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Results from the first intensive dating program for pigment art in the Australian arid zone: insights into recent social complexity

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ABSTRACT

The Canning Stock Route Project (Rock Art and *Jukurrpa*) has yielded the first radiocarbon dates for rock paintings in the Western Desert of Australia. We report on the results of a large-scale project to directly-date both charcoal and inorganic-pigmented pictographs using plasma oxidation combined with accelerator mass spectrometry. This project has yielded the largest number of art dates from any region in the world: one site alone has produced 12 art dates (from 30 collected samples). Our work advances the testing of the dating method through the systematic use of replicates and explores the methodological implications of dating very small samples (10–40 μg carbon). Thirty-six radiocarbon age determinations range from 3000 years ago to Modern. The results contribute to an understanding of art production in the Australian arid zone during a period of extreme cultural dynamism. We have demonstrated for the first time that significant late Holocene changes in discard rates of artefacts and technological organization of the extractive technologies of implements such seed-grinders is matched by a very high level of stylistic heterogeneity in the art – which has been systematically dated within and between dialect groups.

1. Introduction

As part of a broader project to characterize rock art (petroglyphs and pictographs) and *Jukurrpa* (dreaming stories) across the Western Desert of Australia, our study has sought to contextualize rock art production in this part of the arid zone with chronological control. This is the most comprehensive dating project undertaken anywhere in the world for rock paintings from a single cultural locale. As well as recording over 500 rock art sites along the Canning Stock Route, we have collected 71 paint samples for radiocarbon dating. Our aim was to test the dating methodology as well as demonstrate and date stylistic change in art production through the late Holocene. Four of the sites where art samples were collected have also been excavated, allowing comparison of dated occupation sequences and art production results. Unfortunately, a number of these small art samples had insufficient carbon for dating. Nonetheless, we report on 36 radiocarbon dates – the first suite of reliable ages for pigment art in Australia's arid zone.

The team employed a combination of plasma oxidation and accelerator mass spectrometry, which has been used to date rock paintings worldwide (e.g., Russ et al., 1990; Steelman et al., 2005; Rowe, 2009). In contrast to traditional combustion methods, plasma oxidation occurs below the decomposition temperature of carbon-containing minerals such as carbonates and oxalates; therefore, their inclusion in the measured AMS graphite target is avoided for samples with a high mineral content (Russ et al., 1990; Steelman and Rowe, 2012). Thus, extensive acid washes used in conjunction with combustion are not necessary and can be avoided, minimizing the loss of organic material during wet chemical pre-treatment steps. McDonald et al. (1990) and McDonald (2000) observed this problem when using acid–base–acid pre-treatment with combustion, which further reduced the likelihood of achieving reliable dates.

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1.1. Study area: Canning Stock Route, Western Australia

The Canning Stock Route (CSR) is an 1800-km long historic pastoral route punctuated with 50 wells in remote Western Australia. It traverses the Great Sandy, Little Sandy and Gibson Deserts and the Native Title lands of Birriliburu, Martu and Walmajarri peoples, who are all part of the Western Desert cultural bloc. European forays into this part of Australia's arid zone were relatively late, with some of our informants only meeting settlers during the last 50 years.

The archaeology of the Australian arid zone is becoming increasingly well understood (e.g. Veth, 1993, 2000; Veth et al., 2001, 2008; Smith, 2013). Occupation of the Australian deserts is known to have occurred soon after modern humans arrived on the continent (O'Connor et al., 1998; Veth et al., 2009, 2011). Less is known about the rock art styles and techniques of this region (although see McDonald, 2005; McDonald and Veth, 2012; Veth and O'Connor, 2013), and the CSR Project's overall aim was to better understand rock art's role in the recent past, at a time where other archaeological signatures indicate extreme cultural dynamism.

Symbolic behavior is an important cultural component of Australian desert life, and well-documented arid art forms include a diverse body art repertoire (Gould, 1969; Tonkinson, 1978), decorated wooden shields and other objects (Dickens, 1996; Mountford and Tonkinson, 1969) and sand paintings (Munn, 1973). At recent European contact, desert people were occasionally observed producing extensive pigment art galleries (Gould, 1969; Phillip Playford, pers. comm. to Peter Veth in 2013), and today there is a flourishing contemporary art movement (Carty and La Fontaine, 2011). There are various phases of rock art production through time, the earliest we posit as occurring with the initial occupation of the desert (Balme et al., 2009; McDonald and Veth, 2010). This dating project has focused on the most recent pigment art phases, identifiable by superimposition analysis and by the fact that this has visibly more surviving pigment present. We collected several paint samples from older styles, but these samples yielded significantly less viable material and we were unable to successfully date this earlier underlying art.

There is no contact art, identifiable by subject matter, along the Canning Stock Route but claimants speak to certain motifs and panels in most locations, and we have documented how desert rock art is a significant way of curating mythological sagas (McDonald and Veth, 2013a). None of our informants have been involved in production of pigment art, but several remember their parents talking about being involved in, or witnessing the production of rock art, e.g. *Nangapirty*, the father of Brian and Arthur Sampson, was born in the *Jilakurru* Ranges (Tonkinson, 1978) and he told his sons that his hands were stenciled there by his father when he was a boy (AS, pers. comm. 2010). We relocated this art site in 2010. While some Aboriginal people in this part of the Western Desert only made contact with Europeans in the 1960–70s, the recent movement of people out of the desert (Peterson, 1986) would appear to indicate that we are now several generations removed from painting on rock as part of a routine practice.

This project aimed to date pigment art along the entire length of the CSR but there is generally less art at the northern end of the CSR, due to the relative scarcity of suitable geological substrates. Pigment samples were collected from sites as far north as Gravity Lakes (Well 45), but the main focus of sample collection was in four Range systems located along the southern half of the CSR (Table 1, Fig. 1). The *Tipirl* (Diebel) Hills are located south of Well 18; the *Jilakurru* Ranges are at Well 17; the Calvert Ranges (*Kaalp*i) are to the east of Well 16 and the Carnarvon Ranges (*Katjarra*) are located southwest of Well 6.

Table 1
Total Sample collections, sites and dating outcomes.

Locations	Sites	No. of paint samples	No. of backgrounds	Art dates	Background dates
Gravity Lakes	GLM1	2	2	1	
	GLM2	1	1	0	
	GLM3	1	1	0	
Wells 38-37	Nightjar Shelter	1	1	0	
Diebel Hills	Diebel 1	2	1	2	
Jilakurru Ranges/ Durba Hills	DS1	1	1	1	
	DS4	1	1	0	
	DS28	1	1	0	
	DS32	1	1	0	1
	DS55	3	1	1	
	Pinpi 5	30	2	12	
	Pirli 3	1	1	1	
	Biella	1	1	0	
Kaalpi/Calvert Ranges	Kaalpi (V12)	5	2	3	
	M23	4	1	2	1
	P13	2	2	1	
	BTD1	5	3	5	
	PV10	1	0	1	
Katjarra/Carnarvon Ranges	SG1	8	6	5	2
		71	29	35	4

1.2. Dating rock art: plasma oxidation and AMS

Reliably determining the age of rock art assemblages has always posed a major challenge for rock art studies. The development of accelerator mass spectrometry (AMS) and related techniques now makes the chronometric dating of rock art possible (Steelman and Rowe, 2012). AMS requires only very small organic samples (e.g. charcoal, plant fibers), thus largely overcoming ethical dilemmas inherently associated with destructive sample collection processes. Paint must contain organic material temporally related to the painting event – charcoal pigment, or an organic binder added during paint manufacture. Chemical identification of organic materials used in paints (using chromatography and mass spectrometry) is still in its infancy (Rowe, 2001b; Livingston et al., 2009; Mazel et al., 2010; Mori et al., 2006; Vazquez et al., 2008).

Worldwide, pictographs are more frequently made with inorganic pigments. Reds, oranges, browns, and yellows are iron oxide/hydroxide minerals in various oxidation states and degrees of hydration. Similarly, black motifs are often produced using a manganese oxide/hydroxide rather than charcoal. These inorganic minerals cannot be radiocarbon dated because they do not contain carbon related to the production of a painting. If organic material(s) were added to the pigments as part of the paint preparation process, and if enough of that organic material has survived, then measurements can be made with sufficient accuracy and reliability to determine radiocarbon ages for inorganic-pigmented paintings. The plasma oxidation method together with AMS radiocarbon measurement is a direct technique for dating both charcoal and inorganic-pigmented pictographs.

The Steelman laboratory uses a custom-built plasma oxidation apparatus to extract organic material from ancient paint samples for AMS radiocarbon dating (Russ et al., 1990; Rowe, 2001a; Rowe, 2009; Steelman and Rowe, 2012). Plasma oxidation negates the use of extensive acid pretreatments because plasma temperatures (<150 °C) are below the decomposition temperatures of carbonates and oxalate minerals (Russ et al., 1992). The main advantage is that the inorganic rock substrate (sometimes inadvertently collected with the paint sample) does not decompose during exposure to oxygen plasma. Additionally, acid washes may not completely remove oxalate minerals, which are commonly associated with rock surfaces (Armitage et al., 2001; Hedges et al., 1998). Plasma

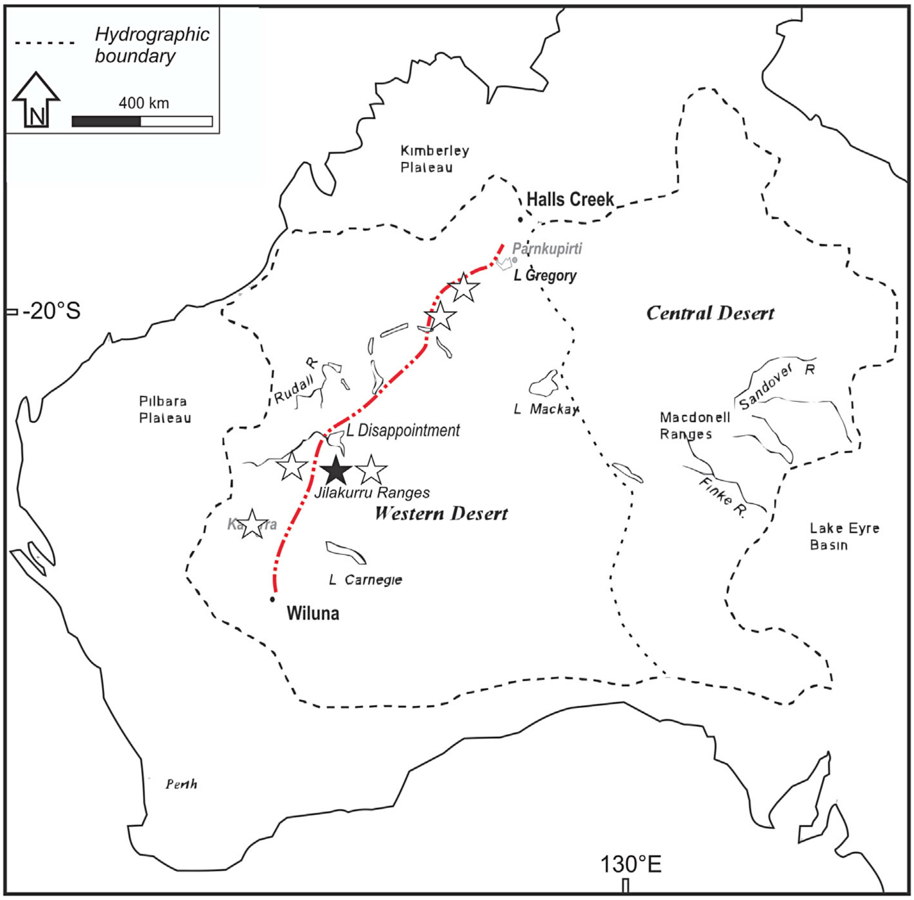

Fig. 1. The Canning Stock Route in the Western Desert. The stars show the locations of the dated rock art provinces. The Jilakurru Ranges (discussed in more detail in Results) is indicated by the black star.

oxidation is ideal for samples with only trace amounts of organic material remaining because it minimizes the loss of organic material, which occurs during wet chemical pretreatment steps.

2. Experimental methods

2.1. Sample collection

Seventy-one paint samples (54 black, 8 red and 9 white) were collected along the Canning Stock Route in collaboration with *Martu* traditional custodians and in accordance with two Section 16 Ministerial Permits (#s 393 & 467) granted by the West Australian Department of Indigenous Affairs. Prior to sampling, all paintings were systematically documented with photographs, drawings, detailed motif descriptions and GPS coordinates. Our sampling regime was focused on methods to crosscheck results: we collected replicates (i.e. multiple samples from a single motif) where possible. In addition, we collected samples where there were superimposed paint layers in the art styles to test for consistency in the stratigraphic chronology.

Sterile scalpel blades were used to remove each paint sample. In general, we targeted a single area from any one motif – confined to a surface area between 0.3 and 1.0 cm^2. When possible, where paintings were observed to be naturally at risk, spalls or flaking paint were collected to minimize the visual impact. Photographs of sampling locations were taken before and after collection for documentation purposes. Photo-enhancement in the field, using D-Stretch software on a Canon PowerShot SX10IS digital camera (Jon Harmon; http://www.dstretch.com), was used to clarify the presence of underlying faded motifs, which were difficult to see with the naked eye but could potentially contaminate the collected sample with multiple painting episodes. Many of the sampling locations are densely painted with numerous superimposed layers. Care was taken to ensure that samples contained only one paint color and, presumably, a single painting event.

Twenty-seven background samples were also collected from adjacent unpainted areas to investigate levels of organic contamination in the rock substrate. It is often difficult to find areas of unpainted rock on these rock art panels due to the high density of paintings. Some of the collected “backgrounds” may in fact not be truly unpainted rock.

All collected samples were placed in aluminum foil squares, folded, and stored in labeled plastic bags, where they remained until commencement of laboratory analysis. Once in the laboratory, samples only came into contact with 500°C-baked glassware, aluminum foil, or sterile scalpel blades in order to avoid laboratory contamination. Even so, latex gloves were worn during sample collection and later in the laboratory during any sample handling.

2.2. Chemical pretreatment

All paint samples and unpainted rock backgrounds were subjected to chemical pretreatment to remove any potential humic contamination. Humic acids, naturally present in soil samples and derived from the decay of organic matter, appear brownish-orange in a basic solution. There has been little investigation as to whether humic acids are present in pictograph samples, as they are in many archaeological artifacts buried in soils (Pace et al., 2000). We routinely use a pretreatment of base as a precaution.

In all but three black samples, we observed wood grains under high magnification confirming that charcoal was the source material used for pigment. At ×40 magnification, samples were examined to remove any visible contaminants. Fibers were removed from approximately half of the samples prior to analysis. Unfortunately, in all cases, these were too small to date separately (i.e. to determine whether they were contaminant plant fibers or rootlets, part of binders used in paint preparation, or pieces of paint brush material).

Samples were weighed (see Tables 2–4 and 6) into sterile centrifuge tubes for chemical pretreatment (2–350 mg). We added 3 mL of 1 Molar sodium hydroxide solution and placed the tubes in an ultrasonic water bath at 50 ± 5 °C for one hour. After centrifuging for 15 minutes, the liquid above most samples was colorless and transparent, indicating that no humic acids were present. After decanting the basic liquid, 4 mL of distilled, de-ionized water was added to each tube. The sample tubes were placed in an ultrasonic water bath at 50 ± 5 °C for another hour. After the tubes were centrifuged, the liquid was decanted and the solid samples were stored in distilled, de-ionized water until filtration onto quartz-fiber filters that had been previously baked at 500 °C. Samples on filters were dried in an oven at 110 °C, then wrapped in foil and stored in a desiccator.

2.3. *Plasma oxidation*

A custom-built plasma oxidation apparatus was used to convert organic material in these samples to carbon dioxide for accelerator mass spectrometry (AMS) radiocarbon dating. The apparatus, kept under vacuum at a pressure of approximately 1×10^{-6} mbar, utilized ultra-high purity (UHP, 99.999%) oxygen and argon gases. Prior to analyzing a sample, the empty sample chamber was cleaned by igniting successive oxygen plasma reactions at 1 torr oxygen gas (1 torr = 1.33 mbar) and either 100 or 150 Watts radio frequency power for one-hour each. These cleaning plasma reactions removed any organic material on the inside of the sample chamber, which may have been introduced by a previous sample or modern contamination from handling.

Next, a sample was loaded into the glass chamber and evacuated overnight using a turbomolecular pump with a scroll forepump to a pressure of $\leq 1 \times 10^{-6}$ mbar. Then, we ignited an argon plasma, at 1.3 mbar and 40 Watts radio frequency power for successive one-hour reactions, to remove adsorbed gasses by surface ablation. Argon is used because it is an inert gas and will not react with organic material in a sample. This continues until a pressure reading of <1 millitorr (corresponding to <0.3 μg C as CO_2) is obtained, indicating that adsorbed gasses have been removed.

Finally, each sample was oxidized with an oxygen plasma at a pressure of 1.3 mbar oxygen and 100 Watts radio frequency power. This converted the organic material in the sample into carbon dioxide and water during a one-hour exposure. The product carbon dioxide was then flame-sealed into a glass tube cooled to liquid nitrogen temperature (−196 °C), after water was removed with a dry ice/ethanol slurry (−72 °C). If sufficient carbon was obtained, the collected glass tube was sent to the Center for Accelerator Mass Spectrometry (CAMS) at Lawrence Livermore National Laboratory for graphitization and AMS radiocarbon measurement.

2.4. *Checks and balances: radiocarbon blanks*

To test our methodology and results, eight samples of USGS coal were processed in the same manner as the paint samples to determine the extent of modern contamination incorporated during laboratory procedures. This is especially important for the small samples analyzed (<50 μg C). USGS Coal is a "dead" radiocarbon standard that is millions of years old with virtually no measureable ^{14}C remaining – an ideal blank for radiocarbon studies. Frequent evaluation of blanks is necessary in order to determine variability arising from different laboratory techniques and equipment. As sample sizes decrease, the effect of contaminant carbon on blanks is potentially more problematic. From eight USGS coal blanks (40 to 250 μg carbon), we introduced ∼1 μg modern carbon during chemical pretreatment, plasma oxidation, graphitization, and AMS measurement. This background level suggests that minimal modern carbon is introduced during laboratory processing. Reported dates for paint samples were calculated using the plasma-oxidized USGS coal samples as process blanks for mass balance corrections of the ages.

3. Results

Thirty-six paint samples had sufficient carbon for AMS radiocarbon measurement. Radiocarbon ages are shown in Tables 2 and 3. Detailed descriptions of the sites and individual motifs dated are to be published elsewhere. Of the 36 age results, 29 are from

Table 2

Radiocarbon results for paint samples with ≥ 50 μg carbon.

Sample	Site	Description	UCA ID	Sample mass (mg)	μg C	CAMS ID	^{14}C Age (years BP)	Calibrated age (2σ, 95.4%)	Calibrated age (2σ, 95.4%)
2005-03	M23	Black SNF	B61	35	50	132481	820 ± 90	1000–1400 cal AD	950–550 cal BP
2005-08	BTD1	Black outline of white snake	B62	28	120	132482	10 ± 35	1700 cal AD–modern	250 cal BP–modern
2005-09	BTD1	Red headdress	B94	49	50	136690	1600 ± 80	300–700 cal AD	1650–1250 cal BP
2005-10	V12	Black outline of SNF	B68	25	240	134001	260 ± 40	1500 cal AD–modern	450 cal BP–modern
2005-17	V12	Black CXNF	B67	13	200	134000	285 ± 35	1500–1800 cal AD	450–150 cal BP
2005-18	SG1	Gray mamu	B69	350	240	134002	285 ± 35	1500-1800 cal AD	450-150 cal BP
2005-19	SG1	Gray mamu	B64	14	130	133998	460 ± 40	1400–1650 cal AD	550–300 cal BP
2005-20	SG1	White SNF of mamu	B95	59	100	136689	795 ± 45	1200–1400 cal AD	750–550 cal BP
2005-24	SG1	Black of bichrome phytomorph	B39	90	80	130499	745 ± 45	1200–1400 cal AD	750–550 cal BP
2005-25	SG1	Black of bichrome phytomorph	B65	14	50	133999	540 ± 80	1250–1650 cal AD	700–300 cal BP
2010-10	DS 1	Black of B&W lizard man	B155	25	180	152983	310 ± 35	1450–1800 cal AD	500–150 cal BP
2010-25	Pinpi 5	Black rejoicing five finger man	B199	279	90	161470	2885 ± 55	1200–800 cal BC	3150–2750 cal BP
2010-26	Pinpi 5	Black oval head man	B168	46	140	152884	1600 ± 40	400–650 cal AD	1550–1300 cal BP
2010-32	Pinpi 5	Black lollipop man	B157	8	60	152984	1320 ± 90	600–1000 cal AD	1350–950 cal BP
2010-37	Pinpi 5	Black CXNF	B184/185/187	33	50	158680	12,970 ± 270	–	–
2010-42	Pinpi 5	Black jila (right side of panel)	B160	6	140	152987	225 ± 40	1600 cal AD–modern	350 cal BP–modern
2010-43	Pirli 3	Black outline of B&W jila	B164	7	60	152882	660 ± 80	1200–1450 cal AD	750–500 cal BP
2010-45	Diebel	Black jila behind fig tree	B163	2	120	152881	290 ± 50	1450–1850 cal AD	500–100 cal BP
2010-52	BTD1	Black of B&W snake	B176	16	70	158668	1450 ± 80	400–850 cal AD	1550–1100 cal BP

Table 3
Experimental radiocarbon results for small μg paint samples.

Sample	Site	Description	UCA ID	Sample mass (mg)	μg C	CAMS ID	^{14}C Age (years BP)	Calibrated age (2 σ, 95.4%)	Calibrated age (2σ, 95.4%)
2005-02	P13	Red phytomorph	B96	27	30	158161	1050 ± 120	700–1300 cal AD	1200–600 cal BP
2005-06	M23	Red of bichrome	B104	116	30	158162	1050 ± 180	600–1300 cal AD	1300–600 cal BP
2009-01	GLM1	Black SNF stylized track	B150	4	20	158163	1510 ± 130	200–900 cal AD	1700–1000 cal BP
2009-02	GLM1	Black CXNF lines	B151	10	40	152980	820 ± 110	1000–1400 cal AD	1000–500 cal BP
2010-02	DS 55	White anthropomorph	B177A	28	20	158168	2690 ± 230	1400–200 cal BC	3400–2100 cal BP
2010-15	Pinpi 5	Black of concentric circles	B175	5	30	158167	1060 ± 130	700–1300 cal AD	1300–600 cal BP
2010-21	Pinpi 5	Black of B&W snake	B167	7	30	158165	650 ± 180	1000–1700 cal AD	1000–300 cal BP
2010-23	Pinpi 5	Black glove head (replicate)	B174	13	30	158166	1160 ± 180	600–1300 cal AD	1400–600 cal BP
2010-30	Pinpi 5	Black of CXNF	B186	10	20	158663	12,620 ± 460	–	–
2010-36	Pinpi 5	Black of bird tracks below CXNF	B161	11	30	152880	5520 ± 290	–	–
2010-46	Diebel	Black right eye	B182	3	40	158662	670 ± 120	1100–1700 cal AD	800–300 cal BP
2010-49	PV10	Black complex pole	B173	3	40	158667	1740 ± 100	100–600 cal AD	1900–1300 cal BP
2010-50	BTD1	Black top "I" on side panel	B177B	8	40	158169	1400 ± 100	400–1000 cal AD	1600–1000 cal BP
2010-51	BTD1	Black sinuous line	B178	8	30	158170	2800 ± 190	1400–400 cal BC	3400–2300 cal BP
2005-14	V12	Red of CXNF	B100	21	<10	136688	3190 ± 610[a]	–	–
2010-28	Pinpi 5	Black of jila	B159	8	<10	–	470 ± 270[a]	–	–
2010-29	Pinpi 5	White of lollipop lizard man	B158	42	<10	–	310 ± 270[a]	–	–

[a] CAMS measured <10 μg C (only for completeness).

charcoal paintings and nine are from inorganic-pigmented paintings. Many of the dated samples were smaller than traditionally considered routine (<50 μg C) for reliable results. Thirty-five samples had (≤10 μg C) and were considered to have insufficient carbon for counting (Table 4). This resulted in 53% of collected charcoal samples having sufficient carbon for AMS measurement, only slightly higher than the 45% success rate achieved for samples from inorganic-pigmented paintings (Table 5). Of the 27 background samples collected (Table 6), six had sufficient carbon for AMS measurement.

Calibration was performed using the OxCal computer program version 4.2.2 (Bronk Ramsey, 2009, 2013) with SHCal04 southern hemisphere atmospheric data from McCormac et al. (2004). Due to the recent age of many of these samples, the calibrated ages have wide ranges. This is because of unavoidable perturbations in the radiocarbon calibration curve (i.e., from natural variations, the industrial revolution, and nuclear bomb testing).

The focus for our most intensive sampling effort was in the centre of the Canning Stock Route at Jilakurru (the Durba Hills) and Kaalpi (the Calvert Ranges). A major rock shelter site at

Table 4
Paint samples with insufficient carbon extracted for radiocarbon dating.

Sample	Site	Description	UCA ID	Sample mass (mg)	μg C
2005-01	P13	Red anthropomorph with spurs on thigh		45	10
2005-05	M23	White of bichrome panel		64	10
2005-07	M23	Red of bichrome panel		95	10
2005-15	V12	Red headdress	B103	83	10
2005-16	V12	White body of mamu	B63	145	<1
2005-21	SG1	White of snake	B93	268	8
2005-23	SG1	Black outline of white snake	B66	18	10
2005-26	SG1	Red of bichrome CNF		206	6
2009-03	GLM2	Black bird track		2	10
2009-04	GLM3	Black of black and yellow arc		3	10
2009-05	Nightjar	Black of black arcs and bird tracks outlined in yellow		2	4
2010-01	DS55	White of M16		30	6
2010-03	DS 55	White of M16 line of arm to right of sample#2		13	10
2010-05	DS4	Black outline of snake	B181	15	<1
2010-11	DS28	Black of b&w SNF 3 prong "Y" shape		2	10
2010-12	DS32	Black anthropomorph		9	10
2010-13	Pinpi 5	Black parallel sinuous lines		5	<1
2010-14	Pinpi 5	Black of b&w concentric circles around cavity		5	10
2010-16	Pinpi 5	Black of b&w headdress figure		2	<1
2010-17	Pinpi 5	Black & white platypus		5	6
2010-18	Pinpi 5	Black glove headed lizardman		4	10
2010-19	Pinpi 5	Black sinuous curve of black jila		12	10
2010-20	Pinpi 5	Black oval		19	10
2010-22	Pinpi 5	Black snake		8	10
2010-24	Pinpi 5	Black glove head (replicate?)		3	6
2010-27	Pinpi 5	Black left eye of lollipop lizard man	B169	8	10
2010-31	Pinpi 5	Black head of concentric circle anthropomorph	B180	8	<1
2010-33	Pinpi 5	Black back of jila		3	9
2010-34	Pinpi 5	White of middle periscope man		7	4
2010-35	Pinpi 5	Black of middle periscope man		10	10
2010-38	Pinpi 5	Black outline anthropomorph		5	8
2010-39	Pinpi 5	Black of 3 headed jila		12	10
2010-40	Pinpi 5	Black of 3 headed jila		5	10
2010-41	Pinpi 5	Black from top curve of jila (above 3 headed jila)		6	10
2010-44	Biella	Black fern figure		3	10

Table 5
Success rate of sampling.

	Black	Inorganic	Total
Successful	27	9	36
Unviable	24	11	35
Total	51	20	71
% Success	53.0	45.0	50.7

Jilakurru (Fig. 1), known as Pinpi 5, was chosen for intensive study because it contains a large assemblage of black and white paintings (Fig. 2).

While Pinpi 5 has numerous phases of art production evident (changing style, subject matter and superimpositions), there is also a suite of motifs that could represent a composition. There is a large Jila (snake), which weaves across this panel amongst numerous headdress figures and other unusual anthropomorphs. Thirty paint samples were collected from this site (several were replicates), making this the most intensively sampled painting site in the world. Sixty percent of the samples were small and, unfortunately, did not have sufficient carbon for dating. We obtained 12 age determinations for nine motifs at this site (Tables 2 and 3).

The age result for the black outline of the large Jila (2010-42) at 225 ± 40 BP is consistent with other black and white snakes at other sites. Interestingly, a <10 μg carbon replicate sample (2010-28) at 470 ± 270 BP statistically agrees with the larger sample, even though the error is sufficiently large to lose resolution. Results from such small samples may be unreliable and should only be considered with other replicate radiocarbon dates, dates found in superimposition, and other archaeological information. A date for another black and white snake (2010-21) at 650 ± 180 BP for only 30 μg carbon is also consistent with other black and white snakes. This motif was painted at various sites from 1550 cal BP to modern times.

Samples from three stylistically dissimilar but proximal motifs reveal an age sequence which shows agreement with the superimpositionning (Fig. 3). Dates for the small black anthropomorph with a concentric-oval head (sample 2010-26) at 1600 ± 40 BP and the adjacent large solid black anthropomorph with a glove-like headdress (sample 2010-23) at 1160 ± 180 BP (30 μg C) overlap at 2σ. A black anthropomorph (sample 2010-25) superimposed underneath the glove-headed anthropomorph was produced earlier 2885 ± 55 BP (180 μg C).

Replicate samples 2010-30 and 2010-37 were collected from a bright black complex non-figurative design associated with two bird tracks (sample 2010-36). The location of this motif, superimposed over all other paintings in its vicinity, clearly identifies these as one of the last produced at the Pinpi 5 site (Fig. 4). Surprisingly, the replicate samples produced statistically indistinguishable older ages of 12,970 ± 270 and 12,620 ± 460 BP, even though one of the samples was only 20 μg carbon. The bird track produced in the same material but located slightly below the CXNF was dated to 5520 ± 290 BP on only 30 μg carbon. In the field, using portable X-ray fluorescence, we had determined that these vibrant black paintings were produced with manganese-pigment (>10,000 ppm Mn) – being different from all other black paintings not only at Pinpi, but at all other studied sites, which contained charcoal. In the laboratory, we confirmed the absence of wood grains under high magnification. In addition, during the plasma oxidation process, the black color of these three samples remained, which would be expected from a mineral pigment. If the pigment had been an organic charcoal, the black color would have turned to white ash as the charcoal was "burned" away. We suspect that these paintings are made with modern paint mixture composed partially of a petroleum-based products (carbide, or perhaps kerosene) resulting in the significantly older ages. It is possible that this was produced by an Aboriginal person (it is traditional motif) during the early droving activity along the CSR. Rowe has observed similar ages for rock art that was wetted with kerosene for photo-enhancement (Chaffee et al., 1994).

The range of age determinations at this site indicates that its assemblage was not produced as a one- off composition, but is the result of multiple phases of art production.

Table 6
Background samples of unpainted rock.

Sample	Site	UCA ID	Sample mass (mg)	μg C	CAMS ID	^{14}C Age (years BP)	Calibrated age (2 σ, 95.4%)	Calibrated age (2σ, 95.4%)
2005-01b	P13	B90	19	130	158672	865 ± 40	1150–1280 cal AD	800–670 cal BP
2005-02b	P13	B97	48	100	158681	1430 ± 45	575–770 cal AD	1375–1180 cal BP
2005-03b	M23	B37	44	40	132479	2100 ± 90	360 cal BC–140 cal AD	2310–1810 cal BP
2005-08b	BTD1	B20	33	20	Broken tube	–		
2005-09b	BTD1		86	20				
2005-15b	V12		41	2				
2005-16b	V12		48	3				
2005-18b	SG1	B32	190	80	133997	3190 ± 60	1610–1210 cal BC	3550–3160 cal BP
2005-19b	SG1	B22	21	8				
2005-20b	SG1	B85	15	30	158160	2050 ± 200	600 cal BC–600 cal AD	2500–1400 cal BP
2005-23b	SG1	B17	23	20	Broken tube	–		
2005-24b	SG1	B21	34	8				
2005-25b	SG1	B24	30	10				
2009-01b	GLM1		8	2				
2009-02b	GLM1		6	2				
2009-03b	GLM2		3	3				
2009-04b	GLM3		2	<1				
2009-05b	Night Jar		2	<1				
2010-10b	DS1		9	10				
2010-11b	DS28		5	6				
2010-12b	DS32	B162	149	40	158164	670 ± 140	1000–1700 cal AD	1000–300 cal BP
2010-13b	Pinpi 5		4	<1				
2010-16b	Pinpi 5		27	7				
2010-43b	Pirli 3		11	2				
2010-44b	Biella		2	<1				
2010-45b	Diebel		2	3				
2010-50/51b	BTD1	B179	5	<1				

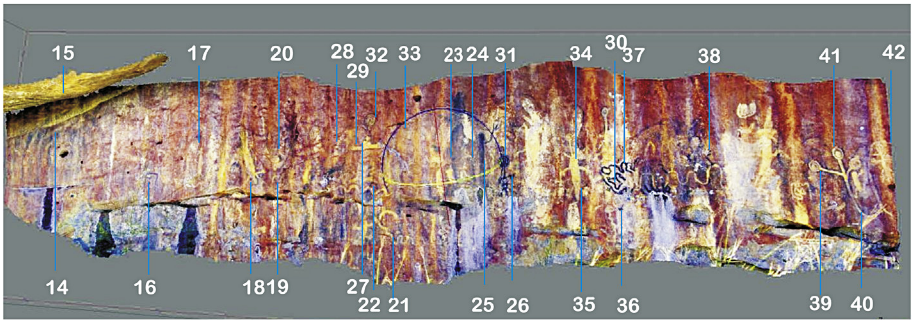

Fig. 2. Pinpi 5 rock shelter showing the 30 sample locations collected from this site. Numbers refer to 2010- sample numbers (see Tables 2–4). Blue lines indicate location where sample was collected.

4. Methodological implications

4.1. Small sample sizes

While the ability to assay small samples is the main technological advantage of AMS, there is still the concern that in many applied contexts there is a limit below which the ultra-small carbon sample cannot be assumed to represent a particular cultural event of interest (Scott and Harkness, 2000; Aubert, 2012). Amounts of organic carbon contained in pictograph paint samples vary from <1 μg to 1 mg, depending upon the type and amount of material removed from a painting. Technologically, counting samples as small as 1 μg is possible, but as archaeologists and chemists we must inevitably question the likely sources of this carbon.

When dates are obtained from an inorganic-pigmented painting, the assays come from non-specified materials. In many cases, sufficient amounts of carbon for ^{14}C measurement are collected from paint samples while negligible amounts of carbon are found in adjacent unpainted rock samples (backgrounds). In these cases, we can assume that the organic material being dated is associated with the paintings. Interestingly, we dated two dark red motifs, which are stylistically older than the predominantly black and white art the team generally targeted. These were found to be significantly older than the overlying charcoal paintings.

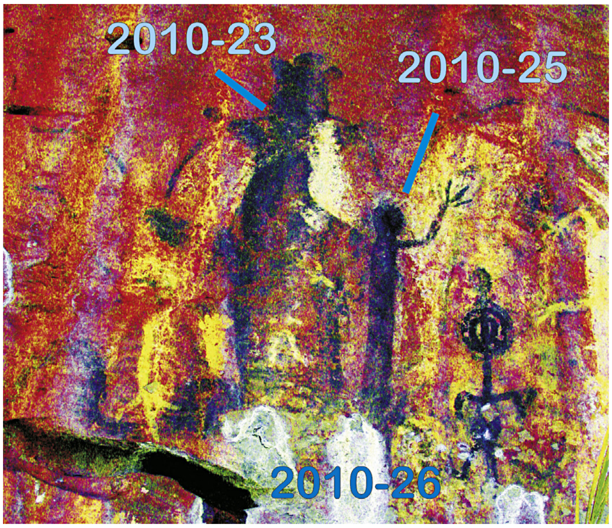

Fig. 3. Pinpi 5 anthropomorphs. Three stylistically dissimilar but proximal motifs which reveal an age sequence in agreement with the complex superimposition sequence (see Tables 2 and 3). D-stretched image (lds).

As in all archaeological applications where charcoal is dated, caution is required in interpreting dates due to *old wood* and *old charcoal* effects (Schiffer, 1986; Bednarik, 1994). In the Australian arid zone, however, the nature of the vegetation (i.e. sparse tree cover) human use of wood fuels, annual wild fires from lightning strikes and wood consuming termites mean that wood is unlikely to sit on surfaces for protracted periods of time: likely decades, but in open contexts very unlikely thousands of years. Pigment art production is embedded within a broader social set of actions – and Aboriginal people exercised both intentionality and structured art production using a specified range of paints. They were decorating shelters that they also lived in, and there is evidence in most excavated shelters that we have cross-dated that there is both contemporaneity of art and occupation as well as episodic art production and habitation.

Because our paint/pigment samples are small, factors such as smoke (from camp and bushfires), bacterial and other growths, and subsequent human contact with the paintings (i.e. touching, rubbing, retouching) are possible sources of contamination. We are still of the opinion that samples yielding less than ~50 μg carbon need to be viewed with caution, unless supported by other data. The focus must remain on identifying the source/nature of the dated material and its association with the event of interest.

Approximately half of the collected samples had sufficient carbon to obtain reliable age determinations (see Tables 4 and 5). Armitage et al. (1998) dated charcoal paintings with samples as small as 15 μg carbon, obtaining statistical agreement with larger replicate samples. While larger samples are always preferable, our

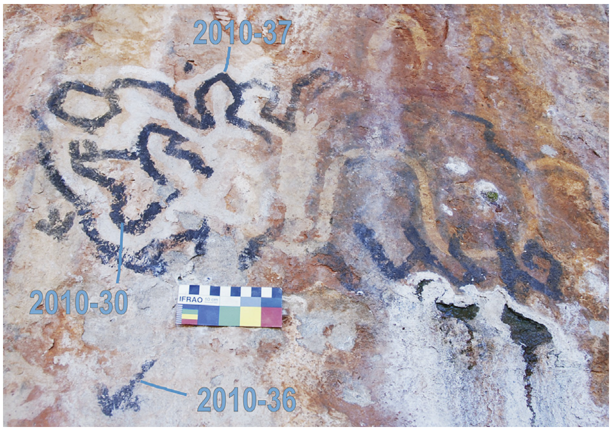

Fig. 4. Pinpi 5 Black Complex-Non-Figurative motif with three samples (-30, -36 and -37) suspected to have been made with modern paint mixture composed partially of a petroleum-based products.

data and the Armitage et al. (1998) study suggest that charcoal samples should contain at least >10 μg carbon for accurate and reliable results. Many of our replicate sample collections did not achieve a comparable result, because one and/or the other had insufficient carbon. Most of the pigment art in the Western Desert has been produced using inorganic pigments. This success rate is similar (c. 50%) to that achieved elsewhere globally for obtaining age determinations from inorganic (red and white) pigments. We do not know whether this is due to insufficient carbon remaining in a sample for reliable radiocarbon measurement or whether there was an absence of organic material being added to the paint in the first place. We are not aware of ethnographic accounts in the Western Desert for paint preparation methods and our results indicate that this is an area worthy of further research. For future work, we will continue to collect multiple samples and target motifs with visibly high quantities of surface pigment.

In this project we concentrated on sampling motifs with black paints, many of which we confirmed in the field – using portable X-ray fluorescence spectroscopy – as likely to contain charcoal pigment (i.e. not manganese). It was surprising that the charcoal samples also only had a c. 50% success rate and many of the dated samples returned values of <50 μg carbon. This is most likely due to the small sample sizes collected (ranging from 20 to 70 mg of paint). In our zeal to be as minimally intrusive as possible, it is possible that we did not collect enough material for dating. When sampling rock art, we tend to err on the side of caution, and preferentially select locations that will not visually impact on the art. Given the relatively high ‘failure’ rate, we perhaps need to be a little more strategic in ensuring that sufficient material is collected to guarantee a large enough yield of carbon. Our results suggest that larger samples need to be harvested – and that our conservatism (in terms of minimizing our impact on the motifs) may be implicated in this result.

4.2. Backgrounds

Of the 27 backgrounds collected, 19 had negligible amounts of organic carbon (Table 6). This suggests that the unpainted rock has insignificant amounts of naturally deposited/generated carbon (i.e. sources of contamination). This situation is ideal as it suggests that the organic material extracted from the paint samples is most likely from organic pigment and/or organic binders added to the paint during its preparation, and not inherent in the rock substrate.

For two of the dated backgrounds (2005-01b and 2010-12b), the corresponding paint samples only had 10 μg carbon and were not dated. For the other five dated backgrounds, it is likely that in these cases the background samples have indeed intersected with older (now not visible) painting episodes. Normally sample backgrounds are not dated, but these background samples posed some interesting research questions. Any difference in ages between the actual paint sample and its associated background could help source contamination, be this from modern carbon (from human contact, recent bush fires and/or biological growth) or older residual carbon (from older bush fires, painting episodes or organic crusts). For example, the backgrounds for samples 2005-3 and 2005-18 were 2000 to 3000 years older, respectively, than the results for the corresponding paint samples. This large age difference suggests that the majority of this potential contamination was likely to have been successfully neutralized by pretreatment of the paint samples prior to analysis. While this older material from the background could potentially skew the result older than the returned date, Sample 2005-18 statistically agrees with other dates received for similar motifs (e.g. sample 2005-19) at Serpent's Glen. This suggests that the age determinations for these motifs with older dated backgrounds are plausible and consistent. Whether this dated material is from organic contamination inherent in the rock substrate or a previous painting episode has not been established and this factor requires consideration in future dating initiatives.

5. Archaeological significance

These results provide the first dates for painting activity in the Australian Western Desert, and this project has yielded more art dates than any other in the world. We have demonstrated that the most recent phases of painting in the Western Desert were made in the last 1500 years (Figs. 5 and 6), and that this painting activity

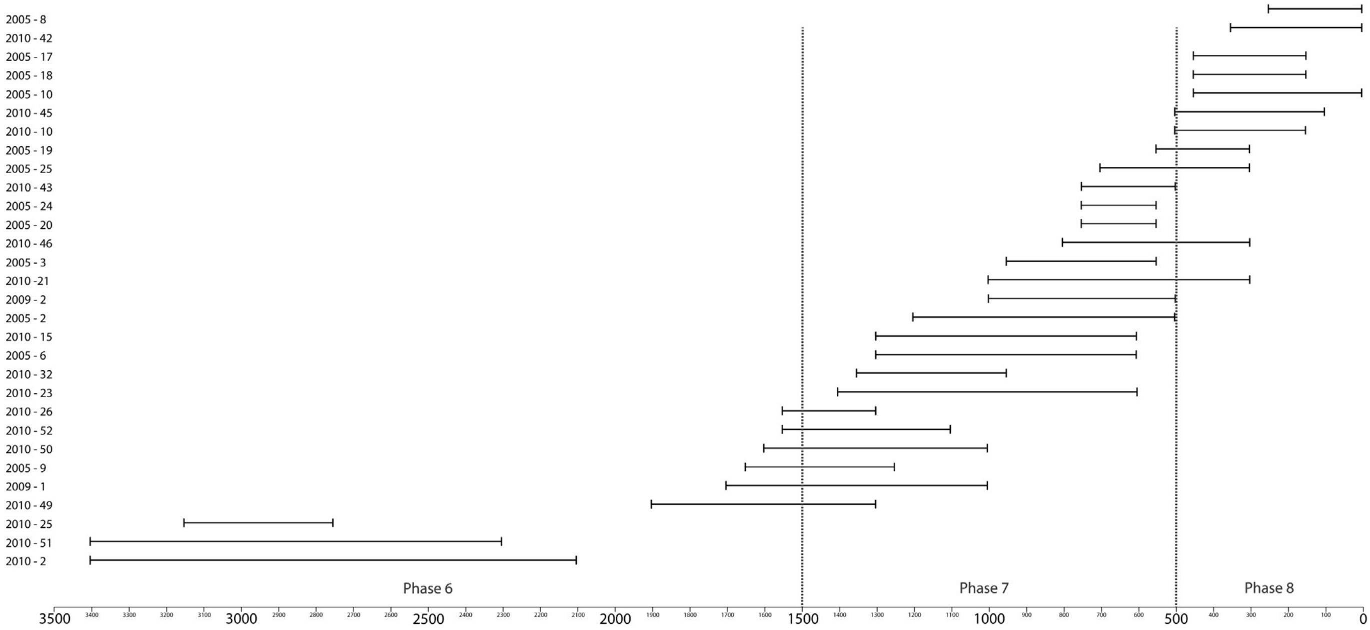

Fig. 5. Calibration curve showing distribution of the received art dates, correlated with archaeological occupation phases (Table 7). Sample numbers are shown on the vertical axis and years (calibrated BP) along the horizontal axis.

Fig. 6. Examples of dated motifs arranged in temporal order from older (at left) to younger (at right) showing relatively late introduction of snakes and variability in anthropomorphic depictions through time (examples are from Katjarra, Kaalpi, Jilakurru and Diebel). Not to scale.

Table 7
Western Desert occupation models with proposed art correlates showing the three most recent phases (adapted from McDonald and Veth, 2013b: Table 1).

Occupation phase	Linguistic correlations	Occupation model	Art correlates
Phase 6 6000–1500 BP	Ngayardic language (Wati) spreads into the Western Desert from the PIlbara	Occupation of all desert ecosystems Development of relatively fixed territorial ranges Regional exchange and information networks established; exotic raw materials (e.g. chalcedony) found in sites after 3500 BP	Pilbara art graphic moves into the WD – social pressures and tensions result from occupation by new groups: rock art used to establish territoriality, with localized style regions evolving
Phase 7 1500–500 BP	Spread of Wati language—Western Desert culture bloc develops: some loan words from northern languages Contact with central Australia	Increased intensity of site occupation Accelerated ritual and ceremonial cycle Increase in long distance exchange; shift from blocky multifunctional informal grindstones towards wet mill stones and formal arenaceous sandstone grindstones after c. 1200 BP	Art is used to negotiate broad-scale and local group identity in distinctive localized style regions Art reveals contacts with the north and centre
Phase 8 500 BP to contact	WD speakers intensify interaction from homelands into central Australia and also westwards into the Pilbara	Increased social dynamism and interaction with social networks in central Australia and Pilbara	Petroglyphs drop out of art repertoire Accelerated use of pigment art to negotiate broad-scale and local group identity. Shared graphic vocabularies from the centre and Pilbara. Pigment art and body painting share graphics

matches an increased intensity of site occupation generally. There are changes within this time period which can be correlated with broader archaeological signals of complexity (Table 7). We also observe significant changes in discard rates of artefacts and technological organization of extractive technologies such as seed-grinders and widespread adoption of tula adzes (Veth et al., 2001). These technological changes are matched by a very high level of stylistic heterogeneity in the art - which has been systematically dated within and between dialect groups for the first time. As a result of this dating programme it is possible to start unravelling the stylistic phases produced in this part of the Western Desert during more broadly defined occupation phases (see Fig. 6). It is clear that there is stylistic diversity within these most recent phases, with these examples of dated motifs coming from Jilakurru, Kaalpi, Diebel and Katjarra.

So far we have not attempted to systematically date earlier pigment phases, predominantly produced in red pigments, or the various petroglyph phases which predate this earlier pigment art. Given that human occupation of the Western Desert started more than 40,000 years ago, the dating of the deeper time art repertoire will require the mobilization of other chronometric techniques (e.g. Uranium Thorium on crusts, cosmic nuclide radiation).

Late Holocene dynamism across the desert is demonstrated by the contemporaneity of rock art production and more recent occupation patterns, confirming the complexity of recent arid zone stylistic behavior. Localized stylistic characteristics in the pigment art are distinctive in the last 500 years with neighboring groups, across dialect boundaries, producing the same subject matter (e.g. snakes, anthropomorphs) in distinctly different styles. This reveals different social interactions across space from the preceding art phase characterized by headdress-figures (presumably more than 1500-BP) which reflects a shared art repertoire, but with distinct intra-dialect group signaling. The so-far undated petroglyph repertoire from the deeper past shows different patterning again, with chains of connections from the Pilbara coast through to central Australia (McDonald, 2005; McDonald and Veth, 2006; McDonald and Veth, 2013b).

The presence of localized style regions in the art suggests greater territorial circumscription in the recent past compared to earlier periods, when we model there were longer-range polities and exchanges between people over very considerable distances, >1000 km (McDonald and Veth, 2013b). Multiple directions of connection (between central Australia, the Western Desert and the Pilbara uplands/coast) are indicated by the graphic traditions found in these Western Desert art provinces, and this project has – for the first time – tested the chronological relationships between these. The increased complexity of social relations across the Australian arid zone in the recent past (Veth, 2005; Smith, 2013) is confirmed by these results, repudiating the earlier model of a deep-time conservative desert culture (Gould, 1990).

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