

AMS DATING OF ALLUVIAL SEDIMENTS ON THE SOUTHERN TABLELANDS OF NEW SOUTH WALES, AUSTRALIA

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ABSTRACT. The dating of alluvial deposits is frequently hampered by a lack of good-quality charcoal or other material for radiocarbon samples. We have dated two sites in southeastern Australia using traditional radiometric methods with minimal pretreatment. Results yielded an inconsistent chronology, affected by contamination with younger humic materials. A more consistent and older chronology was achieved using AMS dating of rigorously pretreated samples of fine-grained charcoal. The results have important implications for the radiocarbon dating of many Late Quaternary stratigraphic sequences with low charcoal abundance.

INTRODUCTION

Alluvial deposits of small drainage basins provide records of the history of erosion that have long been used to determine the controls on denudation and landform evolution. Of particular interest is whether cycles of valley aggradation and degradation are controlled by regional changes in climate or land use, or whether they are controlled by local factors, such as extreme floods or intrinsic instability within each basin (Patton & Schumm 1981; Schick 1974). The radiocarbon chronology of deposition has been central to the discussion. Those invoking climatic changes as the control on denudation refer to synchronous changes in aggradation across many catchments as independent evidence for climatic change (Knox 1983; Williams 1978). In contrast, a lack of regionally synchronous deposition has been used to dismiss environmental change as the control on denudation, and to invoke localized catastrophic events or intrinsic geomorphic changes (Young & Nanson 1982; Prosser 1991). Both arguments rely upon accurate dating of the alluvial sediments, which may not always have been achieved in the past because of incomplete removal of contaminants in the samples or insufficient material for dating.

We report here the problems encountered in dating detrital charcoal and bulk organic sediment from two headwater drainage basins of the Murrumbidgee River, using conventional sample preparation and radiometric dating techniques. In particular, contamination by younger organics in the sediments resulted in an incomplete, and, at times, internally inconsistent chronology of deposition.

The small sample size required by accelerator mass spectrometric (AMS) radiocarbon dating makes more rigorous and destructive sample preparation techniques feasible, and was used here to resolve the conflicting results from earlier radiometric dating. We describe a technique for the removal of contaminants, based upon pollen preparation techniques, together with the implications for the original chronologies.

SITE DETAILS AND STRATIGRAPHY

Wangrah Creek and the Lanyon alluvial fan are located on the Southern Tablelands of New South Wales, 80 and 20 km south of Canberra, respectively (Fig. 1). At Wangrah Creek, a 10-km-long continuous gully has incised to bedrock through a narrow alluvial flat, revealing a sequence of late

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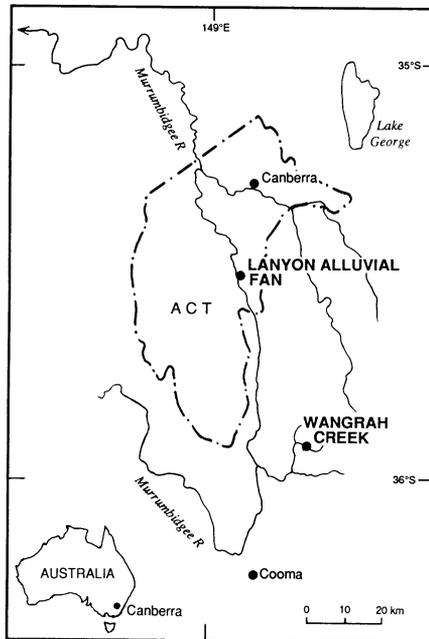


Fig. 1. Location of Lanyon Fan and Wangrah Creek alluvial sites in southeastern Australia

Quaternary alluvial deposits. Prosser (1988) determined the stratigraphy and ^{14}C chronology of the deposits from exposures in the gully walls and cores from the adjacent terrace. A similar situation occurs at Lanyon, where a gully has eroded up the southern side of a 1-km-long, low-angle, alluvial fan adjacent to the Murrumbidgee River, revealing a sequence of fan and valley fill units mapped by Mullins (1985).

Gully walls at Wangrah Creek reveal three major alluvial units, Swampy Meadow Units (SMU) 1 to 3, in order of increasing age. Each unit represents a major phase of aggradation preceded by a phase of gully erosion that removed much of the previously deposited sediment (Prosser 1991). The buried walls of previous gullies can be observed in some sections, but each unit aggraded to a slightly higher level than the previous one, so that, in most sections, the units are arranged as overlying sheets separated by subhorizontal disconformities (Fig. 2). The units consist of uniform, highly bioturbated and mildly organic, fine sandy muds. The sediment is identical to that accumulating in ungullied valleys of the region, where flows thread through a saturated surface densely covered in tussock grasses and sedges; hence, the term, Swampy Meadow Units. Sheets of sand were deposited over SMU 1 at the foot of discontinuous gullies in the initial stages of the present gully erosion.

Mullins (1985) identified six alluvial units at the Lanyon fan, named in order of increasing age. Unit 1 contains benches of sand and gravel confined within the present gully, and Unit 2 consists of discontinuous sand sheets similar to those overlying SMU 1 at Wangrah Creek. Underlying Unit 2 are four major alluvial units, up to 2.0 m thick, which are continuous along the length of gully and separated by subhorizontal disconformities (Fig. 3). Unit 3 is characteristic of fan sediments, containing many planar beds and shallow channels of imbricated gravels and sands, separated by thicker deposits of sandy muds. Units 4, 5 and 6 fine upwards from massive clayey sands and coarse sandy clays at the base to bioturbated, black organic clays at the top, similar to the Swampy Meadow Units of Wangrah Creek. All of the six units on the fan were deposited by fluvial processes.

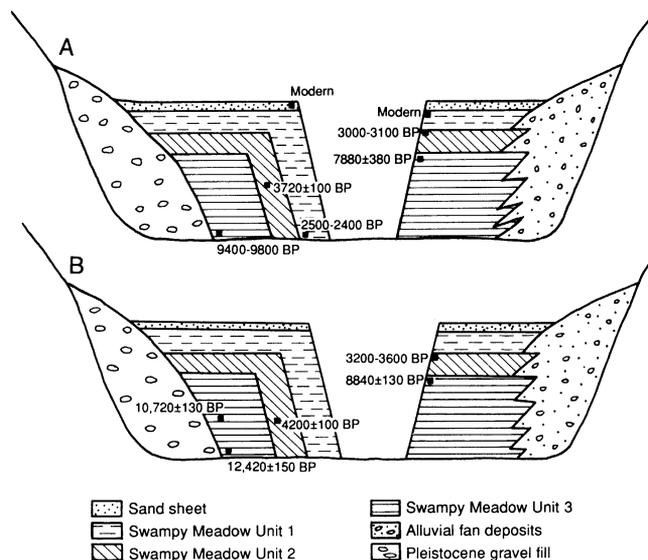


Fig. 2. Wangrah Creek stratigraphy. A. Radiometric chronology; B. AMS chronology.

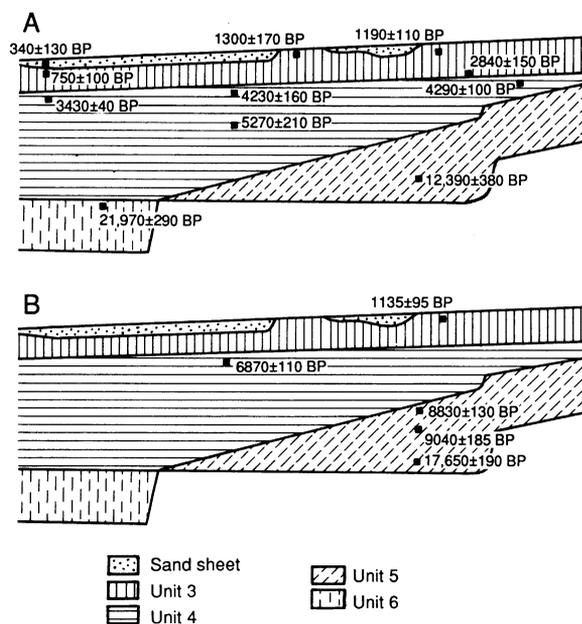


Fig. 3. Lanyon Fan stratigraphy. A. Radiometric chronology; B. AMS chronology.

Vertical movement of material in the older units is evident at both sites, which may have introduced contaminants to the sampled charcoal fragments. Complexes of iron, manganese and organics have precipitated around biotubules in the lower parts of the units and charcoal is absorbing oxides to give a similar appearance. Prosser (1988) and Mullins (1985) noted a general decrease in organic content with increasing age at both sites, which, given the close similarities in the lithology of the units, may reflect gradual loss of organics by leaching, complexing and oxidation. Organic carbon content ranges from 0.2 to 4% at Wangrah Creek and from 0.4 to 1.2% at Lanyon.

SAMPLING AND PRETREATMENT CHEMISTRY

We collected most samples for radiometric dating at Wangrah Creek from lenses of detrital charcoal, containing fragments *ca.* 1 cm in diameter. Other, less satisfactory samples were from smaller, soft charcoal fragments disseminated throughout the sediment. All samples were given minimal pretreatment, which is standard practice for routine radiometric dating, often simply an acid wash. A few samples were also given an alkali extraction (M. J. Head, personal communication). We used both detrital charcoal and bulk samples of organic sediment for radiometric dating at Lanyon. Bulk samples were washed in alkali solution to extract humic acids, and the insoluble residues were then dated. The bulk samples give a minimum age for the sediment, because they probably include organics derived from plant growth after deposition ceased.

AMS samples were collected as bulk sediment lumps from cleaned back exposures in the present creek beds, slurried in water and equilibrated with 1 mol HCl, then washed through standard sieves. For most samples, only the fine fraction passing a 63 μm (230 mesh) sieve was used for the subsequent experiments. The fine fraction was dried at room temperature under reduced pressure, since we have observed that high-temperature drying ($>50^\circ\text{C}$) may lead to the mobile organic components becoming very resistant to dissolution. A sequence of chemical extractions, developed from the preparation of samples for pollen and charcoal particle counting (Gray 1965; Singh 1981; Clark 1983), were used to remove soluble organic materials and most of the mineral components:

1. Cold extraction with 10% KOH, repeated until no further color was present in the solution
2. Hot 40% HF digestion for 2–3 h, repeated until most of the inorganic material dissolved
3. Cold oxidation with 10% NaClO₃ in 35% HNO₃ for 30–60 min
4. Cold extraction with 5% NH₃ solution
5. Reacidification with cold 1 mol HCl.

Each step was followed by washing with distilled water, and centrifuging at 3000 rpm to separate solutions from the residue. The rationale for this sequence is that the alkali solutions remove humic and fulvic acids, the HF destroys clay/organic complexes, and the oxidation renders any remaining organic material soluble in alkali. This sequence of chemical extractions results in the production of residual samples containing mainly pollen and fine charcoal particles. Dates on this material have yielded maximum ages for other fine-grained sedimentary materials (Gillespie *et al.* 1991, 1992).

DISCUSSION OF RADIOMETRIC DATING RESULTS**Wangrah Creek**

Table 1 lists radiometric dates from Wangrah Creek, which show increasing inconsistency in the ¹⁴C results with increasing age of units. The top of SMU 1, the sand sheets and charcoal from deposits in the present gully all have a modern age (ANU-5512, -4452, -5513), consistent with historical records that show that SMU 1 was still being deposited in 1842 (Prosser 1991). There are four consistent samples that show the base of the unit started accumulating between 2.5 and 3 ka BP (ANU-5394, -5364, -5451, -5365), and several consistent ages from various depths within the unit (ANU-5393, -4453, -5454, -4454, -4455, -5367).

Dates from the upper part of SMU 2 are similar to, or overlapping with, the well-controlled age of SMU 1 (ANU-5453, -5317, -5714, -5368). Two poor samples (ANU-5366, -5583) from transect WCT 8 are chronologically inverted (neither offer a feasible age), and the base of the unit is undated, but must be >3.7 ka BP (ANU-5035). The base of SMU 3 was consistently dated at 9.5–10 ka BP from three good lenses of charcoal (ANU-5395, -5510, -5369). Samples from an upstream site, however, gave the same age for the top and bottom of the unit, despite separation by 1 m of

massive sandy mud (ANU-5715, -4457), and another sample from the middle of the unit gave an inconsistent result (ANU-5351). In earlier papers on Wangrah Creek, Prosser (1987, 1990) reported a fourth alluvial unit, older than SMU 3. Reconsideration of the stratigraphic relationships in the field, in the light of the new AMS dates, showed no clear evidence for a fourth alluvial unit, which is now considered to be a part of SMU 3.

TABLE 1. Radiometric Dates From Wangrah Creek

ANU-no.	Location	Sample material	Age (yr BP)
4452	Sandsheet	Coarse charcoal	Modern
5513	Channel	Coarse charcoal	Modern
5512	Top Unit 1	Coarse charcoal	Modern
5393	50 cm from top Unit 1	Coarse charcoal	810 ± 110
4453	40 cm from top Unit 1	Coarse charcoal	870 ± 120
5454	100 cm from top Unit 1	Coarse charcoal	1150 ± 130
4454	Middle Unit 1	Coarse charcoal	1380 ± 90
4455	Middle Unit 1	Coarse charcoal	1370 ± 80
4799	Middle Unit 1	Coarse charcoal	1760 ± 160
5367	Middle Unit 1	Coarse charcoal	2190 ± 100
5394	55 cm above base Unit 1	Coarse charcoal	2480 ± 160
5364	50 cm above base Unit 1	Coarse charcoal	2720 ± 120
5451	Base Unit 1	Coarse charcoal	2910 ± 130
5365	40 cm above base Unit 1	Coarse charcoal	2950 ± 100
5714	Top Unit 2	Coarse charcoal	2140 ± 290
5453	Top Unit 2	Coarse charcoal	3000 ± 150
5317	Top Unit 2	Coarse charcoal	3110 ± 130
5368	Middle Unit 2	Coarse charcoal	3290 ± 120
5035	Middle Unit 2	Coarse charcoal	3720 ± 100
5583	Middle Unit 2	Fine charcoal	9570 ± 170
5366	Base Unit 2	Fine charcoal	2870 ± 160
5351	Middle Unit 3	Fine charcoal	6710 ± 410
5715	Top Unit 3	Coarse charcoal	7880 ± 380
4457	Base Unit 3	Coarse charcoal	7840 ± 100
5395	Base Unit 3	Coarse charcoal	9410 ± 220
5369	Base Unit 3	Coarse charcoal	9840 ± 380

Lanyon Fan

Table 2 lists radiometric dates from Lanyon. Unit 6 was deposited some time before 22 ka BP (ANU-4707) and the upper, organic part of Unit 5 was deposited before 12.4 ka BP (ANU-4709). The minimum age of the top of Unit 4 is 4.2–4.3 ka BP from both a burned root and a bulk sample (ANU-4665, -4711). A third sample further downstream gave a younger age of 3430 ± 40 BP (ANU-4706). Charcoal at the base of Unit 3 was dated at 2840 ± 100 BP (ANU-4710), consistent with two other charcoal samples from further up the unit (ANU-4712, -4668). A bulk sample from the top of the unit showed that deposition had ceased by 750 ± 100 BP (ANU-4705). Charcoal at the apex of the fan and in Unit 2 was dated as modern and 340 ± 130 BP, respectively (ANU-4666, -4667, -4704).

At both sites, determination of a complete ¹⁴C chronology was hampered by inconsistent results and a paucity of coarse detrital charcoal. At least five samples from Units 2 and 3 at Wangrah

Creek gave anomalously young ages (ANU-5714, -5366, -5368, -5351 and -4457), suggesting contamination from organics leached from above. The bulk samples at Lanyon also probably underestimate the age of the sediment. One sample from Wangrah Creek was anomalously old (ANU-5583), possibly a result of reworking of older charcoal into the deposits, a process that Blong and Gillespie (1978) identified for fluvial charcoal. However, reworking is not considered to be a problem at either site, provided coarse charcoal is sampled, because the fragments are extremely fragile. The chronology of both sites supports this interpretation, with the ages of the youngest deposits converging on the known historical age for the gully erosion that terminated deposition.

TABLE 2. Radiometric Dates From Lanyon

ANU-no.	Location	Sample material	Age (yr BP)
4666	Fan apex	Charcoal (tree root)	152.3 ± 2.3 pMC
4667	Fan apex	Charcoal	98.5 ± 0.9 pMC
4704	Unit 2	Charcoal	340 ± 130
4705	Unit 3	Soil	750 ± 100
4668	Upper Unit 3	Charcoal	1190 ± 110
4712	Upper Unit 3	Charcoal	1300 ± 170
4710	Basal Unit 3	Charcoal	2840 ± 150
4706	Unit 4	Soil, alkali washed	3430 ± 140
4711	Unit 4	Soil	4230 ± 160
4665	Top Unit 4	Charcoal (tree root)	4290 ± 100
4713	Middle Unit 4	Soil	5270 ± 210
4709	Unit 5	Soil	12,390 ± 380
4707	Unit 6	Soil	21,970 ± 290

DISCUSSION OF AMS DATING RESULTS

Although the samples used for the AMS dating were not the same as those for the radiometric work, they were selected from the same exposures and, in most cases, at or near the original sites. Thus, the two sets of dates are not strictly equivalent, but can be compared because they are from the same stratigraphic units. Tables 3 and 4 clearly show that the AMS dates on fine-grained charcoal, given the full pretreatment described above, are significantly older than the equivalent radiometric dates on conventionally pretreated macroscopic charcoal (Table 1, 2; Fig. 2, 3).

Taking the set of dates from SMU 2 at Wangrah Creek as an example (Table 3), it is clear that the chlorate oxidation has the effect of releasing more humic material from charcoal than has already been extracted with alkali. Humic acids extracted after oxidation here have about the same age as the traditional alkali-extracted humics, which indicates that charcoal pretreated in the traditional way will not yield the correct age. These dates may result from a size-dependent age effect, as demonstrated for fluvial charcoal by Blong and Gillespie (1978). We think that the major factor involved is the more rigorous chemical pretreatment given to the AMS samples, because the dates on the <63 μm and >300 μm fractions are virtually identical (NZA-753, -746 and -747, -754). In all Wangrah Creek samples measured, the humic acid dates are younger than the residual oxidized charcoal.

At Lanyon, we again find that the fine-grained charcoal given the full pretreatment sequence are significantly older than the minimally pretreated charcoal or bulk organic samples dated radiometrically. Age differences between ^{14}C dates of humic acids and residual charcoal are variable in both magnitude and direction. Unit 3 has variable $\delta^{13}\text{C}$ values, with the acid- and alkali-extracted residue

younger than the humic acid dates. In Unit 4, the oxidized residue is older than the humic acid date, and in Unit 5, there are no consistent age differences or $\delta^{13}\text{C}$ values.

TABLE 3. AMS Dates From Wangrah Creek

NZA-no.	Location	Fraction dated	$\delta^{13}\text{C}$	Age (yr BP)
749	Upper Unit 2	Humic acids	-25.4	2750 ± 100
751	Upper Unit 2	Humic acids after oxidation	-25.6	2970 ± 100
753	Upper Unit 2	Charcoal after oxidation (<63 μm)	-25.3	3225 ± 85
746	Upper Unit 2	Charcoal after oxidation (>300 μm)	-25.5	3550 ± 150
752	Lower Unit 2	Humic acids after oxidation	-25.7	3700 ± 110
750	Lower Unit 2	Humic acids	-25.7	3940 ± 110
747	Lower Unit 2	Charcoal after oxidation (>300 μm)	-25.8	4220 ± 110
754	Lower Unit 2	Charcoal after oxidation (<63 μm)	-25.1	4250 ± 90
481	Top of Unit 3	Humic acids	-24.6	8130 ± 110
566	Top of Unit 3	Residue after oxidation	-24.5	8840 ± 130
500	Middle of Unit 3	Humic acids	-24.5	8890 ± 140
543	Middle of Unit 3	Residue after oxidation	-25.0	10,720 ± 130
497	Middle of Unit 3	Humic acids	-24.4	9550 ± 110
544	Middle of Unit 3	Residue after oxidation	-23.9	11,740 ± 270
482	Low in Unit 3	Humic acids	-24.9	9830 ± 130
545	Low in Unit 3	Residue after oxidation	-23.9	12,420 ± 150

TABLE 4. AMS Dates From Lanyon

NZA-no.	Location	Fraction dated	$\delta^{13}\text{C}$	Age (yr BP)
321	Top of Unit 3	Residue after acid and alkali	-24.7	1135 ± 95
318	Top of Unit 3	Humic acids after oxidation	-24.9	1555 ± 110
244	Top of Unit 3	Humic acids	-21.5	1640 ± 195
317	Middle Unit 3	Humic acids after oxidation	-25.1	1995 ± 175
320	Top of Unit 4	Humic acids	-23.5	6580 ± 115
498	Top of Unit 4	Residue after oxidation	-23.6	6870 ± 110
483	Top of Unit 5	Humic acids	-21.0	7630 ± 120
490	Top of Unit 5	Residue after oxidation	-21.3	8830 ± 130
324	Middle Unit 5	Residue after oxidation	-21.6	9040 ± 185
319	Middle Unit 5	Humic acids	-21.1	9860 ± 150
495	Base of Unit 5	Residue after oxidation	-25.1	17,650 ± 190
501	Base of Unit 5	Humic acids	-25.0	18,290 ± 200

CONCLUSIONS

The more thorough chemical pretreatment used on the AMS samples in this work yields a more consistent chronology, with no inversions or other anomalies at either site studied. The use of oxidation is shown to be necessary for the complete removal of humic materials, even when the traditional alkali extraction has been used. It is intriguing to think that more information may be available from the study of the different humic fractions using both ^{14}C and ^{13}C values, which may lead to a better understanding of the mechanisms by which our original charcoal in sediment is contaminated by younger or older mobile organic carbon. Our results have important implications

for ^{14}C dating all alluvial sediment sequences, because we have shown that the traditional pretreatment chemistry is not adequate. Related work on other sites with different lithologies and sediment characteristics indicates that the more rigorous pretreatment methodology described here is useful for ^{14}C dating of many types of low-carbon environments (Gillespie 1990; Gillespie *et al.* 1991, 1992).

The AMS chronology of Lanyon and Wangrah Creek, together with detailed stratigraphic descriptions, demonstrate that aggradation during the Holocene was relatively continuous over several thousands of years, and at Wangrah Creek, the phases of aggradation were separated by shorter periods of gully erosion. The dominance of aggradation is in contrast to the K-cycle model of rapid valley aggradation during phases of hillslope instability separated by long, stable periods of pedogenesis (Butler 1959, 1967). The stratigraphy at Wangrah Creek suggests that valley aggradation was the result of efficient trapping of sediment in swampy valley floors under stable conditions, and that instability in the landscape was manifest in the periods of gully erosion (Prosser, Chappell & Gillespie, ms.). It is not possible to determine, from the Lanyon and Wangrah Creek sites alone, if aggradation was synchronous across the region, particularly as the sites are from quite different geomorphic positions. This question will have to await accurate ^{14}C dating of additional sites.

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