

AMS RADIOCARBON DATING OF *RATTUS EXULANS* BONE FROM THE KOKOHUIA SITE (NEW ZEALAND)

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ABSTRACT

We AMS radiocarbon dated rat bones (Pacific rat (*Rattus exulans*)) from the archaeological site of Kokohuia, in Northland, New Zealand. An initial series were up to 1200 years too old, but the reason for the offset remained obscure. We considered dietary foodwebs, post-depositional contamination and taphonomy as possible explanations. None provided robust answers. Finally, we identified a laboratory-related error, in which a small amount of old carbon derived from humectants (glycerin) used to moisten ultrafilters prior to the extraction of bone collagen, had become incorporated in the dated collagen. The glycerin humectant was present in amounts averaging 30-40 μ g C per ultrafilter, which ordinarily is too small to significantly affect single dates obtained on bone collagen, but was significant in the case of small rat bones. Redating of bones from identical contexts produced a series of results which were within agreement with the age of the site as determined from other materials, including short-lived identified charcoal, and marine and estuarine shell. These results sound a warning for dating very small bone samples, and emphasise the need to consider all possible contaminants derived from laboratory preparation and chemical processing background. In addition, it emphasises the requirement for utilising standard samples of identical type (eg, bone, charcoal, wood etc) which range from young (1-2 half-lives) to background in age, as well as low and high sample mass, as QA laboratory checks on reproducibility (Bronk Ramsey *et al.*, in press).

INTRODUCTION

The radiocarbon dating of bones of the Pacific rat (*Rattus exulans*) has proven highly controversial in New Zealand. Bones of this species have been excavated from natural cave sites in New Zealand, where they were deposited by Laughing Owl (*Sceloglaux albifacies*) which predate upon them. Because this mammal is a human commensal, absolute dates of rat bone remains in sediments from the lower facies of these sites ought to yield information pertaining to the earliest human presence. An initial series of such dates produced ages spanning a period from European arrival back to around 2000 BP; over 1000 years earlier than current archaeological evidence for first human settlement (Anderson, 1991; Higham and Hogg, 1997; Higham *et al.*, 1999). With the exception of a disputed rat bone reportedly recovered from beneath Taupo Tephra (*c.* 1850 BP)(Holdaway, 1996; Anderson, ???). Yaldwyn, 2002) the hypothesis subsequently outlined by Holdaway (1996; 1999); that transient visitors brought rats which remained and multiplied, rests upon the reliability of Accelerator-based radiocarbon dates. This reliability has been questioned in a series of publications, some of which show dates of rat bone from several archaeological sites produce results which are clearly in error (Anderson, 1996; Smith and Anderson, 1998; Petchey and Higham, 2000; Higham and Petchey, 2000; Anderson and Higham, in press), with one exception; the site of Pauatahanui, which shows good

agreement between rat bone dates and dates from other materials (Beavan Athfield *et al.*, 1999). Explanations for the anomalous results range from laboratory error, to diet and contamination (Anderson, 1996; Hedges, 2000; Higham and Petchey, 2000).

In this paper, we present new rat bone data from Kokohuia, a site in northern New Zealand (Figure 1), which sheds further light on the question of the reliability of rat bone AMS radiocarbon determinations.

THE SITE OF KOKOHUIA

The Kokohuia site is located on a ridge overlooking the Hokianga Harbour in northern New Zealand (Figure 1). Taylor (1995) has excavated five areas of the site. The material analysed in this paper comes from Layers 2, 3, 4 and 5, in Area 3 (a 10 x 6 m excavation) which produced evidence of food preparation, cooking and consumption, and was occupied late in a period designated the “Archaic phase” of New Zealand culture (Taylor 1995:12). One large midden deposit excavated in Area 3 on the ridge was chosen for radiocarbon analysis. A total of thirty-seven radiocarbon determinations have been obtained from this area of the site (Schmidt, 2000; Figure 3), comprising eighteen short-lived charcoal and nineteen shell determinations. Results indicated occupation between *c.* 1400 and 1600 AD, with a 130-230 year (at 95% probability) span of occupation (Figure 7b).

Figure 1. Location of the Kokohuia (O06/317) archaeological site on Waiarohia Point, Hokianga Harbour, Northland, New Zealand (after Schmidt, 2000).

Figure 2. Stratigraphy of the Lower Midden in Area 3 of the Kokohuia site. Samples dated in this paper come from these archaeological contexts (after Schmidt, 2000).

METHOD

We obtained samples of rat bone from Layers 2 to 5 in Area 3 at Kokohuia, which were housed at the Museum of New Zealand, Te Papa Tongarewa (Figure 2).

The bones were initially pretreated to remove surficial soil and detritus with a scalpel and an aluminium oxide shotblaster. They were then powdered using a mortar and pestle, and weighed. Each bone was pretreated using the semi-automated Oxford continuous flow bone pre-treatment system (Law and Hedges, 1989; Bronk Ramsey *et al.*, in press). This involves sequential decalcification using 2% v/v HCl, dehumification using 0.5M NaOH, then acidification using 2% v/v HCl. Each step is interspersed with distilled water rinses. Each sample was then gelatinised in weakly acidic water (pH3) at 75°C in an incubator for 20 hours, and the supernatant recovered using an EziFilter™. The supernatant was ultrafiltered using a Millipore™ ‘Ultrafree’ 30 kD MWCO ultrafilter. The >30 kD fraction was lyophilised and retained for AMS dating (Table 1). The <30kD fraction was similarly treated but yielded no product.

Figure 3: Calibrated radiocarbon determinations obtained from the Kokohuia archaeological site (data from Schmidt, 2000).

Between 0.3—5.0 mg of ultrafiltered gelatin was weighed into tin capsules and combusted in a continuous flow IR mass spectrometer, comprising a Roboprep CHN elemental analyser interfaced with a Europe 20-20 mass spectrometer. This enables the measurement of dual carbon and nitrogen isotopes, C:N ratios, %C and %N (van Klinken, 1999; Bronk Ramsey *et al.*, in press). The samples which were of sufficient yield were then graphitised by catalytic reduction of CO₂ onto iron in an excess hydrogen atmosphere. Smaller samples (<1.6—0.5 mg C) were AMS dated using the Oxford CO₂ gas ion source. The Oxford AMS radiocarbon method and instrumentation is reported by Bronk Ramsey and Hedges (1999), Bronk Ramsey *et al.* (2000) and Bronk Ramsey *et al.* (in press).

Table 1: AMS determinations of ultrafiltered gelatin. %C yield is yield of C after combustion. Gel yield is the yield of gelatin in mg after ultrafiltration and lyophilization. $\delta^{13}\text{C}$ and $\delta^{15}\text{N}$ are reported with reference to VPDB and AIR respectively and expressed in ‰. Measurement error for C isotope ratios is ± 0.2 ‰ and for N isotope ratios is ± 0.3 ‰.

RESULTS

Conventional radiocarbon ages BP and isotopic and analytical data are reported in Table 1. There is a wide range in rat bone gelatin ages which is not apparent in the results of other radiocarbon samples. The Pacific rat is an opportunistic omnivore and Beavan Athfield (this volume) has suggested that due to the potential for fluctuating ¹⁴C levels caused by dietary variation, it is crucial to compare AMS dates of rat with other dated samples from contiguous levels to determine reliability. The radiocarbon dates previously obtained from the site have already been mentioned and range between *c.* 1400 to 1600 AD (Figure 2). All but one of the rat bone determinations were significantly older than this.

Four possible reasons were identified for this unexpected distribution. First, there may be a dietary effect, with rats obtaining depleted carbon through foodwebs not in equilibrium with atmospheric carbon levels. This explanation has already been put forward by Beavan Athfield and Sparks (2001) to explain older than expected radiocarbon dates obtained at the Pleasant River site, in Otago (Smith and Anderson, 1998). Second, there might be contaminants within the site environs which have affected the bones dated. The contaminant must naturally be composed of carbon whose ¹⁴C content is significantly lower than that of the indigenous sample carbon, and is only partially or unremoved by radiocarbon pre-treatment methods. Third, there might be a taphonomic problem at the site, where radiocarbon dates are correct, but the rat bones were redeposited and mixed within a much later archaeological assemblage. Fourth, there may be a laboratory error such that old carbon, derived from routine laboratory preparation, has affected the dates by making them too old.

We initially hoped that the dating of rat bone from Kokohuia would enable us to design a dating protocol that would narrow down the most likely cause, or causes for similar anomalous rat bone determinations.

DIETARY EFFECTS

The ultrafiltered gelatin $\delta^{13}\text{C}$ values obtained (Table 1) appeared to reflect a terrestrial diet with no indication of significant amounts of marine carbon being consumed, despite the extensive archaeological evidence of an abundance of marine resources consumed by human occupants and the location of the site so near to the coast. The $\delta^{15}\text{N}$ values for the rats ranged from 10–13‰. The lower range is indicative of an organism obtaining the majority of its dietary protein from terrestrial meat sources. Unfortunately, a dual stable isotope approach has limitations in determining the uptake of aquatic or freshwater sourced carbon amongst omnivores, since the isotopic range, particularly for nitrogen, overlaps significantly with that of high terrestrial protein consumers. It is apparent when one considers the range in stable isotopes of a limited number of other organisms from the same site (Table 2; Figure 4), that the rat bones are terrestrial in $\delta^{13}\text{C}$ but enriched in $\delta^{15}\text{N}$ which could imply a partial aquatic or freshwater isotopic signature (Ambrose, 1991; Lanting and van der Plicht, 1998; Cook *et al.*, 2002). This possibility has been explored by Beavan-Athfield and Sparks (2001) who found a *c.* 250 yr offset in AMS dated marshland fowl bones from Pleasant River Mouth, with no associated marine-like $\delta^{13}\text{C}$ value. This was interpreted as the result of fowl consuming aquatic plants which, during photosynthesis, may have taken up dissolved organic carbon from the estuarine reservoir. Extending this scenario to Kokohuia, it might be hypothesised that rat bone could be offset from ‘true’ age with no corresponding enrichment in $\delta^{13}\text{C}$, depending on the food sources taken up. In the case of the Pleasant River Mouth fowl, the offset between the bird bone dates and the occupation date of the site was just over half of the amount of the marine reservoir, but Beavan Athfield and Sparks (2001) suggested that more significant and as yet undetected offsets could exist within the foodchain. In the case of Kokohuia, the size of the offset of the dates from their expected age, if it is caused by diet, is significantly larger than at Pleasant River and as in that example a dietary explanation requires a source of carbon significantly depleted in ^{14}C to be identified.

Such offsets in radiocarbon ages, other than those associated with marine reservoirs, can be produced by three principal natural sources of depleted carbon delivered to foodwebs; volcanic sources of fossil CO_2 delivered from magmatic vents (Bruns *et al.*, 1980; Rubin *et al.*, 1987; Pasquier-Cardin *et al.*, 1999; Beavan-Athfield *et al.*, 2001), limestone or hard water sources (Marchenko *et al.*, 1988), and soil organic carbon/humic carbon sources (Schnitzer and Khan, 1972; Bailey *et al.*, 1973, 1975; Kigoshi *et al.*, 1980; Head *et al.*, 1989).

Although Kokohuia is surrounded mainly by consolidated dune sands of Holocene and Pleistocene age, the bedrock of the area is dominated by limestones and other calcareous sediments belonging to the Motatau and Mangakahia Group formations. To the east of the site, the Tangihua Volcanics (Upper Cretaceous in age) dominate (Kear and Hay, 1961; Thompson, 1961). Streams feeding the estuary cut through these calcareous formations. Limestone or hard water effects are therefore of potential significance. One means to test for a hard water effect is to compare radiocarbon determinations of estuarine shell carbonates from archaeological contexts which are in close association with terrestrial materials such as charcoal or wood, of short-lived duration. Schmidt (2000) undertook such an analysis and identified no offsets between marine shell dates and identified charcoal dates after correction for the established New Zealand marine reservoir effect (Higham and Hogg, 1995). This suggests that there is no significant hard water effect in the Hokianga Harbour. Active volcanic sources which could introduce fossil CO_2 are also absent from this region and can be ruled out in the case of Kokohuia.

Explaining the Kokohuia rat bone dates through the mechanism of old carbon being delivered through dietary pathways, then, is not straightforward. It remains difficult to identify a source of depleted ^{14}C derived from food chains which could produce a shift in radiocarbon age of the amount required. In the absence of data to the contrary, we rejected a dietary explanation as the sole cause of the offsets in the radiocarbon dates. However, given Beavan Athfield and Sparks' (2001) results from Pleasant River, in which offsets in ^{14}C from 'true age' for certain aquatic feeding organisms are masked by stable isotope values which are largely terrestrial in value, it is difficult to confidently reject all possibility of a dietary effect.

TAPHONOMIC DISTURBANCE

Invoking a taphonomic reason for older than expected rat bones in this site is considered low in probability (MT to add to)...

POST-DEPOSITIONAL CONTAMINATION:

A range of QA protocols are routinely followed at Oxford to monitor the effectiveness of pretreatments and ensure a sample is reliable for dating. Several analytical parameters are applied to bones for dating. Collagen was within the range 3-9 wt.% showing that between 16-45% of the original gelatin survived. At Oxford, we use a limit of 1% collagen as the threshold for acceptance or failure, and anything below this is rarely, if ever, dated. Generally speaking the % collagen at Kokohuia is lower than sites to the south, as expected given the sub-tropical and high rainfall environment in the Far North of New Zealand (Petchey, 1995). C:N atomic ratios were within acceptable ranges at Oxford (2.9–3.5), though at least two were high compared with our routine experience. In one instance, a sample (OxA-10823) was dated with an acceptable C:N ratio and gave an anomalously old radiocarbon determination (1600 BP). The C:N ratio for this, and for the other ultrafiltrated gelatin samples were within the range of acceptance applied at Oxford (2.9-3.5). If contamination is the cause of the excessively old determination for sample OxA-10841 then it implies within the site with a contaminant of excessive age with a similar C:N to bone collagen. This is considered unlikely. There were two notable exceptions within the sample C:N ratios of the dated batch; two samples (whose run numbers in the laboratory are P13148 and P13149) yielded C:Ns of 4.0 and 4.1 respectively. We AMS dated these samples despite these aberrant C:N's to determine the potential for erroneous determinations under conditions of poor C:Ns. The results obtained were significantly older than expected (c. 1.7 and 1.9 ka). The actual results are not published here because the dates were failed in the laboratory due to poor C:Ns and therefore not given OxA-s. In terms of contamination, this implies the addition of carbon with non-collagen C:N ratios.

LABORATORY CONTAMINATION:

Following the initial results from Kokohuia, we became aware of a problem with the ultra-filtration stage of the ORAU pretreatment, which is designed to remove small molecular weight contaminants (<30 kD in size). Subsequent research found that the ultrafilters used contain a humectant (glycerin) which ensures the filters do not dry out prior to use. The manufacturers

recommend a simple cleanup using distilled water to remove the humectant. The ORAU pre-clean was much more rigorous, but we found a small amount of humectant remains on the filters that could be passed on to the sample gelatin. This humectant is old in age (>35 ka BP) and averages 30-40 μ g remaining per ultrafilter after cleaning, although later analyses imply some variability around this range. It was determined that this extractable carbon is generally insignificant in its effect on very old bones and also for bones of more recent age whose collagen yield is higher (>25-30 mg collagen). However, we found that bones which are close to modern in age, with low amounts of remaining collagen, can, produce ages too old by up to 3-4 centuries. In Figure 5a, the relationship between radiocarbon age and ultrafiltered gelatin (mg) is shown, illustrating the significance of the glycerin contamination.

We have since developed a new protocol for cleaning the filters which overcomes this problem, resulting in reproducible dates and isotopes on bones of known-age (Bronk Ramsey *et al.*, in press). It is worthwhile stressing that the ultrafiltration method has been published and extensively used without obvious problems emerging (Brown *et al.*, 1988), but for small yielding gelatin samples such as rat bones or poorly preserved bones, the humectant must be removed to ensure reliability.

REANALYSIS OF THE RAT BONES

The laboratory-introduced contamination made evaluation of the effect of diet or site contamination problematic. We therefore sought additional rat bone samples from the Kokohuia site for AMS dating. Since little remained of the previous samples, additional material from the same bone clusters was sampled. There is no guarantee that the second series of bones comprise elements from the same individual rat that was dated in the first series, but they are from the same context in the site and therefore subject to the same model of site stratification outlined above.

The samples were gelatinised in the same manner as the first set, but the ultrafiltration step was eliminated due to the small size of the second series of bones. The results are shown in Table 3 and Figure 6. The absence of a relationship between inverse collagen yield and radiocarbon age demonstrates the lack of a significant contaminant effect (Figure 5b).

The new results can be evaluated along with the other non-bone determinations using a model (Figure 7a) which incorporates prior archaeological information within a formalised Bayesian framework. The basis of this approach is outlined in a number of publications to which the reader is referred (Buck *et al.*, 1996; 2000). We used BCal (bcal.shef.ac.uk) and applied the outlier detection method employed within it to test whether any of the rat bone determinations could be considered outliers or not. Prior probabilities of 0.10 were ascribed to each model parameter (^{14}C likelihood), in other words each was given a 1 in 10 chance of being an outlier according to prior supposition. Posterior probabilities were commonly less than 0.10, with some exceptions (Figure 8). The most significant outlier was OxA-12629, which yielded a posterior probability of 0.65. Generally, the outlier analysis suggests the AMS bone results are not significantly different from the ^{14}C activity of the contemporary atmosphere reflected by other radiocarbon dated samples, or, alternatively, that the carbon derived from radiocarbon reservoirs in isotopic disequilibrium with the atmosphere is being taken up by rats in negligible amounts. We ran the calibration model four times to check reproducibility. In one instance, a high dependency factor was noted, this due to the tight

stratigraphic constraints introduced in the model for the site, but with this exception, the models produced reproducible data.

Table 2: Stable isotopes measured for fauna from Kokohuia. Kuri (Maori dog) and kaka bone are from excavated contexts at the Kokohuia site from locations as outlined in column 2. Stable isotope results were from marine organisms collected live from Kokohuia Beach. Flesh was washed in dilute acid to remove carbonates and then lyophilised and combusted for mass spectrometric analysis.

Table 3: AMS dates of rat bones pretreated to filtered gelatin rather than ultrafiltered. See text for details.

Table 4: Highest Posterior Density regions (expressed at 0.95 prob.) for three of the parameters of interest at the Kokohuia site as simulated using BCal. For illustration of the parameters for the model, the reader is referred to Figure 7.

CONCLUSIONS:

Radiocarbon dates of rat bone gelatin from the Kokohuia site initially yielded a range at odds with the previously established chronology. A series of thirty-eight conventional radiocarbon measurements on identified, shortlived charcoal and marine and estuarine species supports a chronology for the entire sequence in the excavated area of the site, of 130-230 years (95% prob.). An initial set of rat bones was obtained within identical stratigraphic layers, therefore we assumed that they should date to the fourteenth century AD. This first set of AMS spanned about 1200 years, and, with the exception of a single result, all were consistently older. Attempts to correlate the range of analytical data obtained with the radiocarbon data showed that no single pair of variables provided a robust fit. Aberrant C:N ratios of two determinations which were excessively old supported the interpretation that there was a contaminant within this area of the site which had a non-collagenous C:N ratio and was old. Other dates, with acceptable C:N ratios, also produced ages which were all uniformly too old to varying degrees. We attempted to correlate possible dietary influences with older radiocarbon-depleted sources of carbon but were unable to identify a link. We reject this as an explanation in this instance whilst acknowledging that it almost certainly applies in other examples.

Redating of all samples was undertaken once it was known that ultrafilters used to isolate >30kD MW components from bone gelatin were in fact adding a small amount of depleted carbon to the bone hydrolysate. Since the bone gelatin extractable from single rat elements is usually very small, the influence of the introduced dead carbon is high. Samples of rat bone from the site were therefore reanalysed without the ultrafiltration step. The results showed good agreement with the previously established chronology.

The experience of dating small rat bones from this site has demonstrated the importance of considering carefully all sources of laboratory-derived contamination, particularly when dating very small bones, since the influence of even small contaminants is magnified. In the case of Kokohuia, the contaminants were of background age and this affected the radiocarbon ages considerably because of the low pre-treatment yield of collagen. Analytical data associated with the affected results was ambiguous because in some cases the CN ratios were within acceptable range. There may be lessons here for others dating very small bone elements. It is critical to evaluate all possible error sources, from within and outside the radiocarbon facility. At ORAU we have introduced a range of new chemistry QA standards and we routinely analyse the samples at low and high mass amounts in order to track small contaminant contributions derived from sample chemistry preparation.

Like Kokohuia, dates of rat bones from other archaeological sites (eg, Shag River Mouth (Anderson, 1996), Pleasant River Mouth (Smith and Anderson, 1998)) have shown initially old and apparently erroneous results. Despite a significant number of reanalyses, reproducing the ages derived during the initial series of AMS dating from Shag River Mouth has not been possible. Instead, work has shown that the old dates appear not to be the result of site contamination or pre-treatment, or poor preservation, although there appears to be some contaminating carbon at the Shag Mouth site (both of a non-collagenous, and a proteinaceous but non-collagenous origin) which is removed by gelatinisation of the bones. None of the recently redated rat bones are older than 1000 BP, even accounting for partial reservoir effects indicated by carbon and nitrogen isotope results. Further evaluation of dietary foodwebs is clearly required to determine the extent to which we can in fact rely on rat radiocarbon dates. It is crucial, therefore, that redating of comparable specimens from other natural sites dated by Holdaway (1996) be undertaken to strengthen the hypotheses put forward concerning rat arrival dates (Holdaway, 1999), or to reject them.

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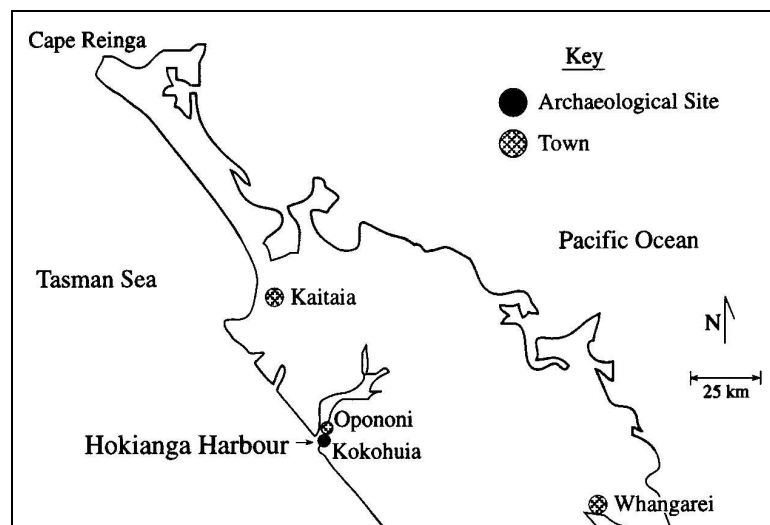
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FIGURES and TABLES



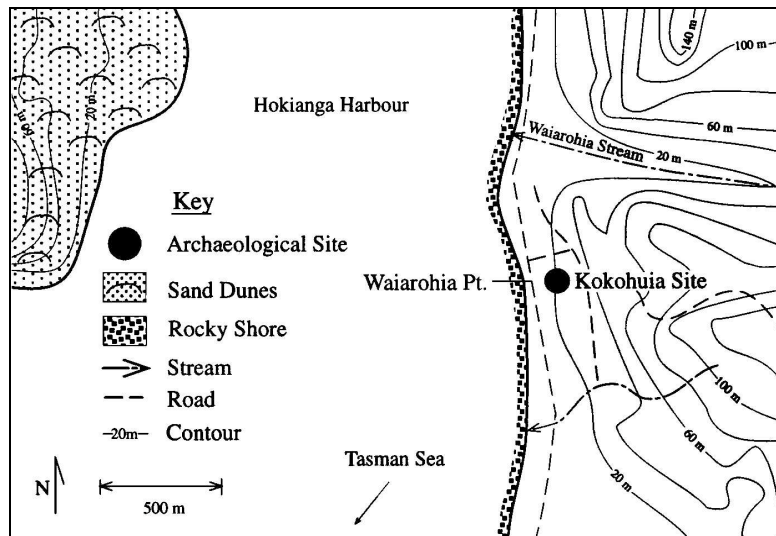


Figure 1. Location of the Kokohuia (O06/317) archaeological site on Waiarohia Point, Hokianga Harbour, Northland, New Zealand.

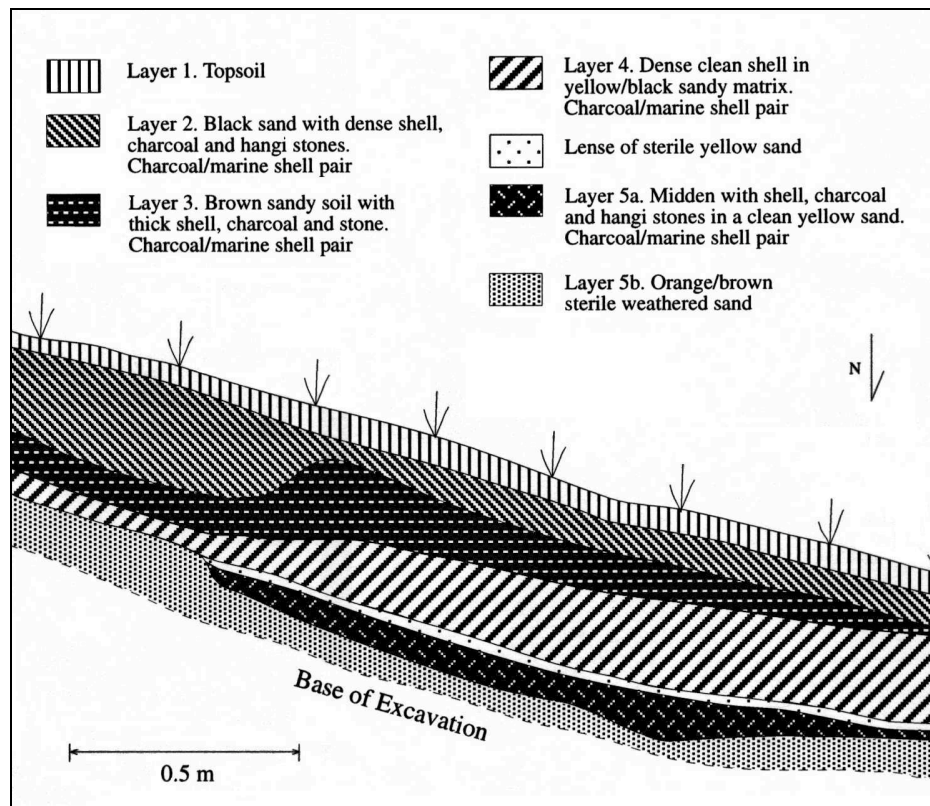


Figure 2. Stratigraphy of the Lower Midden in Area 3 of the Kokohuia (O06/317) archaeological site, Hokianga Harbour, Northland, New Zealand.

Figure 3: Calibrated radiocarbon determinations obtained from the Kokohuia archaeological site (data from Schmidt, 2000).

OxA	Sample reference	Lyr	Material dated	¹⁴ C age BP	Gel. Yield (mg)	% coll	C:N	δ ¹³ C	δ ¹⁵ N	%C yield
10823	J12N/IV/S193/MON2/7	IV	1 R.fem, 1 R. mand., 1 vert.	977 ± 32	3.61	5.9	3.5	-19.0	12.4	43.2
10836	I12N/III/S122/MON2/27	III	1 mandible	885 ± 50	3.1	6.5	3.5	-20.2	13.6	38.5
10837	I11N/IV/S181/MON2/3 9	IV	L+R pelvic fragments	970 ± 50	1.21	3.4	-20.0	12.2	41.6	
10824	I11S/III1/S147/MON2/8 0	III	mandible	705 ± 32	5.07	8.3	3.0	-19.6	12.1	44.2
10841	I11N/Va/S218/MON2/9 1	Va	L. tibia	1605 ± 70	0.72	3.3	3.1	-19.9	12.7	41.3
10774	I10W/IV/S175/MON2/9 3	IV	1 R femur	465 ± 45	2.65	4.4	3.4	-19.4	9.9	41.1

Table 1: AMS determinations of ultrafiltered gelatin. %C yield is yield of C after combustion. Gel yield is the yield of gelatin in mg after ultrafiltration and lyophilization. δ¹³C and δ¹⁵N are reported with reference to VPDB and AIR respectively and expressed in ‰. Measurement error for C isotope ratios is ±0.2 ‰ and for N isotope ratios is ±0.3 ‰.

Code	Provenance	Identification	δ ¹⁵ N	δ ¹³ C
L1	Kokohuia beach	Limpet flesh	9.55	-13.5
C1	Kokohuia beach	Chiton flesh	6.98	-13.7
AL 852	Layer II	Kuri: Right radius, distal portion	14.9	-11.25
AL 853	E17W Layer III	Kuri: Right scapula	13.2	-15.0
AL 854	G13+ F13NZ, Layer IV	Kuri: Left scapula	12.1	-15.75
AL 855	G13 + F12NW Layer V	Kuri: Occiput nuchal crest	15.1	-10.98

AL 856	F10W Layer II	Kaka: Distal right tibiotarsus	3.56	-20.94
AL 857	H11E Layer IIIB	Kaka: Right coracoid	2.35	-20.56
AL 858	E11 Layer IV2	Kaka: Anterior section of sternum	2.42	-18.63
AL 859	E10N Layer V	Kaka: Right coracoid	1.74	-20.43

Table 2: Stable isotopes measured for fauna from Kokohuia. Kuri (Maori dog) and kaka bone are from excavated contexts at the Kokohuia site from locations as outlined in column 2. Stable isotope results were from marine organisms collected live from Kokohuia Beach by FP. Flesh was washed in dilute acid to remove carbonates and then lyophilised and combusted for mass spectrometric analysis.

OxA	Sample reference	Lyr	Material dated	¹⁴ C age BP	Gel. yield (mg)	% coll	C:N	δ ¹³ C	δ ¹⁵ N	%C yield
12394	20/I13E/II-1	II	Mandible	417 ± 38	0.98	2.4	3.4	-19.5	7.1	40.3
12628	28/I13N/II-1	II		398 ± 27	7.5	10.2	3.4	-20.0	13.3	45.8
12629	29/I10N/II 2	II		495 ± 27	15.2	8.6	3.4	-18.6	13.7	42.8
12395	74/I12N/II 1	II		480 ± 45	4.1	12.6	3.5	-19.8	12.8	27.4
12630	76/I11S/III	III		434 ± 27	3.3	14.6	3.4	-19.1	15.5	45.5
12344	H9E/V/S1104	V	R. tibia	483 ± 23	11.6	11.1	3.2	-19.0	6.3	41.4

Table 3: AMS dates of rat bones pretreated to filtered gelatin rather than ultrafiltered. See text for details.

Parameter	HPD region (95%)
μ_1	1420 to 1475 AD (1458 modal value)
μ_4	1575 to 1670 AD (1620 modal value)
$\mu_1 - \mu_4$	121 to 230 (95%) (180 modal value)

Table 4: Highest Posterior Density regions (expressed at 0.95 prob.) for three of the parameters of interest at the Kokohuia site as simulated using BCal. For illustration of the parameters for the model, the reader is referred to Figure 7.

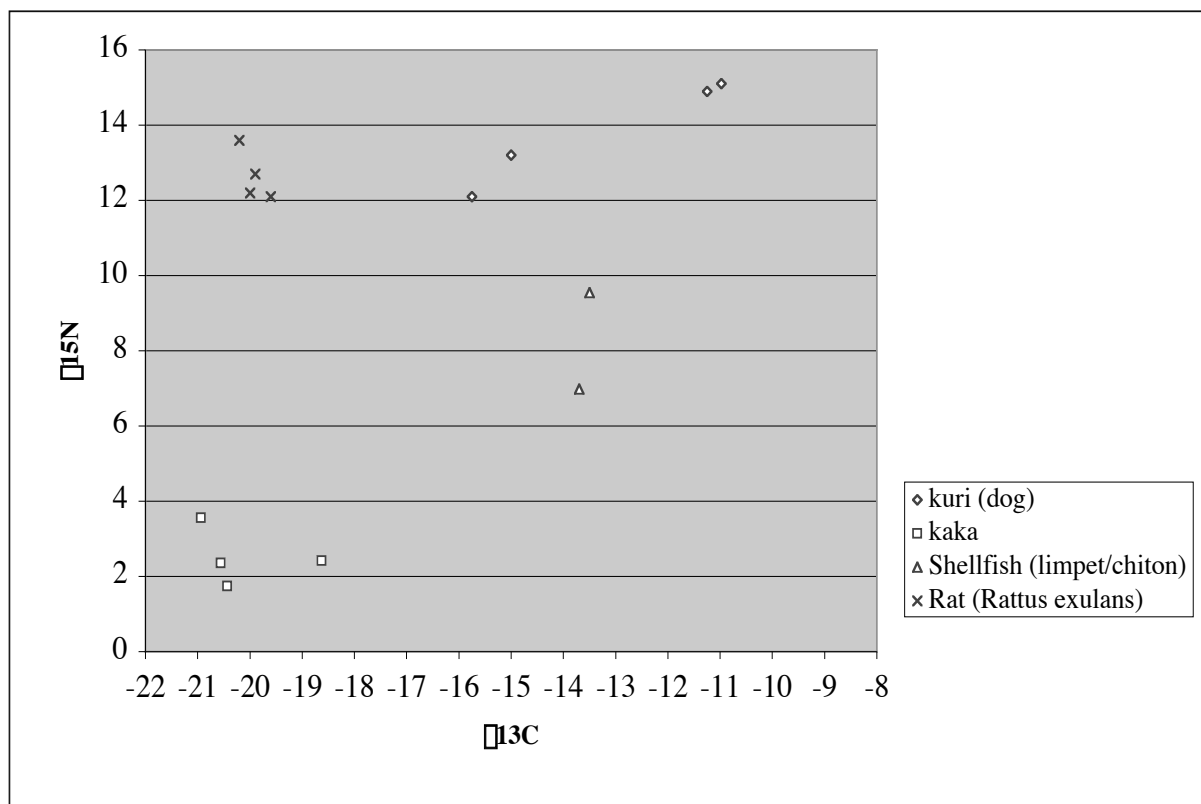
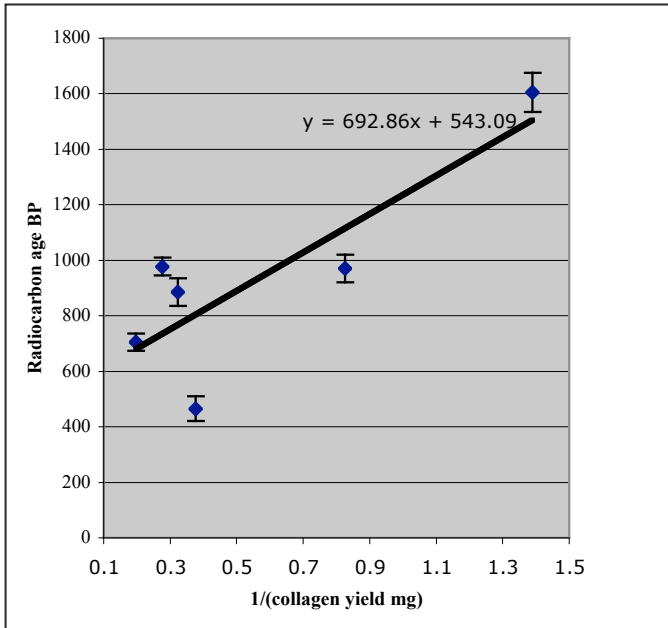
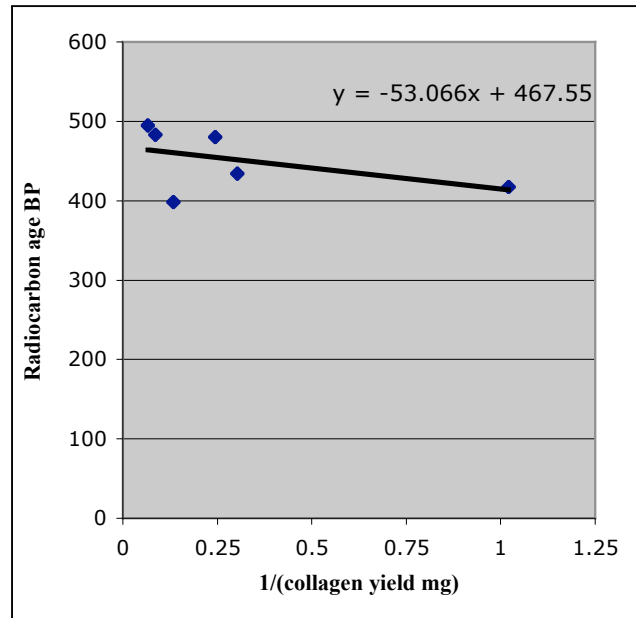


Figure 4: Nitrogen and carbon stable isotopes from Kokohuia fauna. The data is all collected from prehistoric bones, with the exception of the shellfish which were collected live. See text for measurement details. Kuri (dog)-omnivorous scavengers, kaka- terrestrial herbivorous bird eating occasional insects, shellfish-marine/estuarine feeding gastropoda, rat-omnivorous scavenger.



a)



b)

Figure 5:

Comparison of radiocarbon age against the inverse collagen yield (mg) for both series of AMS dates of rat bone from Kokohuia.

- a) Radiocarbon ages of the first series of ultrafiltered samples plotted against the inverse collagen yield (mg). The linear fitted line intercepts the y axis at 543 years, while the gradient line represents a c. 700 year age offset as a function of collagen yield and humectant contamination derived from ultrafilters.
- b) Radiocarbon age BP plotted against the inverse collagen yield for samples listed in Table 2 (gelatin).

Atmospheric data from Stuiver et al. (1998); OxCal v3.6 Bronk Ramsey (2000); cub r:4 sd:12 prob usp[chron]

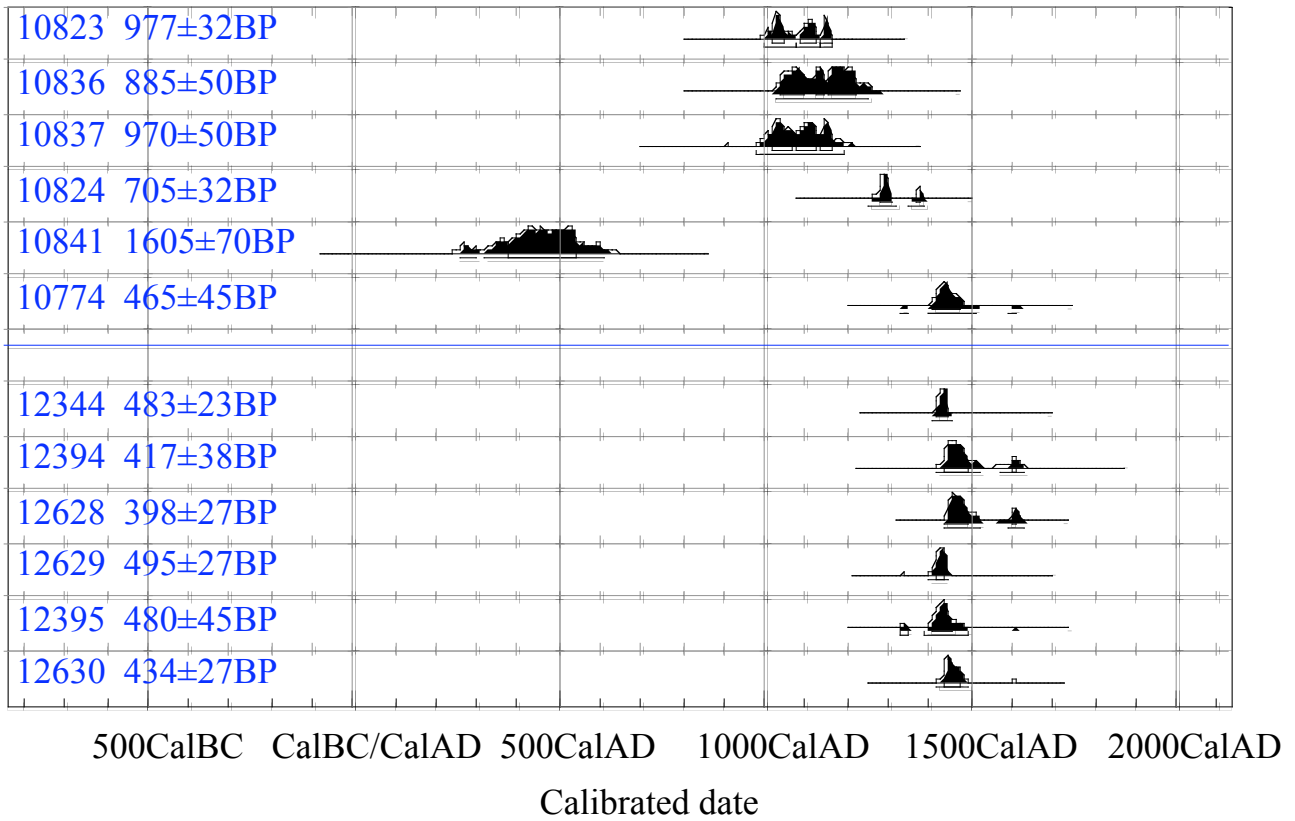


Figure 6: OxA-numbers and calibrated radiocarbon ages for rat bone gelatin AMS dates shown in Tables 1 (upper calibrated likelihoods) & 2 (lower).

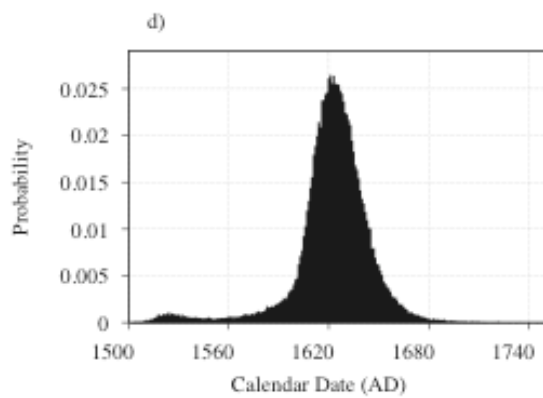
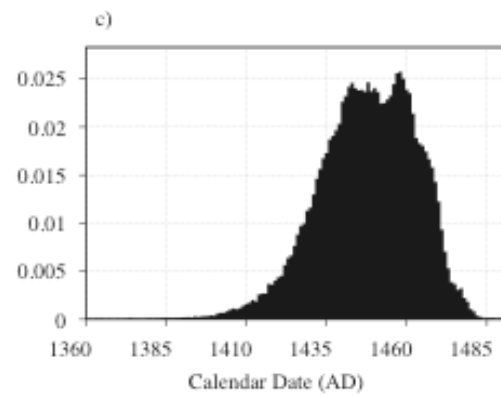
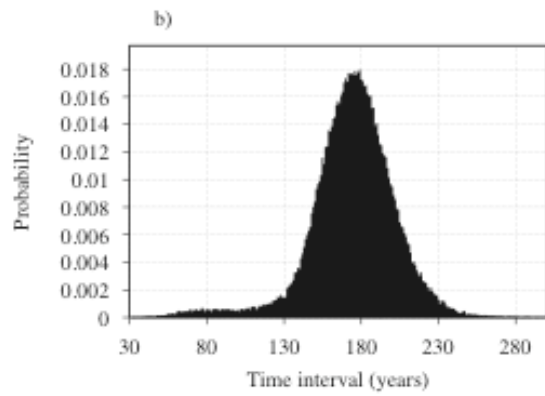
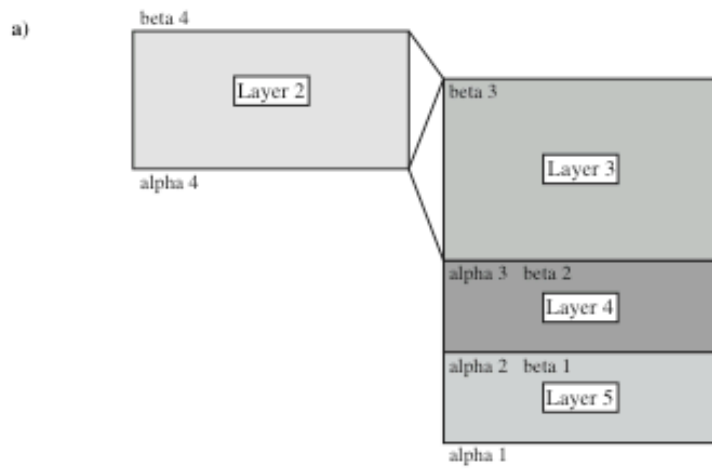


Figure 7: Bayesian model and posterior probability density plots for three parameters used in the calibration model for Kokohuia as simulated in BCal. a) Kokohuia model; mathematical symbols are used to describe the stratigraphic phases and boundaries at the site. \square_n and \square_n represent the beginning and ending dates of phase n .; b) $\square_1 - \square_4$; representing the span of occupation of the site in years. c) \square_1 ; representing the period immediately prior to occupation at Kokohuia. d) \square_4 represents the period immediately after cessation of human occupation. Highest posterior density (HPD) regions are given in Table 4. Modal values correspond with the highest values for the posterior probability.

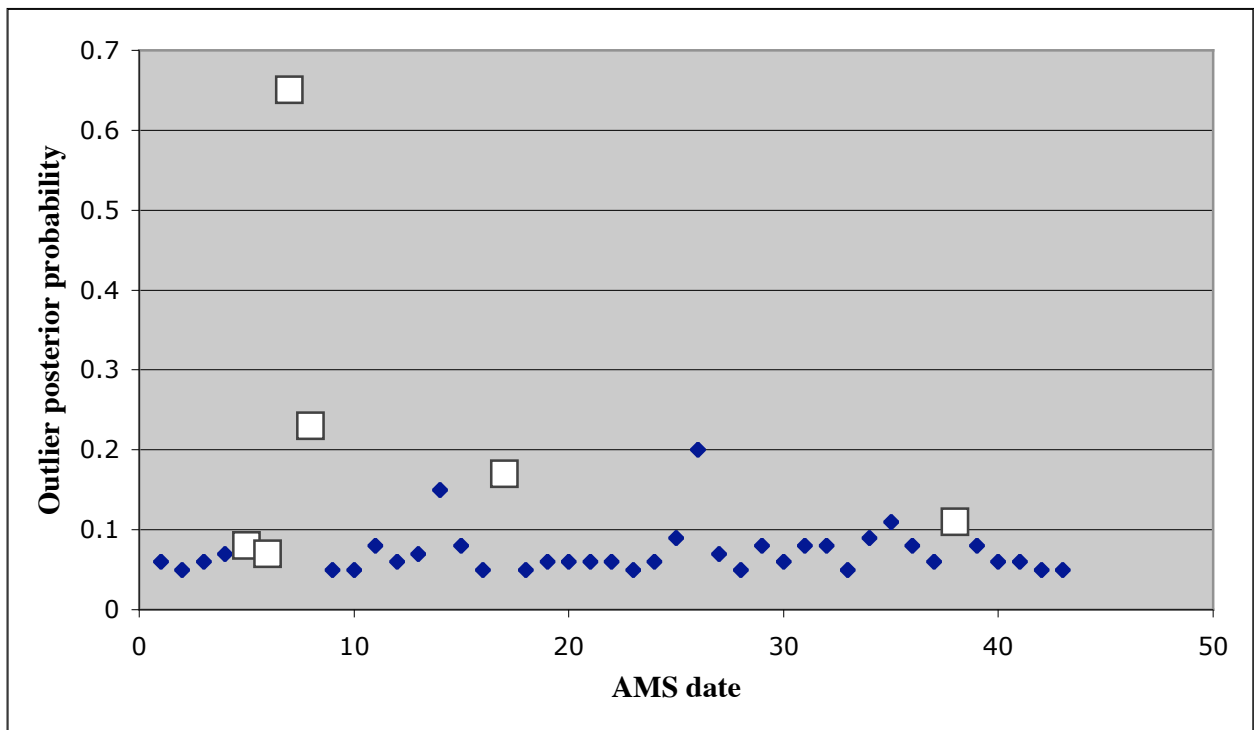


Figure 8: Results of outlier analysis for all radiocarbon determinations from Kokohuia as determined using BCal. A prior probability of 10% was ascribed for all likelihoods. Squares are rat bone posterior

probabilities and the remainder are of other sample types. One result (OxA-12629) indicates a posterior probability of 0.65 of it being an outlier.