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STUDY OF BONE RADIOCARBON DATING ACCURACY AT THE UNIVERSITY OF ARIZONA NSF ACCELERATOR FACILITY FOR RADIOISOTOPE ANALYSIS

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INTRODUCTION

Bone would seem to be an ideal material for ^{14}C dating because this calcified tissue contains 20 weight per cent protein. Fossil bone, however, can lose most of its original organic matter and frequently contains contaminants having different ^{14}C ages. Numerous ^{14}C dates on bone have been available to archaeologists and geologists but many age determinations have been inaccurate despite over 30 years of research in the field following the first ^{14}C age determinations on bone (Arnold & Libby, 1951). This situation remained unchanged until simple pretreatments were abandoned and more bone-specific fractions were isolated. The ideal solution is to use accelerator mass spectrometer ^{14}C dating, which facilitates the use of milligram-sized amounts of highly purified compounds—an approach impossible to pursue using conventional ^{14}C decay-counting methods.

OBJECTIVES

Our principal objective was to determine how bone ^{14}C dates could be made more accurate. Our goal was to improve sample pretreatment chemistry and use TAMS technology to date milligram-sized, highly purified bone constituents. The research was part of a larger study that used stable and ^{14}C isotopes from fossil bones for chronologic, paleoenvironmental, and paleoecologic determinations (Stafford, 1984; Stafford *et al*, 1985).

A secondary objective was to date several bones of unknown age that exhibited a wide range of preservation. The unknown-age fossils provided additional data on the range of ages that would be obtained from various chemical fractions. The accuracy of dates on these bones could be evaluated by knowing whether or not the same fraction dated accurately from the known-age mammoths.

PRESENT KNOWLEDGE OF BONE ^{14}C DATING

Bone is not usually recommended for ^{14}C dating (Libby, 1955; Olson, 1963) because its ^{14}C ages are either discordant with associated charcoal dates or ages for different fractions from the bone are discordant with each other. Numerous methods have been devised to pretreat fossil bones (Olsson *et al*, 1974; El-Daoushy, Olsson & Oro, 1978; Taylor, 1982) but all techniques are minor modifications on methods used to extract either inor-

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ganic carbon (bone apatite carbonate) or organic carbon (bone protein) for ^{14}C dating.

The first bone ^{14}C dates were on total carbon from naturally burned (Arnold & Libby, 1951) and unburned bone (de Vries, 1959). ^{14}C dates on organic fractions used the HCl-insoluble residue from artificially pyrolyzed bone (May, 1955) and later that residue treated with NaOH (Vogel & Waterbolk, 1963). Bone protein (approximately collagen) was extracted by HCl decalcification (Münnich, 1957; Olsson, 1959; Berger, Horney & Libby, 1964; Krueger, 1965; Tamers & Pearson, 1965), with a chelating agent such as EDTA (Berger, Horney & Libby, 1964; Olsson *et al,* 1974; El-Daoushy, Olsson & Oro, 1978) or rarely with H_2SO_4 (Sato *et al,* 1969). Decalcification of bone yields a "weak-acid insoluble residue" that was often contaminated with humates (Vogel & Waterbolk, 1963). Methods to remove humic and fulvic acids from collagen include either NaOH treatment (Berger & Libby, 1966; Haynes, 1967a) or conversion of the collagen to gelatin (Sinex & Farris, 1959; Longin, 1971). The use of both NaOH-leaching of collagen and gelatin extraction was introduced by Protsch (1975). The most rigorous methods for isolating organic carbon fractions are the chromatographic extraction of total collagen-derived amino acids (Ho, Marcus & Berger, 1969) and the isolation of individual amino acids as hydroxyproline and proline (Wand, 1981; Stafford *et al,* 1982; Gillespie & Hedges, 1983; Gillespie, Hedges & Wand, 1984).

Inorganic carbon from fossil bone has been isolated by either acid hydrolysis of untreated bone (Olsson, 1959) or from bone pretreated with acetic acid (Haynes, 1968) or triammonium acetate (Hassan, Termine & Haynes, 1977), two reagents that are used to remove secondary carbonate contamination. Additional techniques for preparing bone carbonate include sequential HCl hydrolysis (Haynes, 1968; Hassan, Termine & Haynes, 1977; Sullivan & Krueger, 1981) and differential thermal release of CO_2 (Haas & Banewicz, 1980).

Many of the inaccurate ages on fossil bone were due to the chemical heterogeneity of the dated fractions. The acid-insoluble residues retain humate contamination that is not removed by any of the described methods. Fortunately, the organic phase is amenable to chemical processing that is specific to the isolation of humates and specific peptides and amino acids. In contrast, inorganic carbon in fossil bones can exchange with environmental carbonate (Hassan, Termine & Haynes, 1977) and it is uncertain whether or not pretreatment methods yield an uncontaminated carbonate phase (Sullivan & Krueger, 1981; Schoeninger & DeNiro, 1982; Haas & Banewicz, 1980). Because no mechanisms are currently known for bone proteins to exchange carbon after burial, we emphasized the dating of the bone's organic phases, which were considered to have the greatest potential for purification and retention of their original ^{14}C integrity.

The following experiments evaluate the efficacy of bone dating by accelerator mass spectrometry. We have evaluated both pre-existing and new chemical procedures and make recommendations for testing the accuracy of ^{14}C dates on bone.

METHODS

Experimental Design

Fifty-eight ^{14}C dates were determined on fractions from 11 fossil bone specimens. Thirteen dates were made on charcoal, shell, or pedogenic carbonates that were associated with fossil bones. Three known-age mammoth bones from Clovis-culture archaeologic sites were initially dated to determine which of several possible fractions would be most reliable for bone ^{14}C dating.

Bone samples of unknown age were chosen for dating because they represented fossils with a range of geologic ages, preservation, and depositional environments. The charcoal and shell samples were dated because they were relevant to interpreting the accuracy of uranium series ages (Bischoff & Rosenbauer, 1981) and ^{14}C dates (Bada *et al,* 1984) on human bone from the Del Mar Man site.

The three known-age bones were all mammoth (*Mammuthus* sp) specimens that were from Clovis Indian mammoth-kill sites that date between 11,000 and 11,500 yr BP (Haynes, 1982). The elephants are independently dated with ^{14}C ages on associated wood or charcoal. The Domebo mammoth was used for the most extensive experimentation and those results were used in determining which fractions would be most suitable for dating from the Dent and Escapule mammoths. Both the Domebo and Dent mammoths had collagen-like amino-acid compositions and contained 0.7%N and 0.8%N, respectively. In contrast, the Escapule mammoth had a non-collagen amino acid composition and contained 0.08%N.

Bones of unknown ages and good to extremely good collagen preservation were a Wisconsinan-age whale preserved in permafrost (Beaufort Sea coast whale), human calvaria from an arid cave (Wilsall/Anzick series), and a horse ramus from a hyper-arid cave (Fishbone Cave series). Humid-cave depositional environments were represented by bird and rodent post-cranial bones from the Puu Naio Lava Tube series, which included specimens with good to very poor collagen preservation. Human bones that had poor to extremely poor protein preservation and which were from leached and oxidized sediments comprised fossils from the Yuha, La Jolla shores, and Wilson-Leonard series. A list of these dates is presented below.

Chemical Pretreatment of Bone

The chemical pretreatment methods used for the bones are summarized in Figures 1 and 2.

Fossil bones are washed in tap water to remove sediments and broken into 1 to 3cm fragments that are ultrasonically cleaned in tap water and distilled water. Physically cleaned bone is ground to $<63\mu m$ or left intact if grinding losses must be avoided. Inorganic carbon is extracted from the OH-apatite phase by hydrolyzing bone powder with 95% H_3PO_4. The bone powder is either untreated or it is extracted for 24hr with 1M acetic acid under line vacuum.

Organic carbon phases are concentrated by decalcifying bone powder in 4°C, 0.6N distilled HCl. The acid insoluble, collagenous residue is sepa-

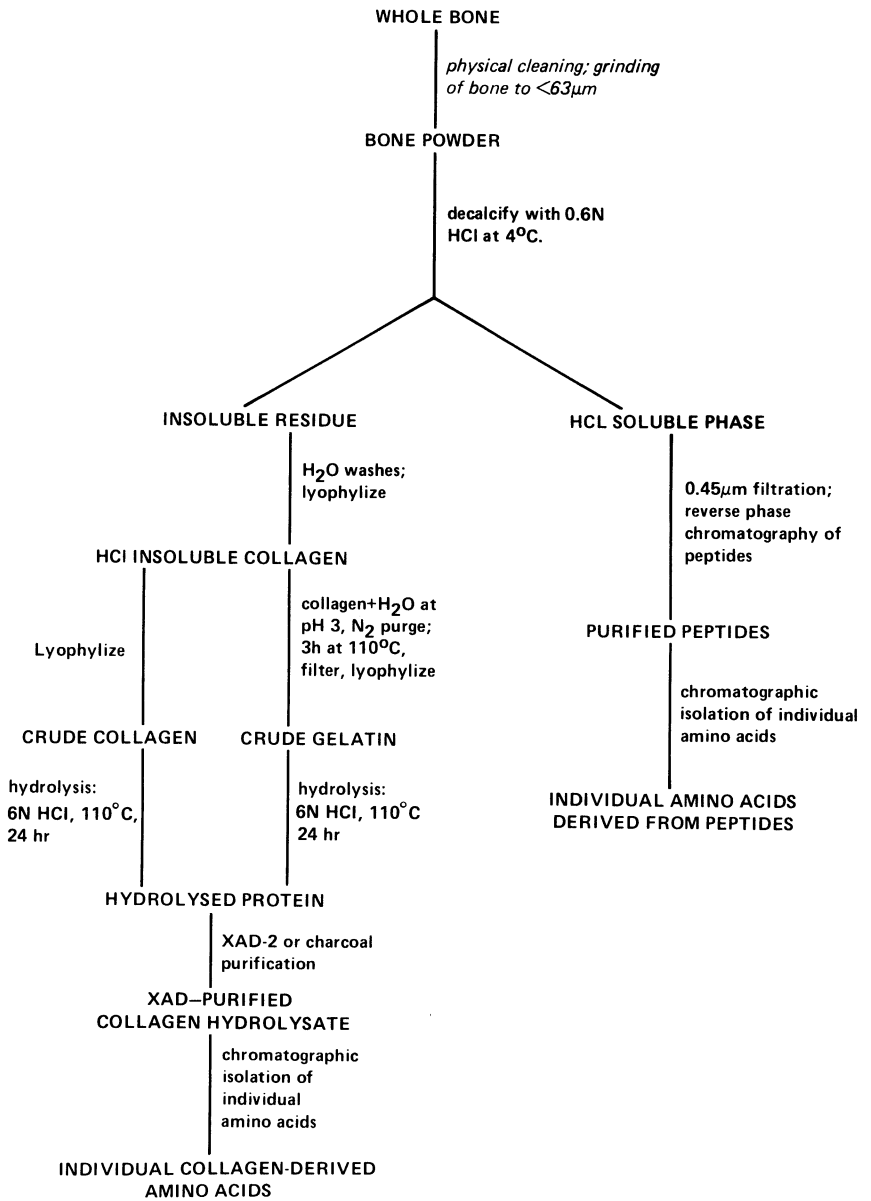

Fig 1. Flow diagram showing pretreatment methods used for ^{14}C dating fossil bones. Well- to moderately-well-preserved bones require the extraction of gelatin or XAD-treated gelatin, whereas the minimum pretreatment for poorly-preserved bones comprises the XAD-treatment of hydrolyzed protein and preferably the isolation of individual amino acids.

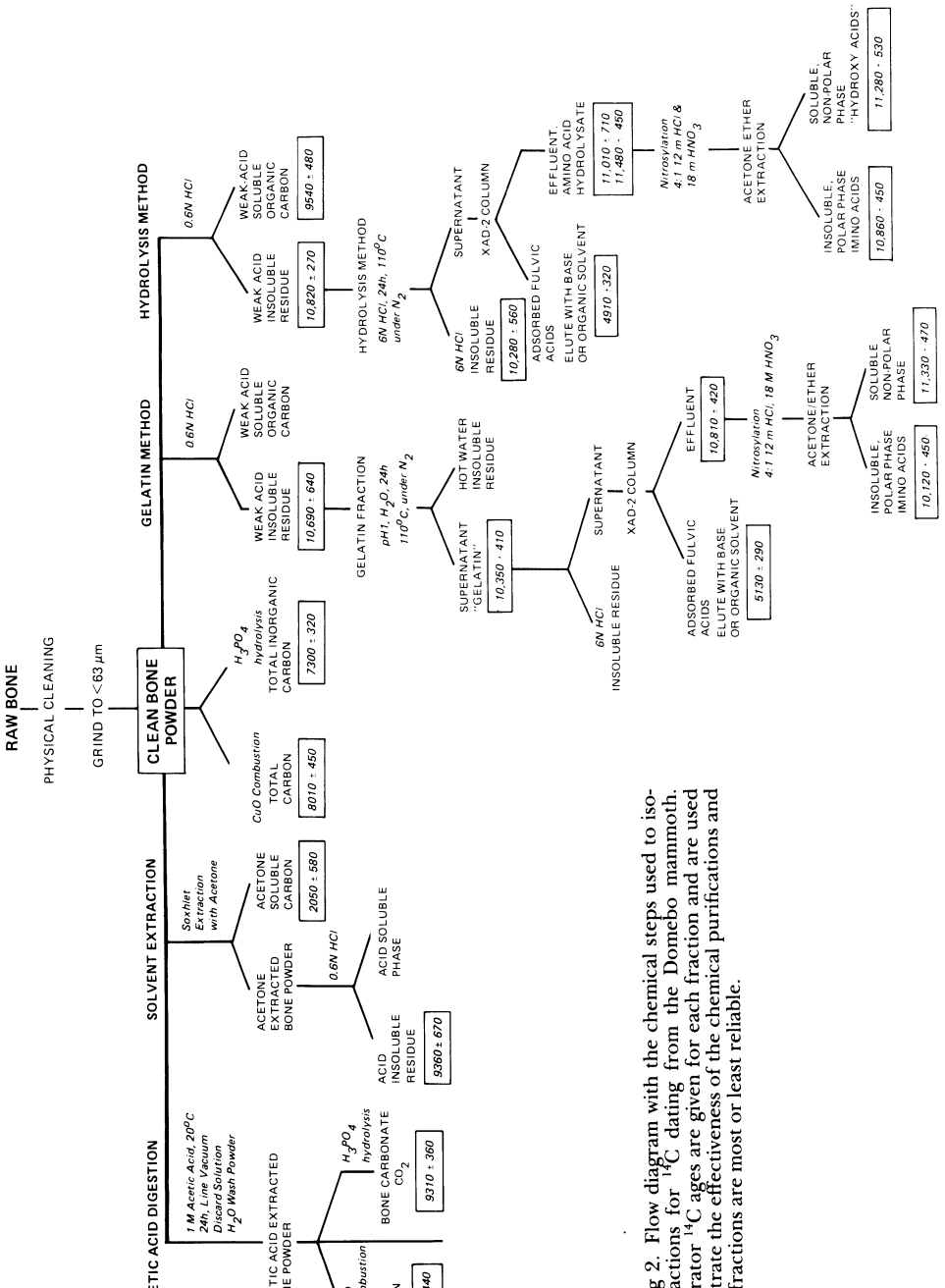

Fig 2. Flow diagram with the chemical steps used to isolate fractions for ^{14}C dating from the Domebo mammoth. Accelerator ^{14}C ages are given for each fraction and are used to illustrate the effectiveness of the chemical purifications and which fractions are most or least reliable.

rated by centrifugation from the acid soluble phase. The acid-soluble fraction is filtered through 0.45μm teflon Millipore membranes and rotary evaporated. The acid-soluble phase can be further purified by passing it through XAD resin, which is used to remove fulvic acids (Stafford *et al*, 1982). The acid-insoluble collagen is lyophylized, then hydrolyzed or converted to gelatin. The protein is hydrolyzed by heating ca 10mg of protein per 1ml distilled 6N HCl for 24hr at 110°C. Teflon-sealed tubes purged with nitrogen are used for the hydrolysis. The hydrolyzate solution is filtered before it is passed over XAD.

Gelatin is extracted from the weak-acid insoluble residue by heating ca 10mg protein and 10ml pH 3 water at 90°C for 3 to 4hr. The hydrolysis tubes are purged with nitrogen prior to sealing. The gelatin solution is centrifuged and filtered before it is lyophylized. The freeze-dried gelatin is hydrolyzed and purified with XAD.

Fulvic acids are removed from the gelatin and collagen hydrolyzates by passing the 6N HCl through a column of XAD resin. From 10 to 50ml of hydrolyzate is passed through a 1X40cm glass column of 20–50 mesh XAD-2 resin. To pretreat the resin for use, ca 500g Sigma Chemical Company XAD-2 resin are washed exhaustively with acetone, methanol, and water before the resin is extracted 3 alternating times with 3N HCl and 3M NaOH. The resin is washed finally with 1N HCl. A bed of resin 20 to 30cm high is poured, capped with glass wool, and equilibrated with 3 bed volumes 6N HCl. The protein hydrolyzate is passed through the resin at 100μl/min or at a flow rate slow enough to adsorb the fulvic acids in the upper 1/3 of the resin bed. The XAD-purified hydrolyzates are filtered and rotary evaporated. Fulvic acids separated from the hydrolyzed protein can be eluted from the resin by washing the resin with distilled water until the eluate pH is between 1 and 2. A 1M NH_4OH solution is used to desorb the fulvic acids, which are immediately acidified with HCl before drying and combustion.

Target Preparation

After isolation of the desired organic fraction, the sample was combusted to CO_2, which was reduced over magnesium to amorphous carbon. One to 2mg carbon was fused with iron powder to form an iron carbide bead that is mounted into a sample wheel for accelerator dating (Jull, Donahue & Zabel, 1983). The purity of CO_2 was increased by combusting the sample with 2g CuO powder that contained 10wt % Ag powder. The oxidant was prepared by mixing AgO powder with CuO powder that had been combusted for 1hr at 800°C. The AgO/CuO powder mixture was finally fired at 600°C for 1hr. The CO_2 gas from one sample, AA-312C, was converted directly to graphitic carbon using the methods of Jull *et al* (1986) and Vogel *et al* (1984).

Radiocarbon Measurement

The $^{14}C/^{13}C$ ratios were measured by TAMS at the University of Arizona. Sample carbon was converted to Fe-C targets according to Jull, Donahue and Zabel (1983). The targets were mounted in a 6-position target wheel, which was placed into the cesium-sputter ion source. Measurements

were made as a series of 4 cycles around the wheel, which contained 4 unknowns, 1 modern standard, and a background sample. For each cycle, a single measurement comprised 15 samplings of ^{14}C for 50 sec and of ^{13}C for 10 sec. Details of the procedure are given in Donahue *et al* (1983).

Achievable ^{14}C precision using the Fe-C targets was between ±150 and ±400 yr for samples <10,000 yr old. The wide variability in precision was caused by variations in the target's $^{12}C^-$ output, which ranged from 0.5 to 3μA (Jull, Donahue & Zabel, 1983). Dates reported here use a background of 2 ± 1% modern ^{14}C. This represents our best estimate of the background for these targets; thus, sample ages may differ slightly from previously published values (Stafford *et al,* 1984; Bada *et al,* 1984; Taylor *et al,* 1985).

All dates reported here were made between February and June 1984. Since then we have developed a procedure for making graphite targets with a modified Vogel *et al* (1984) method. When graphite is used we have been able to attain single-target precisions of ±1%, equivalent to an age precision of ±80 yr or better. Backgrounds for graphite targets are currently ca 0.6% modern ^{14}C. Results for this method of target preparation were reported previously (Jull *et al,* 1986; Linick *et al,* 1986).

Results

The ^{14}C dates are presented in Table 1, which includes only dates for the three known-age mammoths. The elephant ^{14}C dates are listed in order of age for each fraction. The fractions from the Domebo mammoth that were not accurate at 2σ were acetone-soluble organic carbon, fulvic acids, OH-apatite, total bone carbon, and acid insoluble collagen. Inaccurate Dent mammoth dates were from acid-insoluble collagen and gelatin, whereas all three fractions of the Escapule mammoth dated <11,000 yr. Epoxy preservative from the Escapule mammoth had an apparent ^{14}C age (3680 ± 210: GX-11261) that was significantly less than the geologic age of the mammoth.

DISCUSSION AND CONCLUSIONS

Our results show that the accuracy of bone ^{14}C dates depends on the preservation of the bone protein, upon which fractions are dated, and what contaminants are present. Humic and fulvic acids are the predominant contaminants in fossil bone. The degree to which humates affect a bone's ^{14}C age depends on the weight per cent of humates present and the apparent ^{14}C age of the humate fraction. Permafrost-derived and sometimes arid-cave-derived bones have extremely well-preserved collagen that is amenable to standard biochemical isolation techniques. If humates exist, they are usually in negligible amounts and are restricted to the bone's exterior. Although accurate ages can apparently be obtained often on acid-insoluble collagen and untreated gelatin from well-preserved bones, it is recommended that these fractions be purified with XAD resin, which will standardize procedures for humate removal.

Bones that have collagen-like compositions and >0.2% N should be dated using only certain fractions. Recommended fractions are either weak-acid-insoluble collagen or gelatin that is hydrolyzed and purified with XAD resin. Although isolation of individual amino acids may not always be

TABLE 1

Lab no.	Target no.	Sample description	Radiocarbon date (yr BP)
		Domebo Mammoth	
AA-822A	C-783	Acetone soluble organic carbon extracted from bone powder	2050 ± 580
AA-816	C-650	Fulvic acids from hydrolyzed 0.6N HCl insoluble collagen	4810 ± 760
AA-812	C-561	Fulvic acids from hydrolyzed 0.6N HCl insoluble collagen: NH_4OH elution	4910 ± 320
AA-819	C-771	Fulvic acids from hydrolyzed gelatin; acetone elution	5130 ± 290
AA-818	C-662	OH-apatite CO_2; untreated bone	7300 ± 320
AA-801	C-474B	Total inorganic + organic carbon from untreated bone powder	8010 ± 500
AA-815	C-615	OH-apatite CO_2 from acetic acid extracted bone powder	9310 ± 360
AA-822B	C-743	0.6N insoluble collagen extracted from acetone-extracted bone powder	9360 ± 670
AA-802A	C-477	0.6N HCl soluble phase from bone powder	9540 ± 480
AA-810	C-556B	Imino acids from XAD-2 purified hydrolyzed gelatin	10,120 ± 450
AA-802B	C-743	6N HCl insoluble residue from weak-acid insoluble collagen	10,280 ± 560
AA-803	C-480	Unpurified gelatin	10,350 ± 410
AA-814	C-606	0.6N HCl insoluble collagen	10,690 ± 640
AA-804	C-542	Total organic and inorganic carbon after HAc extraction of bone powder	10,760 ± 440
AA-805	C-543	XAD-purified hydrolyzed gelatin	10,810 ± 420
AA-824	C-1002	0.6N HCl insoluble collagen	10,820 ± 270
AA-811	C-559	Imino acids from XAD-purified 0.6N HCl insoluble collagen	10,860 ± 450
AA-806	C-544B	XAD-purified hydrolyzed collagen	11,010 ± 710
AA-808	C-555	Acetone/ether soluble α-amino acids from nitrosylated XAD-purified acid insoluble collagen	11,280 ± 530
AA-807	C-551	Acetone/ether soluble α-amino acids from XAD-purified hydrolyzed gelatin	11,330 ± 470
AA-825	C-1038	XAD-purified 0.6N HCl insoluble collagen	11,480 ± 450
AA-823	C-978	Elm tree stump associated with mammoth (Leonhardy & Anderson, 1966)	11,490 ± 450
		Dent Mammoth	
AA-830	C-1220	0.6N HCl insoluble collagen	8250 ± 520
AA-831	C-1221	Unpurified gelatin	9240 ± 350
AA-832	C-1261	XAD-purified collagen hydrolyzate	10,590 ± 500
AA-833	C-1267	XAD-purified gelatin hydrolyzate	10,950 ± 480
		Escapule Mammoth	
AA-834	C-1259	0.6N HCl insoluble residue from bone powder	8500 ± 470
AA-835	C-1275	Unpurified "gelatin"	5210 ± 270
AA-836	C-1358	XAD-purified, hydrolyzed insoluble residue	4610 ± 280

necessary, their extraction and dating is highly encouraged. HCl insoluble residues, untreated gelatin, and acid-soluble phases may occasionally yield accurate dates, but there are no known chemical criteria for predicting when dates will be spurious on these fractions.

Bones with non-collagen amino acid compositions and <0.2% N do

not date as accurately as bones with substantial amounts of collagen. Even XAD treatment may not be effective in yielding accurate ages on bones that are diagenetically altered. Contamination by exogenous amino acids and epoxy residues are the likely causes of the young ages for the Escapule mammoth's fractions. Accurate dates from degraded bone will probably require the exclusive dating of individual amino acids. The worst fractions to use from poorly preserved bone are weak-acid soluble and insoluble phases and any apatite fraction. Non-specific organic fractions should be used only when further pretreatment would lower carbon to sub-milligram levels and only when a minimum-age estimate is acceptable.

In summary, the fractions that should not be dated from bones are untreated weak-acid insoluble residues, weak-acid soluble phases, untreated gelatin, apatite carbonate, and humic or fulvic acids. XAD treatment to remove humates should become mandatory for acid insoluble collagen and gelatin from all bones. The isolation of individual amino acids is highly encouraged, especially for bones that have lost >90% of their original organic matter during diagenesis.

BONE RADIOCARBON DATES FROM THE ARIZONA NSF ACCELERATOR FACILITY

Beaufort Sea Coast series

Gray whale (*Eschrichtius* sp) rib from marine Flaxman Fm, 20km S of Beaufort Sea coast, Sec 14, T16N, R5W, Teshekpuk C-1 quad, Alaska (70° 44.78′ N, 153° 06.38′ W). Coll Sept 1, 1983 and subm by L D Carter, USGS, Anchorage, Alaska. Date will estimate age of Flaxman transgression.

AA-312A. Beaufort Sea coast **>26,700**

Gray whale rib. *Comment:* weak-HCl insoluble collagen. Target C-1035.

AA-312B. Beaufort Sea coast **>27,300**

Gray whale rib. *Comment:* gelatin phase from weak-HCl insoluble collagen used for AA-312A. Target C-1040.

AA-312C. Beaufort Sea coast **>38,000**

Gray whale rib. *Comment:* graphite made from gelatin fraction.

General Comment: extremely good bone preservation. Bone and its collagen have properties as of modern material. All dates significant at 2σ. (LDC): ^{14}C dates on assoc fossils: marine mollusk shells, 42,600 ± 1500 (USGS-1689), whalebone, 22,530 ± 260 (Beta-6108); seal bone, 19,640 ± 130 (USGS-1515); marine mollusk shells, 20,760 ± 210 (Beta-5869). Whale considered beyond range of ^{14}C dating.

Wilsall (Anzick) series

Homo sapiens sapiens partial calvaria from Wilsall (Anzick) Paleo-Indian site, 0.6km S of Wilsall, Montana (Taylor, 1969; Lahren & Bonnichsen,

1974). One skull fragment was coated with hematite, from 3-to-5-yr-old adolescent. Second sample was bleached-white calvarium.

AA-313A. Wilsall (Anzick) **8690 ± 310**

Hematite stained, 3-to-5-yr-old adolescent calvaria. *Comment:* weak-HCl insoluble collagen dated. Target C-1036.

AA-313B. Wilsall (Anzick) **10,500 ± 400**

Hematite stained, 3-to-5-yr-old adolescent calvaria. *Comment:* gelatin fraction from collagen used for AA-313A. Target C-1037.

AA-313C. Wilsall (Anzick) **8620 ± 340**

Bleached calvaria. *Comment:* weak HCl insoluble collagen. Target C-1039.

AA-313D. Wilsall (Anzick) **8940 ± 370**

Bleached calvaria. *Comment:* gelatin extracted from AA-313C collagen. Target C-1042.

General Comment: both calvaria have physical and chemical properties of modern bone.

Cheek Bone Cave series

Pocket gopher (*Geomys* cf *bursarius*) ramii and unid. larger mammal bone fragments coll Nov 1983 and id by W Klippel; samples from Cheek Bone Cave, 40Mu-261, 13km ESE of Columbia, Maury Co, Tennessee; Stratum 8(2?), 10cm thick, level 42 of 101N 99E. Gopher bones were well perserved and angular, fragments 0.5 to 2cm long.

AA-734. Cheek Bend Cave **14,710 ± 490**

Unid. mammalian cortical bone, CBC no. 1 colln, Sample A. *Comment:* bone analysis: 4.22%C, 0.97%H, 0.95%N. Pale yellow-brown to dark brown, moderately hard, chalky surfaced bone.

AA-735. Cheek Bend Cave **6740 ± 280**

Unid. large mammal cortical bone, CBC no. 1 colln, Sample B. Very pale yellow, very hard, waxy bone with modern physical and chemical properties. C-1125.

General Comment: XAD-2-purified gelatin hydrolyzate dated from all samples. Bones in CBC no. 1 colln range from 0.65% to 1.92%N and are chalky to hard and waxy.

Yuha series

Homo sapiens sapiens post-cranial bone from Yuha cairn burial, W of El Centro, Imperial Co, California. Coll 1971 by M Childers (1974; 1983). Skeletal remains curated in three different collns: Imperial Valley Coll Mus, El Centro, California (IVCM), J L Bishoff, USGS, Menlo Park, California (USGS), and R E Taylor, Univ California, Riverside (UCR).

AA-737. Yuha **3930 ± 270**

Post-cranial bone no. 1. IVCM colln. *Comment:* 0.3N HCl-insoluble fraction dated. Target C-674.

AA-738. Yuha **1750 ± 230**

Post-cranial bone no. 1, IVCM colln. *Comment:* 0.3N HCl soluble fraction. Target C-673.

AA-739. Yuha **2490 ± 300**

Post-cranial bone no. 1, IVCM colln. *Comment:* total inorganic carbon from 95% H_3PO_4 hydrolysis of bone powder. Target C-664. Bone analysis: $^{234}U/^{238}U = 1.24$, $^{230}Th/^{234}U = 0.05$; U/Th date = 5900 + 1000/−800 yr BP (J Bischoff, pers commun, 1983).

AA-740. Yuha **2830 ± 260**

Caliche, 0.3mm thick coating bone no. 1, IVCM colln. *Comment:* CO_2 evolved by H_3PO_4 hydrolysis. Target C-663.

AA-741. Yuha **2460 ± 290**

Caliche, 0.3mm thick coating bone no. 1, IVCM colln. *Comment:* caliche combusted with CuO powder. Target C-684.

AA-742. Yuha **>26,600**

Petrocalcic horizon caliche, 3mm thick, frag no. 1, IVCM colln. *Comment:* CO_2 evolved by H_3PO_4 hydrolysis.

AA-743. Yuha **3030 ± 270**

Petrocalcic horizon caliche, 2mm thick, frag no. 2, IVCM colln. *Comment:* CO_2 evolved by H_3PO_4 hydrolysis.

AA-744. Yuha **2610 ± 200**

Post-cranial bone no. 2, UCR colln. *Comment:* total bone inorganic CO_2 evolved by H_3PO_4 hydrolysis. Target C-748.

AA-745. Yuha **2840 ± 220**

Post-cranial bone no. 3, USGS colln. *Comment:* total 0.3N HCl soluble organic carbon. Specimen was fragment from bone subm to J Bischoff by M Childers and dated by aspartic acid racemization at 23,600 yr (Bischoff & Childers, 1979). Bone analysis: 8.9ppm U, $^{234}U/^{238}U = 1.21$, $^{230}Th/^{234}U = 0.03$ (J Bischoff, pers commun, 1983). Target C-759B.

AA-746. Yuha **2690 ± 200**

Post-cranial bone no. 3, USGS colln. *Comment:* total inorganic CO_2 evolved by H_3PO_4 hydrolysis. Sample as AA-745. Target C-746.

General Comment: for Yuha series ^{14}C dates, see Stafford *et al* (1984). All bones were very poorly preserved. Analyses on unid. cortical fragment from IVCM colln: 7.90%C, 0.32%H, 0.06%N; 8.34%C, 0.26%H, 0.05%N;

and 4.57%C, 0.35%H, 0.08%N. Differences between petrocalcic carbonate dates (AA-742, -743) are probably due to mixing of young and old caliche horizons when body was interred. Caliche ^{14}C dated to 22,125 ± 400 (UCLA-2600 "1854") and ^{230}Th dated to 19,000 ± 3000 (Bischoff *et al,* 1976) probably antedates burial. Caliche-coated cobbles and boulders for cairn were taken from older (Pleistocene) deposits. Caliche 3 to 6mm thick and 25mm across that adhered to bone was ^{14}C-dated to 21,500 ± 1000 (GX-2674) (Bischoff *et al,* 1976). Caliche was probably pre-Holocene carbonates that were later cemented onto bone.

Wilson-Leonard series

Homo sapiens sapiens postcranial bone and assoc charcoal from Wilson Leonard site, 41WM-235, Williamson Co, Texas (3378550N, 617250E) Level 32; bones were in Leanne soil. Coll 1983 by F Weir and subm by FW, State Dept Hwys and Public Transportation.

AA-747. Wilson-Leonard **4650 ± 310**

Homo sapiens sapiens bone fragments. *Comment:* total inorganic CO_2 evolved by H_3PO_4 hydrolysis. Target C-1017.

AA-748. Wilson-Leonard **5940 ± 520**

Homo sapiens sapiens bone fragments. *Comment:* total 0.6N HCl soluble organic carbon. Target C-968.

AA-749. Wilson-Leonard **6700 ± 460**

Homo sapiens sapiens bone fragments. *Comment:* total 0.6N HCl soluble organic carbon. Target C-1117.

AA-751. Wilson-Leonard **5860 ± 270**

Second target from AA-750 carbon. *Comment:* Target C-967.

AA-752. Wilson-Leonard **5440 ± 420**

Homo sapiens sapiens bone fragments. *Comment:* hot-water insoluble organic carbon from 0.6N HCl insoluble residue. Second extraction of this phase. Target C-1116B.

AA-753. Wilson-Leonard **1270 ± 280**

Homo sapiens sapiens bone fragments. *Comment:* gelatin phase dated. Fraction was pale yellow solid resembling inorganic salt. No properties of modern gelatin. Target C-970.

General Comment (TWS): bone collns 2 and 3 contained unid. cortical and cancellous fragments that were combined as 22.7g of cleaned, powdered bone; cortical bone analysis: 4.01%C, 0.54%H, 0.09%N; cancellous bone analysis: 3.48%C, 0.56%H, 0.06%N.

General Comment (FW): ^{14}C dates on charcoal that is apparently stratigraphically higher than burial: 7470 ± 230 (Tx-4798), on charcoal ca 1.5m above burial and 8820 ± 120 (Tx-4784A), 8860 ± 150 (Tx-4784B) and

8940 ± 100 (Tx-4784C) on charcoal ca 1.2m above human skeleton. Leanne soils overlying and enclosing burial had soil ^{14}C dates of 9470 ± 170 (Tx-4787) and 9650 ± 120 (Tx-4793).

La Jolla Shores series

Homo sapiens sapiens long bone fragments coll May to July 1926 by M Rogers, San Diego Mus of Man, California. In dune sands (now leveled) on embayment 1.2km N of La Jolla (32° 51′ 25″ N, 117° 16′ 17″ W) San Diego Mus site W-2, mus specimen no. SDM-16755. Part of colln of Littoral I culture human limb and rib fragments from same white sand stratum yielding partial human cranium SDM-16742. White sand stratum is ca 2m below ground level and ca 5.5m asl. SDM-16755 colln dated by aspartic acid racemization to 28,000 yr BP (Bada, Schroeder & Carter, 1974) and by ^{14}C dating to 1850 ± 200 (UCLA-2368), 1930 ± 200 (UCLA-2384) and 1770 ± 790 (UCR-1511D) (Taylor, 1983). Subm 1982 by R Tyson, San Diego Mus of Man.

AA-754. La Jolla Shores **3640 ± 360**

Homo sapiens sapiens partial radius. *Comment:* total inorganic CO_2 evolved by H_3PO_4 hydrolysis. Target C-658.

AA-755. La Jolla Shores **5290 ± 270**

Homo sapiens sapiens partial radius. *Comments:* total 0.3N HCl insoluble organic carbon. Target C-669.

AA-756. La Jolla Shores **6330 ± 280**

Homo sapiens sapiens partial radius. *Comment:* total 0.3N HCl soluble organic carbon. Target C-675.

A-757. La Jolla Shores **5110 ± 270**

Caliche film coating partial radius. *Comment:* CO_2 evolved by hydrolysis with H_3PO_4. Target C-601.

Fish Bone Cave series

Horse (*Equus* sp) postcranial bone from Fish, Bone Cave, P3e, Winnemuca Lake, Pershing Co, Nevada (40° 12′ 08″ N, 119° 16′ 45″ W). Coll 1956 by P C Orr (1974) and subm 1984 by R Thompson, Univ Arizona.

AA-759. Fish Bone Cave **12,280 ± 520**

Partial *Equus* sp right ramus, Site Pe/4, Nevada State Mus no. 317. *Comment:* extremely well-preserved bone. Sample overlay sagebrush (originally id as juniper-bark mat) that was ^{14}C-dated to 11,200 ± 250 (L-245) (Orr, 1974; Broecker, Kulp & Tucek, 1956). Ramus has chemical and physical properties of modern bone. HCl dissolution yields pseudomorph of bone. Bone washed with acetone twice before decalcification. Gelatin is physically identical to modern gelatin. Target C-1276.

Puu Naio Lava Tube series

Rodent and extinct bird bones from Puu Naio lava tube, Ulupalaku Ranch, Maui, Hawaii (20° 37′ N, 156° 24′ W). Coll Feb 1984 by S Olson and H James, Smithsonian Inst, Washington, D C; subm 1984 by P Martin, Univ Arizona.

AA-760. Puu Naio lava tube **707 ± 350**

Rat (*Rattus exulans*) bones. W12, Unit II. *Comment:* organic carbon extracted from 450mg combined partial pelvis, 2 femora, tibia, 2 radii and ramus bones. Bone analysis: 7.60%C, 1.27% H, 2.08% N. Bones had physical and chemical properties of modern bone. Target C-1271.

AA-761. Puu Naio lava tube **1850 ± 270**

Ibis (*Apteribis* sp) single, complete femur. E24, 10 to 20cm. *Comment:* bone analysis: 9.81% C, 1.74% H, 2.75% N. Bones had physical and chemical properties of modern bone. Target C-1272.

AA-762. Puu Naio lava tube **4340 ± 610**

Extinct goose (*Thambetochen* sp) complete femur. W11, cross-section S face, unit III, subunit I, 50 to 60cm. *Comment:* chalky bone with no spiral breakage possible. Bone analysis: 4.70%C, 0.88%H, 0.3%N. Target C-1273.

AA-763. Puu Naio lava tube **7750 ± 500**

Ibis (*Apteribis* sp) complete tarsomatetarsus. W11, 90 to 100cm. *Comment:* chalky bone, readily disaggregated in HCl. Bone analysis: 7.78%C, 1.34%H, 1.64%N. Target C-1274.

General Comment: XAD-2-purified collagen hydrolyzates were dated from all bones.

Domebo Mammoth series

Postcranial bone from immature mammoth (*Mammuthus* cf *imperator*) id by M Mehl (1966). Mammoth was excavated 1962 at Paleo-Indian Domebo site (34Cd-50), ca 4km E of Stecker, Caddo Co, Oklahoma (NE1/4, SW1/4, SE1/4, sec 29, T6N, R10W). For site report, see Leonhardy (1966). Bone was used as known-age fossil for calibrating bone sample preparations. Mammoth dates 11,000 to 11,500 yr BP by assoc with Clovis culture artifacts (Haynes, 1982, 1984). ^{14}C dates on assoc wood are 11,045 ± 647 (SM-695) (Leonhardy & Anderson, 1966) and 11,490 ± 450 (AA-823), which is accelerator redate of SM-695 wood. Wood 7m above bone level dated to 10,123 ± 280 (SM-610).

AA-801. Domebo mammoth **8010 ± 500**

Bone. *Comment:* total inorganic and organic carbon from powdered bone. CuO combustion. Whole bone ^{14}C date. Target C-474B.

AA-802A. Domebo mammoth **9540 ± 480**

Bone. *Comment:* weak-HCl-soluble organic carbon from bone powder. Target C-477.

AA-802B. Domebo mammoth **10,280 ± 560**

Bone. *Comment:* 6N HCl insoluble organic carbon from hydrolyzate of 0.6N HCl insoluble collagen. Target C-743.

AA-803. Domebo mammoth **10,350 ± 410**

Bone. *Comment:* unpurified gelatin. Target C-480.

AA-804. Domebo mammoth **10,760 ± 440**

Bone. *Comment:* total organic and inorganic carbon from bone powder after leaching with 1M acetic acid. CuO combustion. Target C-542.

AA-805. Domebo mammoth **10,810 ± 420**

Bone. *Comment:* XAD-2-purified gelatin hydrolyzate. Target C-543.

AA-806. Domebo mammoth **11,010 ± 710**

Bone. *Comment:* XAD-2-purified collagen hydrolyzate. Target C-544B.

AA-807. Domebo mammoth **11,330 ± 470**

Bone. *Comment:* alpha-hydroxy acids from nitrosylation of XAD-2-purified gelatin hydrolyzate. Target C-551.

AA-808. Domebo mammoth **11,280 ± 530**

Bone. *Comment:* alpha-hydroxy acids from nitrosylation of XAD-2-purified collagen. Target C-555.

AA-810. Domebo mammoth **10,120 ± 450**

Bone. *Comment:* imino acids (hydroxyproline and proline) isolated from XAD-2-purified gelatin hydrolyzate. Target C-556B.

AA-811. Domebo mammoth **10,860 ± 450**

Bone. *Comment:* imino acids (hydroxyproline and proline) from XAD-2-purified collagen hydrolyzate. Target C-559.

AA-812. Domebo mammoth **4910 ± 320**

Bone. *Comment:* fulvic acids from hydrolyzed collagen. FA eluted from XAD-2 resin with NH_4OH (conc). Target C-561.

AA-814. Domebo mammoth **10,690 ± 640**

Bone. *Comment:* 0.6N HCl insoluble collagen. Target C-606.

AA-815. Domebo mammoth **9310 ± 360**

Bone. *Comment:* hydroxy-apatite (dahllite) CO_2 from bone powder extracted for 24h with 1M acetic acid. H_3PO_4 hydrolysis. Target C-615.

AA-816. Domebo mammoth **4810 ± 760**

Bone. *Comment:* fulvic acids from hydrolzed collagen. Target C-650.

AA-818. Domebo mammoth **7300 ± 320**

Bone. *Comment:* hydroxy-apatite CO_2 from untreated bone powder. H_3PO_4 hydrolysis. Target C-662.

AA-819. Domebo mammoth **5130 ± 290**

Bone. *Comment:* fulvic acids from hydrolyzed gelatin. FA eluted with acetone. Target C-771.

AA-822A. Domebo mammoth **2050 ± 580**

Bone. *Comment:* acetone-soluble organic carbon isolated by soxhlet extraction of bone powder. Target C-783.

AA-822B. Domebo mammoth **9360 ± 670**

Bone. *Comment:* 0.6N HCl insoluble collagen from acetone-extracted bone powder. Target C-743.

AA-823. Domebo mammoth **11,490 ± 450**

Stump of elm (cf *Ulmus elata* assoc with mammoth. *Comment:* outer 10 rings including bark were dated. Total ring count = 94 ± 1 (id by M Thompson). Wood was previously dated to 11,045 ± 647 (SM-695) (Leonhardy & Anderson, 1966, p 24).

AA-824. Domebo mammoth **10,820 ± 270**

Bone. *Comment:* 0.6N HCl insoluble collagen from bone powder. Target C-1002.

AA-825. Domebo mammoth **11,480 ± 450**

Bone. *Comment:* XAD-2-purified collagen hydrolyzate. Target C-1038.

General Comment: fractions of bone previously dated by Leonhardy and Anderson (1966, p 24–25) were untreated tusk: 4952 ± 304 (TBN-311); bone organic carbon soluble in 2N HCl after initial 2% NaOH: 11,220 ± 500 (SI-172) and humic acids extracted after decalcification: 11,200 ± 600 (SI-175). Domebo series represents bone preserved in reduced clay. Analyses of cortical bone (micro-Kjedahl): 0.43%N; cortical bone (CHN analyzer): 5.19%C, 0.69%H, 0.69%N; cancellous bone: 4.37%C, 0.59%H, 0.24%N. Uranium analyses by J Bischoff, USGS: $^{234}U/^{238}U = 1.14 \pm 0.02$, $^{238}U = 4.57 + 0.09$ppm, age = 9512 + 525/−400yr. Uranium series age = 11,500 ± 2000 (Szabo, 1980).

Dent Mammoth series

Postcranial mammoth bone from Clovis culture Dent site, Weld Co, Colorado (40° 19′ N, 104° 49′ W), 1.2km SE of Milliken, Colorado (Figgins, 1933; Wormington, 1959; Haynes, 1974). Coll Oct 1973 by F Frazier. Site was first unquestionable evidence of assoc of humans and mammoths in

North America. Previous ^{14}C date on bone and tusk fragments was 11,200 ± 500 (I-622) (Trautman & Willis, 1966; Haynes, 1967b).

AA-830. Dent mammoth **8250 ± 520**

Bone. *Comment:* weak-HCl-insoluble collagen from bone powder. Target C-1220.

AA-831. Dent mammoth **9240 ± 350**

Bone. *Comment:* unpurified gelatin phase. Target C-1221.

AA-832. Dent mammoth **10,590 ± 500**

Bone. *Comment:* XAD-2-purified hydrolyzed collagen. Target C-1261.

AA-833. Dent mammoth **10,950 ± 480**

Bone. *Comment:* XAD-2-purified gelatin hydrolyzate. Target C-1267.

General Comment: sample was coated with Gelva preservative. Bone powder from cancellous tissue was soxhlet extracted 20hr with ethanol, washed with dist H_2O, extracted 10hr with acetone and wash in H_2O before drying. Extracted powder was used for all subsequent isolations. CHN analysis: 0.83%N (cortical bone); 1.07%N (cancellous bone).

Escapule Mammoth series

Innominate from adult mammoth (*Mammuthus [Parelephas] columbi*) from Escapule site, Clovis culture mammoth kill site (EE:8:28) in Horse Thief Draw, Sec 1, T22S, R21E, Cochise Co, Arizona (Hemmings & Haynes, 1969). Mammoth bones were from erosional contact between Units E and F_2. Fossils were overlain by erosional surface dated at 10,900 ± 40 yr BP; occupational surface overlain by organic carbon-rich horizon dated to 10,800 yr BP (Haynes, 1984). Excavated and coll June 1967 (Hemmings & Haynes, 1969).

AA-834. Escapule mammoth **8500 ± 470**

Bone. *Comment:* 0.6N HCl insoluble residue from pretreated bone powder. Target C-1259.

AA-835. Escapule mammoth **5210 ± 270**

Bone. *Comment:* unpurified gelatin phase. Target C-1275.

AA-836. Escapule mammoth **4610 ± 280**

Bone. *Comment:* XAD-2 purified hydrolyzed weak-HCl-insoluble residue. Target C-1358.

General Comment: cancellous tissue from innominate (6911/UA3404c) was dated. Sample curated at Univ Arizona. Bone was coated with epoxy preservative (2mm thick) that was physically removed. Underlying cancellous tissue was powdered and washed in ethanol and acetone. Haynes (pers commun) noted that bones had been treated with acetone and methyl ethyl ketone before epoxy was applied. Cancellous tissue: 3.64%C, 0.66%H,

0.08%N. Sample was chosen to represent known-age bone with very low organic carbon content and non-collagen amino acid composition; burial was in oxidized clay. Epoxy preservative (2mm thick) was ^{14}C dated to 3680 ± 210 (GX-11261), $\delta^{13}C = -24.2‰$ (PDB). Epoxy was pretreated (by TWS) with 3 successive 6N HCl and 1% NaOH washes.

Del Mar series

Charcoal and *Chione* shell from upper midden (Site W-34) of Del Mar Early Man site (W-34A) on NW point of San Digieto R inlet, Del Mar, San Diego Co, (32° 58′ 36″ N, 117° 16′ 12″ W). Samples coll (1974) by R Tyson, during excavation of upper midden (W-34) adjoining loc W-34A that yielded Del Mar Man skull, SDM-16704.

AA-837. Del Mar **3330 ± 220**

Charcoal from dm 14. *Comment:* target C-1284.

AA-838. Del Mar **3520 ± 330**

Charcoal from dm 12. *Comment:* target C-1285.

AA-839. Del Mar **7000 ± 390**

Charcoal from dm 11. *Comment:* target C-1286.

AA-840. Del Mar **4240 ± 300**

Charcoal from dm 9. *Comment:* target C-1294

AA-846. Del Mar **8680 ± 400**

Chione shell carbonate, dm 11. *Comment:* target C-1300.

AA-847. Del Mar **4720 ± 260**

Chione shell carbonate, dm 12. *Comment:* target C-1359.

AA-848. Del Mar **4880 ± 260**

Chione shell carbonate, dm 14. *Comment:* target C-1360.

AA-849. Del Mar **6610 ± 290**

Chione shell. Comment: target C-1361.

General Comment: individual charcoal fragments weighed 20 to 100mg; 20 to 406mg charcoal were available from each 10cm level. Charcoal was pretreated 3 times each with 3N HCl (60°C) and 1% NaOH (60°C) and finally acidified and washed with dist H_2O. *Chione* shell carbonate was evolved by using 95% H_3PO_4. Outer, chalky shell layers were physically removed and remaining hard core etched to half original thickness with 1N HCl. Previous shell carbonate ^{14}C dates from upper midden(W-34) are 4590 ± 60 (LJ-3175) for dm 3; 5440 ± 70(LJ-3176) for dm 7; 7380 ± 220(LJ-3507) for dm 10 and dm 11, and 9260 ± 100 (LJ-3177) for dm 15; amino acids from *Chione* shell from dm 10 and 11 of W-34 ^{14}C dated to 12,000 ± 1100 (LJ-3631) (Masters & Bada, 1977).

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