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# New Zealand Bone Dating Revisited: A Radiocarbon Discard Protocol for Bone

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#### ABSTRACT

This paper presents a list of <sup>14</sup>C determinations of bone from New Zealand archaeological sites measured at the Institute of Geological and Nuclear Sciences and the Waikato Radiocarbon Dating Laboratory in New Zealand. It discusses problems with these determinations and applies a discard protocol to cull out known problem dates. The results suggest that bone from omnivorous species may be subject to variation caused by reservoir differences. Species identified as suspect include human, dog and rat. The majority of radiocarbon determinations are of moa bone collagen. The data suggest that moa bone can be dated accurately by radiocarbon, though pretreatment methods have not always been successful, especially when the bones were of poor preservation. On the basis of this review, it is recommended that careful assessment of the bone preservation state and contextual relationship, as well as site environment, is necessary to improve the accuracy and security of bone <sup>14</sup>C determinations.

Keywords: BONE, RADIOCARBON DATES, DISCARD PROTOCOL, COLLAGEN, CARBONATE, GELATIN, FIXED CARBON.

#### INTRODUCTION

Debate over colonisation and settlement has dominated archaeological investigations in New Zealand. The interaction of humans with both introduced (e.g., *Rattus exulans*) and indigenous (e.g., moa) fauna has been an important part of that debate (Duff 1950; Lockerbie 1959; Anderson 1989, 1996; Barber 1995; Holdaway 1996). Consequently, the ability to date bone accurately has played a key role in the development of a radiocarbon chronology for the New Zealand prehistoric sequence. Until recently, moa bone was the preferred sample type when dating sites containing moa remains. Currently, however, bone has been considered to be less reliable than other sample types (Anderson 1991: 779; Higham 1993: 97; Schmidt 1996: 9; Anderson, Smith and Higham 1996). Explanations for anomalous bone ages include carbon fractionation due to dietary preferences, inadequate sample pretreatment, varied radiocarbon standards, diagenetic effects and/or contamination (Rafter 1978: 138; Grant-Taylor 1974: 160; Jansen 1984: 17; Caughley 1988: 247; Anderson and McGovern-Wilson 1990: 44–45; Anderson 1991: 777–79, 1998a, 1998b; Anderson, Smith and Higham 1996; 66).

Recent debate over the accuracy of *Rattus exulans* bone gelatin results (Anderson 1996, 1998a, 1998b; Holdaway 1996; Beavan and Sparks 1997; Ladefoged *et al.* 1997; Smith and Anderson 1998; Sparks 1998) suggests, however, that bone remains an important <sup>14</sup>C sample type. Unfortunately, because there has been no comparison of bone determinations with

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other acceptable sample types (cf. Anderson 1991; Spriggs and Anderson 1993; Schmidt 1996), the reliability of this material in the New Zealand situation is not certain. Further, it is apparent that considerable confusion exists. Although several different fractions of bone have been dated in New Zealand, 'collagen' or 'bone' has been used as a generic label for most determinations published. Often the specific pretreatment used for each sample has not been stated and laboratory reports provide only a general indication of the fraction analysed. In addition, mis-identification of the measured fraction has been compounded by the simultaneous analysis of carbonate and collagen (or 'fixed carbon') results at IGNS<sup>2</sup> between 1956 and 1972. Moreover, the first series of carbonate and fixed carbon determinations were given the same laboratory numbers, further complicating this issue. Carbonate results for Tautuku and Papatowai, for example, have been continually misreported as "collagen" (e.g., Anderson 1989: 174; Anderson and McGovern-Wilson 1990: 52) since being first published as "moa bone" determinations by Lockerbie (1959) (though see Hamel 1978 and Caughley 1988). New laboratory numbers have been assigned in some instances to resolve this confusion, but these have not been routinely used in publications. In the past, poor communication between laboratory and submitter has also often led to incorrect reporting of the fraction analysed; for example, NZ-510, a charcoal sample from Paremata, was reported by Sinclair (1977: 158) to be a mix of moa, dog and seal bone. It has since become apparent that charcoal was removed from the mix of moa, dog and seal bone collected by Sinclair (Davidson 1978b: 214).

In this paper, a culling protocol has been devised to appraise comprehensively all bone determinations measured in New Zealand. The aim is to isolate bone radiocarbon determinations that are not obviously suspect, and consider whether there are sufficient grounds for rejecting radiocarbon results of bone in general. This protocol will also provide a framework for future selection of bones for radiocarbon dating and highlight areas of concern. Following the application of this discard protocol, the remaining bone determinations are evaluated by comparison with shell and charcoal results that have been identified as reliable according to the specifications given by Anderson (1991), Higham (1993) and Schmidt (1996).

#### BONE DISCARD PROTOCOL

The University of Waikato and IGNS radiocarbon dating laboratory databases were searched for all known New Zealand archaeological bone determinations run before 1990. Information relating to samples analysed at IGNS was obtained from a preliminary compilation of archaeological determinations by McFadgen, the fossil record forms (FRF), radiocarbon result sheets held by IGNS, and the 'Jansen' database. Most of these radiocarbon measurements have been recalculated with respect to modern standards by IGNS and may, therefore differ from published accounts. A few determinations have not been recalculated because of the obvious inaccuracy of the results. These are instead reported according to the reference cited below Table 1. Some more recently measured bone <sup>14</sup>C determinations were obtained from a search of the literature. The table may, therefore, be incomplete (the list does not include results of bones measured overseas or the fish bone results given in Petchey 1998).

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To distinguish between different pretreatments and fractions isolated, bone determinations in Table 1 are described variously as:

- 1. Fixed carbon: the protein fraction pretreated as outlined by Rafter (1955: 23).
- Collagen: the fraction remaining after an acid wash (using either hydrochloric or phosphoric acid) or a combination of acid and alkali treatment.
- 3. Gelatin: where gelatinisation (i.e., denaturing the collagen fibre by heating in slightly acidic water) has been used to purify the sample.
- 4. Carbonate: the inorganic fraction isolated by acid hydrolysis of whole bone.

In addition, both the laboratory number (Wk-: samples measured at the Waikato Radiocarbon Dating Laboratory; NZ- and NZA-: samples measured at IGNS) and IGNS 'run number' (R-) are supplied, where possible, to assist in the identification of carbonate and protein fractions.

The discard protocol for 'bone' determinations is based on recommendations given by Hedges and van Klinken (1992) and Stafford *et al.* (1991), and the results of bone pretreatment and preservation tests obtained by Petchey (1998). These recommendations are intended principally to aid in the selection of bones, for <sup>14</sup>C analysis, from relatively young (<1000 years) New Zealand archaeological sites. Consequently, the categories discussed deal with areas of specific interest to the archaeological community. The discard protocol is as follows.

#### 1. Carbonate dates

Whole bone or carbonate results are rarely reliable, because the carbonate fraction of bone may exchange with atmospheric CO<sub>2</sub> (e.g., Rafter *et al.* 1972: 638; Rafter 1975: 47; Stafford *et al.* 1991: 62; Hedges and van Klinken 1992: 285). From Table 1 it is apparent that the majority of carbonate radiocarbon dates give younger results than the associated collagen determinations, and around 40% of those are modern. All carbonate ages are therefore rejected. This includes NZ-56 (*R*199), NZ-59 (*R*197), NZ-60, NZ-137 (*R*192/1), NZ-138, NZ-139, NZ-140, NZ-142, NZ-146 (*R*192/2), NZ-460, NZ-480, NZ-424, NZ-425, NZ-514, NZ-558, NZ-751, NZ-753, NZ-755, NZ-757, NZ-759, NZ-765, NZ-784, NZ-917, NZ-927, NZ-929, NZ-931, NZ-1112, NZ-1298, NZ-2464, and NZ-4652.

#### 2. Burnt bone

Burning significantly reduces the protein content in bone and increases the porosity of the mineral phase. Charred bone is, therefore, a problematic sample type for radiocarbon analysis because of the large sample sizes required owing to the loss of collagen, as well as the increased reactivity of the severely degraded collagen with contaminants (see Petchey 1998). In addition, burnt bone tends to yield inconsistent stable isotope and radiocarbon results (Tamers and Pearson 1965: 1055; Polach and Golson 1966: 31; Rafter *et al.* 1972: 643; Stafford *et al.* 1991: 63, table 12).

# TABLE 1

# BONE DETERMINATIONS FROM NEW ZEALAND ARCHAEOLOGICAL SITES MEASURED AT THE WAIKATO RADIOCARBON DATING LABORATORY AND IGNS

	AA3474 <sup>2</sup>					
			Cow bone protein	standard	-29.4	Rafter 1955; Rafter et al.
	AA3474		Cow bone CO <sub>3</sub> <sup>2-</sup>	standard	-16.4	1972:639
Ahuriri Lagoon	NZ-5608 R95541/1		Human collagen	429 ± 55	-16.43	
	NZ-5609 <i>R</i> 9541/2		Human collagen	497 ± 55	-16.60	
Avoca Point	NZ-2717 R4863/3	Cultural layer	Seal collagen	436 ± 156	-23.10	Trotter 1980a:283; Challis 1991:132-133
	NZ-3164 R4857	Cultural layer: Oven	Anomalopteryx didiformis collagen	952 ± 192	-25.29	
	NZ-4155 <i>R</i> 5157/4	Cultural layer: Oven	Anomalopteryx collagen	703 ± 85	-22.90	
Avoca Point. Fyffe Site	NZ-6496 <i>R</i> 9810/1	Base of wall: Early occupation	Anomalopteryx didiformis collagen	529 ± 42	-22.85	Challis 1991:133
	NZ-6566 <i>R</i> 9810/3	Outside wall: Early occupation	Anomalopteryx didiformis collagen	745 ± 59	-22.70	

Awamoa	NZ-4872 R5927/1	Cultural deposit: Test pit	Euryapteryx gravis collagen	671 ± 55	-22.70	Trotter 1980b:185
Cascade Cove, Fiordland	NZ-784 R1526/1A	Floor of cave	Human carbonate	-100 ± 61	-6.29	Begg and Begg 1966:214-215; Moore and Tiller 1976:153; Coutts 1972:527
	NZ-785 R1526/1B	Floor of cave	Human collagen	802 ± 48	-12.91	
	NZ-786 R1526/2B	Floor of cave	Burnt human collagen	720 ± 49	-12.91 assumed	
	NZ-787 <i>R</i> 1526/3B	Floor of cave	Human collagen	574 ± 60	-12.91 assumed	
Cross Creek	NZA-576 R11758/3	Layers 7, 8 and 9	Moa collagen	751 ± 58	-23.45	Sewell 1986:229; 1988:8
False Island	NZ-142 R192/15	Midden	Fish carbonate	516 ± 73	-4.80	Fergusson and Rafter 1959:220-221; Lockerbie 1959:106; Grant-Taylor and Rafter 1963:136
Hampden	NZ-755 R1641/1A	Occupation layer (Sq. F8 and G9)	Euryapteryx gravis carbonate	355 ± 48	-10.67	Trotter 1967b:140; Trotter 1967c:59
	NZ-756 R01641/1B	Occupation layer (Sq. F8 and G9)	Euryapteryx gravis collagen	503 ± 70	-23.34	
	NZ-757 R1641/2A	Occupation layer (Sq. F8 and G9)	Burnt Euryapteryx ? carbonate	437 ± 81	-16.23	

	NZ-758 R1641/2B	Occupation layer (Sq. F8 and G9)	Burnt <i>Eurtyapteryx</i> collagen	519 ± 60	-24.22	
A	NZ-59 AA2550/2 R197	Occupation layer: Oven 1	Burnt <i>Dinornithidae</i> carbonate	470 ± 55 <sup>°</sup>	-13.0	Rafter 1955:36; Fergusson and Rafter 1957:744-745; Lockerbie 1959:106; Grant- Taylor and Rafter 1963:126- 127; Rafter <i>et al.</i> 1972:639
	NZ-59 R198 AA2550/2	Occupation layer: Oven 1	Burnt Dinornithidae "fixed carbon"	440 ± 55 <sup>°</sup>	-25.7	
NZ-60 AB1502	NZ-60 AB1502/15	Occupation layer: Oven 2	Euryapteryx gravis carbonate	293 ± 64		
Hot Water Beach	NZ-1298 R4068/3A	Layer 4	Fish carbonate	-100 ± 104	-4.34	Leahy 1974:71-72
	NZ-1299 <i>R</i> 4068/3B	Layer 4	Fish collagen	647 ± 92	-13.89	
	NZA-583 R11758/1	Layer 6	Moa collagen	549 ± 74	-23.25	
Houhora	NZ-5007 R9107/1	Layer 2c (Sq. D9)	Anomalopteryx didiformis, Euryapteryx curtus, Pachyornis septentrionalis collagen	563 ± 56	-21.10	Millener 1981:847; Anderson and Wallace 1993:10

	NZ-5008 R9107/2	Layer 3b (Sq. D10)	Dinornis struthoides, Anomalopteryx didiformis, Euryapteryx curtus, Pachyornis septentrionalis collagen	585 ± 46	-22.50	
Hurunui River Mouth	NZ-1839 R4745/6	Occupation layer: Oven	Euryapteryx collagen	646 ± 85	-23.60	McCulloch and Trotter 1975a:17; Moore and Tiller 1975:103
Kaupokonui	NZ-3931 R5055/1	Layer 4d (Cassels 1974); Layer 4 (Buist 1963)	Pachyornis sp. collagen	568 ± 49	-22.70	Buist 1963; Cassels nd:9-13; Foley 1980:3, table A1.1
	NZ-3934 R5054/1	Layer 4f (Cassels 1974); Layer 6 (Buist 1963)	Moa collagen	618 ± 57	-25.60	
Lagoon Flat	NZ-1834 <i>R</i> 4745/1	Burial 4	Human collagen	437 ± 57	-16.50	McCulloch and Trotter 1975b:110; 1975a:3, 17; Trotter 1982:97
Makara	NZ-480 <i>R</i> 700/3	Beach midden	Dinornis carbonate	-100 ± 144	-0.44	Davis 1962:149; Rafter <i>et al.</i> 1972:639
Ototara	NZ-753 R1640/A	Occupational layer	Euryapteryx gravis carbonate	308 ± 93	-6.90	Trotter 1965:113; Trotter 1967b:138
	NZ-754 R1640/B	Occupational layer	Euryspterx gravis collagen	435 ± 70	-25.00	

Papatowai	NZ-137 R192/1	Middle layer	<i>Euryapteryx gravis</i> carbonate	309 ± 46	-25.60	Fergusson and Rafter 1959:220-221; Lockerbie 1959:106; Grant-Taylor and Rafter 1963:136; Hamel 1978:54
	NZ-137 (NZ-2688) R192/1A	Middle layer	Euryapteryx gravis "fixed carbon"	707 ± 61	-25.68	
	NZ-138 R192/8	Upper layer	Dinornis maximus carbonate	571 ± 75	-8.50	
	NZ-139 R192/9	Upper layer	Euryapteryx gravis carbonate	399 ± 60	-10.90	
	NZ-140 R192/3	Upper layer	Euryapteryx gravis carbonate	690 ± 59	2.19	
Parker's Midden	NZA-557 R11758/2	Layer 4	Euryapteryx sp. collagen	510 ± 58	-22.57	
Pauatahanui	NZA-7411	Midden 2	Rattus exulans gelatin	$452 \pm 69^{f}$	-	Sparks, Beavan and Redvers- Newton 1997:207
	NZA-7044	Midden 4	Rattus exulans gelatin	$361 \pm 69^{f}$	÷	
	NZA-7410	Midden 9	Rattus exulans gelatin	$439 \pm 71^{f}$		
Pawhetau Pa, Kawakawa	NZ-3903 R2548	Burial	Human crude collagen	350 ± 80 <sup>#</sup>	-	Moore and Tiller 1976:153; Fox 1974:20

Peketa Pa	NZ-4154 R5157/3	Floor of pit house	Dog collagen	508 ± 83	-12.70	Challis 1991:133
	NZ-4296 R5343	Floor of pit house	Dog collagen	222 ± 241	-19.40	
Pleasant River : Trotter 1979 excavation	NZ-5013 <i>R</i> 9096/1	Black soil containing occupational material	Moa collagen	408 ± 56	-24.70	Anderson 1989:222
Pleasant River: Area 1	NZA-6536	Layer 2, Sq. A2	Rattus exulans gelatin	1591 ± 71 <sup>‡</sup>	-22	Smith and Anderson 1998:90
Pleasant River: Area 2	NZA-6532	Layer 4, Sq. C4	Rattus exulans gelatin	$1039 \pm 69^{\ddagger}$	-21.6	Smith and Anderson 1998:90
Pleasant River: Area 7	Wk-5169	Layer 2a, Sq. A3 and B4	<i>Eu. geranoides ?</i> Moa sp. ? gelatin	580 ± 45	-26.2	Petchey 1998
Port Jackson	NZ-4883 <i>R</i> 5965	Deflation hollow under midden	A. didiformis, P.mappini, D. struthoides collagen	606 ± 56	-23.10	Millener 1981:848; Anderson 1991:775
Poukawa	NZ-2464 R2540/A	"Natush property"	Human carbonate	$-100 \pm 45$	-12.04	
	NZ-2467 R2540/B	"Natush property"	Human collagen	315 ± 35	-18.08	
Pounawea	NZ-56 AA2550/1b R199	Middle layer	Seal carbonate	520 ± 55 <sup>-</sup>	-	Rafter 1955: 36; Fergusson and Rafter 1957: 742-743; Lockerbie 1959: 106; Grant- Taylor and Rafter 1963: 126; Anderson 1989: 222

	NZ-56 AA2550/1c R200	Middle layer	Seal "fixed carbon"	550 ± 55~	-		
	NZ-1796 R4451/1	Lowest layer	Dinornis maximus collagen	699 ± 105	-23.73	,	
	NZ-1797 R4451/2	Lowest layer	Moa collagen	668 ± 60	-25.10		
	NZ-1798 R4451/3	Lowest layer, base of oven	Moa collagen	772 ± 66	-26.00		
	NZ-1866 R4544/3	Lowest layer, base of oven	Burnt <i>Dinornis maximus</i> collagen	1465 ± 79	-25.80		
	NZ-4438 <i>R</i> 5437	Lowest deposit	Moa collagen	602 ± 47	-25.60		
Rakaia River Mouth	NZ-927 R2068/1A	Middle layer	Burnt Euryapteryx gravis carbonate	215 ± 120	-16.23	Trotter 1972b:135	
	NZ-930 R2068/1B+ 2B	Middle layer	Burnt moa collagen	556 ± 71	-25.39		
	NZ-929 R2068/2A	Middle layer	White burnt moa carbonate	924 ± 112	-20.42		
	NZ-931 R2068/3A	Middle layer	Euryapteryx ? carbonate	-100 ± 82	-10.77		
	NZ-932 R2068/3B	Middle layer	Euryapteryx ? crude collagen	487 ± 88	-25.10		

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Redcliffs: Hamilton's Deposit	NZ-1112 R1936/2A	Occupation layer	Euryapteryx gravis carbonate	-100 ± 94	-7.55	Trotter 1968:87; 1975c:204
	NZ-1113 R1936/2B	Occupation layer	Euryapteryx gravis collagen	701 ± 60	-20.76	
Redcliffs: Sewer Trench Pit	NZ-1162 R2553/6	Occupational deposit: 9.84 to 19.79 cm depth	Euryapteryx, Emeus and Anomalopteryx collagen	622 ± 44	-23.83	Trotter 1968:87; 1975c:204
	NZ-1376 R2553/7	Occupational depost: 9.84 to 19.79 cm depth	Euryapteryx, Emeus and Anomalopteryx collagen	537 ± 45	-24.91	
Redcliffs: Hine's oven	NZ-460 R1052/2-3	Top of oven	Euryapteryx carbonate	480 ± 84	-13.01	Trotter 1975c:204; Duff 1963:10
Redcliffs: Moa Bone Point Cave	NZ-514 R1090/2	Post fill	Euryapteryx gravis carbonate	515 ± 64	-16.12	Duff 1963:10; Trotter 1967a; 1975c:203
Shag River Mouth: Trotter 1979 testpit	NZ-5016 <i>R</i> 9096/4	Ashy layer below midden	Moa collagen	641 ± 85	-23.50	Anderson 1991:776; Anderson and Smith 1996:9
Shag River Mouth: SM/B:FHA	NZ-7742 R11838/9	Layer 2	Moa collagen	530 ± 36	-23.70	Anderson, Smith and Higham 1996:61-63
Shag River Mouth: SM/A	NZ-7741 R11838/8	Layer 2	Moa collagen	522 ± 37	-25.20	Anderson, Smith and Higham 1996:61-63
Shag River Mouth: SM/C:Dune	NZA-781 R11838/5	Layer 2	Moa collagen	630 ± 82	-24.40	Anderson 1996:179; Anderson, Smith and Higham 1996:61-63

	NZ-7743 R11838/10	Layer 4	Moa collagen	1201 ± 38	-24.90	
	NZA-5719	Layer 4	Rattus exulans gelatin	$1487 \pm 87^{\dagger}$	-21.5	
	Wk-5433	Layer 4	Moa gelatin	$640 \pm 40$	-26.2	Petchey 1998
	NZ-7737 R11838/4	Layer 5	Moa collagen	$1170 \pm 70$	-24.95	
	NZA-5936	Layer 5	Rattus exulans gelatin	$2040\pm68^{\dagger}$	-19.6	
	NZ-7736 R11838/3	Layer 6	Emeus or Euryapteryx collagen	634 ± 58	-24.50	
	NZA-5926	Layer 6	Rattus exulans gelatin	$1578 \pm 88^{\dagger}$	-21.1	
	NZA-780 R11838/2	Layer 7	Moa collagen	509 ± 72	-23.50	
	NZ-7666 R11722	Layer 11	<i>Emeus</i> or <i>Euryapteryx</i> sp. collagen	787 ± 72	-26.88	
	NZA-5720	Layer 11	Rattus exulans gelatin	$1862 \pm 86^{\ddagger}$	-21.2	
Shag River Mouth SM/D:1	NZ-7739 R11838/6	Layer 5	Euryapteryx sp. collagen	370 ± 38	-24.00	Anderson, Smith and Higham 1996:61-63
Shag River Mouth SM/D:3	NZ-7740 R11838/7	Layer 2	Moa collagen	477 ± 53	-24.67	Anderson, Smith and Higham 1996:61-63
Skippers midden	NZA-575 R11758/6	Beach midden	Moa collagen	417 ± 56	-24.19	
Station Bay, Motutapu Island	NZ-4346 <i>R</i> 5407/1	Davidson undefended site	Human crude collagen	451 ± 45	-15.00	Davidson 1972; 1978a:15

	NZ-4347 R5407/2		Human collagen	562 ± 41	-18.20	
	NZ-4348 R5416	Pa site: Burial 1 on floor of kumera pit	Human collagen	367 ± 61	-25.00	
Stewart Island	NZ-424 R780/3	Oven	Euryapteryx carbonate	261 ± 81	-9.6	Rafter et al. 1972:639
	NZ-425 R780/4	Oven	Moa carbonate	1 ± 59	-9.10	
Tairua	NZA-558 R11758/4	Layer 2: oven	Dinornis struthoides collagen	460 ± 55	-24.35	
Tai Rua	NZ-558 R1371/1A	Main occupation (Layers 5, 5a and 6)	Euryapteryx gravis carbonate	$-100 \pm 33$	-10.0	Trotter 1967b:139; 1979:226- 227; Rafter et al. 1972:639
	NZ-559 R1371/1B	Main occupation (Layers 5, 5a and 6)	Euryapteryx gravis collagen	473 ± 37	-25.9	
	NZ-578 R1544/1B	Main occupation (Layers 5, 5a and 6)	Euryapteryx gravis collagen	473 ± 37	-24.7	
	NZ-751 R1639/3A	Main occupation (Layers 5, 5a and 6)	Eurapteryx gravis carbonate	-100 ± 55	-9.21	
	NZ-752 R1639/3B	Main occupation (Layers 5, 5a and 6)	<i>Euryapteryx gravis</i> collagen	513 ± 36	-24.51	
	NZ-765 R1661/1A	Main occupation (Layers 5, 5a and 6)	Euryapteryx gravis carbonate	$-100 \pm 47$	-8.33	

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	NZ-766 R1661/1B	Main occupation (Layers 5, 5a and 6)	<i>Euryapteryx gravis</i> collagen	363 ± 48	-24.22	
Takahanga Pa	NZ-4464 R5492/1	Burial 4	Human collagen	646 ± 83	-13.70	Challis 1991:132; Trotter 1974; 1982:100
	NZ-4465 R5492/2	Cremation K52	Burnt human collagen	171 ± 81	-21.80	
	NZ-4525 R5658/1	Burial 4	Human collagen	$-100 \pm 30$	-13.80	
	NZ-4526 R5658/2	Burial 1	Human collagen	419 ± 45	-14.70	
	NZ-4635 R5742	Burial 4	Human collagen	477 ± 56	-13.70	
Takahe Valley, Rockshelter A	NZA-2227	Layer A: Surface	<i>Megalapteryx didinus</i> collagen	623 ± 39 +	-	O'Regan 1992:174
Tautuku	NZ-146 R192/2	Occupation deposit below human bone	Dinornis torosus carbonate	391 ± 59	-9.50	Fergusson and Rafter 1959:220-221; Lockerbie 1959:106; Grant-Taylor and Rafter 1963:136
	NZ-146 (NZ-2684) R192/2A	Occupation deposit below human bone	Dinornis torosus "fixed carbon"	530 ± 67	-25.78	
Fimpendean, Weka Pass	NZ-917 R2251/1A	Lower occupation level	Euryapteryx gravis carbonate	252 ± 86	-8.04	McCulloch and Trotter 1975a:5: 1975b:110; Trotter 1972a:45, 49; Moore and Tiller 1975:103

	NZ-918 R2251/1B	Lower occupation level	Euryapteryx gravis collagen	1192 ± 62	-24.81	
Titirangi Beach	NZ-4236 R5159/1	Lowest occupational deposit (240 cm depth)	Euryapteryx geranoides collagen	792 ± 148	-22.80	Trotter 1977:9; 1982:90-91; Challis 1991:130
Tumbledown Bay	NZA-825 R11690/3	Layer 3	Dinornis novae- zealandiae collagen	307 ± 85	-21.90	
Waimataitai Mouth	NZ-5015 R9096/3	Eroded section of main occupational deposit	Emeus crassus collagen	686 ± 173	-24.10	Anderson 1991:776
Wairau Bar	NZ-1835 R4745/2	Burial 42	Human collagen	700 ± 142	-20.90	Moore and Tiller 1975: 103; McCulloch and Trotter 1975a: 12; Trotter 1975a: 80; 1975b: 90; Challis 1991: 131
	NZ-1838 R4745/5	Main phase of occupation	Euryapteryx collagen	547 ± 58	-23.90	
	NZ-4442 R5433/1	Burial 3	Human collagen	575 ± 45	-19.70	
	NZ-4443 R5433/2	Burial 5	Human collagen	598 ± 56	-18.70	
	NZ-4444 R5433/3	Burial 35	Human collagen	329 ± 46	-23.10	
Wakanui	NZ-1766 R4002/1	Oven, in ash matrix	Burnt <i>Euryapteryx gravis</i> collagen	629 ± 58	-25.58	Trotter 1975b:90; McCulloch and Trotter 1975a:6
	NZ-1767 R4002/2	Oven, in ash matrix	Euryapteryx gravis calcined bone	558 ± 69	-24.71	

	NZ-1768 R4002/3	Oven, in ash matrix	Euryapteryx gravis collagen	383 ± 58	-24.70	
Whakamoenga Cave	NZA-577 R11758/5	Occupation 1: Period 1	Euryapteryx curtus collagen	4750 ± 81	-24.78	McFadgen 1989
Woolshed Flat	NZ-759 R1692/1A	Occupation layer	Pachyornis and Euryapteryx sp. carbonate	-100 ± 47	-13.20	Trotter 1967b:139; 1968:87; McCulloch and Trotter 1975a:6
	NZ-760 R1692/1B	Occupation layer	Pachyornis and Euryapteryx sp. collagen	544 ± 70	-20.03	

<sup>1</sup>Conventional Radiocarbon Age; <sup>2</sup>AA and AB are early IGNS reference numbers; <sup>^</sup>Rafter *et al.* (1972: 639); <sup>#</sup>Fox (1974: 20); <sup>^</sup>Fergusson and Rafter (1957: 126) includes 120yr industrial correction; <sup>\*</sup>O'Regan (1992: 174); <sup>f</sup>Sparks, Beavan and Redvers-Newton (1997: 207); <sup>‡</sup>Smith and Anderson (1998: 90); <sup>†</sup>Anderson (1996: 179).

Radiocarbon determinations of samples that have been burnt in prehistory are therefore rejected. This includes NZ-59 (*R*198) from Hawksburn; NZ-758 from Hampden Beach; NZ-930 from Rakaia River Mouth; NZ-1866 from Pounawea; and NZ-1766 and NZ-1767 from Wakanui.

There may be additional <sup>14</sup>C determinations of burnt samples which have not been identified by either the submitter or laboratory. It is therefore recommended that determinations should be interpreted with caution when they are of samples selected from firescoops or ovens, or from sites where burning has been noted in the associated layer generally.

#### 3. Unsuitable species

A number of different species have been dated by radiocarbon, including dog, seal, human and rat. Several of these may be unsuitable for <sup>14</sup>C analysis because of dietary or reservoir influences (see below).

Radiocarbon results of marine species known to migrate into depleted Antarctic waters are rejected because of the possibility of wide variations in reservoir correction (i.e., up to 1400 years; Law 1981: 234–35; Ambrose and Norr 1993: 31; Gordon and Harkness 1993). Similarly, species that feed in the deep ocean are rejected because of the possible incorporation of "old carbon" (Pearcy and Stuiver 1983; Williams *et al.* 1987). This includes NZ-56 (*R*200), a fixed carbon seal bone determination from Pounawea, and NZ-2717, a collagen measurement from Avoca Point. Both of these radiocarbon results are unexpectedly too young. This is likely to be the result of inadequate pretreatment (see below). The reliability of seal bone cannot therefore be adequately assessed at present.

Some fish can also range outside New Zealand waters, or into depleted waters (below 200 m). Determinations of fish species which have not been identified as coastal are therefore discarded. This includes NZ-1299 from Hot Water Beach.

Two dog bone <sup>14</sup>C determinations from contiguous contexts at Peketa Pa, Kaikoura, differ both in radiocarbon age and  $\delta^{13}$ C value (NZ-4154: 570±90 BP,  $\delta^{13}$ C = -12.7%; and NZ-4296: 270±60 BP,  $\delta^{13}$ C = -19.4‰)<sup>3</sup> (Trotter 1976). The  $\delta^{13}$ C result for NZ-4154 implies a marine dietary influence (see Schoeninger and DeNiro 1984) and the older than expected radiocarbon result supports a marine <sup>14</sup>C component. Results for both NZ-4154 and NZ-4296 are therefore considered to be erroneous.

The range of  $\delta^{13}$ C values (from -12.91‰ to -25.00‰) evident for human bone radiocarbon determinations suggests that a number of anomalous results are likely to have been caused by a varied diet (Jansen 1984: 24). All human bone determinations are therefore discarded, including NZ-785, NZ-786 and NZ-787 from Cascade Cove; NZ-2467 from Poukawa; NZ-3903 from Pawhetau Pa; NZ-1834 from Lagoon Flat; NZ-5608 and NZ-5609 from Ahuriri Lagoon; NZ-4346 from Station Bay N38/37; NZ-4347 from Station Bay N38/30; NZ-4348 from Station Bay N38/25; NZ-4464, NZ-4465, NZ-4525, NZ-4526 and NZ-4635 from Takahanga Pa; and NZ-1835, NZ-4442, NZ-4443 and NZ-4444 from Wairau Bar.

 $<sup>^3</sup>$  Shell samples from same deposit, NZ-4152 and NZ-4153, gave ages of 290 ± 50 BP and 350 ± 50 BP respectively.

Anomalous  $\delta^{13}$ C,  $\delta^{15}$ N, and  $\Delta^{14}$ C values for modern samples of *Rattus exulans* gelatin imply variation introduced through diet. This is especially evident at Pleasant River Mouth (e.g., OxA-6743 dated to 950±60 BP yielded a δ13C of 19.2% and δ15N of 16.90%. OxA-6744, on the other hand, produced a more reasonable result of 515±55 BP with a  $\delta^{13}$ C of -20.0% and 815N of 11.29%) (Smith and Anderson 1998: 90, table 1). Inconsistencies between duplicate rat gelatin determinations also suggest a lack of reproducibility (e.g., NZA-6532 produced a result more than 500 radiocarbon years different [1039±69 BP] from OxA-6744 [above] which is considered to be of the same individual) (Smith and Anderson 1998: 89). Laboratory-induced contamination or inadequate pretreatment of small AMS samples have also been suggested as possible causes for the lack of reproducibility in archaeological bone determinations from Pleasant River (Smith and Anderson 1998) and Shag River Mouth (Anderson 1996, 1998a, 1998b; Petchey and Higham in press). All rat bone radiocarbon results are therefore rejected. This includes NZA-5719, NZA-5620, NZA-5926, and NZA-5936 from Shag River Mouth; NZA-6536 and NZA-6532 from Pleasant River, Areas 1 and 2 respectively; and NZA-7411, NZA-7044 and NZA-7410 from Pauatahanui. Consequently, the rat bone determinations from natural sites (Holdaway 1996), which have important implications for the New Zealand prehistoric sequence, are also suspect.

All remaining <sup>14</sup>C determinations are of moa bone. Because moa feed mainly on C3 plants (e.g., trees, flowers and grasses) (Anderson 1991: 777), they are considered to have been in equilibrium with atmospheric  $\Delta^{14}$ C values. Variation introduced by dietary  $\Delta^{14}$ C should not, therefore, significantly influence these radiocarbon results.

#### 4. Contamination (pretreatment and preservation state)

Inadequate pretreatment and variable preservation state have been suggested as possible reasons for anomalous collagen determinations (Anderson 1991: 777, 779; Anderson, Smith and Higham 1996: 64; Beavan and Sparks 1997: 8). Neither possibility has been adequately assessed in New Zealand, although a variety of bone pretreatment methods have been applied at IGNS and the Waikato Radiocarbon Dating Laboratory. Research overseas (Stafford *et al.* 1991: 62–64; van Klinken and Hedges 1995) and in New Zealand (Redvers-Newton 1995; Redvers-Newton and Coote 1994; Petchey 1998) suggests, however, that many of these pretreatments are not of equal validity.

Rafter (1955) devised a pretreatment technique to isolate both the protein (fixed carbon) and carbonate portions of bone. The fixed carbon fraction is, in effect, the residue remaining following decalcification of bone. The success of such fixed carbon determinations is therefore likely to be similar to that of those that have undergone an acid wash (see below).

The fixed carbon method was superseded at IGNS by a more conventional acid wash pretreatment (Rafter 1965; Rafter *et al.* 1972; Jansen 1984). An acid wash (hydrochloric or phosphoric acid) can leave a considerable amount of contamination (>15%) (van Klinken and Hedges 1995: 268). For a sample of 600 BP this could result in a error of >80 years when contaminated by modern carbon, and >1300 years when contaminated by <sup>14</sup>C free carbon. However, both scenarios are unlikely, because contamination may originate from different sources of disparate age. Typically, bone pretreated in this manner will give a minimum age (Stafford *et al.* 1991: 63, table 12). The presence of unremoved contamination can clearly be seen in the infrared spectra of some acid-insoluble fractions given in Petchey (1998).

In the late 1980s, a sodium hydroxide wash was added to the routine bone pretreatment method at IGNS (McFadgen and Manning 1989). An alkali washed, acid-insoluble fraction may leave up to 10–15% humic contamination (van Klinken and Hedges 1995: 268). This equates to a possible error of 55–80 years in a sample 600 years old, when contaminated by modern carbon. In most cases, an error of this magnitude would go undetected.

A gelatinisation step was added to the pretreatment at IGNS in 1993 (Redvers-Newton and Coote 1994). Van Klinken and Hedges (1995: 268) have demonstrated that gelatinisation of heavily contaminated collagen can leave around 8% humic contamination. This can result in an error of around 42 years younger in a sample 600 years old, or about 700 years too old when contaminated with <sup>14</sup>C free carbon. It is generally agreed, however, that such pretreatment improves the possibility of an accurate radiocarbon determination on well preserved bone (>20% collagen remaining) (Stafford *et al.* 1991: 64, table 12; Hedges and van Klinken 1992: 286). Redvers-Newton (1995: 113) concluded that gelatin pretreatment would not give accurate results for bones with 0.9–0.4% nitrogen remaining (*c.* 10–20% collagen remaining<sup>4</sup>).

A sodium hydroxide wash before gelatinisation can improve the accuracy of radiocarbon measurements, depending on the level and type of contamination (Gurfinkel 1987: 51; Arslanov and Svezhentsev 1993: 389). All samples from the Waikato Radiocarbon Dating laboratory (Wk-) were treated with sodium hydroxide before gelatinisation. This method has been shown to be successful in most cases for bones with >40% extractable protein (Petchey 1998), and should reduce contamination to levels below the possible 8% of gelatinisation alone.

The majority of bone determinations in Table 1 were pretreated with either an acid or acid/alkali wash. Under these circumstances, the influence of contamination (10 to >15%) on bone that was very well preserved may not be statistically significant at the level of precision used to date most New Zealand archaeological sites. Where the bone is of 'poor' or 'transitional' preservation (<20% collagen remaining), however, it is likely that significant contamination will remain following the pretreatment and be responsible for anomalous results. Very few of these determinations are accompanied by data pertinent to preservation state and their accuracy is therefore difficult to assess. This is dealt with in greater detail in the discussion below.

At present, radiocarbon determinations have only been excluded if the sample analysed has been noted by the laboratory or researcher to have been at risk of being contaminated. This includes NZ-766 from Tai Rua, which was contaminated with dieldrin — a carbon compound (Trotter 1967d); and NZ-7666 and Wk-5433 from Shag River Mouth. NZ-7666 was contaminated with "electronegative impurities" and required further purification through a "molecular sieve" to prevent interference with the gas counting method used (Anderson, Smith and Higham 1996: 64). The exact method of purification is unclear and NZ-7666 is considered to be questionable. The preservation state of Wk-5433 was anomalous when compared to other bones from Shag River Mouth. Excessive recrystallisation of the hydroxyapatite fraction (Petchey 1998) implies that this sample was not in primary deposition and may be a sub-fossil bone (cf. NZ-7743 and NZ-7737 from SM/C:Dune discussed below).

<sup>&</sup>lt;sup>4</sup> Around 30% of modern dry bone is collagen.

## 5. Old dates

Old <sup>14</sup>C determinations, removed by more than  $2\sigma$  from results of samples belonging to stratigraphically identical horizons, have been variously identified as the result of contamination or a consequence of dating sub-fossil bone. The recognition of sub-fossil bone is a major problem with <sup>14</sup>C analysis, despite the use of markers such as preservation state (Trotter and Malthus 1967: 151), butchery marks (Scarlett 1974: 3), and bone articulation (McFadgen 1982: 387, table 5), which can all be easily mis-interpreted (Andrews 1995: 148). This problem is compounded where archaeological sites are found on sub-fossil bone deposits, or where sub-fossil bone has been imported into a site for tool manufacture (Anderson 1989: 112; Millener 1981: 240), or stock piled for fuel (Kooyman 1985: 99, 116). Even avoiding bones from known problem areas (e.g., dune sands, cave sites, and archaeological sites with industrial bone) (Millener 1981: 239-44; Anderson 1989: 53, 55, 111; Anderson and McGovern-Wilson 1990: 44) has not always been successful, and in some cases a sub-fossil origin for the bone has only been suggested following an anomalously old radiocarbon result. Large deviations from the expected age have also been attributed to laboratory or pretreatment inadequacies (Anderson 1996, 1998a, 1998b). This is extremely difficult, if not impossible, to prove after the sample has been pretreated and a radiocarbon determination obtained. An attempt is made here to clarify this situation. Determinations significantly older (removed by more than  $2\sigma$ ) than results of associated samples are discarded. On the basis of available site and sample information, a possible cause of any anomalous results (e.g., the result of contamination or the inclusion of subfossil remains) is given below.

Trotter initially submitted the bone for NZ-918 from Weka Pass/Timpendean because fractures appeared to have been applied when fresh (Trotter 1969). The estimated date for this sample (1192±62 BP) is, however, older than the anticipated fifteenth century occupation. Trotter (1972a: 45) later suggested that the bone analysed was of suspect cultural origin, because sub-fossil moa remains were discovered in the cave. NZ-918 is therefore excluded.

NZA-557 from Whakamoenga Cave was considered initially to date the remains of a moa brought into the cave by humans. A result of  $4750\pm81$  BP raised doubt about this interpretation. McFadgen (1989: 257) has since suggested that NZA-557 dates either a dead bird washed into the lake when water levels were higher, or an old moa nesting site. NZA-557 is excluded from further analysis.

NZ-7743 (1201±39 BP) and NZ-7737 (1170±70 BP) from Shag River Mouth (SM/C:Dune) both yielded results approximately 600 years older than the established date of occupation. These anomalous results have been attributed to either variability in the rates of preservation or inadequate pretreatment. The use of sub-fossil bone was considered to be less likely, given the range of difficulties associated with bone <sup>14</sup>C determinations (Anderson, Smith and Higham, 1996: 66) and because both samples were carefully selected on the basis that they exhibited butchery marks (Anderson, Smith and Higham, 1996: 60, 64; I. Smith, pers. comm. 30/6/1997). Indeed, one of these samples (NZ-7743) was lightly burnt (i.e. "blackened on outside, but still robust": I. Smith pers. comm. 30/6/1997), a factor that could have affected the radiocarbon result. In addition, percent carbon values for samples of whole moa bone from Shag River Mouth indicated a low organic content (Melhuish 1990). Analysis of material from SM/C:Dune (Petchey 1998; Petchey and Higham in press) suggests, however, that bone from this location was generally well preserved, and a shift in age of 600 years, which would require around 8% contamination

by <sup>14</sup>C free carbon, is unlikely in such radiometric samples. Consequently, the possibility that NZ-7743 and NZ-7737 were sub-fossil bones, collected in prehistory and subsequently discarded within the site, should not be dismissed at present. Clearly, further research is required into the reliability of different pretreatments, preservation state and the identification of sub-fossil remains.

#### 6. Uncertain provenance

All acceptable radiocarbon determinations must come from samples in direct stratigraphic association with an archaeological event. Where displacement has occurred, either by a natural occurrence or human disturbance, the <sup>14</sup>C result is discarded because the provenance and chronological integrity of the sample cannot be proven.

A number of anomalous moa collagen determinations were obtained on material from Lake Poukawa. Unfortunately, cultural and natural moa deposits became mixed as a result of shrinkage following drying out of the Poukawa lake bed (McFadgen 1978: 177). The majority of moa bone crude collagen determinations from Poukawa are undoubtedly from natural deposits. These results have not been included in Table 1.

NZ-4236 from Titirangi Beach is significantly older ( $792\pm148$  BP) than three shell determinations which suggest a sixteenth century occupation. All three samples come from the lower occupation deposit, 90 cm above the moa bone sample. It is therefore possible that different events are being dated. NZ-4236 is excluded from further discussion, as this sample could belong to sub-fossil moa remains, and because the large standard error makes assessment difficult.

NZ-4883 from Port Jackson came from a deflation hollow under a midden (FRF *R*5965). This sample is discarded because of its questionable cultural association.

Wk-5169 from Pleasant River Area 7, Layer 2a, yielded a conventional radiocarbon age closer to that expected for the lower occupation deposit at this location (Layer 2b). Because there is evidence of disturbance at the site (I. Smith pers. comm. 23/10/1996), contextual mis-placement either from modern or prehistoric disturbance is likely (see Petchey 1998).

NZ-5013, also from Pleasant River (Area D), was submitted by Trotter in 1979. The exact stratigraphic and chronological relationship between this and other excavated areas at Pleasant River is unclear (I. Smith, pers. comm. 23/4/1998). Given the possibility of disturbance elsewhere at Pleasant River (Higham 1993: 143, 157; Higham and Hogg 1997: 153), the reliability of this sample cannot be adequately evaluated.

NZ-6496 and NZ-6566 from Fyffes, Avoca Point, are discarded. These samples came from outside the concentration of prehistoric debris identified by Trotter (1980a: 278–79) as undisturbed. Schmidt (1996: 51–52) has previously discussed shell sample NZ-6525 (800 $\pm$ 32 BP, Cal AD 1459–1508 at 1 $\sigma$ ) from this provenance and concluded that disturbance may be responsible for this anomalously young result. This is based on McFadgen's (1987) analysis, which suggested that sub-fossil material from a natural beach ridge formation at the site may have become mixed with cultural remains. Trotter (1980a: 281) also noted natural faunal remains to the west of the *in situ* deposit. The older of the two moa collagen determinations, NZ-6566 (745 $\pm$ 59 BP), may therefore be of sub-fossil material.

NZ-1838 is of moa bone obtained from early excavations at Wairau Bar (Duff 1950). The exact provenance of this sample is unknown, but it appears to have come from Layer 4 (the "main layer") which, according to Wilkes (1964: 4), was in places disturbed by subsequent agricultural activity. This sample is discarded, given the present uncertainties surrounding its precise context.

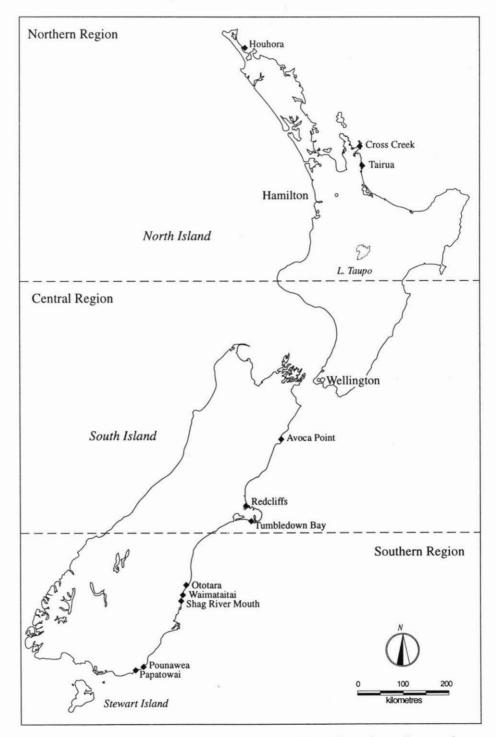


Figure 1: New Zealand Archaeological Sites with bone and matching charcoal or marine shell radiocarbon determinations.

#### 7. Single radiocarbon determinations

Because of the uncertainties with all bone determinations listed in this paper, <sup>14</sup>C results are only accepted where they can be validated through comparison with stratigraphically associated samples which are acceptable. Non-paired bone determinations that are rejected include NZ-146 (*R*192/2A) from Tautuku, which has been re-numbered NZ-2684; NZ-756 from Hampden; NZ-932 from Rakaia River Mouth; NZ-1162 and NZ-1376 from Sewers Trench, Redcliffs; NZ-1839 from Hurunui River Mouth; NZA-557 from Parker's Midden; NZA-575 from Skipper's Midden; NZ-1768 from Wakanui; and NZA-583 from Hot Water Beach, Layer 6.

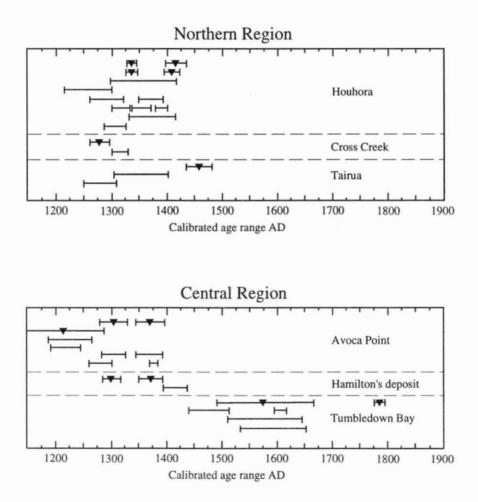


Figure 2: Calibrated age range for sites from the Northern and Central regions of New Zealand (see Table 2 for details).

Radiocarbon ages are also excluded from further analysis where the matching charcoal or shell pairs can be rejected following the protocols given in Anderson (1991), Higham (1993), Schmidt (1996: 161–62) and Higham *et al.* (in prep.), or where the reliability of the sample type is unknown (see Petchey 1998: Appendix 6 for list of associated <sup>14</sup>C determinations and discard protocol). This includes NZ-559, NZ-578 and NZ-752 from Tai Rua; NZ-760 from Woolshed Flat; NZA-2227 from Takahe Valley; NZ-4872 from Awamoa; and NZ-3934 and NZ-3931 from Layer 4, Kaupokonui.

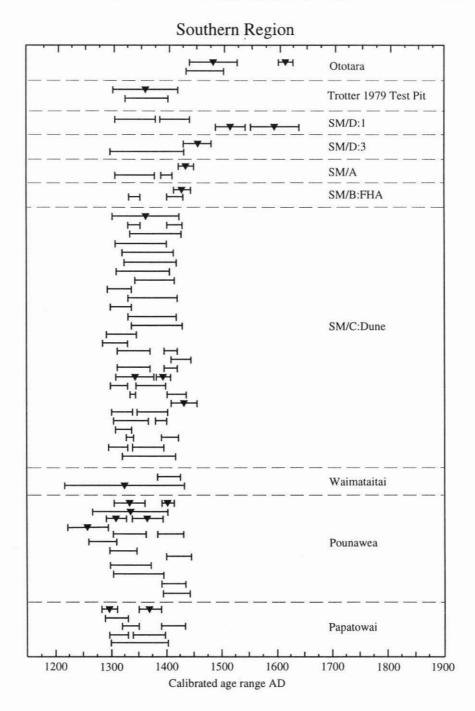
#### RESULTS

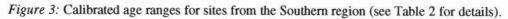
The <sup>14</sup>C determinations remaining following application of the discard protocol were calibrated with OxCal v3.0 using the intercepts method (Bronk-Ramsey 1995, 1998). For terrestrial samples,  $27\pm5$  years was subtracted from the conventional radiocarbon age to allow for the southern hemispheric offset (McCormac *et al.* 1998). The decadal curve (Stuiver *et al.* 1998) was used to calibrate results on moa bone collagen and identified charcoal. Shell results were calibrated using the marine curve of Stuiver, Reimer and Braziunas (1998) with  $\Delta R$  set at  $-25 \pm 15$  years (Higham and Hogg 1995).

A total of 11 sites (Fig. 1) with bone and matching charcoal and/or marine shell determinations, out of 46 sites with bone results, remain after application of the discard protocol. The calibrated <sup>14</sup>C results are shown in Table 2 and Figures 2 and 3.

These results were evaluated using OxCal combine probabilities method (Bronk-Ramsey 1995). Using this method, <sup>14</sup>C determinations are calibrated, combined, and then assessed in the light of the combined data. This enables uncertainties with all calibration curves to be taken into account and allows direct combinations of radiocarbon determinations on material from different reservoirs (e.g., marine *versus* terrestrial). An agreement index is calculated which should not fall below 60.0% (<A'c) (an unaltered index = 100%), but can be tested by calculating an overall agreement index for all determinations. A<sub>overall</sub> is the calculated agreement index and An is the value (dependant on n) below which it should not fall. If A<sub>overall</sub> falls below 60.0%, the combination of sample determinations should be questioned. In this paper, an overall agreement index was calculated for each sample type (e.g., marine shell, charcoal, and bone) and for the <sup>14</sup>C determinations for the site as a whole. Because of the untested nature of these bone results, bone, charcoal and marine shell determinations remaining after the discard protocol are only considered to be acceptable (at the level of precision encountered) when they are statistically identical and overlap with the calibrated age ranges of other sample types at one standard deviation (1 $\sigma$ ).

The overall agreement statistics for each of the 11 sites are given in Appendix 1. Bone determinations from the following sites are in overall agreement and overlap with results of associated sample types: Houhora, Shag River Mouth (Trotter's 1979 test pit, SM/B:FHA, SM/C:Dune and SM/D:3), Pounawea, Papatowai, Tumbledown Bay, Ototara and Waimataitai. A number of determinations are in poor agreement, or do not overlap with associated radiocarbon determinations. These include Cross Creek, Tairua, Avoca Point, Shag River Mouth (SM/A and SM/D:1), and Hamilton's deposit, Redcliffs.





# TABLE 2

#### BONE, CHARCOAL AND MARINE SHELL RADIOCARBON AGES FROM NEW ZEALAND ARCHAEOLOGICAL SITES REMAINING FOLLOWING APPLICATION OF DISCARD PROTOCOL

Site name	Lab no Run no.	Provenance	Sample type	CRA	Cal 95% (AD)
Mt Camel/ Houhora	NZ-5007	Layer 2c	Moa collagen	$563 \pm 56$	1328-1344 1394-1435
	NZ-5008	Layer 3b	Moa collagen	$585 \pm 46$	1325-1348 1391-1421
	NZA-2436	Layer 2b	Charcoal	$632 \pm 86$	1293-1417
	NZA-2437	Layer 2b	Charcoal	774 ± 87	1217-1299
	NZA-2438	Layer 3	Charcoal	727 ± 86	1261-1322
					1350-1390
	Wk-5485	Layer 3b	Charcoal	$640 \pm 40$	1300-1334
					1338-1373
					1378-1400
	Wk-5034	Layer 2b	Shell	$960 \pm 40$	1332-1420
	Wk-5035	Layer 2b	Shell	$1060 \pm 45$	1281-1327
Cross Creek	NZA-576	Layers 7,8,9	Moa collagen	751 ± 58	1262-1298
	NZ-6800	Layer 7	Shell	$1035 \pm 28$	1300-1332
Tairua	NZA-558	Layer 2, oven	Moa collagen	$460 \pm 55$	1431-1483
	Wk-5444	Layer 2	Shell	$1000 \pm 50$	1306-1404
	Wk-5445	Layer 2	Shell	$1090 \pm 50$	1250-1313
Avoca Point	NZ-4155	Cultural layer	Moa collagen	$703 \pm 85$	1276-1329
		Oven			1343-1396
	NZ-3164	Cultural layer	Moa collagen	$952 \pm 192$	900-919
		Oven			959-1282
	NZ-2719	Cultural layer	Shell	$1174 \pm 33$	1184-1264
	NZ-2718	Cultural layer	Shell	$1183 \pm 29$	1190-1244
	Wk-4000	Cultural layer	Eggshell	-	1280-1326†
					1347-1392
	Wk-4001	Cultural layer	Eggshell	-	1262-1302 <sup>†</sup>
					1370-1382
Redcliffs	NZ-1113	Occupation layer	Moa collagen	$701 \pm 60$	1282-1322
Hamilton's			1414 1211		1350-1390
Deposit	NZ-1111		Shell	$924 \pm 42$	1390-1440
Tumbledown	NZA-825	Layer 3 lower	Moa collagen	307 ± 85	1487-1670
Bay		· · · · · · · · · · · · · · · · · · ·		1999 - 1999 -	1780-1797
					1942-1945
	NZ-7656	Layer 3 hut site	Charcoal	$418 \pm 47$	1443-1518
					1596-1620
	NZ-7654	Layer 3	Shell	$706 \pm 50$	1513-1648
	NZ-7745	Layer 3 lower	Shell	$686 \pm 38$	1535-1653
	5004000 1050 S 1050	100000 10000 0000 00000000000000000000	2012/01/2012/2012		

Ototara	NZ-754 NZ-560	Occupation Layer	Moa collagen Shell	435 ± 70 838 ± 59	1434-1520 1593-1624 1429-1498
Shag River Mouth: SM/C:Dune	NZA-781	Layer 2	Moa collagen	630 ± 82	1294-1416
	NZ-7758	Layer 2	Charcoal	$580 \pm 47$	1326-1347 1392-1424
	Wk-2751	Layer 4	Shell	960 ± 45	1329-1423
	Wk-2410	Layer 4	Shell	1020 ±50	1298-1392
	Wk-2411	Layer 4	Shell	990 ± 45	1312-1407
	Wk-2412	Layer 4	Shell	980 ± 45	1318-1412
	Wk-2362	Layer 4	Shell	$1010 \pm 50$	1302-1398
	NZ-7805	Layer 4	Shell	965 ± 26	1336-1411
	Wk-2508	Layer 4	Shell	1060 ±45	1281-1327
	Wk-2632	Layer 4	Shell	980 ± 40	1321-1410
	Wk-2752	Layer 4	Shell	1040 ±45	1291-1338
	Wk-2856	Layer 4	Shell	$980 \pm 40$	1321-1410
	Wk-2857	Layer 4	Shell	950 ± 45	1334-1428
	Wk-2440	Layer 4	Shell	$1050 \pm 50$	1284-1335
	Wk-2441	Layer 4	Shell	1070 ±45	1273-1322
	NZ-7761	Layer 4	Charcoal	$600 \pm 50$	1306-1365 1386-1416
	NZ-7757	Layer 5	Charcoal	537 ± 44	1404-1438
	Wk-2416	Layer 5	Eggshell	$600 \pm 50$	1306-1365 1386-1416
	NZ-7736	Layer 6	Moa collagen	624 ± 58	1300-1374 1378-1410
	NZ-7756	Layer 6	Charcoal	$670 \pm 47$	1293-1328 1344-1394
	Wk-2417	Layer 6	Eggshell	$560 \pm 45$	1331-1341 1398-1433
	NZA-780	Layer 7	Moa collagen	509 ± 72	1404-1452

Petchey: A radiocarbon discard protocol for bone

	NZ-7755	Layer 7	Charcoal	646 ± 47	1298-1334 1338-1400
	Wk-2589	Layer 7	Charcoal	630 ± 35	1302-1369 1383-1402
	NZ-7806	Layer 7	Shell	1022 ±29	1305-1341
	Wk-2604	Layer 8	Eggshell	570 ± 45	1329-1343 1395-1428
	NZ-7771	Layer 11	Charcoal	660 ± 46	1296-1330 1342-1397
	NZA-1175	Layer 11	Shell	974 ± 49	1320-1417
Shag River Mouth: SM/D:1	NZA-888	Layer 3	Charcoal	585 ± 93	1300-1372 1380-1438
	NZ-7739	Layer 5	Moa collagen	370 ± 38	1480-1532 1541-1636
Shag River Mouth: SM/D:3	NZ-7740	Layer 2	Moa collagen	477 ± 53	1424-1474
	NZA-887	Layer 2	Charcoal	626 ± 95	1292-1425
Shag River Mouth: SM/A	NZ-7741	Layer 2	Moa collagen	522 ± 37	1411-1440
	NZ-7759	Layer 2	Charcoal	$627 \pm 40$	1302-1370 1382-1404
Shag River Mouth SM/B:FHA	NZ-7742	Layer 2	Moa collagen	530 ± 36	1408-1438
	NZ-7760	Layer 2	Charcoal	582 ± 47	1326-1348 1392-1424
Shag River Mouth:	NZ-7760 NZ-5016	Layer 2 Ashy layer below midden	Charcoal Moa collagen	582 ± 47 641 ± 85	
		Ashy layer	Moa		1392-1424

	NZ-5015		Moa collagen	686 ±173	1218-1433
Pounawea	NZ-4438	Lowest layer	Moa collagen	602 ± 47	1306-1365 1387-1414
	NZ-1796	Lowest layer	Moa collagen	699 ±105	1264-1402
	NZ-1797	Lowest layer	Moa collagen	668 ± 60	1289-1331 1341-1398
	NZ-1798	Lowest layer, base of oven	Moa collagen	772 ± 66	1223-1296
	NZ-5031	Layer 1, Sq. H21	Charcoal	582 ± 77	1304-1368 1384-1436
	NZ-1864	Lower layer	Shell	1095 ±41	1254-1307
	NZ-1867	Lower layer	Shell	1028 ±41	1298-1349
	NZ-1868	Middle layer	Shell	905 ± 41	1402-1448
	NZ-1869	Middle layer	Shell	1025 ±41	1300-1363
	NZ-1870	Lower layer	Shell	1012 ±41	1305-1392
	NZ-1871	Lower layer	Shell	926 ± 41	1390-1439
	NZ-1872	Lower layer	Shell	919 ± 41	1394-1442
Papatowai	NZ-137 NZ-2688 R192/1A	Middle layer	Moa "fixed carbon"	707 ± 61	1280-1318 1352-1388
	NZ-1333	Bottom layer	Shell	1051 ±45	1286-1332
	NZA-1415	Upper shell layer	Charcoal	570 ± 66	1324-1350 1390-1436
	Wk-1761	Lower cultural layer	Charcoal	$650 \pm 45$	1298-1332 1340-1399
	Wk-1762	Lower cultural layer	Charcoal	640 ± 45	1299-1402

<sup>†</sup> Unpublished data courtesy Dr. T.F.G. Higham.

#### DISCUSSION

All bone determinations from these sites are of an acid or an acid/alkali washed fraction. Given that all remaining determinations could have 10 to >15% contamination, those samples which were of poorer preservation are likely to be clearly erroneous. It should, therefore, be possible to evaluate under which conditions problematic bone determinations may be encountered. Factors which influence collagen preservation vary from site to site but generally they depend on biological activity, temperature, pH of the surrounding matrix, the presence of water in the immediate environment and drainage (Linse 1992; Hedges and Millard 1995). Consequently, soil conditions, climate and cultural influences play a major role in the survival of bone (Henderson 1987: 46), and may be more important than age. Unfortunately, much of this information is not available for the sites listed in Table 2. Regional climatic data are given in Appendix 2.

Statistical variation may also play a part in explaining some of the smaller discrepancies identified between the bone determinations and more reliable sample types. Van Klinken and Hedges (1998), for instance, have suggested that a shift in age of  $1-2\sigma$  between identical repeat dated samples may be due to the effects of contamination or inbuilt age (growth age or storage age), or might reflect simple statistical variation in a Gaussian distribution. A shift in age of  $3\sigma$  or greater is a clearer indication of contamination. Difficulty in reliably assessing the accuracy of many of the determinations discussed below is further exacerbated by larger standard errors.

The agreement between the acceptable moa bone determinations and other sample types from Shag River Mouth, SM/C:Dune suggests that the near neutral pH (6.77–8.35) of the soil matrix (Anderson, Worthy and McGovern-Wilson 1996: 208, table 14.6), low rainfall and temperature (508–635 mm and 10.6–11.1°C) (Appendix 2), and good drainage of the sand dune were conducive to long-term survival of bones. This conclusion is also supported by recent research into fish bone remains from this area (Petchey 1998; Petchey and Higham in press). Similar conditions are expected for Trotter's 1979 test pit located on the southern margin of the Shag River Mouth site (Anderson and Smith 1996: 9).

The remaining areas at Shag River Mouth (SM/D:1, SM/A, SMD:3 and SMB:FHA) are a different matter. Bone determinations from both SM/D:1 and SM/A are not in overall agreement with associated samples, and although results from SM/D:3 and SM/B:FHA are, they are considered to be suspect. First, all four areas have only one other determination for comparison, limiting assessment of accuracy. Second, bone collagen results from SM/D:3 and SM/B:FHA point to a later occupation than the fourteenth century suggested by Anderson, Smith and Higham (1996: 67) for the site as a whole. Third, the sample from SM/D:3 was collected from the base of an oven (Smith 1996: 57-58) and it is possible that it may have been burnt. Indeed, a higher degree of burning was noted at this location, with just under 50% of bone blackened (considered to be the result of being buried in oven rakeout), and just under 10% completely calcined (Anderson, Worthy and McGovern-Wilson 1996: 210). Fourth, SM/A, SM/D:3 and SM/B are close to the Salicornia mud flats (Allingham and Anderson 1996: 35, figure 4.1; Smith 1996: 51), where bone preservation may have been influenced by a higher water table. Consequently, bone from these areas may have been of poorer preservation despite all submitted bone samples being robust and relatively dense, with no obvious visual sign of weathering (I. Smith, pers. comm. 30/6/1997). The possibility of poor preservation is supported by Anderson, Worthy and McGovern-Wilson's (1996: 207-09) observation that 51% of the bones from SM/D:3 belonged to Weathering Stage 3 (Anderson, Worthy and McGovern-Wilson 1996: 207, 209,

210)<sup>5</sup>. According to Anderson, Worthy and McGovern-Wilson (1996: 207, 209, 210), SM/A and SM/B:FHA are also characterised by a higher degree of weathering and burning compared to material from the dunes. Similarly, SM/D:1 is located on the edge of a waveeroded bank, close to the mud flats (Smith 1996: 52). Anderson, Worthy and McGovern-Wilson (1996: 207–09) suggest, however, that bone weathering in this area was limited and the matrix of SM/D:1 was similar to the high dunes. This is not supported by the large sample of bone required for radiocarbon measurement, which implies lower carbon content, and therefore possible poorer preservation for this sample (i.e., 349.18 g compared to around 200 g [see Anderson, Smith and Higham 1996] for other conventional collagen determinations from Shag Mouth with comparable standard errors). Overall, this suggests that the hand specimen identification techniques used in the selection of bone may not have been sufficient to detect problem samples (cf. Nicholson 1996, 1998; Petchey 1998; Petchey and Higham in press). Clearly, the variation in preservation state suggested for bone from different areas of Shag River Mouth requires further investigation.

The main cultural contexts at Waimataitai Mouth were also continually damp, being only approximately 5 cm above the water level of the lagoon (Trotter 1955: 296). It is possible, therefore, that this sample was of poorer preservation, despite the apparent acceptability of the overall agreement index. This is difficult to assess given the limited number of radiocarbon determinations involved.

There are two moa bone collagen results for Avoca Point, NZ-4155 and NZ-3164. Unfortunately, NZ-3164 has a very large standard error, making any conclusion regarding accuracy problematic. Again, poor preservation may be an issue, as the occupation layer at Avoca Point is located at the edge of swampy ground into which material had been deliberately dumped at the time of occupation (Trotter 1980a: 281, 283). In addition, at the eastern end of the site there was a natural accumulation of faunal remains. Therefore, although the moa bones collected for analysis came from squares several metres away from the natural accumulations and swamp deposits (Trotter 1980a: 283), it should be kept in mind that poorly preserved or sub-fossil material may have been selected.

Similarly, swampy conditions on Redcliffs flat (Trotter 1975c: 190) may be responsible for the discrepancy in calendrical ages between moa collagen and shell determinations. In addition, the possibility of disturbance at the site cannot be ruled out, because both samples were collected in 1966 from the approximate location of a previous excavation (Trotter 1975c: 192, 197).

Papatowai and Pounawea are located in a high annual rainfall and low temperature region (Appendix 2). Generally, both sites appear to be well drained, though the lowest layers at Pounawea, from where all bone samples were collected, lie below the high tide mark (Lockerbie 1959: 82). Radiocarbon determinations of all three sample types dated at Pounawea support Hamel's (1980: 16) suggestion of a short period of occupation some time in the fourteenth century. There is no indication of a sequence that may support multiple occupation, as implied by Anderson (1991: 787), who suggested that only the basal layer of Pounawea was inhabited prior to the thirteenth century. The sequence of <sup>14</sup>C determinations from Papatowai also supports Anderson and Smith's (1992: 151) suggestion of a short period of occupation in the fourteenth century. It would appear, therefore, that the

<sup>&</sup>lt;sup>5</sup> Stage 1 being unweathered bone, while Stage 5 bone is on the point of disintegration (Anderson, Worthy and McGovern-Wilson 1996: 207).

bone from these sites was well enough preserved for the influence of contamination to be minor.

Further north, radiocarbon results of bone from Cross Creek and Tairua are suspect. The various determinations from Tairua are in poor agreement, and the single bone determination from Cross Creek does not overlap at  $1\sigma$  with the calibrated result of the contemporary shell sample (NZ-6800). The Tairua bone calibrated ages are also young, suggesting a fifteenth century occupation, compared to the shell results which indicate a late thirteenth to early fourteenth century occupation (Schmidt and Higham 1998). At Cross Creek, the slightly older moa determination could support Anderson's (1989: 111–12) suggestion that the moa bone had been imported for industrial purposes. Because Cross Creek and Tairua are located in regions with high annual rainfall and temperature (see Appendix 2), however, it is likely that these bone samples were of 'poor' preservation and the erroneous <sup>14</sup>C determinations are the result of contamination left behind after the acid wash (see Petchey 1998).

Although the radiocarbon determinations from Houhora are acceptable at the level of precision encountered (Appendix 1), the moa collagen determinations are in poor agreement with other sample types (A=52.1%, <A'c= 60.0%). Again, this may be a result of insufficient pretreatment and poorer bone preservation caused by higher annual temperatures and rainfall at the top of the North Island (Appendix 2).

The remaining sites of Tumbledown Bay and Ototara are both young, occupied around 400 to 300 years ago. Because ten percent contamination is unlikely to be statistically significant in either of these cases, the bone determinations (NZA-825 and NZ-754 respectively) cannot be adequately assessed at this level of precision. The fifteenth century result for Ototara is, however, in keeping with Trotter's (1965: 113) description of the site as having remains intermediate between "typical" Archaic and Classic Maori. The location of the Ototara midden in a low rainfall and temperature region (508–635 mm and 10.6–11.1°C) (see Appendix 2) under a limestone overhang (Trotter 1965: 109), also suggests that this should be an accurate radiocarbon measurement. However, there is some doubt whether NZ-754 dates a butchered moa since, according to Trotter (1965), there is evidence of industrial bone at the site. Similarly, Tumbledown Bay is located in a well drained sand dune environment (FRF), in a region of low annual rainfall and temperature (635–762 mm and 11.1–11.7°C) (see Appendix 2). Collagen determinations from this site should therefore be reliable.

# CONCLUSIONS AND RECOMMENDATIONS

The results presented in this paper suggest that bone dating in New Zealand has been hampered by a number of variables, rather than being inherently unreliable. First, there have been no, or few, comprehensive tests of bone pretreatment, species reliability or the influence of contamination. Second, confusion has arisen about the effectiveness of the varied radiocarbon pretreatments available, in part because of the complexity of some methods. This has been compounded by the incorrect reporting of fractions isolated for <sup>14</sup>C measurement. Third, inadequate sample selection procedures have resulted in burnt bone, sub-fossil bone and severely degraded bone, each with intrinsically different chemistries, being submitted for radiocarbon assay. Fourth, insecure provenance of samples or associated samples to be dated mean that few comparisons of bone reliability could have been, or can be, made. Fifth, publication of results and procedures have been limited or incorrect. Finally,

there has been limited research into the radiocarbon measurement of bone in New Zealand because of preconceived ideas about the reliability of bone determinations.

- Although comprehensive tests of bone from sites with multiple <sup>14</sup>C determinations are necessary before the reliability of bone can be adequately assessed, the following conclusions can be drawn from this review.
- Moa bone protein can be dated by radiocarbon, but preservation state and pretreatment inadequacies are responsible for discrepancies with many radiocarbon determinations.
- An acid or acid/alkali treatment does not remove sufficient contamination in most bone determinations, except where the bone is well preserved.
- Such well preserved bone appears to be more common in well drained sites in regions
  that have low average rainfall and temperature. Therefore, bone preservation is likely
  to be better in sites located along the east coast of the South Island.
- Sites located in or near swamps or in high temperature and rainfall areas of the North Island may yield bone of poorer preservation.
- All bone samples should be carefully assessed by one or preferably more techniques such as amino acid, infrared, percentage gelatin yield or whole bone N% in order to choose an appropriate pretreatment.
- Hand specimen selection alone is not a suitable method for choosing samples for radiocarbon analysis, and can be misleading.
- · Identification of sub-fossil moa bone may be difficult.
- Gelatinisation following an alkali wash should remove better than 8% contamination in bone with >40% collagen remaining.
- Bones of poor or intermediate preservation state (<20% collagen) may require additional purification.
- Potential anomalies caused by the dietary and habitat peculiarities of some species also need to be carefully considered, especially when dealing with novel sample types.

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# **APPENDIX 1**

# STATISTICAL COMPARISON OF <sup>14</sup>C DETERMINATIONS FROM SITES FOLLOWING APPLICATION OF THE DISCARD PROTOCOL

Site	Agreement Index	Combined calibrated age range at $1\sigma$
Houhora	$A_{overall} = 60.1\%$ (An = 40.8%, n=3)	AD 1315-1332 and 1340- 1364
Cross Creek	$A_{overall} = 79.9\%$ (An = 50.0%, n=2)	AD 1283-1325 and 1372- 1379
Tairua	$A_{overall} = 15.8\%$ (An = 50.0%, n=2)	-
Avoca Pt	$A_{overall} = 47.7\%$ (An = 40.8%, n=3)	
Hamilton's deposit	$A_{overall} = 83.6\%$ (An = 50.0%, n=2)	AD 1351-1395
Tumbledown Bay	$A_{overall} = 98.0\%$ (An = 40.8%, n=3)	AD 1564-1628
Ototara	$A_{overall} = 128.5\%$ (An = 50.0%, n=2)	AD 1436-1494
Test pit, Trotter (1979)	$A_{overall} = 113.1\%$ (An = 50.0%, n=2)	AD 1324-1390
SM/A	$A_{overall} = 53.2\%$ (An = 50.0%, n=2)	
SM/B:FHA	$A_{overall} = 108.1\%$ (An = 50.0%, n=2)	AD 1404-1431
SM/C:Dune	$A_{overall} = 135.9\%$ (An = 35.4%, n=4)	AD 1330-1341
SM/D:1	$A_{overall} = 28.0\%$ (An = 50.0%, n=2)	-
SM/D:3	$A_{overall} = 79.2\%$ (An = 50.0%, n=2)	AD 1404-1448
Waimataitai Mouth	$A_{overall} = 124.1\%$ (An = 50.0%, n=2)	AD 1358-1422
Pounawea	$A_{overall} = 99.4\%$ (An = 40.8%, n=3)	AD 1346-1372 and 1380- 1396
Papatowai	$A_{overall} = 107.5\%$ (An = 40.8%, n=3)	AD 1300-1330 and 1348- 1356

## **APPENDIX 2**

# CLIMATIC INFORMATION FOR SITES WITH BONE DETERMINATIONS FOLLOWING APPLICATION OF DISCARD PROTOCOL

Site	Annual rainfall (mm) <sup>,</sup>	Mean annual temperature (°C) <sup>,</sup>
Houhora	1016-1270	14.4-15.0
Cross Creek	1270-1524	13.9-14.4
Tairua	1524-2032	13.9-14.4
Avoca Point	1016-1270	11.7-12.2
Redcliffs	635-762	11.1-11.7
Tumbledown Bay	635-762	11.1-11.7
Ototara	508-635	10.6-11.1
Shag River Mouth	508-635	10.6-11.1
Waimataitai Mouth	508-635	10.6-11.1
Pounawea	1270-1524	9.4-10
Papatowai	1016-1270	9.4-10

<sup>†</sup>Rainfall and temperature at sea level (from McLintock 1960:map 8).

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