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Radiocarbon Determinations on Chillagoe Rock Paintings: Small Sample Accelerator Mass Spectrometry

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ABSTRACT. Indirect dating methods have previously been applied to the rock paintings of north Queensland, utilising patterns of superimposition, depictions of material items and animals of known antiquity, the use of fragile paints such as mud and white kaolinite, and *in-situ* pigment stratigraphies. These patterns suggest that the vast majority of Chillagoe rock paintings are relatively young, likely less than 3500 years old. We directly analysed radiocarbon in the charcoal pigments in several of the Chillagoe rock paintings. Preliminary radiocarbon results at three sites confirm that these charcoal paintings are less than 3500 years old, as predicted. A change in the geographical distribution of rock art styles across north Queensland—from widespread non-figurative forms (as evident in surviving petroglyphs) to regionally distinctive motifs—suggests a regionalisation of artistic conventions starting around 3500 years B.P. Such a regionalisation implies that major cultural changes accompanied the changes in rock painting styles.

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Behavioural diversity is one aspect of the human condition that makes archaeology possible; it is the desire to characterise, model and understand both spatial variation in human behaviour, and its short- and long-term dynamics through space and time, that underlies this project. This is also the source of our methodological anguishes: how can we measure or otherwise describe past human activities, their spatial variability, and their trends at various temporal scales?

This paper is an exploration of method firmly rooted in this broader problem. Specifically, rock art is a manifestation of cultural activity. It is a cultural expression embedded in the social, political, economic and psychological circumstances of its creator(s). Each picture, or part thereof, was created some time in the past, and through time certain artistic conventions changed while others remained the same. While representing the actions of particular individuals at specific places and times, individual items of rock art thus also articulate cultural conventions of those times. Rock art is, therefore, not only amenable to spatial and temporal analyses, but through space and time it is also suitable for the investigation of cultural patterning and dynamics.

However, rock art is also notoriously difficult to date; thus it is not easily situated archaeologically. Individual items of rock art were usually created either by adding specific substances to rock surfaces (pictographs, such as paintings), or by taking away parts of the rock (petroglyphs, such as abrasions or peckings). It should be possible, therefore, to date the age of a particular artistic creation by either dating the addition of materials to a surface, or its subtraction; in both cases it is the event which we would like to date. In the case of the former, pigments can themselves consist of organic compounds, such as charcoal or other plant colours, which are amenable to radiocarbon dating. In other cases, the presence of organic binders or other intrusions (e.g., filaments from paint brushes) may offer the opportunity to date the pigments directly. In the case of petroglyphs, organic materials caught both beneath and above the modified surface may give maximum and minimum ages to the artistic event.

Unfortunately, the dating of the artistic event is more complex than this, as there are various potential factors complicating the issues. Some of these concern the antiquity of the organic materials when they were put on the wall, for example, the "old wood problem" in the case of charcoal drawings. Others involve sources of contamination, both in the form of fungi, algae and other organic activity, and in the intrusion of modern or ancient carbon through the activity of water, wind and the like. There is also the problem of retouching in ancient times, potentially adding layers of pigment through time. And last but not least, there is the real problem of extracting minute amounts of datable materials from a given rock surface.

This paper is a methodological contribution to the dating of rock art. As such, it is a contribution aimed at further exploring the ability of accelerator mass spectrometry (AMS) radiocarbon dating method to date charcoal drawings. It also aims to help elucidate the types of problems (and solutions) one may be confronted with when "directly" dating rock art. Although the case study presented here also forms an early stage in an attempt to understand the antiquity of the rock art of the Chillagoe region of northern Australia, at this stage the promising results obtained should be interpreted as much in terms of a methodological investigation as of a regional study.

The Chillagoe case study: background

Rock art is commonly found in many parts of northern Australia. Chillagoe is one such region, located some 120 km west of Cairns in north Queensland (Fig. 1). Archaeologically, Chillagoe can be divided into two temporal units (at a broad temporal scale): the period before ≈ 3500 years B.P., and that since (David & Chant, 1995).

Little is known of the earlier period. Of relevance here, however, is what appears to be a relatively homogeneous artistic tradition consisting of abstract motifs, including "star bursts", circles, pits, single lines as well as sets of lines. Non-figurative motifs such as these were clearly engraved on rock surfaces during this early period. Campbell & Mardaga-Campbell (1993: 59) have reported a single AMS radiocarbon age > 7085 ± 135 years B.P. for one pecked "sunburst" from Walkunder Arch Cave in the Chillagoe region, indicating the presence of very old motif forms similar to late Holocene paintings for the region. The degree to which such motifs may also have been painted or drawn is not yet clear. Campbell & Mardaga-Campbell (1993) and Watchman & Hatte (1996) have reported evidence of earth pigments laminated between ancient rock crusts at various sites around Chillagoe dating back to $28,100 \pm 400$ years B.P. The nature of the pictographs is yet unknown as they were revealed in crosssectional studies of encrusted rock surfaces-are they paintings, prints or stencils? In addition, uncertainties still exist concerning the anthropogenic nature of these pigments and the sources of the dated carbon (extracted by laser ablation). However, this evidence implies that pigment art has been created in the Chillagoe region ever since people first arrived in the region despite their more common occurrence after 3500 years B.P.

Certainly, ochre crayons and other small fragments of ochre are far more common within excavated, stratified deposits after ≈ 3500 years B.P. than they were from earlier deposits. All in all, it appears that before ≈ 3500 years B.P. rock art in Chillagoe and elsewhere in north Queensland consisted principally of engraved, non-figurative motifs, although pigment art was also practised. The early rock art of Chillagoe is believed to be very similar both formally and technically to that of other regions in north Queensland, including the Mitchell-Palmer limestone zone, Laura and the Koolburra Plateau > 100 km to the north (David & Chant, 1995; see also Rosenfeld *et al.*, 1981).

The period after \approx 3500 years B.P. saw major changes in the archaeological record of all parts of north Queensland. This was the time when dingoes first arrived in Australia; it was also the time when new stone tool types and technologies first appeared (e.g., systematic seed grinding stones, burren adzes, blades) (David & Lourandos, two manuscripts in press: *a*, *b*). Deposition rates of cultural items increased many-fold within individual sites, and the number of occupied sites across the landscape also increased markedly. Along with these changes came a major increase in the amounts of ochre dropped on ancient

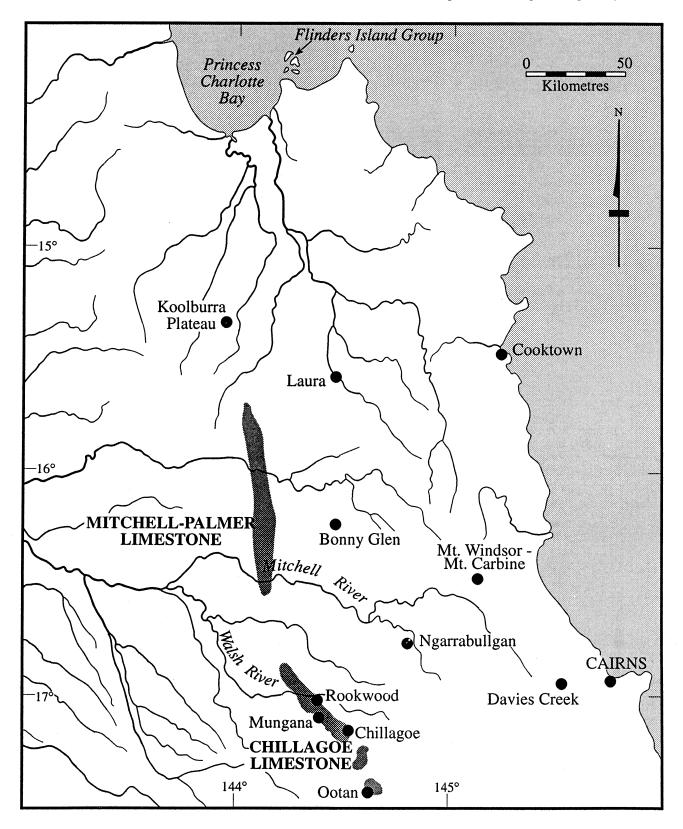


Figure 1. Map of northeastern Australia (Queensland) including the Chillagoe region.

288 Records of the Australian Museum (1998) Vol. 50

floor levels within individual sites; the implication is that painting, drawing, stencilling and/or printing activity increased significantly. Together, these various lines of evidence, often indicating quantitative increases in the activities of people on the landscape, have been interpreted by archaeologists to indicate (among other things) probable and noticeable increases in intensities of land use during the mid to late Holocene. This was probably associated with population increases (e.g., David & Chant, 1995; Lourandos, 1997), although we are still uncertain about what caused these changes (but see David & Lourandos, in press, *b*).

But this quantitative evidence does not stand in isolation. While at Chillagoe motif forms do not appear to have changed noticeably after ≈ 3500 years B.P., in neighbouring regions to the north they did. At Laura, a unique set of conventions commonly referred to as "Quinkan" art probably dates to the last 3500 years (and especially to the last 2000 years) (Cole et al., 1995; David & Lourandos, in press, b). In the Mitchell-Palmer limestone zone between Laura and Chillagoe, an extremely homogeneous body of paintings consisting almost entirely of monochrome or bichrome anthropomorphs emerged around this time (David & Chant, 1995; David et al., 1997). In the Koolburra Plateau nearby, painting conventions were similar to those at Laura, but here endemic "human-echidna" pictures began to be painted (David & Chant, 1995; Flood, 1987). In each of these regions the archaeological evidence points to ages for the paintings during the mid to late Holocene. The indication here is that in addition to the quantitative changes noted above, the rock art attained a more regionalised configuration; the cultural landscape appears to have become divided in space in a way not previously evident. The implications for cultural (including social, political and economic) dynamics in north Oueensland prehistory are major (David & Lourandos, in press, a).

Our understanding of the antiquity of the art is based, however, on rather poor, circumstantial evidence, although the various lines of evidence support rather than contradict each other (see David & Chant, 1995, for details). These include:

- 1. the occurrence of animals of known antiquity (e.g., dingoes, animals introduced by Europeans);
- 2. the presence of fragile pigments (e.g., white kaolinite, brown mud) in the art purported to be of late Holocene age;
- 3. peak deposition rates of ochre in the late Holocene layers of excavated sites with purported late Holocene rock art. The indications here are that most of Chillagoe's surviving paintings date to the last ≈ 3500 years B.P. It also implies that earlier times witnessed markedly lower levels of painting activity throughout north Queensland;
- 4. the few radiocarbon dates currently available for the art are in line with the above temporal model. All eleven radiocarbon dates for paintings and drawings obtained so far have revealed late Holocene ages. Late Holocene, as well as older dates, have been obtained for some engravings, indicating that engraving activity is both older than and contemporaneous with the period of

painting/drawing activity (Campbell & Mardaga-Campbell, 1993; Morwood & Hobbs, 1995; Watchman & Cole, 1993) (see above);

5. patterns of superimposition are in agreement with the relative ages as outlined above (that is, paintings are always superimposed over *patinated* engravings; some unpatinated engravings, however, occur over paintings, indicating a continuity of engraving traditions until recent times). This is in line with 4, above.

To test the above temporal model, we initiated a systematic AMS radiocarbon dating programme on paintings and drawings of both Chillagoe and the Mitchell-Palmer regions. This testing is a high priority for a regional archaeology of north Queensland, given the major implications of the apparent changes in distribution of rock art forms across space in north Queensland (and indeed Australian) prehistory (see David & Chant, 1995, for an elaboration of these implications). If the model is sustained, a regionalisation of land use, territorial structures and interregional interaction may be implied. Qualitative changes in the organisation of Aboriginal people on the ground may be implicated for the mid to late Holocene; this restructuring of cultural landscapes may well be associated with population increases, given the quantitative changes outlined above.

We report below on our results from the Chillagoe study.

Experimental procedure

Samples were collected by BD and MWR from five separate charcoal rock paintings in five rock shelters in the Chillagoe region. All samples came from limestone outcrops, except for sample CM75-1, which came from a painting on a granite rock near Ootan towards the southern edge of the Chillagoe region. Sample CM2-1 is from a set of 14 radiating lines ("sunburst") (Fig. 2); CM55-1 is from a set of concentric arcs; CM56-1 is from a set of enclosed parallel lines; CM62-1 is from a set of sub-parallel lines immediately underlying an elongated red linear design (the latter hence post-dates the radiocarbon-dated design); and CM75-1 is from seven radiating lines ("sunburst"). Photographs were taken before and after sample removal. Particular care was taken to keep the visual effects of sampling to a minimum. A small area of each drawing was scraped onto a square of aluminium foil, each time using a sterile scalpel blade. The resulting powder was a mixture of limestone (CaCO₃) substrate, charcoal pigment and overlying accretion. New latex gloves were worn during collection of each sample and when handling the foil to prevent contamination. Enclosed in the foil, each sample was placed in a zipper-seal polyethylene bag, labelled and taken to the plasma-chemical laboratory at Texas A&M University. There they were photographed under magnification and weighed after foreign matter was removed: CM55-2, three short, white fibres; CM55-4, four white fibres; CM55-1, one black and one white fibre; and CM75-1, one fibre (no colour recorded).

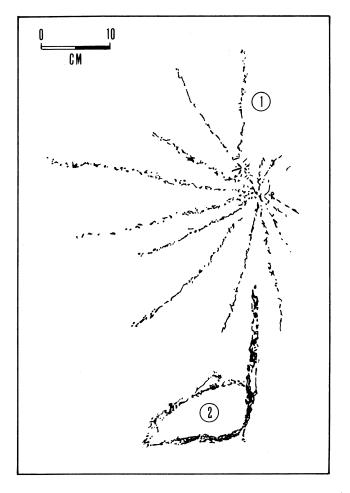


Figure 2. Tracing of the CM2-1 (top) and CM2-2 (bottom) rock drawings from Chillagoe. AMS radiocarbon results for CM2-1 are reported in Table 1.

Weights of total material ranged from 9 to 66 mg, with one exceptional sample that included large pieces of substrate limestone weighing 263 mg (CM62-1).

We use a radio frequency generated low-temperature ($\approx 150^{\circ}$ C), low-pressure (≈ 1 torr) oxygen plasma, coupled with high vacuum techniques, to remove organic matter in the rock painting sample; remaining carbonate or oxalate accretions do not decompose under these conditions (Ilger *et al.*, 1996 and references therein). Early studies in the Texas A&M University plasma-chemical laboratory established the efficacy for cleaning the plasma extraction system with oxygen plasmas before sample insertion to rid surfaces of organic contamination that may have been trapped in the chamber, as well as adsorbed CO₂ (Chaffee *et al.*, 1993).

A standard alkali (1 M NaOH) pretreatment was performed to remove possible contaminating humic acids from the charcoal pigments. Approximately 1 mL of 1 M NaOH was added to the sample in an ultracentrifuge tube and sonicated for 60 minutes at 50°C. After decanting off the alkaline solution, the remaining solid was sonicated at 50°C in dilute acid (0.1 M HCl) to reduce adsorption of atmospheric CO₂ and then rinsed in deionised distilled

water. Solid remaining after this treatment was transferred to aluminium foil and dried overnight at 100°C. The treatment solutions from both steps were combined and saved; these solutions contained visible amounts of solid from the decanting step. After plasma treatment of the solids, the remaining solutions were vacuum filtered to recover as much charcoal as possible. Before use, the binder-free, glass-fibre filters were baked at 500°C overnight to remove organic contamination. The filtered material was then plasma-chemically treated separately to provide replicate analyses of the sampled material. The filters with the charcoal, background limestone and accretion were dried at 100°C prior to plasma chemical treatment: these vacuum-filtered samples are referred to as "filtrates". Samples CM55-1, CM55-1 filtrate and CM56-1 yielded less than 10 µg carbon and were not analysed for radiocarbon. Sample CM56-1 filtrate, yielded 70 µg carbon and was sent to Australian Nuclear Science and Technology Organisation (ANSTO), but gave no output (negligible ¹⁴C counts and negligible ¹³C current) on AMS analysis. This may have been a result of poor graphitisation.

The charcoal samples isolated from the Chillagoe rock paintings were removed from the filter and placed in the plasma reaction chamber under ultra-high purity (99.999%) argon maintained above ambient pressure. The system was sealed and the samples subjected to sequential low power radio-frequency argon plasmas. Though unreactive, the energetic argon atoms in the plasma remove surfaceadsorbed CO₂ through inelastic collisions. When less than one μg carbon as CO₂ is desorbed via this process, the system is evacuated using oil-free sorption and ion pumps to $\approx 2 \times 10^{-7}$ torr. After this pressure has been maintained overnight, all pumping is stopped and any rise in the system pressure is recorded over time. If the pressure increase were all from CO₂, as we assume to provide a worst-case scenario, it can be converted using the ideal gas law to an equivalent weight of carbon. The maximum rise in pressure during a vacuum integrity check occurred with sample CM62-1; after 60 minutes, the system pressure rose to 1.0 \times 10⁻⁴ torr. This corresponds to < 0.2 µg carbon even with the unrealistic assumption that all the pressure rise is due to CO₂. Because this corresponded to less than background levels of carbon from graphitisation (typically an upper limit of 1.1 µg carbon), they were always considered to be negligible. Plasma-chemical oxidation followed these vacuum integrity checks, using ultra-high purity (99.999%) oxygen passed through a liquid nitrogen-cooled finger to remove any contaminants from the gas fill line. The CO₂ and H₂O produced was collected in a 6 mm glass tube cooled to liquid nitrogen temperature (-194°C). Water was removed from the CO₂ using an ethanol-liquid nitrogen slush prior to refreezing the finger in liquid nitrogen; the tube was then removed by flame-sealing. The Chillagoe paint samples yielded from less than 10 up to 110 µg carbon as CO_2 (Table 1). Samples yielding less than 10 µg carbon were stored at Texas A&M University, as they are too close to the expected AMS background for reliability. Radiocarbon analysis was conducted on the other samples to investigate the reliability and accuracy of radiocarbon dates obtained on very small samples of carbon. The sizes

sample	carbon (micrograms)	AMS ID	modern carbon $\%$	¹⁴ C age (years B.P.)
CM2-1	45	OZB758	86.11 ± 4.69	1200 ± 440
CM2-1 filtrate	40	OZB783	65.93 ± 2.81	3350 ± 350
CM55-1	< 10	too small	not measured	_
CM55-1 filtrate	< 10	too small	not measured	_
CM56-1	< 10	too small	not measured	_
CM56-1 filtrate	70	OZB764	no output	_
CM62-1	15	OZB586	88.46 ± 5.33	990 ± 485
CM62-1 filtrate	95	OZB765	85.42 ± 2.59	1270 ± 250
CM75-1	75	OZB587	71.72 ± 2.6	2670 ± 290
CM75-1 filtrate	25	OZB782	77.96 ± 4.92	2000 ± 510

Table 1. Radiocarbon results for replicate analyses of five Chillagoe rock paintings.

of our organic carbon samples analysed by the AMS (Table 1) were often less than the 100 μ g typically needed for a reliable AMS analysis. The sealed glass tubes containing the CO₂ extracted from the paintings were shipped to the ANTARES AMS facility at ANSTO. The carbon dioxide was converted to graphite using a modification of the method of Jull *et al.* (1986). In a single tube, the carbon dioxide was reduced by iron catalyst (at 600°C) in the presence of zinc (400°C) and a small amount of hydrogen. The resulting graphite/iron mixture was loaded into aluminium holders for AMS measurement.

Results

Radiocarbon results are shown in Table 1. Filtrate samples provided additional results from each of the three samples analysed (CM2-1, CM62-1 and CM75-1). Large uncertainties reflect the poor statistical precision resulting from the low output (14C counts and ¹³C current) of small samples. We emphasise that the dates listed in Table 1 are dates for the charcoal; as always in archaeology, the charcoal age does not necessarily date the archaeological event desired, in this case the time of painting of the motif (the "old wood problem" [Schiffer, 1986]). However, in the Chillagoe region we argue that while it is possible that old charcoal was picked-up from ancient floor surfaces rather than created afresh for rock drawing, the "old wood problem" as conventionally defined-that is, that the wood itself was old before burning—is unlikely to be a major problem, given that in this part of north Queensland no species of tree lives more than a few hundred years. Once dead, plant material (including wood) does not last very long on the ground (again, a maximum of tens of years), given the tropical nature of the area and the activity of termites. Therefore, the difference between the age of the archaeological event and the time of burning of the wood may be of a few hundred years, but it is even more likely to be of the order of tens of years only.

More problematic in our case is the possibility that ancient charcoal was picked-up from old occupational surfaces, or even dug-up from below the ground within previously occupied rock shelters. This is a very difficult phenomenon to measure, although one noticeable characteristic of deposits prior to the late Holocene is a marked paucity of charcoal (along with lower amounts of all types of cultural materials). In many excavated sites in north Queensland (e.g., Fern Cave, Hearth Cave, Mitchell River Cave), charcoal is either extremely rare or totally absent from deposits dating to before ≈ 3500 years B.P. (David & Chant, 1995). Also, most archaeological rock shelters throughout north Queensland only have mid to late Holocene deposits; that is, there is no evidence for earlier occupation. Indeed, this is a common phenomenon across Australia (David & Chant, 1995). Furthermore, if pre-existing charcoal was picked-up from the old floor surface at the time of painting or drawing, it would date to the immediately previous time of occupation. Given the more or less continuous nature of sediment deposition on inhabited floor surfaces (albeit at varying rates), exposed floor surfaces are unlikely to be much older than the events that took place on them. Exceptions to this are generally relatively identifiable geomorphologically, such as in the southwest Tasmanian rainforests where ancient floor surfaces have remained exposed for many thousands of years (e.g., Porch & Allen, 1995). But even here lack of sediment deposition during "recent" times was due to an absence of human occupation during this time. There is no evidence of such sedimentation trends, cultural or geomorphological, in any of the sites investigated at Chillagoe, the Mitchell-Palmer, Laura or the Koolburra Plateau (David & Chant, 1995; Morwood & Hobbs, 1995). On the contrary, in each of the excavated sites the character of mid to late Holocene occupation appears to be more or less continuous, as measured at an archaeological time scale (i.e. one occupational event leads into another up the sequence; these are not easily differentiated in time via radiocarbon analyses). Given the above, we conclude that it is extremely unlikely that the charcoal examined from the rock drawings incorporate any significant degree (i.e. of more than ≈ 300 years at the most) of old wood problems, in any of the various senses described above.

Corrections were made to the radiocarbon ages for the background from graphitisation at ANSTO. We have conducted radiocarbon analyses of ¹⁴C-free materials oxidised with the plasma-chemical technique, obtaining an average radiocarbon age of > 42,000 years B.P. The evidence suggests that negligible modern carbon is added by the plasma treatment (Ilger *et al.*, 1995).

To compare the two radiocarbon ages (the original solid left after decanting the NaOH solution and the solid filtered from the solution) for each of the three sets of samples, we used the statistical method described in Ward & Wilson (1978). In that method, the two ages are tested for difference using equation 1.

$$T = \sum (A_i - A_c)^2 / \sigma_i^2 \tag{1}$$

(1) where T is the test statistic with χ^2 distribution for two dates, $\chi^2 = 3.841$. Thus if T is < 3.841, then the two dates are statistically indistinguishable and can be combined according to equation 2.

$$A_c = \sum (A_i / \sigma_i^2) / \sum (1 / \sigma_i^2)$$
(2)

(2) where A_c is the combined (pooled) date, A_i are the individual radiocarbon ages and *i* values are 1 and 2, and σ_i are the uncertainties of the measurements. Thus the combined or pooled age is weighted as the inverse of the squares of the uncertainties of the measurements.

CM62-1 yielded an age of 990 ± 485 years B.P.; the solid remaining in the rinse solution (CM62-1 filtrate) yielded an age of 1270 ± 250 years B.P. Ward & Wilson analysis indicated that ages for these two replicates of CM62-1 were statistically indistinguishable. Agreement is striking in view of the fact that there was only 15 µg carbon in the smaller of the two. The combined age for CM62-1 calculated from equation 2 is 1210 ± 220 radiocarbon years B.P. CM75-1 was dated to 2670 ± 290 years B.P. as compared to 2000 ± 510 years B.P. for CM75-1 filtrate, again statistically indistinguishable. The combined age for CM75-1 is 2500 ± 250 radiocarbon years B.P. The ages for the two replicates of CM2-1 were different statistically (1200 \pm 440 years B.P. for CM2-1 and 3350 \pm 350 years B.P. for CM2-1 filtrate) and cannot be combined. We assume that the older of the two ages is more accurate based on the observation in the Texas A&M University laboratory that extremely small samples (such as our 15 µg samples from CM62-1) are more likely to be significantly affected than larger ones by minute amounts of modern contamination and yield younger results.

Conclusion

Although the number of dated samples is too small to carry statistical significance, these preliminary radiocarbon analyses confirm the expectation that the Chillagoe paintings studied here were painted within the past 3500 years (David & Chant, 1995). While we are encouraged that two of the three coupled samples reported here agree within statistical expectations with one another, we remain cautious and conclude that much more work remains before we can say that such small samples can be reliably analysed by AMS at ANSTO.

292 Records of the Australian Museum (1998) Vol. 50

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