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How old is X-ray art? Minimum age determinations for early X-ray rock art from the 'Red Lily' (Wulk) Lagoon rock art precinct, western Arnhem Land

Tristen Jones, Vladimir Levchenko and Daryl Wesley

Introduction

A central and fundamental issue in rock art research is where the art is placed in space and time (David et al. 2013). Discovering and applying new techniques to understand motif styles and their chronology will provide us with this information. Here we use a 'cabled' methodology (see Chippindale and Taçon 1998:93), where multiple lines of evidence are developed together, by combining absolute and relative dating techniques. Absolute radiocarbon dates are made on two different substances that have been related with relative dates derived by assessing motif superimpositions, the stylistic analysis of motifs and degrees of preservation. Combined, the absolute and relative methods provide reliable dates for the painted motifs on a rock art panel at Red Lily Lagoon Site 3 (see Figure 2.1). Radiocarbon dates were obtained for mineral accretions suspected to contain the minerals whewellite and whedellite (both are hydrated forms of calcium oxalate CaC_2O_4, and called hereafter 'calcium oxalate'), and from preserved non-reactive organics contained within ancient mud wasp nest stumps. This is the first attempt to apply radiocarbon dating to these two different materials, calcium oxalate and mud wasp nests, directly associated with the same rock art.

A combination of radiocarbon dating and optically stimulated luminescence (OSL) dating on mud wasp nests has proved fruitful in dating rock art in the Kimberley (see Roberts et al. 1997). A previous Australian study used radiocarbon determinations for a calcium oxalate crust encasing rockfall within excavated deposits. Those results were compared with radiocarbon ages for charcoal samples in the same stratigraphic units (Watchman et al. 2005). These studies show the potential of both calcium oxalate and mud wasp nests for radiocarbon dating of rock art. While both calcium oxalate and mud wasp nest samples each have limitations for radiocarbon dating (cf. Aubert 2012; Bednarik 1996, 2000, 2002, 2007; David et al. 2013; Gillespie 1997; Rosenfeld and Smith 1997), our study has found that multiple radiocarbon age determinations from these two different substances can together generate robust dates for rock art.

Red Lily Lagoon Site 3

Red Lily (Wulk) is a coastal freshwater lagoon within the vast floodplains of the East Alligator River catchment area. The lagoon is found c. 5 km northeast of Cahill's Crossing on the main Oenpelli Road (Figure 2.1). The region falls within the territory of the Gagudju/Erre/Mangereridju language group, and forms part of the Manilakarr clan estate. Red Lily Lagoon is bordered on its eastern side by the western Arnhem Land Proterozoic Kombolgie Sandstone massif. This most western section of the plateau converges with Red Lily Lagoon, and the adjacent Kakadu wetlands, as a steep escarpment, varying in elevation from 50 m to 400 m above sea level (ASL).

The floodplains that surround the escarpment are annually inundated, re-filling permanent lagoons and billabongs, and at times submersing the majority of the land surface. It is within this landscape that the major archaeological site complex of Red Lily is situated (for a more comprehensive environmental and historical background for Red Lily Lagoon, see Chapter 2).

Red Lily Lagoon Site 3 is situated on top of the plateau at an approximate elevation of 70 m ASL, 700 m west of Red Lily Lagoon. The site is a major sandstone rock stack. On all sides of the rock stack, intensive weathering has generated deep overhanging shelters with rock floors, some with shallow sandy deposits, and isolated sandstone boulders. (For a description of the formation of rockshelters in Arnhem Land's quartzitic sandstone stacks, see Chapter 13.)

Each side of the rock stack is reported as a separate section, termed Areas A–D; each is a distinct and major rock art shelter in its own right. As field time was limited, all art panels were mapped, but not all individual motifs recorded. Overall, the Red Lily Site 3 rockshelters contain over 700 rock art images in a range of different art styles. These span the Early, Middle and Late periods outlined in Chippindale and Taçon's regional stylistic chronology (Chippindale and Taçon 1998:107). For example, rock art includes hand and foot stencils, 'Large Naturalistic' figures, Northern Running/Mountford figures, early X-ray and representations of the Complete Figure Style, including stick-figures, simple energetic figures, full figures and recent X-ray figures. There is also European contact–period imagery. Painting methods, forms and pigment types are variable. Motifs range from full-bodied, intricately detailed figurative representations of animals, applied with fine brushwork, to rudimentary-lined human figures. Pigment colours include red, orange, black, white and yellow, with many complex images bichromatic or polychromatic. Notable motifs include four painted in Reckitt's Blue pigment in Area B, and a high number of hand, forearm and foot stencils in Area C. Reckitt's Blue is the commercial name given to a laundry product used to whiten clothing (Chaloupka 1993:84). The use of Reckitt's Blue as a paint was first reported in the Alligator Rivers region in 1912 by Baldwin Spencer (1928:831). Chaloupka (1993:84) suggests that the blue pigment found widespread use after the introduction of Reckitt's Blue by Oenpelli missionaries in 1925.

The panel here studied is in Area D (Figure 7.1). Area D consists of three panels, with Panel 2 containing the majority of rock art images. The rock wall surface, facing northwest, measures 6 m in width by 1.2 m in height. The rock surface is well sheltered from the elements, with the dripline more than 1 m from the panel surface. However, the condition of the panel is quite poor: it is affected by both mud wasp nests and termites; mineral crusts and some parts of the rock face are spalling. The recording team perceive about 40 highly weathered rock paintings, some superimposed on the rock wall. Nonetheless, some images, and a basic sequence of superimposition of the clearest motifs on the rock wall, can be deciphered (Figure 7.2).

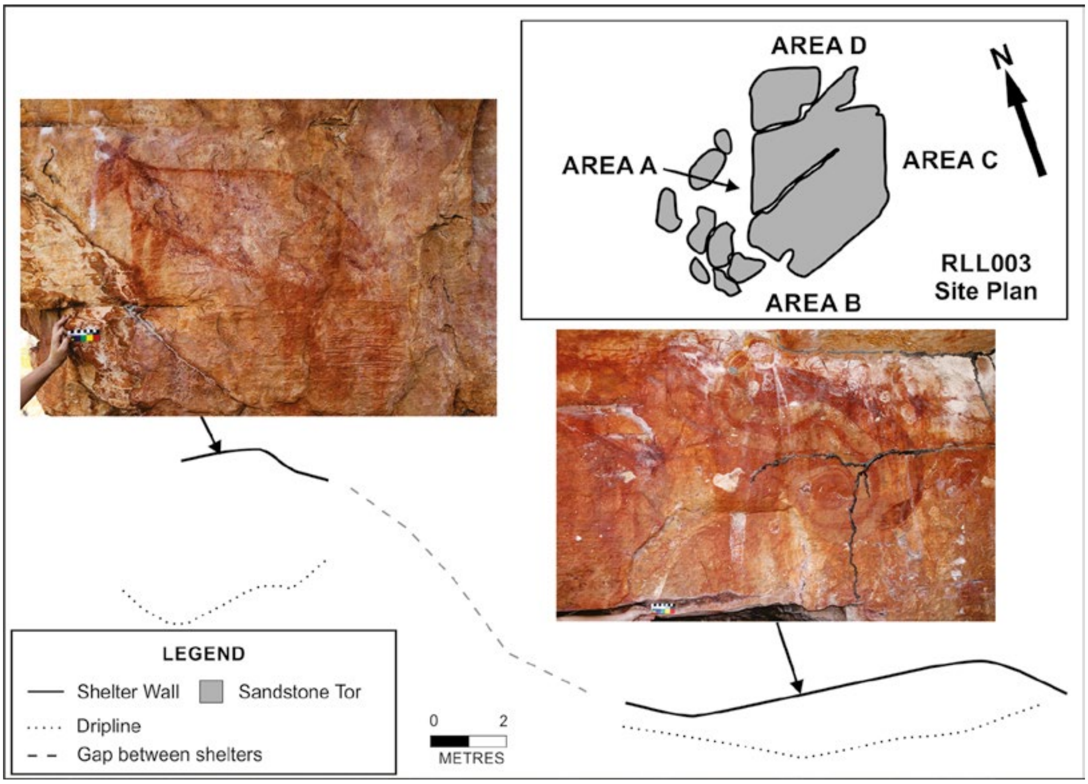

Figure 7.1 Plan of Red Lily Lagoon Site 3, Area D.

Source: Rose Whitau and Daryl Wesley.

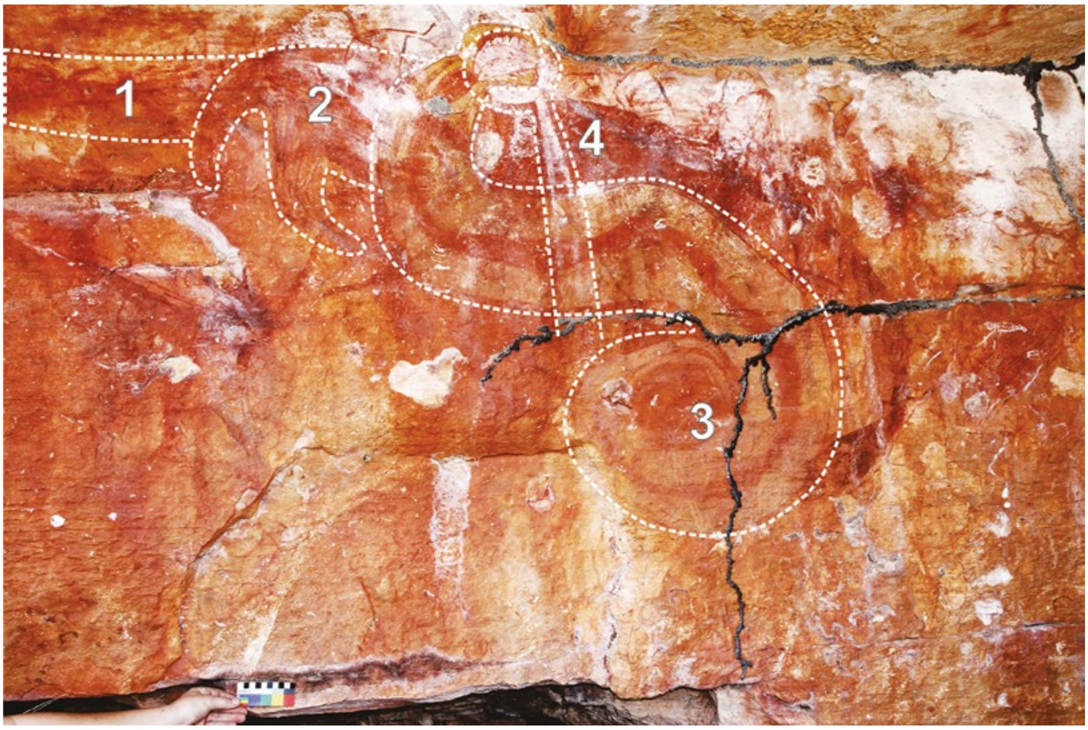

Figure 7.2 Red Lily Lagoon Site 3, Area D Panel 2, highlighting the sequence of rock paintings.

The numbers of the highlighted sections 1–4 refer to the motifs 1–4 contained within them.

Source: Photograph by Damien Finch with line drawing by Daryl Wesley.

Motif 1

Motif 1 is a very large red motif, composed of distinct curved lines. It spans much of the rock wall. Parts of the motif are solid infilled with protruding lines that are suggestive of spines. The motif spans left to right, across and down the rock surface. It is unknown where this motif lies in the relative sequence of artworks on this panel. The image is not clear enough to determine its exact shape, or indeed its corresponding style. The 'spines' are reminiscent of those common in 'Yam'-style Rainbow Serpents (Taçon et al. 1996). The image is superimposed by others, as outlined below.

Motif 2

Motif 2, in the upper left-hand section of the panel, is a red unidentifiable animal with hind limbs and the remnants of a tail. It is delineated by a solid line and decorated with the linear infill characteristic of 'Large Naturalistic' figures (Chaloupka 1993:94). Motif 2 overlies Motif 1.

Motif 3

Motif 3, the predominant image on the panel, is the best preserved. It is a red-outlined bichrome S-shaped motif, clearly depicting a snake. The motif is large, measuring 110 cm in length and 90 cm in width. The red pigment is better preserved than the yellow. The head of the snake is segmented, and is red and solid. Throughout the length of the body, a solid red segment also features, outlined by yellow pigment on either side. Starting from the head of the snake, two parallel red-lined segments following the curved S-shape of the snake border the main solid infill. The decoration features areas of solid infill, creating a block yellow to red patterning. This feature is lost throughout most of the snake's body due to weathering. There are also early X-ray features, such as a characteristic main body cavity with a thick line running through it representing a backbone (e.g. Taçon 1989: Figure 18; see Figure 7.3). Motif 3 overlies Motif 2.

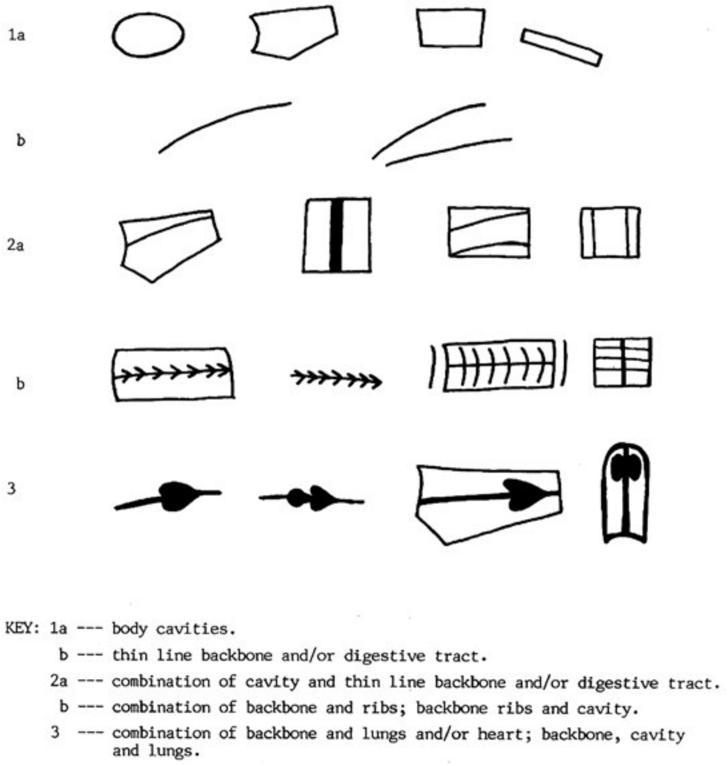

Figure 7.3 Early X-ray attributes, some of which are depicted in Motif 3.

Source: Taçon 1989: Figure 18, reproduced with permission.

Motif 4

The topmost layer in the painting sequence is comprised of a white anthropomorphic figure. Extensive weathering has resulted in only part of the motif remaining on the rock wall, that of the head and upper body. The bichrome figure consists of a red outline, infilled with white pigment. The figure is depicted in profile, as is common in human-like representations, with the head a hollowed C-shape. Chaloupka (1993:148–149) refers to such depictions as 'hooked face' figures. These types of human figures have been categorised as part of the 'Complete Figure Style' (Taçon 1992:204–205). Motif 4 overlies Motif 3.

Style and preservation as indicators of antiquity

For many decades, rock art researchers have utilised the method of grouping rock art motifs into 'styles' or manners of depiction, creating a relative sequence of change in artistic depiction over time. For western Arnhem Land, the original stylistic sequence proposed by George Chaloupka in various publications, including his hallmark *Journey in Time* (Chaloupka 1993:89), was modified and further developed by Chippindale and Taçon (1998:107), which is referenced in this study.

The classification of motifs according to style at Red Lily Lagoon Site 3 Area D Panel 2 is problematic. While the shape of Motif 1 is uncertain, the presence of spines suggesting a backbone signals some kind of zoomorph. While the decorative attribute of spines has previously been categorised as typifying 'early X-ray' art (Figure 7.3), the spines feature on the outside of the painting. Therefore the spines are more likely to indicate decorative features seen in 'Yam'-style Rainbow Serpents (Taçon et al. 1996). Taçon (1989) defines the internal attributes that constitute the 'early X-ray' style (see Figure 7.3), some of which are present in Motif 1, but also occur in Motif 3 of Panel 2.

Motif 2 is a depiction of a naturalistic macropod, as the motif is drawn with a free-flowing outline and is textured with linear infill (Chaloupka 1993:94). Chaloupka's categorisation of 'Large Naturalistic' animals as one discrete category of painting type that only appears at the beginning of the Arnhem Land sequence is inaccurate. Lewis succinctly summarises the issue as follows: 'The style of large naturalistic animals and humans is ill-defined and it may actually consist of a number of similar styles present throughout the entire sequence of Arnhem Land art' (1988:72). Indeed, the depictions of large animals are very common, many researchers having identified paintings in a 'Large Naturalistic' manner throughout the entire sequence, particularly throughout the Early and Intermediate period (cf. the Large Fauna Style of Chippindale and Taçon 1998).

The predominant motif in Panel 2 – Motif 3 – is a large snake. This motif is better preserved than any other rock painting on the panel. The segmentation of the head and the inclusion of both solid and patterned infilled segments clearly denote this image as an X-ray painting. The X-ray painting tradition has been argued to span some 8000 years (Taçon 1989, 1992), and therefore the categorisation of the motif according to 'style' does not provide a high resolution age estimation for the motif. The use of two colours, red and yellow in combination, suggests that the motif may have been painted in the late Holocene. Instances of bichromatic early X-ray are uncommon, accounting for only 5.7 per cent of the total number of paintings recorded by Taçon (1989:121–122). On the other hand, in recent X-ray art, which Taçon (1989:124) defines as occurring from 3000 BP to present, bichromatic representations are the most common, making up 60.2 per cent of the samples. Additionally, the combination of red and yellow pigment is the second-most popular extant pigment combination after red and white in recent X-ray rock art (Taçon 1989:126). Differentiation of motifs as either early or recent X-ray in style may also rely on the subject matter of the motif. Many researchers have argued that changes in faunal depictions through time signal changing environmental conditions (Chaloupka 1993:88). Taçon rightly notes that freshwater animal species begin to predominate in early X-ray paintings. As such, the commencement of early X-ray art is thought by many researchers to correspond with mid to late Holocene environmental conditions, such as are evident in the Kakadu wetlands of today (Brockwell 1996; Taçon and Brockwell 1995). As the subject matter of Motif 3 is formally a generic depiction of a snake, and as snakes are known to exist in both wet and dry environments, the faunal taxon cannot itself be used as an indicator of the painting's age. The snake's depiction in this instance does not have any characteristic features of a 'Rainbow Serpent'. Rainbow Serpents are commonly depicted with elaborately detailed tails, and plant and animal appendages, macropod-like heads and cross-hatching decorative features (Taçon et al. 1996). Motif 3 does not feature any of these attributes.

'Indirect' dating methods place Motif 4 in the late Holocene. While the full diversity of human-like representations in western Arnhem Land art remains a topic of ongoing research (Chippindale and Taçon 1993), the attribute of a hooked face, or C-shaped head, is akin to the facial features of 'Energetic' stick-figures. 'Energetic' stick-figures are commonly depicted in the rock art record of the wider Red Lily Lagoon area. Typically, these figures are frequently painted with material culture, particularly weaponry (Chaloupka 1993:148–149). The presence of weaponry in rock art paintings has previously been used as an indicator of the relative age of depictions (Lewis 1988). However, poor preservation of the painting has eliminated any potential evidence of material culture, and as such cannot be used to assist in estimating the age of Motif 4. It is assumed that the condition is due to the fact that the motif is painted in white pigment, which is acknowledged to have the least permanency of all pigment types perhaps apart from charcoal (Chippindale and Taçon 1998:103–104). According to Taçon, 'Energetic' stick-figures are a sub-category of the 'Complete Figure Style'. This style incorporates many different forms of art, including 'full figures', 'stick-figures', stencils, beeswax images and prints. Taçon (1992:204–206) argues that the 'Complete Figure Style' co-existed with 'Recent X-Ray' and is assigned an age range from 3000–2000 BP to present. On this basis, we assume that Motif 4 was painted during this period and has a maximum age of 3000 BP.

Sample selection and methodology

The rock surfaces and panels of Area D were surveyed for radiocarbon sampling, with particular attention concentrated on the white accretions and mud wasp nests present along sections of the rock wall. The presence of a mineral coating possibly containing calcium oxalate (whewellite $CaC_2O_4.H_2O$ and weddellite $CaC_2O_4.2H_2O$) was identified and in areas the whitish growth was covering part of Motif 3. Additionally, remnants of an ancient mud wasp nest were also present in close proximity to the mineral crust and located adjacent to Motif 3 (Figures 7.4 and 7.5). Three samples of coating material were collected from the rock surface (Sample 3-4-1, Sample 3-4-2 and Sample 3-4-3) (Figures 7.4 and 7.5). Sample 3-4-1 is of the mineral accretion that overlies part of Motif 3. The accretion covers the red and yellow segment patterning depicted in the upper neck of the snake (Figure 7.5). Sample 3-4-2 is from the remnant mud wasp nest. The mud wasp nest is situated adjacent to the right of the main body of the snake painting (Figure 7.5). Sample 3-4-3 is a mineral accretion that overlies the red outline of the mid-body of the snake painting (Figure 7.4). All samples were removed using a diamond disk Dremel® drill, producing bulk crust removal in powdered form. Powder was captured in clean individual sheets of aluminium foil, and emptied into clean centrifuge tubes.

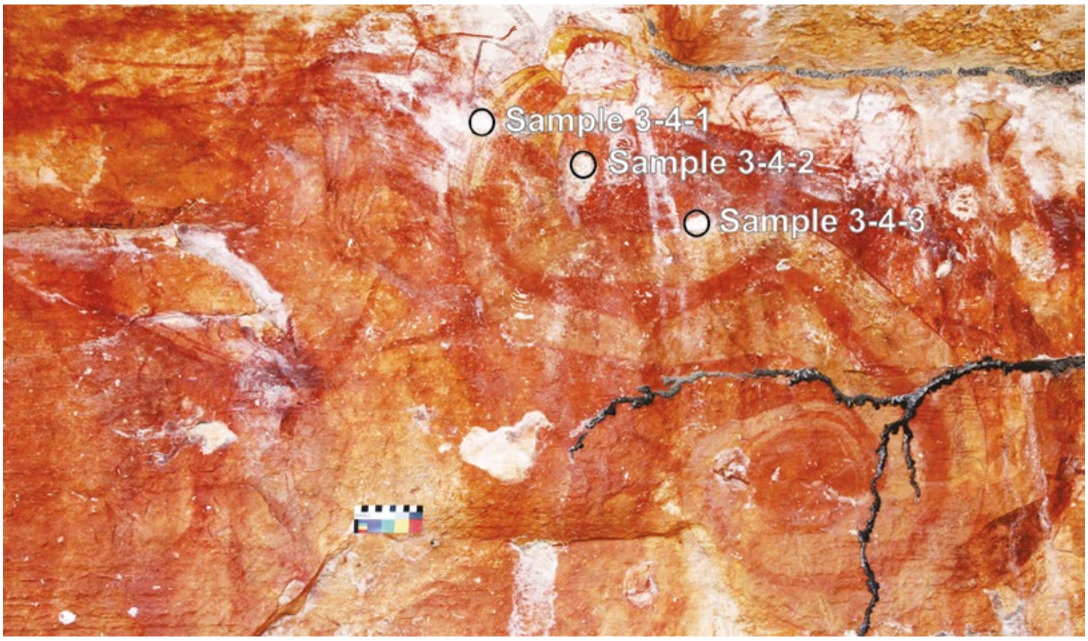

Figure 7.4 Panel 2 showing sample locations for radiocarbon age determinations.

Source: Photograph by Damien Finch.

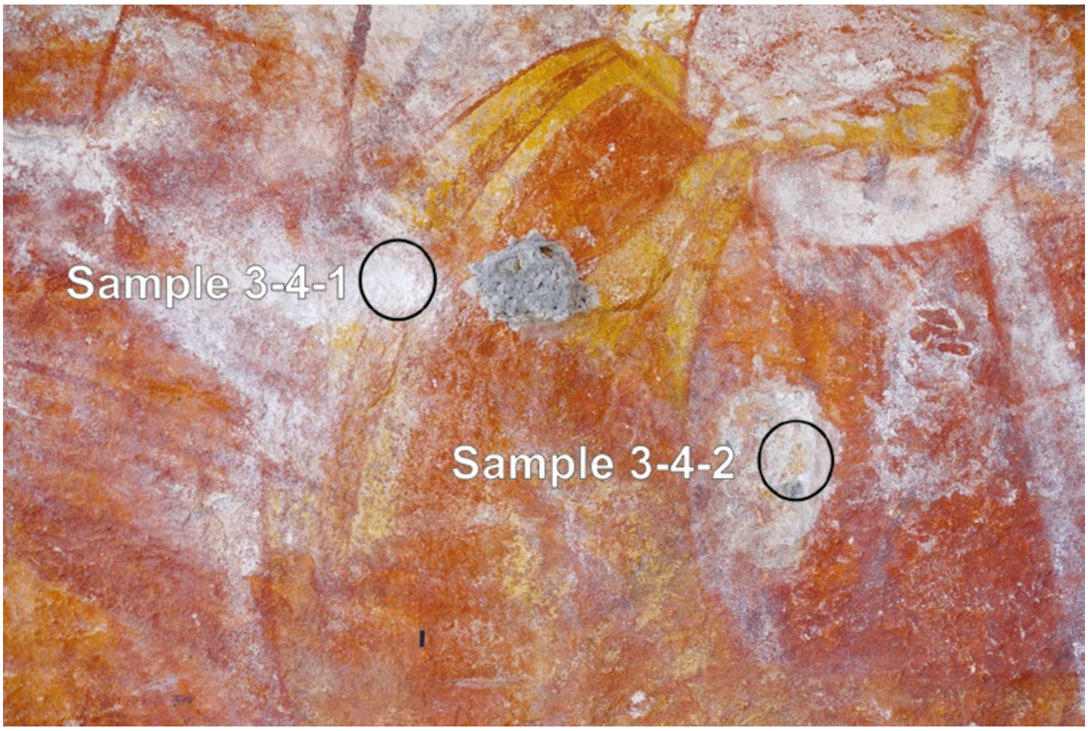

Figure 7.5 Location of samples collected for radiocarbon dating.

Source: Photograph by Vladimir Levchenko.

Once removed from the field, all samples were taken to the Australian Nuclear Science and Technology Organisation (ANSTO) research laboratories. Samples were registered and weighed, and aliquots taken from those expected to be of oxalate origin (RLL3-4-1 and RLL3-4-3). Aliquots were sent to The Australian National University's (ANU) Research School of Earth Sciences laboratories to test for the presence of calcium oxalate using Fourier Transform Infrared Spectroscopy (FTIR) and X-Ray Diffraction (XRD). The latter two mineralogical methods did not detect calcium oxalate suggesting that any oxalate that might be present was below the level of a few per cent. Additionally, the FTIR and XRD results confirmed the presence of minor phosphates (tinsleyite, taranakite), sulfate (gypsum) and natural weathering products such as amorphous materials, quartz and mica/kaolinite. The samples from Red Lily 3 have comparable mineral compositions to other rock art dating samples analysed from the study area (King et al. 2017)

Sample RLL3-4-1 was processed at ANSTO. The sample was pre-treated to remove potential carbon-bearing contaminants according to the following procedure. The powdered material was placed in a centrifuge tube with excess of 5 per cent NaOH solution at 60°C for one hour. At this step, various organic acids and non-acid soluble paint solids were transferred into the solution. Solution was centrifuged and the supernates decanted and retained in case of further analysis. After rinsing with Milli-Q® water, the samples were treated with 6M HCl at 60°C, to remove possible carbonates and to dissolve whewellite and whedellite minerals. After one hour, the solution was centrifuged and supernates decanted and saved. This time the precipitates, possibly containing various more or less inert solids (silica, pollen, charcoal, some non-reactive organic matter) were retained for separate analyses; these are referred to as the 'residue' in this chapter. The decanted solutions containing oxalic and HCl acids and their salts were dried down (HCl volatilises), sealed in quartz combustion tubes and combusted at 900°C, as described elsewhere (Hua et al. 2001). Separate residue fractions were rinsed in Milli-Q® water multiple times, dried and also sealed in combustion tubes and correspondingly combusted in the same conditions. Evolved CO_2 was cryogenically purified and collected and its yield determined. Then it was converted to graphite following standard graphitisation procedures (Hua et al. 2004).

Sample RLL3-4-2, the remnants of the mud wasp nest, was pre-treated at ANSTO following standard ABA procedures (Hatté et al. 2001). As per the following, the weighed powder was placed in a centrifuge tube and treated with 2M HCl for two hours at 60°C, followed after centrifuging by multiple Milli-Q® rinses. The next step included multiple treatments of 0.5 per cent up to 10 per cent NaOH for 2 hours each at 60°C (until two consecutive treatment solutions remained clear). After another Milli-Q® rinse, the third step consisted of 2M HCl at room temperature overnight and multiple Milli-Q® rinses until a near-neutral pH of the solution was achieved. The sample was oven-dried at 60°C. This treatment effectively removes all carbonates, humic and fulvic acids, fats and lipids, leaving behind pollen, charcoal dust (soot) and possible powdered macrofossils material. The dried sample was placed in a sealed quartz combustion tube similar to the oxalate sample, combusted to CO_2, which was again cryogenically purified and collected and converted to graphite.

Graphite targets were pressed into aluminium cathodes, and the $^{12}C/^{14}C$ ratio determined on ANSTO's STAR and ANTARES accelerator mass spectrometry (AMS) installations (Fink et al. 2004). In parallel with real samples, a set of chemistry procedural blanks was prepared to determine the level of possible contamination through the preparation process and corrected for this in data evaluations. Smaller samples are more likely to be contaminated, resulting in relatively large error bars of radiocarbon determinations for the smallest sample sizes.

Determinations of δ^{13}C were done on the residue of graphite targets derived from the studied fractions after the radiocarbon measurements were completed. Measurements were performed on a separate elemental analyser Elementar varioMICRO CUBE coupled to a Micromass Isoprime IRMS machine. If graphite residues were too small for a measurement, the average value for the same type of samples from the area was used for isotope fractionation correction.

Radiocarbon age determinations

The radiocarbon calibrated age determinations of the calcium oxalate and mud wasp nest samples fall within c. 210 years between their median values. They both indicate an early to mid-Holocene age of somewhere between 5068 cal BP and 6636 cal BP, taking the 95.5 per cent probability calibrated age ranges into account (Table 7.1). That the ages of two different dated material types converge – calcium oxalate and the mud wasp nest – is very encouraging. Each of these sample types has a different kind of formation history, has been subject to different pre-treatments and has a different dated fraction. This suggests the robustness of radiocarbon method for both of the dated materials, particularly in the utilisation of calcium oxalate minerals for radiocarbon dating.

Table 7.1 Radiocarbon determinations.

ANSTO code	Sample type	Submitter ID	Extracted carbon μg	δ^{13}C ‰	Radiocarbon pMC ($\pm 1\sigma$)	Radiocarbon age BP	Cal BP age (2σ range)	Median probability
OZR994U1	Mineral crust: oxalate	RLL3-4-1	9	−10.0[*]	52.40 ± 2.02	5190 ± 310	5068–6636	5906
OZR994U2	Mineral crust: residue	RLL3-4-1	23	−11.3[*]	57.94 ± 1.15	4380 ± 160	4445–5446	4953
OZR995	Mud wasp nest	RLL3-4-2	520	−6.7 ± 0.1	53.56 ± 0. 21	5015 ± 35	5605–5875	5697

[*] Results have been calibrated using CALIB 7.1 (Stuiver and Reimer 1993) using Sothern Hemisphere 2013 calibration curve (ShCal13) (Hogg et al. 2013). The value of δ^{13}C is assumed from determinations of similar type samples. A measured value is not available due to the small size of samples. For explanations of two dated fractions for mineral crust sample, see text.

Source: Authors' data.

In this study, we have reported two radiocarbon age determinations for sample RLL3-4-1. The first date is produced from the carbon contained within the calcium oxalate mineral only, isolated in sample pre-treatment. The second radiocarbon age determination is produced from the mineral crust residues (inert solids such as silica, pollen, charcoal, some non-reactive organic matter) contained within the sample. The calcium oxalate mineral crust returned an age of 5068–6636 cal BP, which, judging by the median age probability, precedes the mud wasp nest formation on the rock wall by a few centuries (Table 7.1). The calcium oxalate mineral crust has produced the oldest age, and as such it can be considered the *terminus ante quem* for the underlying snake image (Motif 3). The radiocarbon determination for the calcium oxalate sample was produced as a bulk measurement. Therefore all calcium oxalate mineral deposits that relate to mineral formation events and are stratified temporally in the crust have been combined into one sample. Hence, the produced age of the calcium oxalate represents an averaged result.

The mineral crust residue radiocarbon age – 4445–5446 cal BP – is determined from the carbon contaminates contained within and on the mineral crust (Table 7.1). The younger age of the residue may be due to the continual integration of carbon contaminants throughout the growth history of the crust, in conjunction with a build-up of carbon pollutants on the rock surface. Mineral crusts are known to grow sporadically over a period of time dependent on microenvironmental conditions. Considering the age spread between the coating minerals and the mineral crust residue, we can assume that the calcium oxalate mineral has been growing over a considerable length of time (Hassiba et al. 2012).

The radiocarbon age determination for the mud wasp nest is 5605–5875 cal BP (Table 7.1). This date is interpreted as representing the moment of mud wasp nest construction, as the organic components, such as pollen, spores, phytoliths and charcoal dust, are gathered and integrated into the structure of the nest by mud dauber wasps during the construction process (Bednarik 2014; Roberts et al. 1997). While some mud dauber wasps prefer to construct nests on the remnants of pre-existing nests, visual inspection upon sampling the mud wasp nest residue in the field indicated that in this instance there was only a single nest-building event. Therefore, carbon age averaging from multiple nest-building events is unlikely. Contrary to the calcium oxalate sample, the mud-wasp nest does not overlie Motif 3 or Motif 4. The mud wasp nest is located adjacent to the snake motif. As the mud wasp nest is younger than the calcium oxalate crust, it must have developed on the rock surface after the painting of Motif 3.

Discussion

A paucity of chronometric ages directly dating rock art remains an ongoing issue in rock art research worldwide (Aubert 2012; David et al. 2013). In Australia, a recent review of direct dates revealed that while there is a substantial number of age determinations for rock art, particularly in the Northern Territory (244 age determinations accounting for 56.4 per cent of the national data-set), the majority (74 per cent) of the age determinations are from beeswax rock art designs (Langley and Taçon 2010). The ages produced from beeswax figures are predominately of late Holocene antiquity; indeed, 81 per cent of the age determinations generated from beeswax samples in the Northern Territory are less than 500 years old (Langley and Taçon 2010). The predominance of chronometric age determinations from beeswax motifs is largely due to the fact that the material generally contains the most ^{14}C for radiocarbon dating. Additionally, dating beeswax motifs produces age determinations for the actual art object and the material is far less likely to be affected by carbon contamination, carbon recycling and sampling issues that plague the use of calcium oxalate and mud wasp nest substances for radiocarbon dating (Bednarik 2012). Langley and Taçon (2010:71) conclude of western Arnhem Land: 'The dominance of dated beeswax figures in this region means that while the chronology of this medium is now quite well understood, three remaining media (paintings, engravings and cupules) remain largely disarticulated from a regional chronology'.

Concerns in the literature regarding the use of calcium oxalate minerals for radiocarbon dating rock art have focused on the unclear nature of the mineral's developmental pathway, the unknown rate of mineral formation and the technical sample processing limitations in the separation of carbon-bearing components. These issues have previously stalled the widespread use of calcium oxalate as a substance for radiocarbon dating and acceptance of published radiocarbon age determinations generated from calcium oxalate minerals. The sample preparation methodology utilised in this study is a novel carbon compound-specific separation technique, which effectively isolates the carbon compound in the calcium oxalate minerals in sample pre-treatment (refer to methodology above). The result is two separate age determinations produced from the calcium oxalate mineral and the carbon 'residues' contained within the mineral crust. The residue carbon is assumed to be integrated into the formation of the mineral growth throughout the mineral crust's life span, and may also incorporate carbon pollutants residing on the mineral crust's surface. The refinement of this sampling procedure and the ramifications for the interpretation of radiocarbon age determinations is the subject of ongoing research and will be discussed in future publications (Jones et al. 2017). For this study, however, the considerably younger age of the mineral crust residue is proposed to be a result of the continuing build-up of carbon pollutants on the crust's surface. While the calcium oxalate mineral crust at some unknown point in time ceased growing, accumulation of carbon pollutants has remained ongoing to the present day.

The younger age of the mineral crust residue demonstrates the efficiency of the sampling pre-treatment cleaning procedures and compound-specific isolation method employed in this study. Previous radiocarbon age determinations using traditional acid-base-acid (ABA) sampling pre-treatment techniques may have failed to remove some carbon contaminants present in calcium oxalate mineral crust samples, such as charcoal dust, pollen, etc. This previously unresolved reliable pre-treatment issue has been the cause of much of the critique of the calcium oxalate radiocarbon dating results. The radiocarbon age determinations produced in this study highlight the potentially significant impact that carbon pollutants can have on radiocarbon age determinations. In this example, it can be assumed that without the separation of the mineral crust residues from the calcium oxalate, the mineral crust radiocarbon age determination would have been significantly younger.

Recent discussions in rock art dating have noted the rich organics contained within mud wasp nests, and their suitability for radiocarbon dating has remained an under-exploited resource in rock art dating (Bednarik 2014). While previous studies have assessed radiocarbon ages from organics within mud wasp nests in comparison to OSL ages (Roberts et al. 1997, 2000), the potential to exploit other methods, such as calcium oxalate radiocarbon dating, for comparative analysis in determining age estimations for rock art has been identified as a growing area of research (Bednarik 2014). The fact remains that problems persist for all 'direct' dating methods in rock art research, placing significant constraints on our ability to generate chronometric age estimates for rock art. However, while we have only presented three age determinations in this study, the results demonstrate that robust age determinations using both radiocarbon dating approaches can be produced. The resolution of questions surrounding the validity of methods in this case is in the combination of multiple radiocarbon age determinations generated from different substances. This methodology has been suggested previously on many occasions by rock art researchers (e.g. Bednarik 2012). In this instance, generating age determinations from multiple sources also lends weight to the potential of dating both calcium oxalate and mud wasp nests, and advocates for the ongoing adoption of both approaches. It also highlights the substantial opportunity for rock art researchers to date both materials more frequently, due to the common occurrence of both calcium oxalate mineral accretions and mud wasp nests overlying rock art in northern Australia.

The radiocarbon ages indicate that the minimum age (median probability) for the snake painting (Motif 3) is 5906 cal BP (Table 7.1). The painting has been interpreted as being of the early X-ray style, due to the presence of linear and segmented infill features. These decorative features are highlighted by the use of yellow and red pigments. While the poor preservation of both Motif 1 and Motif 2 render it difficult for a complete description to be established for either motif, they must be older than Motif 3 as they occur beneath it.

According to Chippindale and Taçon's stylistic regional chronology, early X-ray style is positioned between the Intermediate and Late period, and is assumed to appear c. 6000 years ago, co-existing with 'Simple Figures', 'Yam Figures', 'Large Human' and 'Large Fauna' styles. The radiocarbon age determinations produced in this study supports the assumed ages for this art style in the relative regional chronology. Furthermore, our new age determinations complement the previous proposition by Taçon (1989:119, 1992:203), who, by utilising motif superimpositions and stylistic analysis, estimated an upper age limit for the introduction of the early X-ray technique to between 8000–6000 BP. His reasoning for this time frame was the existence of early X-ray attributes appearing underneath the ‘Yam’ and ‘Simple Figures with Boomerang’ styles, while acknowledging that the upper age limit may be open to further investigation. It is important to note that the radiocarbon age of Sample 3-4-1 is a minimum age for the underlying painting, as the calcium oxalate mineral crust overlies the snake painting (Motif 3). The age gap between the Motifs 1–3 painting events, and both the growth of the calcium oxalate mineral crust and mud wasp nest, are not known and the actual painting event may have taken place many years before.

Most rock art researchers have discussed the problems associated with generating age estimates for rock art by employing indirect dating methods. Yet in-depth discussions addressing the implications of *underestimating* style age ranges has been missing in the literature to date. The radiocarbon age determinations produced in this study demonstrate that X-ray attributes occur in rock art motifs from the mid-Holocene. In fact, they may potentially occur much earlier. In this instance, the painted rock art surface (Panel 2) occurring at Red Lily Lagoon Site 3 is well protected from environmental decay, particularly water erosion. The dripline above Panel 2 is at some points nearly 2 m from the painted rock wall, providing a micro-environment that has produced greater conservation outcomes than normally would occur at other rock art sites.

It must be noted that the appearance of early X-ray rock art during the early to mid-Holocene corresponds with major environmental and climatic changes that occur in the East Alligator River region after sea-level stabilisation c. 8000 to 6000 BP (for further discussion, see Chapter 2, this volume). Significant changes occur in archaeological assemblages during this period, such as the appearance of shell middens at Ngarradj Warde Djobkeng (Allen and Barton 1989; Kamminga and Allen 1973), major changes in stone artefact technologies (Hiscock 1999, 2011) and the occupation of Birriwilk to the south of Red Lily Lagoon Site 3 (Shine et al. 2013). Taçon and Brockwell (1995) and Taçon et al. (1996) suggest that these archaeological changes in the early to mid-Holocene parallel the development of new rock art traditions. The radiocarbon age determinations produced in this study support that proposition.

Conclusion

This study has undertaken radiocarbon dating on different types of materials in order to complement and test relative methods of assessing motif antiquity. First, the relative methods of motif superimposition, stylistic analysis of motifs and motif preservation were employed. Then radiocarbon dating was utilised on mineral accretions containing calcium oxalate and carbon-bearing material contained within ancient mud wasp nests. This is the first attempt in rock art research to apply radiocarbon dating utilising these two different types of datable materials, calcium oxalate and mud wasp nests, directly associated with a single item of rock art. Novel developments in sampling pre-treatment for calcium oxalate mineral crusts have resolved some of the previous criticisms of this method of radiocarbon dating, increasing the confidence of age determinations. Combined with supplementary radiocarbon ages produced from mud wasp nests, the results of this study have demonstrated that the adoption of multiple radiocarbon age determinations generated from different substances can greatly assist rock art researchers to generate robust radiometric data regarding the antiquity of rock art. Coupled with an ‘indirect’ dating analysis, evaluating the painting sequence, stylistic and preservation attributes of the rock art, this study reliably proposes a minimum age of 5068–6636 cal BP for the introduction of early X-ray art in western Arnhem Land rock art. This mid-Holocene age determination is supported by previous chronological schemas, and parallels the changes evident in other archaeological assemblages occurring during this period in the region.

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