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PXRF ANALYSIS OF A YELLOW OCHRE QUARRY AND ROCK ART MOTIFS IN THE CENTRAL PILBARA

Lynley A. Wallis1,2, Jillian Huntley1,3, Michael Marsh4,5, Alan Watchman1,6, Allan Ewen4 and Annunziata Strano4

Abstract

The significance of ochre in Indigenous Australia is well documented. Several large, well-known quarries containing ochre that is highly sought after have been described in the archaeological literature, however less attention has been paid to smaller, regionally and locally significant quarries. In this paper a small yellow ochre quarry (VSTA_20140611_1) from the Central Pilbara, where evidence of paint preparation is preserved in the form of residues in two in situ grinding hollows, is described in order to address this oversight. Portable XRF (pXRF) analysis of the pigment in the quarry itself and the paint in the hollows was undertaken to understand the chemistry of the pigmentaceous minerals, to explore the taphonomy of the ochre seam, and to gauge variation within the source. Chemistry indicates that the VSTA_20140611_1 quarry is composed of an iron mineral (likely a hydroxide such as goethite), with Fe abundances consistently between 10.7 and 30%. Typical of the regional geology, the yellow pigment is consistently siliceous, with an Si abundance of between 5.8 and 20.4%. As there are no painted motifs in the rockshelter containing the VSTA_20140611_1 quarry, nor on the suitable BIF surfaces nearby, it is considered highly likely that the surviving paint produced on-site was used for either body decoration or the adornment of artefacts, rather than for rock art production. Nevertheless, the similarity in chemical composition between the VSTA_20140611_1 pigments and that of motifs painted in sites a few kilometres away suggests that, in addition to immediate processing and use at the site, ochre from this quarry may have been transported to rockshelters in the vicinity and used for rock art production.
Introduction

High quality ochres were (and still are) traded widely throughout Australia (Chaloupka 1993; McBryde 1987; McCarthy 1939a, 1939b; Smith 2000:665). Some of the largest and most widely known quarries include Bookartoo (aka Parachilna) in the Flinders Ranges, South Australia, Karrku, Ulpunyali and Lawa in Central Australia, Wilgie Mia and Little Wilgie in the Weld Ranges of Western Australia, and Tooumbunner in Tasmania (Broughton n.d.; Clarke 1976; Davidson 1952:82–83; Jones 1984; Morwood 2002:111–112; Peterson and Lampert 1985; Sagona 1994; Smith 2000; Smith and Fankhauser 2009; Smith et al. 1998). Archaeological studies of ochres have typically concentrated on the aforementioned quarries (Creagh et al. 2007; Green and Watling 2007; Popelka-Filcoff et al. 2012; Scadding et al. 2015; Smith et al. 1998). Outside of these, many other smaller, regionally or locally significant sources of pigment exist. With a few exceptions (e.g., David et al. 1993; Huntley et al. 2015; Mulvaney 1996), limited information about these smaller pigment sources and quarries is available in the published literature. Consequently, data for the small yellow ochre quarry described in this paper adds significant information to the available literature.

The term ochre is derived from the Greek word ochrós, meaning yellow (Barnetta 2006:446; Miriello et al. 2010:59). Most archaeological definitions of ochre restrict the term to secondary weathering products consisting predominantly of iron-oxide minerals (Bonneau et al. 2012; MacDonald et al. 2013; Popelka-Filcoff et al. 2007; Watts 2010; Zipkin et al. 2015), but also including a proportion (~30%) of additional matrix materials, typically clays, and other crystals like quartz and feldspar (Ambers 2004:770; Popelka-Filcoff et al. 2007; Rosso et al. 2014:86). Restricting ochre to iron-oxides limits the colour

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1 In using the terms ‘quarry’ and ‘source’ in this article, the definitions provided by David et al. (1993) are followed, whereby the latter refers to a location of available raw material, while the former refers to a demonstrably exploited raw material location.
palette to red hues including mulberry, orange and yellow, occasionally incorporating black and brown (Ford et al. 1994; Hodgskiss 2013; Jercher et al. 1998). Scientific studies of historical works of art refer to ‘earth’ pigments, defined coarsely by their physical structure as ochres and/or clays (Hradil and Hradilová 2012:86). Indigenous Australians make no such distinctions and include a variety of iron-oxide, iron-sulphide, manganese-oxide, clay, calcite and carbonate minerals in their use of the term ochre, incorporating hues from red, to black/brown, grey, blue and white (Attenbrow 2002; Clarke and North 1976; Cole and Watchman 1993; Crawford and Clarke 1976; Mosby 1993; Ward et al. 2001). Here we adopt this more inclusive meaning, defining ochres as natural pigmentaceous minerals that have been anthropogenically collected and/or modified.

A focus on red-coloured, iron-oxide minerals has led some archaeologists to use the terms ochre and haematite interchangeably. Caution should be employed in indiscriminately applying these terms, as a small amount of a coloured mineral—which may not be an iron-oxide at all—can account for a samples’ colour streak3 (Ford et al. 1994; Huntley 2012; Huntley et al. 2015; Ward et al. 2001). Further, focus on iron-oxides misrepresents the mineralogical diversity of ochres and the material properties (such as greasiness, shine, small particle size and clay content; cf. Cole and Watchman 1996; Morphy 1989) that influence their selection by Indigenous peoples.

The importance of ochre in Indigenous Australia is well documented, its use having been recognised in the earliest European accounts of Aboriginal life-ways (Horton 1994:820; Smith 2000; Taçon 2004:33). A particular ochre source might be important for its cultural affiliation (Blundell 1974; Morphy 1989, 1991; Mosby 1993; Vinnicombe 1997) and therefore used despite the availability of closer, local sources (McBryde 1987; Smith et al. 1998). In addition to producing rock art, pigments were used for body art, especially as an important component of ceremony and dance (Taçon 2004:35–36), and also for the decoration of many items of material culture, both secular and sacred (Jones 1984:6; Morwood 2002:101; Rifkin 2011, 2012;

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2 By pigmentaceous, we mean minerals that produce a colour streak.

3 Refer to Watts (2010) for a definition of colour streak.
Rifkin et al. 2015; Taçon 2004:37). Beyond decorative functions ochre had multiple utilitarian uses, including for covering balls of dessicated fruit (Smith 2000) and for medicinal purposes (Jones 1984:6; Velo 1984).

VSTA_20140611_1

The VSTA_20140611_1 quarry was initially reported during a reconnaissance survey by Marsh et al. (2014:13), and subsequently recorded in detail by Wallis et al. (2016). It is located in Banjima Country, approximately 120 km northwest of the township of Newman, in the Central Pilbara (Figure 1). The underlying geology of the area is the Mount Newman Member of the Marra Mamba Formation, which has a major ore-bearing horizon consisting of a banded iron formation (BIF) with interbedded carbonates and shales (Geological Survey of WA 1990; Kneeshaw 2008:13; Thorne and Tyler 1997; Trendall 1990:163). By and large, these shale beds were observed as the source of pigments throughout the study area (Wallis et al. 2016). The VSTA_20140611_1 quarry is located in an overhang at a small convergence between a southerly-orientated gully and the main east-west northern gully, on the eastern side of the junction. The nearest known instance of painted rock art is found 3 km to the northwest in a different gully system, with more abundant motifs in multiple rockshelters occurring 4 km to the southwest in the same gully system.

The dimensions of the overhang within which the quarry is located are 45 m in length and 10 m in height, with a northwest aspect. The overhang contains a distinct stratum of yellow coloured shale positioned 2–3 m above the gully floor (Figures 2 and 3). This yellow stratum is generally hard but a softer area of this rock is weathering into an alcove on the southwest side of the overhang by a combination of physical processes, the most important of which is water flowing down two sub-vertical joints and along the upper bedding plane. Subsequent to this has been thermal expansion of the pigment horizon, combined with chemical weathering in the form of granular disintegration caused by evaporation of water and
crystallisation of salts. While there is no apparent archaeological deposit associated with the overhang, a single proximal flake was noted on the floor amongst heat-shattered BIF and other angular gravel.

Figure 1 Map showing the location of the general study area in the Pilbara, Western Australia.

The dimensions of the quarry are 2 m wide, 1.75 m deep and 0.75 m high. There are few visible scratch or other gouge marks in the pigment, though its friable nature means evidence of such marks would likely not survive for extended periods of time. The angular faces of the pigment, together with the amount of material removed, is conspicuous compared to other naturally weathered pigmentaceous strata in the vicinity, indicating a large amount of material had been deliberately extracted from it. This observation is further supported by the absence of pigment debris at the base of the seam—something that was often present in association with smaller pigmentaceous strata, particularly where chemical and physical weathering (i.e., granular disintegration) resulted in the formation of powdery sediment-like deposits. A second, less weathered expression of the same shale seam is present in an alcove several metres to the east of the main VSTA_20140611_1 quarry. In the latter, where there was no indication of deliberate pigment extraction, was also observed and geochemically characterised.
Figure 2 General view of the VSTA_20140611_1 quarry, with Allan Ewan (left) and Nunzi Strano (right) in view. Above the quarry are two joints down which water has flowed to intersect the shale bed and initiate alteration and weathering.

Figure 3 (left) Exfoliated fragment of pigment from the primary seam crushed to test hardness and colour streak; (right) detailed view of the interior of the quarry showing natural flake or ‘onion’ exfoliation produced by thermal expansion, along with powdered residue produced by chemical weathering.
Of particular interest, two small hollows were present in the bedrock directly below the pigment seam (Figure 4). The dimensions of the eastern hollow are approximately 27 x 20 cm, while those of the western hollow are approximately 26 x 15 cm. Both show evidence of polish, and clearly still contain thick, dried residues of yellow paint (Figure 5). The extent of paint in the eastern hollow measures ca 13 x 11 cm, while that in the western hollow measures ca 11 x 7.5 cm.

In addition to the grinding hollows, several lines of dry applied yellow pigment were observed on a vertical rock surface below the quarry and above the grinding hollows (Figure 6). The lines are up to 10 cm in length and approximately 0.5 cm wide. They bear some resemblance to ‘tally marks’ seen in rock art sites in the region, though the latter are typically either painted or comprise large, rather than small, numbers of drawn parallel lines (Wallis et al. 2015; Wright 1968). As such, and given their proximity to the quarry, we suggest they were produced as a by-product of someone ‘testing’ the quality and colour of the extracted pigment rather than having been created by someone deliberately marking the rock surface (as in rock art production) per se.

Figure 4 View of two grinding surfaces with yellow pigment present at VSTA_20140611_1.
Figure 5 (left) Detailed view of the western grinding surface at VSTA_20140611_1; (right) Detailed view of the eastern grinding surface at VSTA_20140611_1.

Figure 6 View of several lines executed in dry applied yellow pigment at VSTA_20140611_1.
pXRF Analysis: Methods

pXRF analysis of the pigment in the quarry itself, the second smaller seam, and the paint in the grinding hollows was undertaken. In addition, assays were collected from elsewhere in the study area on yellow painted and drawn motifs in nearby shelters, other potential quarries/pigment sources, and the fabric of sites (i.e., the geological substrate of rockshelters and any geological weathering products such as mineral accretions/skins) in order to ensure a comprehensive context for interpreting element profiles. The decision to use pXRF, rather than some other fully quantitative/highly sensitive laboratory-based technique, was to ensure analysis would be non-destructive, in line with the wishes of the Banjima Native Title holders.

Analyses were undertaken using a Bruker Titan S1 800 instrument with a silicon drift detector, Rh target (max voltage 50 kV) and an automated filter changer running two phase parameters. Given the time constraints associated with recording, the decision was made to collect one 45 analytic second assay consecutively for each beam parameter for all sampled areas (as opposed to the more time consuming analytic protocol described in Huntley [2012, 2014; Huntley et al. 2015] designed for a different instrument).

A total of 34 elements were measured over the two pXRF ‘beam phases’/instrument parameters, of which 23 are present in the VSTA_20140611_1 quarry (reported in Table 1), including 12 elements routinely measured in ochres by desktop XRF and seven by bulk techniques such as neutron activation analysis (Popelka-Filcoff et al. 2007). Phase 1 was set to target high z elements at 45 kV, 10.45 μA with a Ti 25 μm, Al 300 μm filter in the beam path. Phase 2 was set to target low z elements at 15kV, 31.55 μA with no filter in the beam path. To ensure the X-ray tube had reached the required temperature, no assays were collected until the instrument had been running for at least

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4 This term is used interchangeably with ‘chemical profile/spectrum’ to describe the semi-quantitative results of the pXRF analyses performed. These assays have unique identification numbers to differentiate replicate spectra.

5 An ‘analytic second’ is the time taken to send a primary X-ray into the sample and receive a fluorescent X-ray back into the detector; it is not always equivalent to a second in time.
three minutes. During each analytic session assays were collected on two Certified Reference Materials (CRMs) that served as internal standards for the dataset, including monitoring for instrumental drift (cf. Johnson 2012) (CRMs are reported in detail in Wallis et al. 2016, including the quantitative wet chemical data).

Element abundances were calculated using the manufacture’s on-board fundamental parameters calculations, corrected for error terms and limits of detection. Multivariate statistical comparisons of pXRF data from the VSTA_20140611_1 quarry and local painted rock art were undertaken in JMP13 software, using Principle Components Analysis (PCA) based on a correlation matrix.

**pXRF Analysis: Results**

As shown in Figure 7 and Table 1, characterisations revealed variation within the ochre quarry. Nonetheless, the semi-quantitative chemistry from pXRF indicates the VSTA_20140611_1 quarry is rich in iron-oxide with Fe abundances consistently between 10.7 and 30% (Table 1). As is typical of weathered BIF environments, the yellow pigment is consistently siliceous, with Si abundances from 5.8–20.4%. Consistently high silicon abundances may represent the redeposition of silica in solution at the surface of the quarry and the BIF surrounding it. Similar processes were observed to deposit vitriform mineral accretions in rock art sites in the region (Wallis et al. 2016). If present, silica accretions would reduce X-ray yields within the light element suites (cf. Huntley 2012). Evidence for silica surface depositions within the ochre quarry could be reflected in the elevated Ca concentrations of 10.6% and 3.9% measured at the two locations where Si was low. These higher Ca concentrations correlate with the S measurement of 15%, which we suggest indicates the presence of sulphate salts such as gypsum (again, similar chemical concentrations were observed in the rock art panels at other sites in the region). We presume other geological salts are also present at the surface of the ochre seam, particularly associated
with granular disintegration in the secondary eastern expression of the pigment seam where an accumulation of powder was analysed (shown in the far right of Figure 3; assay reported in Table 1) evident by a consistently present low concentration of Cl. This is interpreted as evidence of physical weathering acting on the seam; geological salts at certain locations within the seam likely represent areas of higher water precipitation.

Table 1 Relative element abundances (weight %) recorded by pXRF at the VSTA_20140611_1 quarry.
Figure 7 pXRF element profiles for the VSTA_20140611_1 samples, relative abundance in parts per million (ppm) scaled to Log10.

The chemistry indicates that the pigment seam is a combination of iron and clay/mica minerals. An absence of elevated Sr (a surrogate of Ca), combined with persistent Al and K, indicate a clay matrix for this pigment. While the consistent elevated Fe abundance point to an iron-bearing oxide mineral producing the yellow colour; though sulfur minerals are well described in local geology, S abundance is not consistent nor elevated enough to account for the ochre’s colour (Table 1). Given its colour, the mineralogy of VSTA_20140611 is likely a hydrated iron-oxide mineral such as goethite and/or one of its polymorphs, probably within a kaolinitic matrix (though this is yet to be confirmed with structural analyses). It would be informative to know the exact mineralogy of the seam and quantify within source mineral variation; this could be easily and inexpensively achieved through future scientific studies such as X-ray diffraction of both the ochres and the pigment motifs in rock art in the Central Pilbara. However, further higher resolution scientific analyses...
would require (small amounts of) destructive sampling for work in a laboratory environment; permission for such a study has not yet been sought from the Banjima Native Title holders.

The preliminary, non-invasive pXRF analyses described in this paper were undertaken as part of a broader study to contextualise the painted rock art of the Central Pilbara (Wallis et al. 2015, 2016). Generally speaking, we found that the pXRF element profiles of potential pigment sources (white, grey, red, yellow and orange weathered seams and secondary deposits of alluvial creek gravels) in the Central Pilbara correlated to parent geology, and are heavily influenced by their geomorphic context (weathering processes), rather than grouping based on colour streak (though we acknowledge the surficial nature of the technique used in these preliminary characterisations—see Wallis et al. 2016 for further details). As part of this work we have compared the element profiles from the VSTA_20140611 quarry to 48 assays from yellow motifs at eight rock art sites in the Central Pilbara, including three assays from a potential weathered yellow pigment seam with sediment build up from granular disintegration elsewhere in the study region. The first three components of the PCA, represented in Figure 8, accounted for 34.75% of variation in the dataset (eigenvalues of 3.9631, 3.8660 and 2.9429, respectively). This comparison shows similarities between discrete assays from the quarry and discrete rock art motifs at three sites (BHP 60918 [4.4 km away], BHP 14579 [33.5 km away], and BHP 54449 [8 km away]). In contrast, the potential pigment seam from elsewhere (represented by the circles in Figure 8) does not group with any of the painted motif samples or those from VSTA_20140611.
**Figure 8** Two rotated projections of the first three principle components of multivariate comparison between the VSTA_20140611_1 quarry, rock art sites in the region and an unmodified pigmentaceous seam in the Central Pilbara. Key: Open diamonds = VSTA_20140611_1 quarry; Solid diamonds = VSTA_20140611_1 grinding hollow paints; Asterisks = yellow motifs in other rock art sites; Circles = the potential yellow pigment seam elsewhere in the study area.

**Discussion**

While pigment sources have previously been recorded in the Central Pilbara (see Centre for Rock Art Research + Management 2013; Wallis et al. 2016), this report constitutes the first documentation of an indisputable quarry. While this quarry is not of the same scale as ethnographically well known sources such as Wilgie Mia (located some 550 km to the south), the VSTA_20140611_1 quarry is a rare regional site type and affords a glimpse into the use of local pigment sources, and also the methods used for processing the pigments.
With BIF being typically reddish in colour, across the Central Pilbara region there is a definite preference towards yellow for the production of rock art motifs: approximately 66% of all motifs (n=1214) across 30 sites recorded by Wallis et al. (2015, 2016) were yellow. Despite this, very few yellow pigment sources or quarries have been reported in the region or further afield.

When used for the production of ‘paint’, pigments require some form of preparation, usually grinding to a powder followed by mixing with liquid (presumed mostly to be water alone, as other binders have rarely been reported, though see Cole and Watchman 1992). Paint production is often assumed to have taken place on bedrock surfaces; however, ethnographic evidence from northern Australia suggests that ground bedrock surfaces in rockshelters were primarily used for the processing of plant food stuffs and sometimes animal parts (Mulvaney 1996:17). The presence of the two fixed (i.e., not portable), ground hollows at this site, which still retain clear yellow paint residue (Figure 4), make VSTA_20140611_1 a unique find in the Central Pilbara region.

Peterson and Lampert (1985:6) described ochres from both Parachilna and Central Australian quarries (such as Karrku) being crushed and then mixed with water to form ‘cakes’, that were sometimes wrapped in bark for easier transport (see Curr 1886:70; Smith and Fankhauser 2009). It is possible that the ochre from VSTA_20140611_1 was prepared in a similar way, although the apparent impressions of ‘brushes’ can be seen in the dried pigment in the hollows, suggesting the paint was used immediately, at least in the most recent instance. Given the more exposed surfaces of the rockshelter preserve drawing/test ing of dry pigment (Figure 6) and the preparation of paint (Figures 4 and 5), we consider the absence of painted yellow motifs (or indeed any rock art) at this site to be a cultural pattern, as opposed to being due to taphonomy—it seems unlikely that the paint at VSTA_20140611_1 could have been used for rock art production or we would see evidence of that on the shelter walls. Rather, it is considered it more likely that the paint was used for either body decoration or the adornment of artefacts. Today Banjima people still carry out ceremonies during which they will adorn their bodies with paint, and high quality pigments are sought for this purpose.
Nevertheless, the similarity in chemical composition between the VSTA_20140611_1 pigments and rock art motifs in the region suggests a multiplicity of uses for local ochre sources and that lumps of ochre from this quarry may have been transported for use in artistic practices at least 5 km, and up to 33.5 km, away.

Finally, the chemistry of the VSTA_20140611_1 ochre quarry is consistent with previous studies of yellow rock art in Australia (Table 2) that have identified hydrated iron oxide minerals with clay matrices. Using semi-quantitative chemistry from field-based pXRF, it has been shown that local ochre sources were important to the artists of the Central Pilbara. Further targeted scientific analysis may quantify the spatial extent of ochre use, as well as confirm any purported correlations between ochre sources and site/motif specific features.

**Table 2** Minerals previously described in yellow rock art in Australia, adapted from Watchman (1990).

<table>
<thead>
<tr>
<th>Mineral Pigment</th>
<th>Chemical Compound</th>
<th>Citation</th>
</tr>
</thead>
<tbody>
<tr>
<td>Goethite</td>
<td>Fe$^{3+}$O(OH)</td>
<td>Cook et al. (1990); Walston and Dolanski (1976)</td>
</tr>
<tr>
<td>Lepidocrocite</td>
<td>Fe$^{3+}$O(OH) – a less stable polymorph of goethite</td>
<td>Walston and Dolanski (1976)</td>
</tr>
<tr>
<td>Limonite</td>
<td>FeO(OH) nH$_2$O</td>
<td>Walston and Dolanski (1976)</td>
</tr>
<tr>
<td>Jarosite</td>
<td>KFe$^{3+}_3$(SO$_4$)$_2$(OH)$_6$</td>
<td>Clarke and North (1989); Ward et al. (2001)</td>
</tr>
<tr>
<td>Carphosiderite</td>
<td>(H$_2$O)Fe$^{3+}_3$(SO$_4$)$_2$(OH)$_6$ – a synonym of hydroniumjarosite</td>
<td>Nobbs (1984)</td>
</tr>
<tr>
<td>Sulphur-Rich Kaolinite</td>
<td>Al$_2$Si$_2$O$_5$(OH)$_4$ – with a high abundance of sulphur</td>
<td>Huntley et al. (2011)</td>
</tr>
<tr>
<td>Whewellite</td>
<td>CaC$_2$O$_4$(H$_2$O)</td>
<td>Ford et al. (1994)</td>
</tr>
</tbody>
</table>
Conclusions

There are limited studies of pictograms in the Pilbara, with most attention having been paid to the extensive assemblages of petroglyphs in the region. As Wallis et al. (2015, 2016) have demonstrated, there are in fact many more pigment art sites in the Central Pilbara than have been previously recognised. There must also be a concomitantly larger number of pigment quarries and sources than have been documented to date, especially given the highly pigmentaceous nature of the regional geology. One such yellow quarry site has been described in this paper that, contrary to typical findings, includes ground hollows retaining yellow paint. pXRF analysis of the paints has confirmed that they are chemically consistent with that of the pigment in the immediately adjacent quarry. The absence of painted yellow rock art motifs in the immediate vicinity strongly suggests that, in this site, the paint was used for non-rock art production purposes, possibly body decoration or the adornment of artefacts. Nevertheless, the chemical composition of yellow painted motifs from some painted rock art sites in the Central Pilbara region is similar to the VSTA_20140611_1 ochre quarry, demonstrating that local sources were important in artistic practices also. Patterning in the comparisons between the quarry and local rock art motifs indicates that further high resolution scientific analyses could establish if local ochre sources were the basis of the paints used.

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