

## 7.0 CHAPTER SEVEN



### **Summary: Field Based Archaeometric Pigment Characterisation**

Our knowledge of the ochres used by Aboriginal people is rudimentary, not only in respect of their materiality (physical characteristics), but also their association in cultural landscapes. Characterising the geochemistry of rock art provides an alternate perspective within regional studies, one that can focus on intentional interactions between people and the landscape rather than cultural diffusion, if we relinquish the goal of assigning provenance to pigments. The archaeometric investigation of ochre undertaken in this thesis offers a means of integrating site specific data into the examination of regional scale cultural systems (Smith et al. 1998) and in turn, an opportunity to integrate the geological scale landscapes represented by pigment sources into regional archaeologies (Eiselt et al 2011; Linse 1993; MacDonald et al. 2012). This thesis has also demonstrated the conservation and management implications of characterising the geological environments which house rock art (**Chapters Two and Three**; Ford et al. 1996; Ogburn et al. 2013).

In the 1990s, seminal research demonstrated that ochre characterisation can provide valuable evidence of past cultural systems (Smith et al. 1998), hinted at the potential of nationwide provenance research through the analysis of ethnographically known ochre mines (Jercher et al. 1998; Smith and Fankhauser 2009, 1996) and archaeological specimens (David, 1993; Goodall, 1996), and documented the limitations of unsuccessful methods trialed (Smith et al. 1997; Smith et al. 2002). Around twenty years on, archaeometric pigment analyses have rarely been integrated into regional archaeological studies (cf. MacDonald et al. 2011) and provenance investigations languish, still concerned with evaluating techniques (Green and Watling 2007; Popelka-Filcoff et al 2012a, 2012b; Scadding et al. in press 2014). In 2005, Newman and Leondorf demonstrated the archaeological value of qualitative pigment analysis by describing the major mineral constituents of rock art paint and highlighting the cost-effective nature of recent generation commercially produced portable spectrometers. Yet, ten years on, only a handful of pigment studies have

analysed rock art using these instruments (Bedford 2013; Bonneau et al. 2012; Huntley 2012; Huntley et al. in press; Huntley and Freeman in press; Koenig et al. 2013; McDonald et al. 2014; Olivares et al. 2013; Prinsloo et al. 2013; Roldan et al. 2010; Sepulveda et al. 2012; Tourniè et al. 2010; Velliky 2013; Wesley et al. 2014).

Despite early progress in Australia, archaeometric studies of ochre have been fundamentally constrained by the expense of large-scale analytic programs. In addition, the sampling requirements of destructive and/or composition altering techniques have inhibited the scientific analysis of rock art. The advent of portable spectrographic techniques some thirty years ago began to alleviate these issues; however, the accessibility of instruments driven by recent generation commercial production has seen a flurry of compositional analysis in archaeological assemblages including lithic technologies, ceramics, buildings, excavated sediments and metallic artefacts. The scarcity of pXRF investigations of ochre, and rock art pigment in particular, despite the accessibility and ubiquity of instrumentation, can be attributed to an absence of critical scrutiny, explanation and specific methodologies for this application. The papers presented in this thesis have gone some way to redressing that gap; providing a means by which large-scale studies of the elemental character of rock art and other archaeological ochres can be undertaken. Explicit consideration of the complex taphonomy of rock art panels (**Chapters Two, Three, Five and Six**), surface finds of archaeological ochres (**Chapter Three**) and pigments from excavated contexts (**Chapter Five**) also sets this work apart, as prior studies had not considered nor addressed the chemical indices of post-depositional processes in such a comprehensive manner. By separating characterisation from the assignment of provenance, this research not only outlines the technical means for applying pXRF to rock art, but offers a fresh conceptual framework for archaeological pigment research (**Chapter One – Section 1.2.1**).

This research began with three interconnected aims, a) provide insight into the cultural context of rock art production and b) thereby complementary strands of evidence, and more fine-grained data to assist in the development of a more robust chronology for rock art assemblages, while c) demonstrating how archaeologically informative qualitative and semi-quantitative geochemical characterisation of archaeological ochre, especially rock art pigments, can be by illustrating alternate chronological and behavioural perspective within regional studies. The following two sections summaries the archaeological findings of the Sydney Basin and northwest Kimberley case studies. These case studies demonstrate the archaeological significance of qualitative and semi-quantitative geochemical characterisations and how describing the elemental composition of pigment can offer an alternate strand of behavioural and chronological evidence, showing the sorts of insights into the cultural context of rock art production that can be gleaned from this style of research. I also draw together the evaluation of pXRF and set out what I consider the important caveats for rock art applications. I restate the three novel methods for pXRF analysis of rock art presented in this thesis, discuss the future of pigment research in Australia and the conclusions drawn by this research program.

### 7.1.1 Archaeological implications: the Sydney Basin

The archaeologically significant findings of the Sydney Basin Case study were:

- Demonstrating that there is no geochemical evidence to support contemporaneity of rock art and excavated pigment at D&HA and Y1 rockshelters (**Chapter Five**). This is a significant finding not only in the context of the Sydney Basin case study, but for all regional prehistories. It shows that pXRF can be used as a means of testing the indirect evidence used to argue for temporal frameworks of rock art production where age estimates associated with ochres from subsurface contexts are extrapolated to chronological frameworks for rock art of the same colour. That pXRF can show there is no relationship in these circumstances makes available a readily accessible and inexpensive way to test hypotheses of this sort. Where similar pigment characteristics are noted, pXRF provides a valuable screening tool with which to direct further archaeometric work—able to select samples for subsequent high resolution analyses that are known to be similar in geochemical composition.
- pXRF analyses were able to replicate the geochemical trends observed in previous PIXE/PIGE and XRD datasets from BR29 on the southern Woronora Plateau. This is significant because it demonstrates how it is possible to turn the results of prior analyses into hypotheses testable by pXRF and therefore to exploit the non-invasive and rapid nature of the technique within well designed field programs. The findings of this case study give confidence to the further use of pXRF in testing the hypothesis that rock art paints in the region are composite clay based recipes with a preference for local sources (within the Winatamagga group shales and the Illawarra Coal measures). pXRF affords the opportunity to test this hypothesis at different spatial scales, within art panels across a site, sites within a complex and sites across the region.
- **Chapter Five** has reported the first known use of calcium matrix archaeological ochres in the Sydney Basin at Y1 rockshelter. Though used since the mid-Holocene, the increasing archaeological visibility of these pigments in the recent period suggests increasing exploitation of calcium sources at a time when increased social contact between groups is proposed (McDonald 2008). The use of clay matrix red pigments is seen throughout the archaeological sequences suggesting that local sources, including detrital lenses of local sandstone, continued to be exploited through time. Although these findings are based on a small number of samples, the different patterns evident in white and red ochres illustrates the more nuanced picture which begins to immerge when the whole ochre assemblage is analysed. This case study also illustrates that patterning of archaeological interest is clearly evident in qualitative characterisation of archaeological pigments (indicating major mineral constituents).

### 7.1.2 Archaeological implications: the northwest Kimberley

The archaeologically significant findings of the northwest Kimberley case study were:

- The development of a method for differentiating between major mineral constituents in distinctive mulberry rock art paints found across tropical northern Australian (known colloquially as the top end) (**Chapter Six**). This is a significant contribution to the prehistory of the region as pXRF could be used to identify distinctive mineralogy within the geographic and stylistic rock art provinces described across the top end, and as the example of Gwion motifs shows, within typologically coherent rock art assemblages (stylistic sequences).
- This work has documented the first archaeologically described, geochemically analysed mulberry ochre quarry in northern Australia (**Chapter Six**). This is a significant contribution to the regional archaeology of the Kimberley and broader top end because it shows the type of geomorphic and archaeological context in which mulberry ochre were being procured. Mulberry pigments are available locally within the siltstone beds occurring throughout the sandstone geology of the northern Kimberley. Mulberry pigment sources are also found within sites that contain large and diverse rock art assemblages, surface finds of ochre and diverse subsurface ochre assemblages.
- Archaeologists have long understood the importance of climate and prevailing environmental conditions in influencing human behaviour. The structural analysis of a mineral accretion from a rock art panel in the northwest Kimberley presented in the  $\mu$ -CT reconstructions of **Chapter Three** and SEM micrograph presented in **Chapter Six** indicates accretions to be seasonally episodic and rapidly forming. These skins may therefore provide valuable archives for palaeoenvironmental information and information relevant to the taphonomy and conservation and management of rock art.
- **Chapter Three** has shown that leaching of ochre matrix minerals is more rapid and therefore more advanced in surface finds of archaeological pigment when compared to those from subsurface archaeological deposits. This leaching has implications for the success of ochre provenancing investigations. Further, the leaching of matrix minerals in subsurface ochres may provide a proxy dataset for the seasonal water inundation of archaeological deposits in the monsoonal environment of the Australian top end—in turn a taphonomic proxy for the preservation of archaeological finds.

## 7.2 Evaluation of pXRF for Archaeology and Rock Art Research

Does pXRF work? Of course it does, but it can only do what it can do. Like any other scientific techniques, pXRF is the product of physics (Huntley 2012—**Chapter Two**) and mechanics (Potts 2008; Shackley 2011). Like other portable spectrographic techniques such as Raman and Fourier Transform Infrared, recent generation commercially produced hand held pXRF's are not equivalent to laboratory instruments and therefore cannot replace them in an analytic program. Portable spectrometers operate using the same physical principles to measure the same

phenomena as their laboratory counterparts; however, the mechanics of field portability, such as the miniaturisation of X-ray tubes in pXRF, limits their precision through reduced power output. It remains the case that successful archaeometric studies rely on selecting the correct technique to measure the archaeological phenomena of interest. You need the right tool for the job. There is simply no point in trying to use a screw driver if you need a hammer.

It would be easy to be blind to the archaeological potential of pXRF because of the complexities and limitations it, and any *in situ* spectrographic technique, encounters when examining archaeological pigments and specifically rock art. As I have outlined in **Chapters Two, Three, and Five** a myriad of matrix effects are re-introduced when analysing unprepared materials. Added to these complexities, field based pXRF sampling may introduce a number of X-ray attenuation effects. As discussed in **Chapter One**, obsidian sources/lithic technologies are exemplar materials for pXRF analysis because of their homogeneous, fine grained nature that suffers minimal (if any) mineralogical segregation. Each flow (obsidian source) has highly distinctive chemical characteristics that express differentiation in the suite of minor and trace elements measured by pXRF. If obsidian is shooting the proverbial fish in a barrel, the field based analysis of rock art pigments with pXRF is analogous to shooting fish in the open ocean, with a low powered rifle, from the deck of a moving ship. Yet this thesis has shown it is not only possible to analyse rock art with pXRF, a range of scientifically robust and archaeological useful data can be generated.

pXRF can measure the relative abundance most elements of interest in rock art research and a lot of elements that are of interest in conservation and management (**Chapters Two, Three and Five**). For instance, I measured eight transition metals with pXRF in this research (**Chapter Five**), commensurate with laboratory studies of archaeological ochres that have measured six transition metals by Instrument Neutron Activation and six transition metals by laboratory based energy dispersive XRF (Popelka-Filcoff et al. 2007a:22). pXRF can identify the chemical constituents of cavernous geological weathering before salts and precipitate mineral skins are visible on the surface of a panel, an important finding for rock art conservation and management (**Chapter Three**). pXRF can also measure elements indicative of past rock art recording techniques, such as the application of chalk (**Chapter Five**). Able to differentiate between ochre matrix minerals (clay and calcite phases), pXRF can provide information regarding the geomorphic context of pigment procurement locations (**Chapters Five and Six**). Matrix mineralogy distinguishes ochres from different sources and therefore, irrespective of the assignment of provenance, can show change or continuity of ochre procurement through time and across space. However, **Chapter Four** serves as a reminder that pXRF also has the potential to effect concurrent, and possibly subsequent, archaeometric investigations.

### 7.3 Caveats for *in situ* pXRF Analysis of Rock Art

Successful archaeometric research needs to recognise and understand machine bias, method bias, and observer bias, in order that these factors are not interpreted as a cultural patterns (Plog 1982:70). The intensive field program of rock art analysis conducted in the Sydney Basin culminated in a protocol for data collection that was subsequently used in the northwest Kimberley (**Appendix A**). Rather than setting out a prescriptive protocol relevant to only one instrument or manufacturer, I outline below a set of considerations for successfully gathering archaeological data, in the field, via pXRF.

#### 7.3.1 Selecting Analyte Locations

The single most important decision directing the collection of any evidence is where to look. The unprecedented sampling freedom afforded by field portable, non-invasive spectrometry has facilitated almost unrestricted sampling opportunities. It is therefore vital to stop and critically consider what environmental or cultural phenomena you want to measure before selecting analyte locations prior to data collection.

A well designed sampling strategy should account for the following:

- Select areas for geochemical characterisation that address the aims of research. As discussed in **Chapter One**, chemical characterisation must be archaeologically valid. Selecting analyte locations with defined research questions in mind will produce archaeologically meaningful data.
- For materials to be comparable they must have the same relative density (Karydas 2005; Karydas et al. 2005), therefore field based analysis should be conducted following several weeks of dry weather ensuring the moisture content of the stone massive is even.
- Ensure that any other scientific analyses have been conducted prior to pXRF, unless you are certain that the ionising radiation delivered by the technique will not affect these. Although pXRF is touted as non-destructive, it irradiates the surface of the sample and may have short to medium term effects on other scientific techniques (**Chapter Five**). Analysts should also be mindful that ionising radiation can be detrimental to organic materials such as the micro-flora that may be present on the rockshelter/cave surfaces (lichens, etc.) (**Chapter Three**).
- Avoid superimposed pigments, mineral accretions and sites of geological weathering (precipitous mineralisation, case hardening, salt efflorescence etc.) – unless these are the attributes targeted by the investigation as maybe the case for conservation research. In other words, try to get a ‘clean shot’ of the phenomena you are trying to measure.
- Make detailed observations about analyte locations in order to contextualise the interpretation of element profiles. These observations should include not only the attributes of the rock art panel that are likely to produce chemical characteristics incorporated into element profiles, but also anything that might attenuate X-rays resulting in loss of fluorescent yields, such as biofilms.



- Select areas for analysis that contain the thickest extant pigment. The more pigment by volume in the analyte, the stronger the chemical signature of the paint in the spectra generated.
- Understand the rockface. The chemistry of the stone substrate will inevitably be incorporated into a dataset. Understanding mineral variance in the stone massive and the likely chemical signatures of weathering processes such as case hardening, mineral accretions/skins and precipitous mineralisation such as iron-oxides, silica/calcites and salts is critical to separating the chemical signatures of environmental and cultural processes. Analysis of rock surfaces containing no applied pigment will qualify the chemistry of the rockface. However, it is not feasible to merely 'subtract' the chemistry of the rock as a background within pXRF spectra as has been suggested (Olivares et al. 2013) because ochres are themselves products of geological weathering (Rowe 2001; Watchman 1990). Both the rock substrate and rock art pigments have a degree of naturally occurring geochemical variation, the amplitude of which should be qualified. In the northwest Kimberley (manuscripts in preparation) I deliberately targeted areas of the rockface with mineral accretions, case hardening and fresh flake scars in order to understand the chemical expression of geological weathering, rather than simply analysing the adjacent rock matrix as previous studies have done (Koenig et al. 2014; Leondorf and Leondorf 2013; Newman and Leondorf 2005; Nuevo et al. 2012; Olivares et al. 2013; Rowe et al. 2011; Velliky 2013; Wesley et al. 2014).

### 7.3.2 Instrument Parameters

Much of the literature in relation to the application of pXRF in archaeology has focused on evaluating the scientific merit and rigor of the technique. These studies have stressed the importance of fundamental scientific practices such as instrument calibration and inter-laboratory replication (Craig et al. 2007; Heginbotham et al. 2010; Speakman and Shakley 2013; Speakman et al. 2011) as well as highlighting sources of potential error specific to the technique (Johnson 2012). In this regard, the following should be noted during field based data collection and subsequent data processing/analysis:

- It is desirable to collect both light and heavy element optimised data sets for rock art analysis (**Chapter Five**). Diagenetic and post-depositional chemical alteration are chiefly expressed in light elements as these are the most mobile (Ogburn et al. 2013:1823). Whereas diagenetic chemical expression is observed in transition metal and rare earth elements (MacDonald et al. 2012:12).
- The precision achieved through longer spectral acquisition is considered adequate to minimise the effects of instrumental drift as pXRF pulses stabilize during longer assays (Johnson 2013:6; Erdem 2008:2489).

- The effects of S-Drift in light element datasets appear to be within the order of magnitude of the detection limits of the instrument (Johnson 2012:7-8). These can be removed by the exclusion of minimum detection limits in the datasets, however data close to detection limits may be desirable and can proceed but with caution (Speakman and Shakley 2013).

### 7.3.3 Data Processing

Several scholars have pointed out the 'black box' nature of fundamental parameters calculations for XRF (Heginbotham et al. 2010). It is clear from the case study presented in **Chapter Two** (Huntley 2012) that pXRF cannot readily exceed a semi-quantitative resolution when analysing rock art. However, this is no cause for analytic apathy. Semi-quantitative or qualitative data still demands rigorous scrutiny to remove any spurious measurements.

- It is important to recognise minimum detection limits and understand the amplitude of variance in pXRF measurement for different materials. Amplitude of analytical error increases with proximity to the limits of detection for each element measured (Johnson, 2012; Speakman and Shakley 2013:1438-9). With replicate analyses I was able to confidently analyse low abundance light elements (**Chapter Two**) and this may be desirable, particularly in relation conservation research.
- Systematic collection of an internal standard for each dataset, one that is compatible with the chemical characteristics of the rock art paint, is highly recommended (I have used Ohio Red Clay). The standard should be analysed at the same time during each session and recent studies suggest that interspersed standard spectra during the analytic session are desirable (see Johnson 2012).
- With any calculation of concentrations, including semi-quantitative measures/relative abundances, it is critical that the parameters used to arrive at the 'number' are reported. This is especially so for fundamental parameters calculations that are notoriously a bit of a 'black box' (Heginbotham et al. 2010). I have reported the variables selected within the manufacturer supplied software (number of corrections cycles and energy range) in all methodology sections (**Chapters Two, Four and Six**) to ensure replicability.

## 7.4 Novel methods for *in situ* pXRF analysis of Rock Art

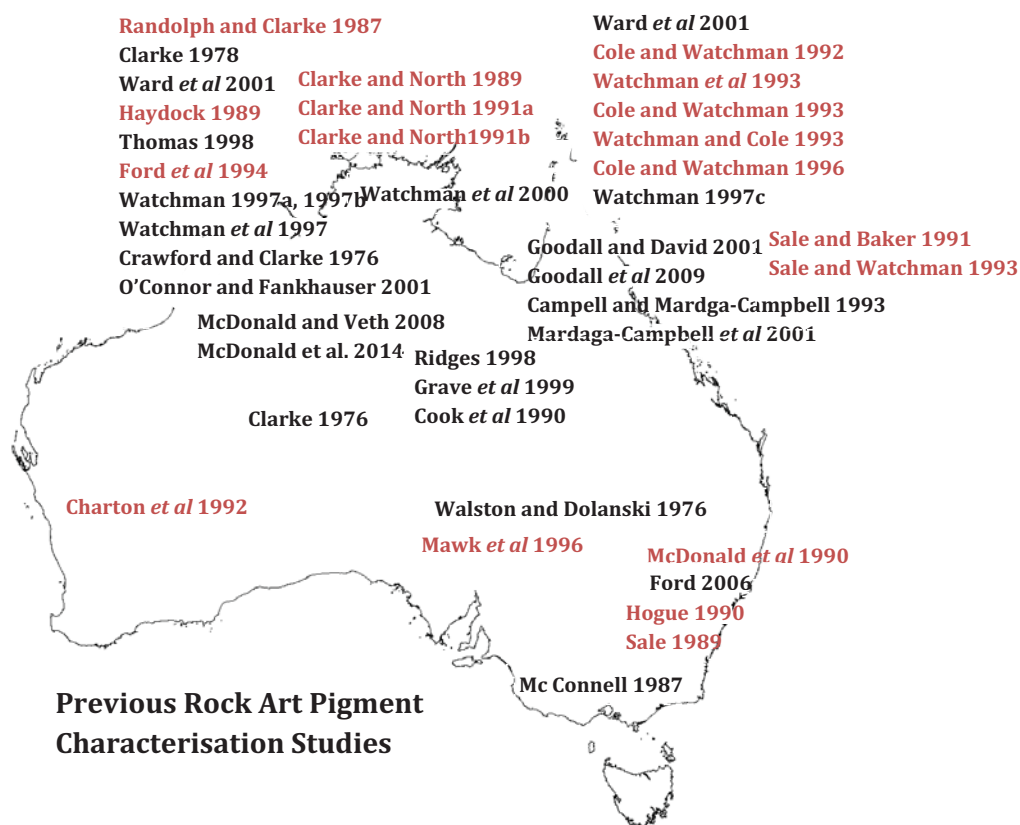
The journal articles included as **Chapter Two** and **PART II** of this thesis each outline a different, innovative method for the pXRF analysis of rock art. These methods set out ways of characterising archaeological information using pXRF. The application of these novel methods demonstrates how pigment characterisations can generate alternative perspectives within regional studies and provide insights into past Aboriginal cultural landscape use. The novel methods of data treatment and analysis included in **Chapters Three, Four and Six** also address the third aim of this research by demonstrating the conservation/management value of qualitative and semi-quantitative geochemical data.



- **Chapter Two** outlines a method whereby groups of elements that represent different cultural and taphonomic phenomena are identified and scrutinised in comparison with a spatial transect of the underlying rock matrix. The revolution of this method is that it is prefaced upon identifying the chemical signatures of rockshelter taphonomy, something not adequately considered or addressed to date in published studies (David et al. 2013; d’Errico et al. 2012; Koenig et al. 2014; Leondorf and Leondorf 2013; McDonald et al. 2008, 2014; Newman and Leondorf 2005; Nuevo et al. 2012; Olivares et al. 2013; Prinsloo et al. 2013; Rold’an et al. 2010; Rowe et al. 2011; Rifkin 2011, 2012; Tourniè et al. 2010; Wesley et al. 2014) or graduate research (Bedford 2013; Velliky 2013).
- **Chapter Five** has set out a method that compares rock art and excavated pigments, to determine if there is geochemical evidence in support the assertion made by archaeologists during previous work, that age determinations associated with excavated pigment can be extrapolated to inform the chronology of the rock art assemblages. The method sets out instrument parametres, data collection protocols and chemometric procedures. The innovation of this method is in identifying elements in rock art that represent ochre digenesis, elements in excavated pigments that represent rockshelter taphonomy and in offering a means to distinguish between these geological environs. The use of standard cheometric procedures such as iron normalisation is also novel in this study because not all the ochre or rock art analysed are red. As a scale reduction method iron normalisation is successful in comparing trends across the dataset. Further, ratioing elements to iron assists in recognising environmental processing such as case hardening.
- Previous studies have identified the presence of different mineral pigments using the characterisation of major chemistry (heamatitie—Koenig et al. 4, Nuevo et al. 2012; furchsite—Newman and Leondorf 2005; clay, maganise-oxide, iron-oxide— Rold’an et al. 2010). The innovation of the research documented in **Chapter Six** is that it has differentiated rock art of the same colour between species of iron minerals (hematite and jarosite).

## 7.5 The Future for Archaeological Pigment Studies in Australia

Between 1986 and 1998 the Australian Institute of Aboriginal and Torres Strait Islander Studies (AIATSIS) Rock Art Protection Program funded 174 projects including major rock art conservation science and chronometric investigations (Ward 2011:10, his Table 2). This national scheme, with additional support from state government conservation programs and supplementary institutional funding for the experts involved, was responsible for the bulk of previous archaeometric works conducted in Australia (**Figure 7.1**).



**Figure 7.1.** Map illustrating the location of pervious projects. Red text shows studies funded by the AIATSIS Rock Art Protection Program.

Until the research documented herein (including **Appendices C and D**), no archaeological science focused works had occurred on the rock art of either case study region subsequent to the end of the AIATSIS Rock Art Protection Program. Although AIATSIS remains interested in furthering rock art research as one part of their mandate (Taylor and Veth 2008), the absence of dedicated schemes that target rock art conservation is evident in the lack of of archaeometric investigations undertaken following the end of the dedicated funding stream. There is time pressure to stimulating further material science and conservation focused rock art research in Australia. The experts who undertook prior investigations are reaching/have reached retirement or have already moved on from these specific pursuits. Although published works and detailed unpublished reports are available from the AIATSIS Rock Art Protection Program projects, there is a heavy practical aspect to rock art research and materials science. Without stimulus to involve younger generations with the required expertise alongside the archaeologists, conservators and scientists who undertook these seminal projects, we are in danger of losing the intensive, multi-faceted corporate knowledge accumulated.

The future of archaeological pigment research in Australia requires projects that bring to bear the legacy of archaeological, provenance and conservation science research from the 1990s, together with the advantageous attributes of recent generation, non-invasive field based analysis. Archaeologists have a variety of analytic tools at their disposal to investigate past ochre

use. There is a body of technical literature that has appraised these techniques, outlining their limitations and potential specifically as tools for examining pigment. This thesis has built on the advantageous attributes of cost-effective, non-invasive geochemical analysis afforded by current generation, commercially produced pXRF, explaining and evaluating the technique, and offering several innovative methods for applying it to rock art. The case studies in **Chapters Two, Three, Five and Six** have shown the types of archaeological information that can be generated and most importantly how these can inform our understanding of past regional systems. What is required to advance this area of research in Australia is the use of these scientific tools in archaeological investigations.

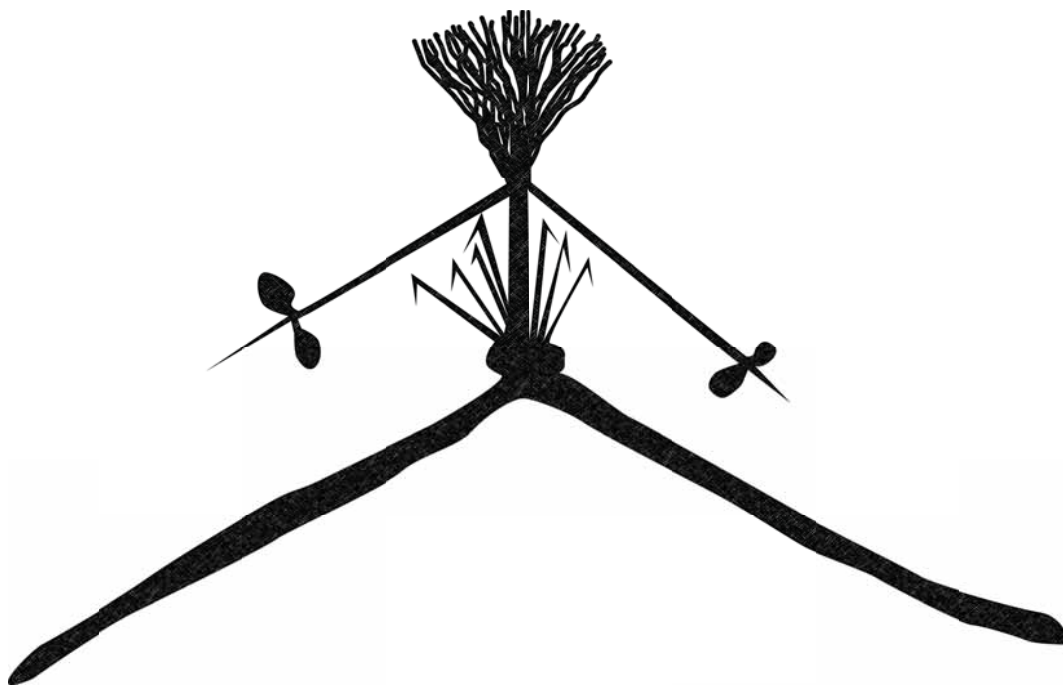
## 7.6 Conclusion

In conclusion, it is not always the highest resolution scientific data that produces the most insightful archaeological findings. Simply knowing what mineral was selected, collected and subsequently used to make rock art and/or observing patterning in the preference for some minerals over others within ochre assemblages, through time and across space, can inform us about the lives of people in the past and the ways they interacted intentionally with each other and with the landscape. In Australia, indigenous peoples have always held a close spiritual connection to the physical landscape. Rock art pigments offer unique archives into the fluidity or stability of that relationship. It is not straightforward to either collect or interpret geochemical profiles with pXRF, however it is possible to generate scientifically robust datasets, to separate cultural from taphonomic signatures and this thesis has shown, it is archaeologically rewarding to do so.

What I have proposed here is not only a technical means with which large-scale investigations of archaeological ochres can be undertaken. I have advocated a paradigm shift from a focus on provenance to characterisation, from explanatory frameworks that imply diffusion to those that illuminate intention, and from concentrating on archaeological evidence which is mobile (ochre pieces) to the incorporation of archaeological evidence which is stationary (rock art). By expanding archaeological ochre investigations in these ways the advantages of recent non-invasive spectrographic instrumentation can be harnessed to develop more multifaceted understandings of prehistoric pigment use. Qualitative and semi-quantitative geochemical data from ochres are capable of providing alternate and additional strands of behavioural and chronological evidence in regional investigations. The non-invasive methods presented here offer unprecedented potential, whether used as a screening tool within a broader archaeometric program, or as a means of providing regional or even intra-regional scale datasets. As the evaluation phase associated with the use of pXRF in archaeology draws to a close, the parameters for scientifically and archaeologically robust investigations having been established, it is time to get down to the business of doing archaeology. I look forward to the prudent adoption of pXRF and other archaeometric techniques within regional archaeological investigations, to ochre analyses becoming routine within these, and to the new insights archaeological pigment studies will provide, no longer constrained by the assignment of provenance.



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## Appendix A

### Protocol for *in situ* pXRF analysis of rock art using a Bruker Tracer III-V I spectrometer

By the end of the intensive Sydney Basin field program chronicled in this thesis, the following data collection protocol was adopted:

1. Start instrument (must wait <five minutes for the X-Ray tube to come to temperature).
2. Select analyte locations, minimising matrix effects and attenuation where possible. When selecting analyte locations ensure an understanding of environmental phenomena that may be captured in the composite spectra. This may include mineral accretions, micro-organics and rock substrate surfaces (case hardened and fresh surfaces, plus a representative spatial sample to explore chemical variance within the substrate). Where sampling rock art: avoid superimposed pigments if possible and select locations of thickest pigment to increase the pigment volume in pXRF spectra.
3. (Once instrument is 'warmed up') Begin collecting analytes on the internal standard for the dataset. Collect both heavy and light element optimised data. Collect 3 consecutive replicate analytes of 300 analytic seconds duration each at FWHM between 150 and 185. Light z-element parameters - 12 keV, 20  $\mu$ A, using a 25  $\mu$ m titanium filter in the X-Ray path, under vacuum. Parameters for heavy z-element analytes - 40 keV, 13  $\mu$ A, using a 76  $\mu$ m copper/305  $\mu$ m aluminium/6  $\mu$ m titanium filter in the X-Ray path, no vacuum. *Note: recent research suggests the collection of spectra on the internal standard during the use session to help identify (and quantify) instrument drift.*

4. Monitor spectra acquisition to look for any irregularities in instrument performance, X-Ray attenuation or spectral response (secondary fluorescence or other phenomena).
5. Record rock art panel including anything that might be included in the chemical profiles generated, or might attenuate the X-Ray signal. This includes the incident angle of the beam if mounting the instrument aperture against the rock art panel is complicated.
6. Select an analyte location. Collect three consecutive, replicate analytes of 300 count seconds each using light element parameters, followed by 3 consecutive replicate analytes using heavy element parameters. To ensure consistency do not move the instrument between consecutive analytes. Ensure the same contact between the aperture and panel when switching between light/heavy parameters (after changing the filter). Make specific notes about the analyte location.
7. Ensure the collection of a 'rock matrix blank'. That is, select at least one analyte location on the panel without applied pigment. Where possible I collected element profiles on freshly exposed surfaces such as spall flake scars, and on case hardened surfaces, with additional analyte locations over mineral skins (where present). Again, try and ensure a representative spatial sample to explore chemical variation in the substrate.
8. Note any irregularities that could result in instrument or observer bias during the analytic session. For instance, if the aperture window fails under vacuum, the vacuum losses suction, the instrument lead come loose, the tripod shifts or the battery of either the pXRF or operating system (PDA or laptop) fails during the session.

**Tips:**

1. Select areas for analysis that can answer your research question. This might seem trite, however, it is important that spectral acquisition has a focus and that the research questions posed are well enough resolved that they can be answered by the analyses conducted.
2. The manufacturer's vacuum windows are expensive. I used 10  $\mu\text{m}$  Mylar mounted by duck tape, cutting the circle into tape with a commercial, hand held leatherwork punch that was larger than the pXRF instrument aperture.
3. Post-processing – select settings for the fundamental parameters calculation that address the dataset and be consistent. For instance, one set of parameters should be applied to any data that will be compared. Think broadly about the data beyond the immediate investigation. It is worth calculating relative abundances for any robustly measured element within the spectra.
4. Clearly record the instrument and processing parameters.
5. Clearly record analyte locations and deposit these recordings with agency site cards, institutional reports etc. Remember that pXRF irradiates the analysed area and may have unforeseen consequences for high sensitivity scientific methods into the future (we have already demonstrated consequences for surface luminescence age determinations).