56 | Kinahan, 2018 |
| **Geister höhle Shelter** | -21,85 | 15,07 | Wendt, 1972 |
| **Girafe Shelter** | -21,53 | 15,56 | Pleurdeau unpublished |
| **Hasenbild** | -20,58 | 14,38 | Vogelsang, 1998 |
| **Leopard Cave** | -21,57 | 15,56 | This study |
| **Maguams Terrace** | -25,53 | 16,87 | Wendt, 1972 |
| **Messum Shelter 2** | -21,37 | 14,28 | Vogelsang, 1998 |
| **Omungunda 99/1** | -17,78 | 13,68 | Vogelsang & Eichorn, 2011 |
| **Philipps Cave** | -21,80 | 15,64 | Wendt, 1972 |
| **Pockenbank** | -27,22 | 16,52 | Wendt, 1972 |
| **Rain Cloud** | -21,52 | 15,58 | Pleurdeau unpublished |
| **Seal shelter** | -21,58 | 15,54 | Pleurdeau unpublished |
| **Striped Giraffe Shelter** | -21,77 | 15,70 | Sandelowsky & Viereck, 1969 |
| **Tiras 5** | -26,20 | 16,58 | Wendt 1970 |
| **Umuab 21** | -21,05 | 14,35 | Breunig, 2003 |
| **Ururu** | -20,50 | 14,05 | Wendt, 1972 |

Future studies should subject more archaeological samples from Leopard Cave to non-destructive analyses such as external beam PIXE to provide new information on the ochre fragments that are anthropogenically modified. Such studies should include other Erongo LSA rock art sites that have delivered ochre fragments like those from Facketlräger, where a large amount of hematite was recovered within archaeological layers contemporary with the LSA occupations of Leopard Cave between 5000 and 2000 BP (Richter, 1991; Mauran et al, 2020c). In doing so, it would be interesting to consider sites that have delivered ochre fragments in the Spitzkoppe, Brandberg, near Twyfelfontein and in the Rhino Desert to compare the ochre procurement strategies between these areas and to determine whether some traditions, social interactions and the procurement strategies were similar in the distinct cluster. In this regard, the reference collection presented in this article provides the initial step for ochre provenance studies in Namibia. This research will therefore serve as a reference for archaeological ochre assemblages from other sites in Namibia. Combining both rock art studies and ochre provenance would provide key information about prehistoric hunter-gatherer land-use in the region and the possible interactions between the distinct groups. Enlarging these studies over southern Africa would also permit the confirmation or rejection of the existence of long-distance networks during the LSA.

## 5. **Conclusion**

Multivariate statistics of geological ochre compositional data demonstrate the efficiency of our micro-sample ICP-OES and ICP-MS/ MS protocol to distinguish ochre sources and they confirm the Provenance Postulate to be met for north-central Namibian ochres.

We consider that the present study is a critical first step in developing micro-destructive LA-ICP-MS/MS approaches for the analysis of ochre fragments, successfully applied elsewhere to distinguish ochre samples in different contexts (Green & Watling, 2007; Gil et al., 2009; Scadding et al., 2015; Zipkin et al., 2017; 2020) and to analyze rock art pictorial layers (Green & Watling, 2007; Resano et al., 2007; Bu et al., 2013; Scadding et al., 2015). Therefore, our study presents the first Namibian analysis of ochre and it serves to study rock art pigment and to correlate Namibian rock art pictorial layers with excavated ochre fragments (Mauran et al., 2020a). Such correlation could be achieved by in situ measurements with methods such as pXRF but due to the fact that pigments might have been mixed with other raw materials or might have become altered, these analytical methods analyze both the pigment and the substrate (Mauran et al., 2019).

Future LA-ICP-MS/MS studies should address the representativity of the micro-samples compared to larger ones. As strongly emphasized in the literature, ochres are heterogeneous materials. Therefore, it is necessaryto question the representativity of micro-sample analyses and to determine the numbers of analyses required to ensure that LA-ICP-MS/ MS analyses performed are representative of the whole sample analyzed. We believe that our micro-sample liquid ICP-MS/MS method could help to address these questions. As other issues include in the lack of adequate standard matching ochre matrix, we also think our method could be of great help in analysing and providing important data sets for some references as like those created by Salomon & Chalmin (2019). Indeed, our method provides precise compositional data from a small number of samples allowing to us to perform more analyses and provide robust data sets for future studies.

Ochre reference collection from north-central Namibia built up during our provenance study of archaeological ochre from Leopard Cave, paves the way for future studies on Namibian ochre archaeological assemblages. Our micro-sample ICP-OES and ICP-MS/MS protocol allowed us to use our modern ochre reference collection to analyze 41 archaeological ochres from Leopard Cave, Namibia. Therefore, our results provide crucial archaeological data because they have securely determined the provenance of 23 archaeological ochre fragments. We have shown that the LSA populations who occupied Leopard Cave collected ochre both from local and regional outcrops. Regarding the socio-cultural behaviours of the LSA populations, these results confirm people's mobility within the Namibian Karoo biome at the edge of the Namib Desert. Wheter the material diversity is due to an opportunistic procurement strategy or a large raw material exchanged network remains in question, and this should be further investigated. To do so both local geological surveys within the Erongo Mountains will be carried out and future provenance studies should include other sites in the region, including Fackelträger, Rain Cloud and Seal Shelter all located less than 10 km from

Leopard Cave. Our initial work and the sampling of additional sites should be integrated with analyses of ochre chaînes opératoires to consider the connection between the primary and secondary outcrops and hence gain a better understanding of the LSA population ochre procurement strategies. Our ochre provenance data reveal the existence of interactions between the people who made the distinct rock art clusters of north-central Namibia during the LSA. This has implications for other similar studies, within the sub-continent. Future geological surveys and LSA ochre provenance studies will be of utmost importance for the study of potential long-range networks through southern Africa.

## 6. Lay Summary

Namibian prehistoric rock paintings suggest a possible technical link between different key rock art sites in central Namibia. We collected raw modern ochres and used a technique to identify their geochemical fingerprints. We then applied statistics to evaluate the origin of ochre samples. The application of this method to ochres possibly used for ancient paintings confirmed that past communities collected ochres originating from different Namibian regions rich in rock paintings.

## Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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## Supplementary data

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