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Chemical Pretreatment and Radiocarbon Dating of Samples from the Prehistoric Site of Killermont #2, Mackenzie Basin, New Zealand

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ABSTRACT

We present the first radiocarbon results from the prehistoric moa butchery camp of Killermont #2 (H39/19) in the Mackenzie Basin, South Canterbury. Killermont is the first moa hunting site to be excavated and dated in this region. Identified charcoal from one area has been analysed for post-depositional contamination. Chemical fractions of charcoal have been extracted and radiocarbon dated at the Radiocarbon Dating Laboratory, University of Waikato. We compare the results with the date of an untreated sample to assess the extent of post-depositional contamination. A radiocarbon date of the root material within the sample indicates it is a contaminant, but amongst the chemically extracted fractions, no statistically significant contamination is found. Other radiocarbon dates from different areas of the site suggest that occupation was brief and restricted to the fourteenth century AD. The dates obtained are very similar to those from the recently dated coastal Archaic sites of Otago such as Shag Mouth, Papatowai, Pleasant River and Warrington.

Keywords: KILLERMONT, MOA HUNTING, RADIOCARBON DATING, ARCHAIC, CHEMICAL, PRETREATMENT, HUMIC ACIDS.

INTRODUCTION

Since the development of the radiocarbon dating technique in the late 1940s, both archaeologists and chemists have been confronted by the problem of sample contamination. Many physical and chemical methods have been developed to remove the various contaminating substances affecting samples during their post-depositional history. Routine radiocarbon dating by the two laboratories in New Zealand has usually incorporated standard pretreatment techniques for removing contamination. Many New Zealand archaeologists have assumed that the influence of contamination upon radiocarbon samples is less important than it is in contexts with more extensive chronologies. This is because of the comparatively recent arrival of humans (*c.* 700 yr) and the fact that contamination becomes more significant for older radiocarbon samples. This assumption may have had validity in the past few decades, but the development of optimised ^{14}C measuring equipment has

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enabled increased dating precision, requiring the validation of radiocarbon dating accuracy. In addition, the methods of chemical pretreatment have become more sophisticated (Hedges 1994).

In this paper, we assess the accuracy of radiocarbon measurements of samples from the Killermont #2 site[3] in the Mackenzie Basin, South Canterbury, by examining the extent of post-depositional sample contamination in the charcoal remains from the site. We have isolated certain chemical fractions from the charcoal which may contain carbon contaminants derived from decayed plant and organic matter. We dated the extracted fractions to consider the extent of contamination in them as part of a larger project examining post-depositional contamination in different archaeological dating materials in New Zealand (Higham 1993). By comparing an untreated potential sample with chemically treated fractions, the magnitude of error may be considered. If the dates of sample and contaminant are close, the associated error will be insignificant. If there is a major difference, however, the dating error will be increased. Where different extracted fractions give statistically identical results to the untreated material, it may be concluded that no significant contamination has occurred.

KILLERMONT #2 SITE

The Killermont #2 site is located on the banks of the Ahuriri river in the Mackenzie Basin, South Canterbury (Figs 1, 2). Little is known of the prehistoric sequence in this region. In 1992, the site was excavated by one of us (RMcG-W), who found evidence of moa butchery and processing. Three areas of the site were identified (KLM areas A, B and C) consisting of large ovens, represented by hāngi stones in a stained charcoal matrix, with silcrete blades, moa bones and artefacts in association (Fig. 3). This is the first site excavated in the Mackenzie Basin which contains evidence for moa hunting. Thus, as one of a handful of major moa butchery and processing sites excavated in the interior of the southern South Island, it is important both archaeologically and chronologically (McGovern-Wilson 1993).

MATERIALS AND METHODS

PHYSICAL AND CHEMICAL PRETREATMENTS

Analysis of the sections and stratigraphy of the site revealed no apparent evidence of leaching. However, this has not been confirmed by a pedologist. Rainfall in the Mackenzie Basin is low (according to Ryan [1987], 526 mm per annum on average from 1951–1980 at Tara Hills station, Omarama); therefore leaching is likely to be minimal compared to sites west of the Southern Alps. The soils on the fluvial terraces (and adjacent fan deposits) in the area of study are developed typically in relatively thick silt or sand-rich loess (aeolian) deposits approximately 0.6–1.2 m deep, overlying coarse gravelly or bouldery alluvium. The soils are likely to be members of the Curraghmore series (previously mapped in the Grampians set) (McIntosh *et al.* 1990) and are provisionally classed in the New Zealand Soil

[3]New Zealand Archaeological Association metric Site Record number H39/19 (grid reference H39/593 283).

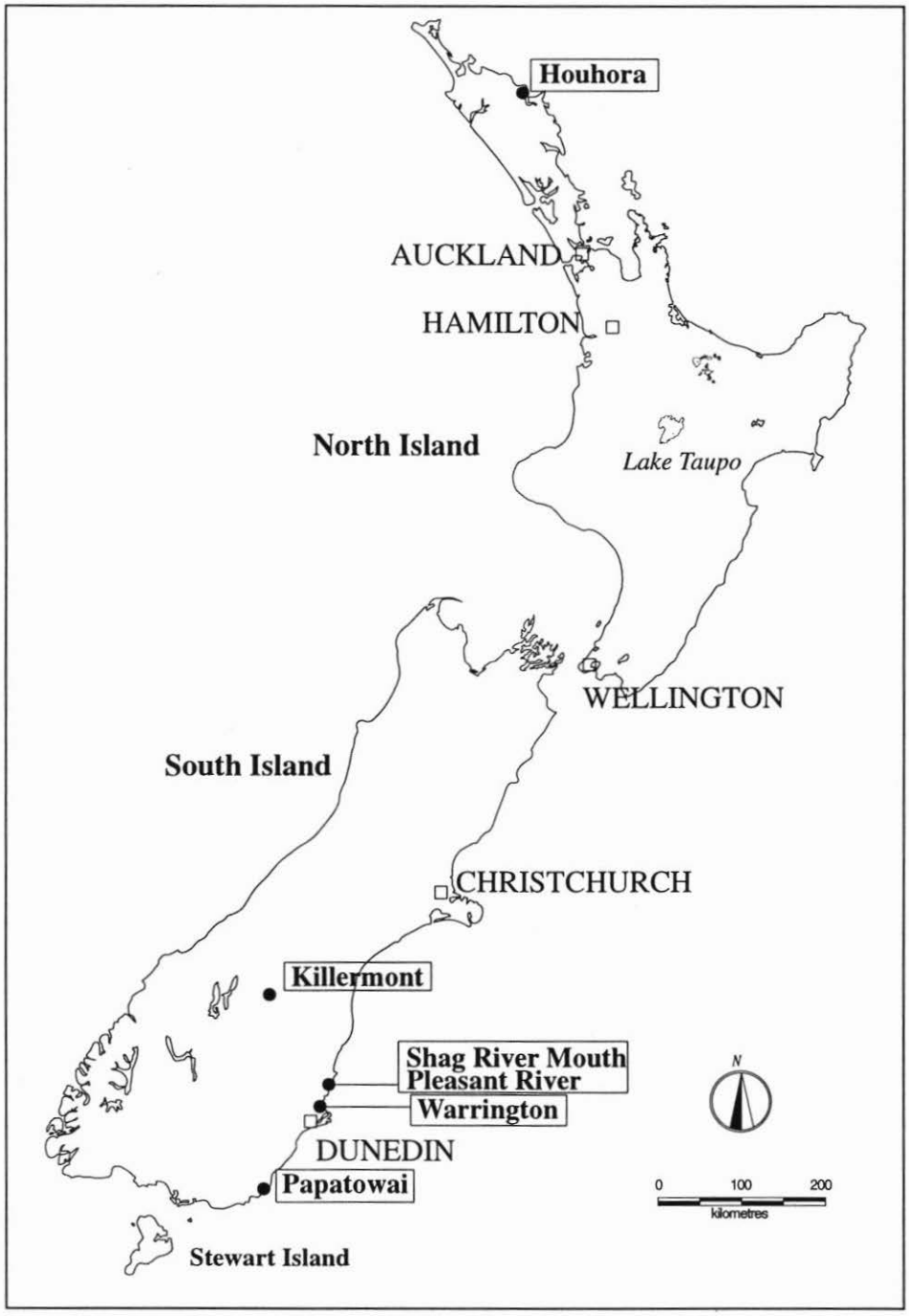

Figure 1: Locality map for Killermont #2 site. Archaeological sites mentioned in the text are represented by small black circles.

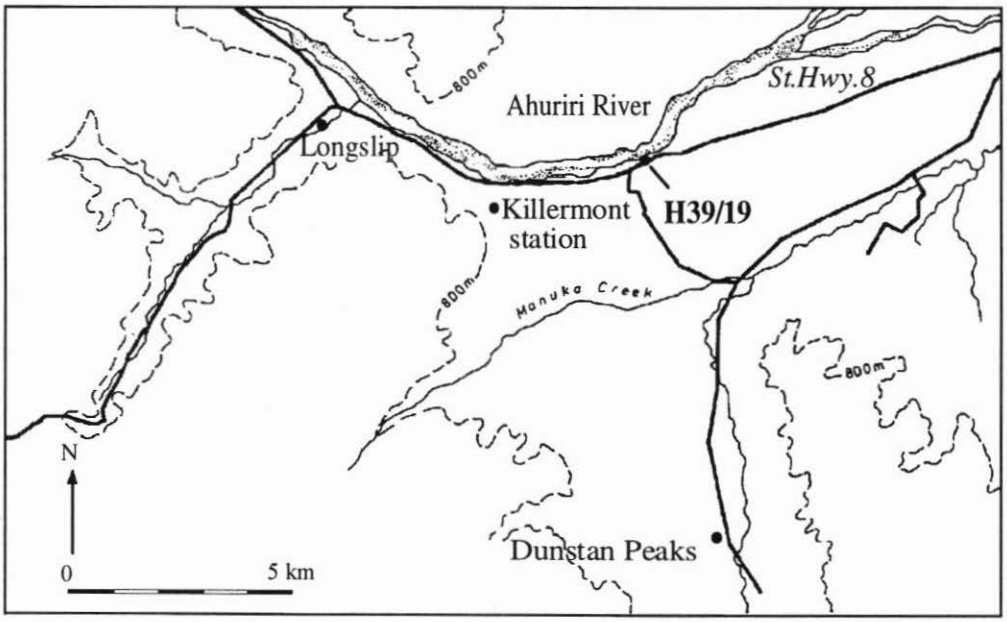

Figure 2: Location of the site of Killermont #2 (H39/19 Grid reference H39/ 593 283), Mackenzie Basin, New Zealand.

Classification (Hewitt 1992) as Typic Argillic Pallic Soils (approximately equivalent to Haplustalfs in Soil Taxonomy [Soil Survey Staff 1996]).

Profiles on the terraces are generalised as follows: 0.2 m-thick greyish-brown (2.5 Y 5/2 moist) A horizon, silt textured, moderately developed polyhedral structure; 0.3 m-thick pale olive (near 5Y 6/4 moist) Bt horizon, silt-loam textured (more clay than A horizon), weak prismatic structure; 0.7 m-thick pale olive (5Y 6/3 moist) to pale yellow (5Y 7/3 dry) BC horizon grading to Cu horizon, loamy-sand textured, non-calcareous; on boulders and gravelly alluvium (Fig. 4). There was evidence of extensive root penetration into the cultural layer that was identified and much of the charcoal excavated contained rootlets.

Trotter (1968), McFadgen (1982), Law (1984), Anderson (1989), Anderson and McGovern-Wilson (1990), Caughley (1988), Anderson (1991) and McFadgen *et al.* (1994) have cautioned against dating wood or wood charcoal whose species composition is unknown, or which contains material derived from long-lived trees. All of the charcoal remains from the site were identified and indicate that the palaeoenvironment was dominated by shrub species. Wallace (pers. comm. 1993[4]) has suggested on the basis of the charcoal remains that "the ecological picture presented in this data reveals a pure shrubland association". *Olearia* sp., *Hebe* sp., *Archeria traversii*, *Coprosma* sp., *Pittosporum* sp.,

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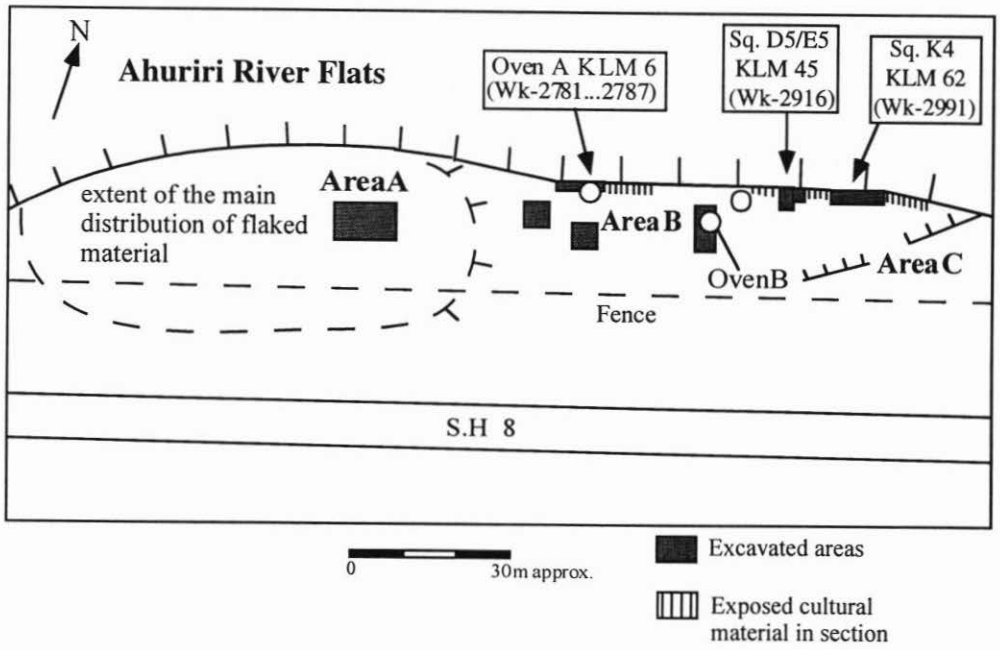

Figure 3: Areas excavated at the site of Killermont #2, Mackenzie Basin, New Zealand showing location of samples obtained for radiocarbon dating.

Myrsine australis, *Discaria toumatou* and one other unknown shrub species were identified. According to McFadgen *et al.* (1994), the remains are well suited for dating because they are from short-lived species and thus are more likely to date the prehistoric event closely. One possible source of error is the low rainfall which characterises the Mackenzie Basin. Wood may survive for longer on the ground than it does elsewhere because of the drier conditions and therefore may have an inbuilt age before use in prehistory. We plan to investigate this further by dating samples of moa bone gelatin from the site as a check on inbuilt age.

Previous research

Archaeological materials may be affected in a variety of ways by natural processes within their depositional environment (Schiffer 1987). There are two major contaminants present in soils which may affect radiocarbon dating of organic samples from archaeological sites: humic acids and fulvic acids. Both are similar organic compounds derived from the decayed remains of plants in the surface layers of the soil. Their presence in archaeological charcoals may constitute error of unknown magnitude. Humus substances within the soil have been classified according to the ease with which they can be removed from soils using alkaline solutions (Head 1987). Humic acid is defined as the fraction extracted by alkaline solution which becomes insoluble after acidification (Head 1987: 144). Fulvic acids are soluble in both acid and alkaline solutions (Head 1987). The residue soluble and insoluble in alkaline

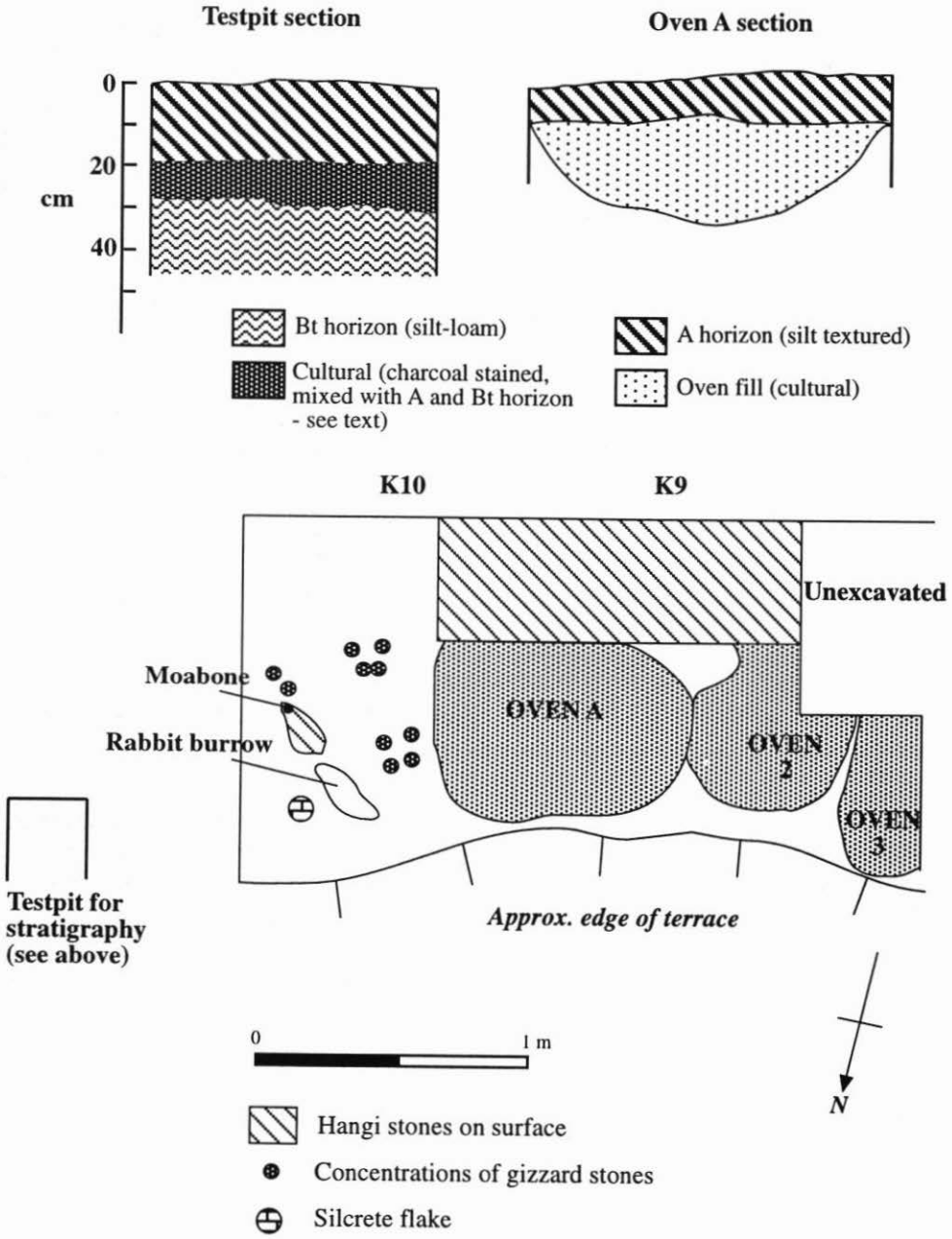

Figure 4: The excavation plan and stratigraphy of squares K10 and K9, Area B, excavated at the site of Killermont #2, Mackenzie Basin, New Zealand. The charcoal used in the pretreatment experiments described in this paper came from Oven A.

solutions has been termed humin. This is usually the fraction targeted for radiocarbon dating.

The presence and effects of humic acids in carbonaceous samples in New Zealand has been investigated by researchers including Bailey and Lee (1972), Bailey *et al.* (1973), Bailey *et al.* (1975), Goh (1978), Goh and Molloy (1972, 1979), Goh *et al.* (1978), Hammond *et al.* (1991) and Gillespie *et al.* (1992). Most of this research concerned problems in dating soil and peat samples, but charcoals were also investigated and much of the work assumes relevance to archaeological dating. Goh and Molloy (1979), for instance, analysed a series of charcoal samples from archaeological and geological sites and from commercial laboratory charcoals, to estimate the humic and fulvic acid fraction. Fourteen archaeological samples, mainly from Canterbury, were amongst those analysed. Charcoals may be especially prone to humic contamination because of their absorbent properties. Goh and Molloy (1979) estimated total oxidisable carbon for each sample, then extracted humic and fulvic acids. Amongst the archaeological charcoals, the total oxidisable carbon averaged 27% of the sample. Goh and Molloy (1979) found that amongst these charcoals, the majority of the total oxidisable carbon in each sample was non-extractable, or 'humin' carbon (mean = 75%) with the remainder comprising a variable size and proportion of the extractable humic and fulvic acid fractions.

Bailey and Lee (1972) dated the humic acid and humin fractions derived from NaOH treatment from a series of seven New Zealand charcoals ranging in age from 1 ka to Rotoehu Ash age (*c.* 64 ka, Lowe and Hogg 1995) and compared them to dates of untreated charcoal. They found that two of seven samples yielded ages for the humics which varied significantly from the other dated samples. Two of the charcoal samples dated were <10 ka and neither produced variable ages.

Bailey *et al.* (1975) found that, in general, the pretreatment of charcoal using NaOH did not result in significant changes in the ages of dated charcoals. They compared the ^{14}C ages of humic acids and residues with untreated charcoals and found that although in some instances the humic acid extractive did produce an earlier result, it did not affect the ^{14}C measurement of the residue in a significant way. This is probably because of the fractional size of the extracted humic acids.

Goh *et al.* (1978) examined the ^{14}C ages of peats from Timaru Downs and reported significant differences between fractions extracted from peat samples using a range of different pretreatment techniques. The ages of the two untreated peat samples examined were *c.* 9.5 ka and *c.* 34 ka, and both ages became older as the rigour of the pretreatments applied to them increased. The younger peat changed in age by 490 ^{14}C yr and the older by >15,300 ^{14}C yr after extensive pretreatment.

Hammond *et al.* (1991) found at the site of Graham's Terrace, in North Westland, that the maximum age of dated soil and peat samples was associated with the most rigorous pretreatment method and they suggested therefore that untreated ^{14}C ages were probably anomalously young. Isochronic tephra layers were used in the analysis of the ^{14}C ages, including the Kawakawa Tephra (*c.* 22,590 BP, Wilson *et al.* 1988). The erroneous ^{14}C determinations were influenced by the high rainfall and leaching combined with the Gley Podzol soil horizons at the site where young organics were washed down the profile. Hammond *et al.* (1991) recommended that caution must be applied to dating soil, peat and pollen samples from this type of environment using radiocarbon, unless rigorous pretreatments were undertaken. Gillespie *et al.* (1992) reached similar conclusions.

Methods

A charcoal sample (*c.* 5,600 g in size) from Area B (Oven A, squares K9/K10; Fig. 4) at the Killermont site was used in a series of pretreatment experiments designed to test the extent to which post-depositional contaminants affected radiocarbon analyses. The chemical steps followed in this experiment are shown in a flow diagram (Fig. 5). The sample was mixed before analysis to ensure that it was homogeneous. An untreated sub-sample (Wk-2782) was retained for dating to serve as a reference for comparison with the treated samples. Radiocarbon samples can be contaminated both chemically and physically. This sample was physically contaminated by extensive rootlet penetration. The removal and quantification of this contaminating fraction was both problematic and time consuming. To attempt to quantify the rootlets, a sub-sample of charcoal (55 g) was removed. The sample was examined rigorously for root hairs under a microscope after sieving in three sieves measuring 63, 250 and 1000 μm. Certain portions were subjected to flotation but this proved only moderately successful. Tweezers were used to remove the majority of root hairs within the sample. From the 55 g analysed in this manner, 0.55 g of root hairs were removed. Because some of the rootlets were minute in size, it is likely that this is an underestimation of the total rootlet contaminant. We estimate, therefore, that it comprised between 0.8 and 1.2% by weight of the total charcoal sample. We calculated total carbon content by combustion and found that the charcoals dated from Killermont had a similar carbon content to the rootlets (*c.* 38% for the rootlets and *c.* 38–41% for the charcoals). The rootlets are thus minimal in terms of their fractional size and therefore are probably not a significant source of error in an untreated sample. The charcoal sample rigorously screened for rootlets (Wk-2783) was dated as the "physically pretreated fraction".

The remaining bulk charcoal sample was pretreated by the removal of obvious adhering rootlets and crushing with a mortar and pestle, further removal of rootlets, followed by oven drying at 90°C prior to further chemical pretreatment.

We used the acid-base-acid method (ABA) to treat the Killermont charcoal. We heated the samples in 10% w/v HCl at 70°C for approximately one hour and then rinsed them to neutral pH with distilled water. We retained a portion of the acid insoluble residue for radiocarbon assay (Wk-2791). After drying, we boiled the sample in a 5% w/v NaOH solution for approximately one hour and rinsed it again. The NaOH treatment produces two fractions, base soluble and insoluble. The former (humic acid) was retained for dating by acidification and rinsed and dried (Wk-2785). The base insoluble fraction was also acidified because the NaOH used in the pretreatment absorbs CO_2 from the surrounding air (Goh and Molloy 1972: 575). The final acid wash ensures that the majority of this contamination is removed. The insoluble fraction usually contains the sample minus the contaminant and is often the most reliable datable component. This residue was retained for dating (Wk-2784).

A portion of the remaining base insoluble fraction was retained and split into two fractions: one for a repeat and identical ABA pretreatment (with 5% w/v NaOH) and one for pretreatment with 70% conc. HNO_3. A second base soluble fraction (Wk-2787) and base insoluble fraction (Wk-2786) were retained from these pretreatments. This repeat base pretreatment is similar to the method used by Kigoshi *et al.* (1980) who separated up to three humic acid fractions from samples of clay soil and peat collected from soils in Japan. They identified contamination due to leaching of young organics down the profile above a non-permeable layer, because some of the extracted humic acids yielded divergent ages from one another, and from humin.

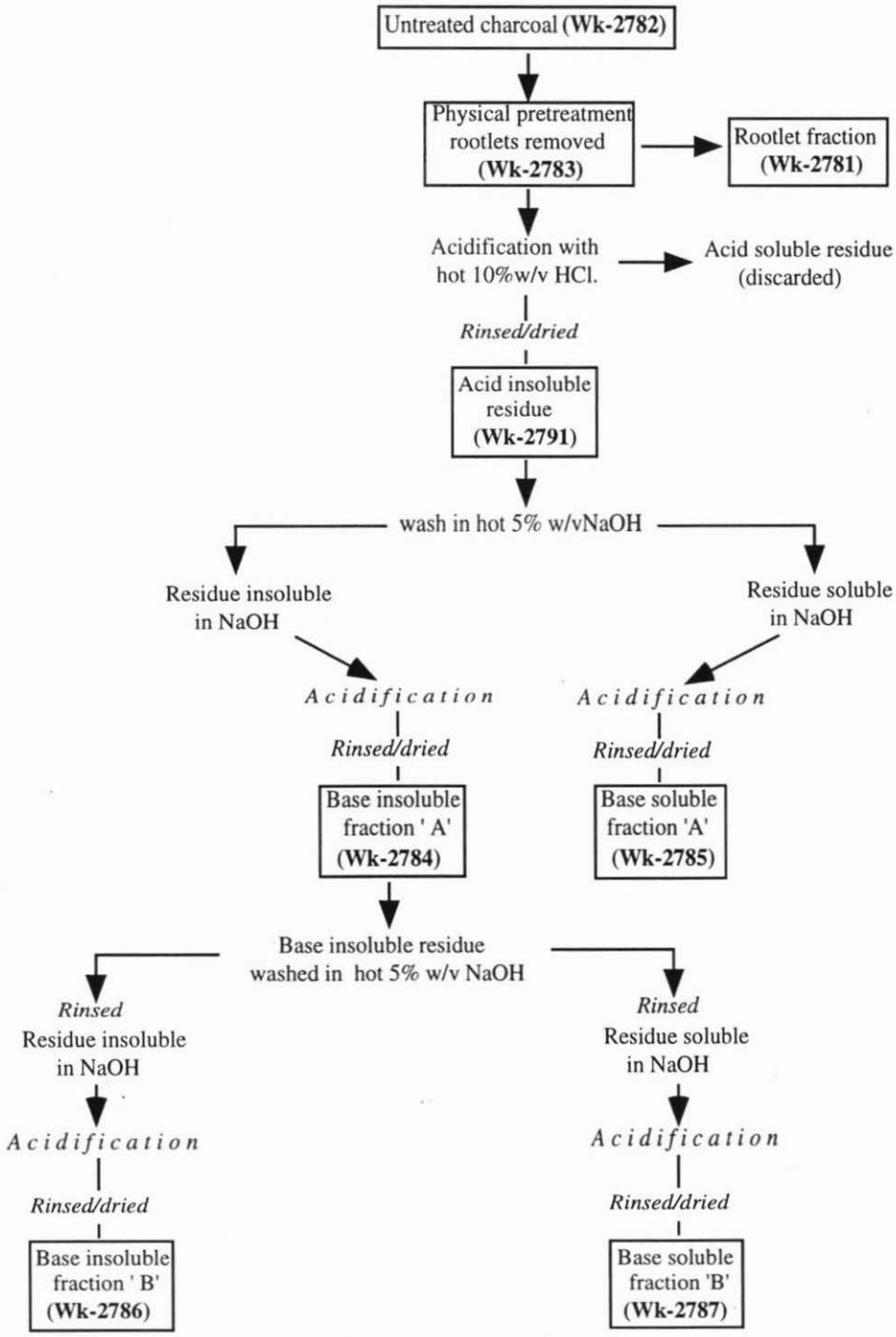

Figure 5: Flow diagram showing the pretreatment chemistry applied to the charcoal remains from Killermont.

RADIOCARBON ASSAY

The radiocarbon determinations from the pretreated fractions were dated at the Radiocarbon Dating Laboratory at the University of Waikato. Details of the Liquid Scintillation Counting (LSC) method employed are described elsewhere (Hogg *et al.* 1987; Higham 1993; Higham and Hogg 1997). All dates presented here are conventional radiocarbon ages BP, calculated according to the conventions given in Stuiver and Polach (1977).

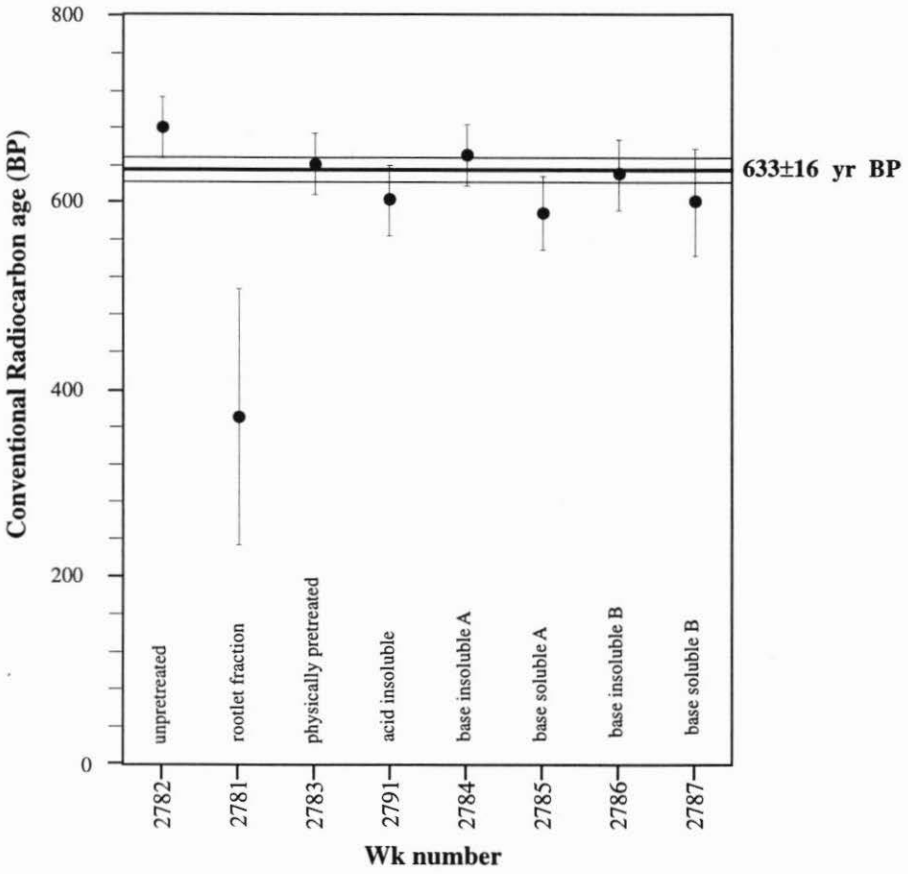

Figure 6: Radiocarbon determinations of the pretreated Killermont charcoal showing pooled mean value ± one standard deviation.

RESULTS AND DISCUSSION

The radiocarbon dates for the treated and untreated fractions are shown in Table 1 and Figure 6. With the exception of the rootlet fraction, all results were statistically indistinguishable from one another using the Case II method described by Ward and Wilson (1978) (error weighted mean ($A'(p)$) is 633±16 BP (T'=3.98; $\chi^2_{6:0.05}$=12.59). There are no statistical grounds for considering the dates as representing anything outside a normal radiocarbon counting distribution. It is concluded that, with the exception of the rootlet fraction (Wk-2781), there is no significant contamination present within the matrix at the Killermont site. Again, the rootlets are considered minimal in terms of their effect because of their small fractional size. The sample pretreated with 70% conc. HNO_3 was not dated in the light of the results accumulated for the existing chemical pretreatments.

We radiocarbon dated other ovens at the Killermont site to consider contemporaneity and ascertain whether there might have been multiple periods of occupation. We analysed a sample from square D5/E5 (Wk-2916; Sample KLM 45) in Area C, which yielded a conventional radiocarbon age of 660±45 BP. A sample from an oven in Area C (Wk-2991; Sample KLM 62) gave a measurement of 630±45 BP. Both samples were analysed without rigorous chemical pretreatment in the light of the experiments described above. Both were physically pretreated and acid washed. The pooled mean date ($A'(p)$) for these samples and Wk-2791 (the acid insoluble fraction) from sample KLM 6 is 628±25 BP (T'=1.00; $\chi^2_{2:0.05}$=5.99). We calibrated these three determinations and the pooled mean, using the high precision curve of Stuiver and Becker (1993) with a -27 yr correction for the Southern hemisphere offset incorporated (McCormac *et al.*, n.d.)(Table 2). We applied the formula $(\sigma^2_{age} + \sigma^2_{cal\ curve})^{0.5}$ to increase the standard deviation for each ^{14}C age prior to calibration, to account for the standard errors inherent in the calibration curve ($\sigma_{cal\ curve}$ = ±16 ^{14}C yr at 640 BP; Stuiver and Becker 1993)(Ward and Wilson 1978). The calibrated result for the error weighted mean suggests an occupation date for the Killermont site within the fourteenth century AD (AD 1325–1353, 1359–1367 and 1388–1419).

The radiocarbon results presented here are the first for prehistoric Maori occupation sites in the Mackenzie Basin. Radiocarbon dates from other Archaic sites in the Otago region, which reveal a similar material culture and subsistence strategy based upon big game and in particular remains of now extinct moa (Dinornithiformes), mirror those from Killermont. The radiocarbon series from this site is indistinguishable statistically from some of the well dated early southern Archaic sites. Recent radiocarbon dating at Shag River Mouth, for instance, has yielded a mean age based upon thirteen identified charcoal dates of 620±13 BP (Anderson *et al.* 1996). This result is confirmed by dates of other sample types. Re-excavation and dating at the Papatowai site in South Otago yielded an occupation of a similar antiquity, with a mean of 631±32 BP, although this is based on fewer dates (Anderson and Smith 1992). Smith (pers. comm. 1995[5]) and Higham (1993, 1994) have obtained similar dates from early contexts at the Pleasant River site. At Warrington, basal contexts similarly imply settlement by 600 BP (Higham 1993). The difficulty in differentiating more accurately between the ages of these sites occurs because of the age

[5] Dr I.W.G. Smith, Department of Anthropology, University of Otago, P.O Box 56, Dunedin.

spread of the calibrated radiocarbon ranges, which usually yield multiple age ranges due to the 'wiggle' in the calibration curve at *c.* 600 BP (cf. McFadgen *et al.* 1994).

TABLE 1

Radiocarbon dating results from pretreatment experiments using material excavated from the Killermont #2 site, Mackenzie Basin. The charcoal sample was excavated from Oven A in Area B (sq. K9/K10). All samples are 5 ml benzene samples except Wk-2787 (3 ml) and Wk-2781 (0.35 ml) (Higham 1993).

Lab No	**Sample fraction and pretreatment**	**Conventional Radiocarbon Age (BP)**	**$\delta^{13}C$ (‰)**
Wk-2781	Rootlets extracted by flotation and tweezers.	370 ± 140	-26.2
Wk-2782	Untreated charcoal sample.	680 ± 35	-25.5
Wk-2783	Charcoal with rootlets removed (physically pretreated).	640 ± 35	-25.3
Wk-2791	Charcoal residue acid insoluble in 10% w/v HCl.	600 ± 40	-25.5
Wk-2784	Base insoluble A fraction. Residue insoluble after treatment with 5% w/v NaOH and washing with 10% w/v HCl.	650 ± 35	-25.0
Wk-2785	Base soluble A fraction. Residue soluble after treatment with 5% w/v NaOH. Washed with 10% w/v HCl.	590 ± 45	-26.0
Wk-2786	Base insoluble B fraction. Residue of Wk-2784 insoluble after treatment with 5% w/v NaOH and washing with 10% w/v HCl.	630 ± 40	-25.5
Wk-2787	Base soluble B fraction. Residue of Wk-2784 soluble after treatment with 5% w/v NaOH and washing with 10% w/v HCl.	600 ± 60	-25.7
	Residue remaining after washing in 70% conc. HNO_3.	not dated	

TABLE 2

Calibrated age ranges of radiocarbon determinations from Areas B and C at the Killermont #2 site, and their pooled mean. Calibrations made using the high precision curves of Stuiver and Becker (1993) with a -27 yr correction for the Southern hemisphere offset (McCormac et al. n.d.).

Laboratory Number	**Site number**	**Conventional Radiocarbon Age (BP)**	**δ13C (‰)**	**Calibrated age ranges AD (1 σ)**	**Calibrated age ranges AD (2 σ)**
Wk-2791	KLM 6 Area B (Oven A)	600 ± 40	-25.5	1327 - 1351 1364 - 1366 1389 - 1416	1297 - 1436
Wk-2991	KLM 62 Area C (Sq. K4)	630 ± 45	-24.8	1301 - 1374 1379 - 1410	1289 - 1429
Wk-2916	KLM 45 Area C (Sq. D5/E5)	660 ± 45	-25.5	1299 - 1329 1348 - 1392	1284 - 1408
Pooled	Mean (A' (p))	628 ± 25	-	1325 - 1353 1359 - 1367 1388 - 1419	1301 - 1373 1379 - 1432

Anderson and Wallace (1993) recently reanalysed the prehistoric chronology of Houhora, Mount Camel, Northland and found it to have been occupied for a short period within the fourteenth century AD. Their conclusion was that the similarity of the radiocarbon determinations from sites which can be described as early or Archaic, based upon their material culture and faunal remains, could represent a swift expansion period in the settlement phase of prehistory. The results from the Killermont site presented here appear to favour this conclusion. The pretreatment experiments validate the accuracy of these measurements.

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