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Comparative AMS ^{14}C dating of plant macrofossils, beetles and pollen preparations from two late Pleistocene sites in southeastern Australia

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Introduction

In the Northern Hemisphere, late Quaternary chronologies are commonly constructed using AMS ^{14}C dated plant macrofossils because they are generally argued to provide the most reliable chronology (MacDonald et al. 1991; Törnqvist et al. 1992; Snyder et al. 1994; Birks and Birks 2000; Hatté and Jull 2007). Although terrestrial macrofossils are often relatively abundant and well preserved in a variety of site types, they are still potentially subject to a range of complications that include reworking, movement of dissolved organic carbon, contamination by modern carbon due to inappropriate storage and analysis, and potentially, measurement effects relating to small sample sizes (Wohlfarth et al. 1998; Turney et al. 2000; Nilsson et al. 2001; Oswald et al. 2005; Hatté and Jull 2007).

Insects, likewise, are common and often abundant in a range of sediment types (Elias 1994; Porch 2008). Examination of their utility as dating materials has focused on beetle sclerites (Elias and Toolin 1989; Elias et al. 1991; Cong et al. 1996; Hodgins et al. 2001; Walker et al. 2001; Hormes et al. 2004; Tripp et al. 2004) and chironomid head capsules (Jones et al. 1993; Wolfe et al. 2001; Fallu et al. 2004; Hormes et al. 2007). Results suggest that insect determinations may, in some cases, be younger than determinations on other fractions, including plant macrofossils (Hodgins et al. 2001; Walker et al. 2001), bulk sediments – gyttja (Fallu et al. 2004) and humic acid fractions (Wolfe et al. 2001). The results of Child and Werner (1999) comparing bulk sediment, chironomid/cladoceran and plant macrofossil ages indicated concordance of bulk sediment and invertebrate ages

with, conversely, younger macrofossil ages. In contrast, Tripp et al. (2004) report similar ages from both uncharred and charred plant macrofossils and beetle sclerites. In essence, there is a general assumption that insect sclerites may not be reliable for AMS radiocarbon dating, although this is poorly founded. Site-specific differences in insect dates and dates from other fractions suggest the observed variation may relate to the taphonomy of the various components of the sedimentary column rather than specific problems with any particular fraction.

Although the perceived advantages of dating pollen preparations (ubiquity and chemical stability), rather than bulk sediment organics, have been made explicit (Brown et al. 1989, 1992; Regnéll and Everitt 1996; Prior and Chester 2001; Vandergoes and Prior 2003), little consideration has been given to assessing the general suitability of pollen preparations in a variety of contexts. In Australia and New Zealand, there have been limited efforts to examine late Quaternary plant or insect macrofossils for dating potential. However, there has been a trend towards dating lake, swamp and playa sequences using pollen preparations or bulk sediment dates on relatively inorganic materials (e.g. Gillespie et al. 1991; Magee 1995; Chester and Prior 2004; Vandergoes et al. 2005; Cupper 2006; Kershaw et al. 2007; Newnham et al. 2007). In the latter case, the carbon being dated in organic poor sediments is likely to be effectively similar to a pollen preparation concentrate.

Almost all the studies comparing sediment or pollen and plant or insect macrofossil ages have been based on late glacial or Holocene material from extended lake and/or peat-bog sequences. In contrast, many important palaeoecological sites in the Australian region are near-surface exposures, characterised by slow rates of sediment accumulation, or relatively inorganic. Depositionally old sediments may be preserved in such contexts. Here we report on the results of a dating exercise in which subfossil seeds, beetle sclerites and 'pollen preparations' from two such sites, Spring Creek in western Victoria and Pulbeena Swamp in northwestern Tasmania, were dated. Results suggest that pollen preparations from these two sites may yield aberrantly young ages and that both beetle and plant macrofossils give more comparable and potentially more reliable results. Although sample sizes utilised here are much larger than usually available for most Quaternary sequences, they reflect the requirements of sampling for insect assemblages rather than material for radiocarbon dating. Plant and insect macrofossils are frequently preserved and often abundant in many late-Quaternary records from mesic Australia (Porch pers obs.) and may, in some cases, provide a more reliable approach for establishing robust radiocarbon-based chronologies.

Site descriptions and previous research

Spring Creek

Spring Creek, western Victoria, is a mega-fauna-bearing site, initially argued to provide evidence for late survival of a number of extinct taxa (Flannery and Gott 1984). Fossil bone has been recovered and still erodes from the edge of a thin exposure of heterogeneous gravelly silt. Flannery and Gott (1984:391) reported a single conventional radiocarbon determination of $19,800 \pm 390$ (Teledyne 1-11,018) on 'plant remains (leaf, seed and stem fragments)' from sediments associated with the extinct form of the macropod *Macropus giganteus*, and seven other extinct vertebrate taxa. Subsequently, four AMS radiocarbon determinations (White and Flannery 1995) were obtained on bone to test the concern that the original radiocarbon determination was not indicative of the age of megafauna – two determinations on two macropod bones: $27,268 \pm 330$ BP (NZA 3202) and $27,863 \pm 333$ BP (NZA 3201), and two further determinations on halves of a longitudinally sectioned humerus of an extinct mega-fauna taxon, *Palorchestes*, the marsupial tapir: $36,500 \pm 2400$ BP (NZA 3871) and $25,440 \pm 150$ BP (CAMS 9559/BETA 67231). Results were interpreted by White and Flannery

(1995:16) to indicate contamination of the bone samples, although the pair made no further attempt to date the depositional context of the fauna.

Pulbeena Swamp

Pulbeena Swamp, northwestern Tasmania, has been the subject of a number of palaeoecological research projects, including analysis of vertebrate fossils (Banks et al. 1976), pollen and chronology (Colhoun et al. 1982), ostracods (De Deckker 1982) and non-marine molluscs. More recently, it has become a focus of subfossil insect studies (Porch 2007). The swamp is characterised by inter-bedded peaty and marly sediments and generally very slow rates of sediment accumulation (<<10 cm/1000 years). Peats have been argued to be deposited during periods of low groundwater flux resulting from regionally reduced effective precipitation, and conversely, marls have been argued to represent periods of high groundwater flow (Colhoun et al. 1982). Several laterally continuous peat beds within the range of radiocarbon dating occur in the upper 1-1.5 m of the Pulbeena sequence.

Sample descriptions

Spring Creek SC97/5

A sample of approximately 9 kg of laminated, organic sandy silt derived from a short sequence of bedded silts immediately downstream from the concentration of bone. It represents an equivalent of the disturbed-bone-containing layers upstream, but is difficult, because of cover, to physically trace laterally: both layers at least rest upon Miocene marl and are overlain by the distinctive ferruginous gravel referred to above. The sample is 40-50 mm in thickness. Four samples taken from above SC97/5 are in total 160-180 mm in thickness, and another 150 mm of coarse relatively inorganic gravels were removed before beginning sampling. A further 250 mm of bedded organic silts underlie the sample.

Spring Creek SC97/6

A 5 cm thick, approximately 11 kg sample of heterogeneous gravelly, organic, clayey, sandy silts excavated upstream from the sampling locality of SC97/1-5. Here, there is no evidence of bedding; rather, a suggestion of mixing of several sediment types at some time in the past, although these sediments are capped with a distinctive ferruginous gravel. On top of the Tertiary marls lies a layer (50-200 mm) of silty and sandy gravel containing large rock clasts, mega-fauna bones, occasional wood and material from the marl below. Overlying this are blocky structured, gravelly, clayey, silty sands that contain occasional mega-fauna bone fragments. Interpretation of these deposits suggests they may represent several depositional and reworking episodes, almost certainly within a relatively short period, given the excellent preservation of most of the mega-fauna material before being covered by gravels and, later, Holocene alluvium.

Depositional environment of the Spring Creek samples

Based on the nature of the sediments and interpretation of the insect and plant macrofossil fauna, both Spring Creek samples were deposited at the head of a pool in a flowing creek, probably very similar to the arrangement that occurs at the site today. Fossil material in the deposit is almost certainly derived from the creek catchment above the waterfall. Significantly, the pollen and macrofossil floras and the insect assemblages, to be described elsewhere, are almost identical in both samples.

Pulbeena Swamp P3/D

Sample P3/D is a 13 kg sample of moderately humified, shelly peat from 104-107 cm below the present-day surface. The top of this peat layer elsewhere in the site has been dated previously. The resulting determination was $27,300 \pm 200$ (Beta-70065). Three further conventional determinations were obtained on the sample sequence referred to here. These dates, which are essentially identical because of the wide errors, range from $29,820 \pm 1350$ (GX-25690) at the top of the peat bed, to $30,400 \pm 1450$ (GX-25691) for the middle and $29,380 \pm 1280$ (GX-25692) for the base on the bed. There is no obvious evidence of physical disturbance of the sequence.

Depositional environment of the Pulbeena sample

The abundance of both aquatic and terrestrial shells in the Pulbeena sample, its organic matrix, the presence of sedge seeds and the remains of micro-sclerophyllous Myrtaceae (*Leptospermum* and/or *Melaleuca*) all imply deposition in a shallow swamp with flowing water. The snail assemblage is dominated by the small aquatic snail *Fluvidona* (Hydrobiidae), commonly found in springs and creeks. Terrestrial snails, especially the presence of *Victaphanta milligani*, indicate there was a vegetation canopy over the swamp surface.

Sample preparation

Plant and insect macrofossils were recovered by disaggregating approximately 10 kg of sediment in sodium pyrophosphate and washing the residues through a series of sieve screens; the Pulbeena Swamp sample was treated with hydrochloric acid before pyrophosphate to remove carbonate. Material for dating was picked from the 1 mm sieve, dried and stored in glass vials. Pollen samples measuring 5 cm^3 were processed using standard pollen preparation methods, although without acetolysis, which has the potential to introduce modern carbon. Samples were dated at ANSTO using conventional acid-base-acid pre-treatment, graphitisation and analysis procedures.

Results

All samples yielded sufficient carbon for AMS radiocarbon dating and the results are presented in Table 1. Because the majority of samples are from a context that is at the limit of the radiocarbon method and beyond the range of standard calibration curves, the results are presented in radiocarbon years BP. Two samples from Spring Creek gave results that were not different from background (OZD380 on beetle sclerites and OZD387 on *Pimelia* seeds). The other beetle-sclerites determination from Spring Creek yielded an age $>44,100$ BP. All other samples gave finite results.

Table 1. Results of comparative AMS ¹⁴C dating of seeds, insects and pollen from Spring Creek (Series 1 and 2) and Pulbeena Swamp (Series 3)

Lab. code	Material	Sample series and identity	pmc±1SD	¹⁴ C yr BP±1SD	δ(¹³ C) per mil
Spring Creek					
OZD379	<i>Pimelia sp.</i> seeds	1-SC97/6A	0.37 ± 0.16	44,900 ± 3,600	-26.86
OZD380	beetle sclerites	1-SC97/6B	0.10 ± 0.11	NDFB ¹	-23.6
OZD381	pollen preparation	1-SC97/6C	0.72 ± 0.12	39,600 ± 1,400	-27.4
OZD382	Asteraceae seeds	1-SC97/6D	0.45 ± 0.11	43,400 ± 2,100	-27.9
Pulbeena Swamp					
OZD383	Cyperaceae seeds	3-P3D/A	3.33 ± 0.11	27,350 ± 260	-26.5
OZD384	beetle sclerites	3-P3D/B	3.03 ± 0.16	28,100 ± 410	-26.0
OZD385	pollen preparation	3-P3D/C	5.05 ± 0.18	24,000 ± 300	-26.1

¹Not Distinguishable from Background (NDFB) – background for this sample is 43,400 years

²Not Distinguishable from Background (NDFB) – background for this sample is 46,300 years

Discussion

Reliability of macrofossil versus pollen preparations

The trend in the results of the ¹⁴C AMS determinations is the general concordance of the plant and beetle macrofossil determinations and the fact that pollen preparations gave finite or younger ages; in the case of sample Series 1 from Spring Creek (SC97/6A-D in Table 1), the large errors of the two plant-macrofossil samples mean, however, they overlap the pollen age at two standard deviations. Given that the two Spring Creek samples represent laterally equivalent layers and that the flora and fauna from the samples is very similar, the two series can be considered as dating the same unit. If taken together, there are two finite dates on seeds that are close to background (OZD379, OZD382), two dates not different from background (OZD380, OZD386), a single date >44,100 (OZD387) and two pollen dates markedly younger than background. It is therefore likely the two finite plant macrofossil ages from Spring Creek are minimum ages rather than reflecting the real age of the Spring Creek sequence. For the Pulbeena Swamp sequence, the difference in the macrofossil ages relative to the single pollen age is much clearer.

In Australia, the use of pollen preparations for dating late-Quaternary sequences often stems from the apparent lack of terrestrial macrofossils in sites, particularly during the last glacial. In some sites, this absence may, however, be more apparent than real, with few Australian researchers actively searching for macrofossils (see, however, Kershaw et al. 2007). The potential problems of dating pollen samples need to be assessed, and the data presented here points to one of them, contamination by modern or younger fine organic material. Another, contamination by derived older charcoal or uncharred organics, is more likely to be significant in lacustrine sites dominated by minerogenic sedimentation, especially during periods of instability (Gillespie et al. 1991).

In the Australian context, few pollen preparations derived from Quaternary sediments are composed exclusively or even principally of pollen. Much of the material remaining after physical and chemical pollen separation techniques consists of resistant plant fragments, including fine charcoal, even after extensive treatment. The Spring Creek samples are estimated to contain <20% pollen and Pulbeena Swamp <10%, the majority of the remaining material being fine,

uncarbonised plant matter, and to a lesser extent, opaque organics, possibly charcoal. For the determinations reported here, the pollen samples are biased towards fine insoluble organics other than pollen. If contamination problems by older organic material from the catchment, or younger penetrative root contamination are to be avoided, methods for the production of relatively pure pollen samples should be routinely employed (Long et al. 1992; Regnéll 1992; Richardson and Hall 1994; Regnéll and Everitt 1996; Prior 1998). Alternatively, more attention needs to be paid to sampling and processing for terrestrial macrofossils.

It is possible the finite ages for the Spring Creek pollen samples reflect contamination by modern and/or Holocene root penetration, a factor noted by Newham et al. (2007) to be a potential problem in bog contexts. A small amount of fine modern organic matter added to the Spring Creek samples would be enough to give a finite age. However, in the case of Pulbeena Swamp, a significantly greater amount of contamination would be required. The samples at both sites are derived from near-surface contexts, and in the case of Pulbeena Swamp, swamp forest occurred on the site before clearing in the late 19th century. Presumably, such contamination issues become less problematic in more rapidly accumulated sites and sites dominated by organic rather than minerogenic sedimentation, where dates on bulk organics or pollen are less likely to be influenced by the inclusion of younger carbon.

Utility of insect sclerites for AMS dating

The most interesting result from this study was the apparent consistency of the AMS ages of beetle sclerites in relation to the plant-macrofossil ages. The Spring Creek results were background (OZD380) and infinite (OZD387), in line with the postulated age of the Spring Creek sequence, noted above. The Pulbeena Swamp determination (OZD384) was completely consistent with the plant-macrofossil age from the same sample and the conventional peat age from the same layer (GX-25690 29,820 ± 1350 BP: Porch unpublished) – all dates overlap at two standard deviations.

Elsewhere, comparative AMS radiocarbon dating of insect sclerites has shown they can yield younger dates (than other materials), which have remained unexplained (Hodgins et al. 2001; Walker et al. 2001), or younger ages that have been assumed to be accurate relative to the older, contaminated, bulk sediment or humic fractions (Wolfe et al. 2001; Fallu et al. 2004), or in the case of the palaeosol dating of Hormes et al. (2004), reflect the extended period of palaeosol formation and accumulation of its constituent fractions over an extended period. Other studies have demonstrated that beetle sclerites yield ages that are comparable to ages from plant macrofossils, in line with the results reported here (Tripp et al. 2004). Similarly, there are conflicting results when comparing simple pre-treatments like ABA with more complex pre-treatments involving the isolation and purification of chitin or its derivative, chitosan. Finally, Tripp et al. (2004), however, reported no difference in ages derived from fractions that had only received a simple acid-wash pre-treatment and those that had been chemically purified to almost pure chitin.

There is clearly a need for more research into the utility of insect sclerites for AMS radiocarbon dating. This research should examine alternative pre-treatment methods, fossil insect taphonomy, whether taxonomic differences in ecology are related to differences in age, and whether these issues can explain differences between sites in the comparability of insect, plant and other determinations. These issues are important because direct dating of insect sclerites is an ideal way to demonstrate whether selected taxa are indigenous or introduced under contentious circumstances and to demonstrate the age of insect sclerites in sites with complex depositional histories.

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