UCRL-JRNL-206429

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September 7, 2004

American Antiquity

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Plasma oxidation was used to obtain radiocarbon dates on six different materials from a naturally mummified baby bundle from the Lower Pecos River region of southwest Texas. This bundle was selected because it was thought to represent a single event and would illustrate the accuracy and precision of the plasma oxidation method. Five of the materials were clearly components of the original bundle with 13 dates combined to yield a weighted average of 2135 ± 11 B.P. Six dates from a wooden stick of Desert Ash averaged 939 ± 14 B.P., indicating that this artifact was not part of the original burial.

Plasma oxidation is shown to be a virtually non-destructive alternative to combustion. Because only sub-milligram amounts of material are removed from an artifact over its exposed surface, no visible change in fragile materials has been observed, even under magnification. The method is best applied when natural organic contamination is unlikely and serious consideration of this issue is needed in all cases. If organic contamination is present, it will have to be removed *before* plasma oxidation to obtain accurate radiocarbon dates.

La oxidación de plasma, una alternativa no destructiva a la combustión, se usó para obtener muestras de radiocarbono de seis materiales distintos de un envoltorio de bebé naturalmente momificado de la región del bajo Río Pecos del sudoeste de Texas. Este bulto se escogió porque representa un hecho único y ilustra la exactitud y precisión del método de oxidación de plasma. Cinco tipos de materiales que eran claramente componentes del bulto rindió 13 fechas que se combinaron para rendir un promedio con un valor asignado de 2135 ± 11 A.P. Seis muestras del palillo de madera promediaron 939 ± 14 A.P., mostrando que este artefacto no era parte del entierro original.

La oxidación de plasma promete ser un método no destructivo para obtener fechas de radiocarbón de artefactos orgánicos perecederos. Porque solamente menos de miligramos son eliminados de la superficie expuesta de un artefacto, ningún cambio visible se observó en los materiales frágiles después de la oxidación de plasma, aun con amplicación. El métado se aplica mejor cuando no existe la posibilidad de contaminación orgánica, este factor necesita ser seriamente considerado en todos los casos. Si hay contaminación orgánica presente esta debe ser removida *antes* de la oxidación de plasma para obtener fechas de radiocarbón exactas.

When an archaeological sample is radiocarbon dated, it typically undergoes three separate steps: (1) chemical pretreatment to remove contamination or isolation of sample-specific chemical compounds; (2) conversion to a measurable form; and (3) measurement of ^{14}C to determine age. This current study demonstrates that step 2, conversion to a measurable form, is a virtually non-destructive process using plasma oxidation. Combustion has been the traditional method for converting a sample to carbon dioxide, which is then reduced to graphite for accelerator mass spectrometry (AMS). Because chemical pretreatment and combustion are destructive, small samples are removed from artifacts for radiocarbon dating. As an alternative to combustion, plasma oxidation is a virtually non-destructive method for converting solid organic carbon to carbon dioxide. An oxygen plasma, electrically excited oxygen gas, gently removes organic carbon from the surface of a whole artifact at low temperatures (~50°C). Since only 100 _g of carbon is needed for AMS measurement, the effect upon an artifact is negligible.

Plasma oxidation has the potential to resolve one of the major problems facing archaeologists working with rare, unique, or sacred objects. The need to place these artifacts in a secure chronological context is often offset by a reluctance to destroy even the small part that must be combusted for current dating methods. The preservation of normally perishable organic material in the dry rock shelters of southwest Texas provided an opportunity to test the method on a suite of

4

materials that were thought to be from a single event—the mortuary furnishings of an infant grave. Using plasma oxidation, along with traditional combustion, we have now determined 19 radiocarbon dates on six materials from an infant burial bundle found in a dry shelter in the Lower Pecos River region of southwest Texas. Two clusters of AMS dates demonstrate that the process clearly differentiated between artifacts of different ages and that the results were replicable.

Plasma Oxidation

Plasma-chemical extraction was originally used by our chemistry laboratory at Texas A&M University to radiocarbon date ancient rock paintings (e.g., see Rowe 2001 and references therein; Russ et al. 1990). The primary advantage of this process is that the low temperatures of the plasma gas (50-150ºC depending upon our operating conditions) are below the decomposition temperatures of carbon-containing minerals. This allows the plasma to selectively remove only organic carbon from a sample and leave accompanying carbonates and oxalates intact (Chaffee et al. 1994; Russ et al. 1992).

More recently, we employed plasma oxidation on perishable organic materials to obtain *non-destructive* radiocarbon dates (Steelman and Rowe 2002, 2003). Low-temperature (~50-150ºC) oxygen plasmas, electrically excited ionized gas, were used to extract sub-milligram amounts of organic carbon from surfaces of archaeological artifacts for radiocarbon dating. It is possible to collect multiple aliquots of carbon dioxide from a single material for replicate accelerator mass

spectrometry (AMS) and carbon stable isotope measurements without any observable change in an artifact's appearance. In contrast, when combustion is used to collect carbon for dating, milligram-size or often larger samples must be physically removed from an artifact and destroyed.

In some cases, it is undesirable to remove even a small portion of an artifact for destructive analysis. Non-destructive plasma oxidation should be employed when: (1) the amount of material needed for combustion is a large fraction of artifact size; (2) the information content of sample structure should be preserved; (3) an object is rare or has intrinsic value; and (4) an artifact is a sensitive item that might not otherwise be dated. Plasma oxidation may also be preferable for any type of sample containing significant amounts of carbon-containing minerals. Two previous studies found that in unusual circumstances, acid treatment is insufficient to remove offending calcium oxalate minerals (Hedges et al. 1998; Armitage et al. 2001). The characteristics of plasma extraction have great potential for the study of artifacts associated with burials, where the least intrusive techniques are especially critical. Museum artifacts such as basketry, textiles, cordage/twine, and paleobotanical samples would also benefit from this application of the plasma oxidation process.

NATURALLY MUMMIFIED INFANT BUNDLE

All samples used in this study were taken from a bundled baby burial that was unsystematically removed from a dry cave on the Pecos River in the 1950s (Figure 1). After 40 years of storage in a closed compartment, the bundle was brought to the attention of professional archeologists in 1989 by a local resident who described the original context of the burial. He provided a number of details about the find and pointed to the location of the cave on a USGS topographic map. To him, this was the old Marcos place, but it had been registered in the state trinomial system as Hinds Cave, 41VV456.

Texas A&M University carried out excavations at Hinds Cave for years, contributing several master's theses and doctoral dissertations on environmental data gleaned from fauna, flora, and coprolites (Dering 1979; Lord 1984; Stock 1983; Williams-Dean 1978; Woltz 1998). An NSF report on Hinds Cave was submitted by Shafer and Bryant (1977). Others have also studied various aspects of the archaeology of the cave (Andrews and Adovasio 1980; Bement and Turpin 1987). No burials were reported from the Texas A&M excavations, but many of the details provided by the donor of the infant burial bundle are consistent with the layout of the Hinds Cave site. The exact provenience of the baby burial is not critical to the current project since it is indisputable that the bundle came from a dry cave on the Pecos River and therefore belongs within the cultural matrix devised from over seven decades of archeological research (e.g., Shafer 1986; Turpin 1991).

Cultural Context of Bundled Burial

The cultural chronology of the Lower Pecos region generally follows the
quadripartite sequence used throughout Texas, but hundreds of radiocarbon dates
generated by excavations permit subdivision of these long periods into more
precise subperiods (Turpin 1991). Although very few burials have been dated, it
can be assumed that the practice of bundle burial in dry rock shelter deposits is
predominantly a Late Archaic trait and more specifically one attributable to the
Flanders-Blue Hills subperiods (2300-1300 B.P.). These periods follow
immediately after a mesic interlude (Cibola Subperiod) in which the Great Plains
habitat expanded into the Lower Pecos region for a few hundred years. The
resumption of a general trend toward aridity that had been in effect since the end
of the Pleistocene presaged the return to a desert-adapted economic strategy and
settlement pattern, possibly by an influx of people from northern Mexico. Rock
shelter occupation regained favor and the dead were often buried in the refuse left
by the living. The dry climate and sheltered environment contributed to excellent
preservation of perishables and occasionally led to natural mummification,
especially of infants such as the one selected for this study.

Bundled Infant Burials

Despite the irregularities in their collection, the baby burials of the Lower Pecos
region share certain similarities that are indicative of a structured mortuary
pattern, at least in so far as evidenced by interments in dry rock shelters. With few

exceptions, the infant corpse is wrapped in a skin shroud, such as a rabbit fur robe, coyote skin, antelope hide, fawn skin, or unidentified membrane or in a finely woven mat (Banks and Rutenberg 1982; Butler 1948, Martin 1933; Pearce and Jackson 1933; Turpin 1988:Table 3). This bundle is then placed in some form of nest or cradle. Examples are pillows of grass held in place by wooden stakes, a pit lined with twigs, a finely woven basket or net, a crude cradle made of crossed sticks, or their broken cradleboards. Next, up to three mats are placed over or wrapped around the bundle. The sequence invariably begins with the most finely woven mat on the interior and the most coarse on the exterior. The interior mats are often small and intact, suggesting they were specially made for the baby, but the exterior coarser checkerweaves can be folded segments of larger pieces. The entire burial is then usually covered with a metate, limestone slab, or in one instance, a large stick that held the bundle down. Occasionally, exotic offerings are included in the bundle; examples are woven fiber artifacts decorated with red designs, an olivella shell necklace with coyote teeth pendants, mussel shell plaques, and human hair cords or ropes.

Although very similar bundled infant burials have been reported, and others are known to be in private collections, only two have been radiocarbon dated (Turpin 1991:Table 1)—the Hinds Cave bundle that is the subject of this report and a bundle that was exhumed from a dry cave on the Pecos River by a relic hunter. For the latter bundle, the collector opted to rebury the infant but donated

9

the grave goods. This baby had been wrapped in a rabbit skin robe and buried in its broken cradleboard. The grave was covered with three mats in ascending order of crudity and capped by a metate. A fragment of one of the sotol mats produced an uncorrected, uncalibrated radiocarbon date of 2270 ± 50 B.P. (Turpin 1991:Table 1; TX-6166).

Hinds Cave Bundled Infant Burial

The Hinds Cave bundled infant is typical of the pattern that emerged from professional and amateur excavations (Turpin et al. 1986). The tiny corpse was wrapped in a rabbit skin robe that is now reduced to spirals of leather and connecting twine that lay over the torso. Where the robe had been pulled up to cover the baby's head, matted fur still crowns the skull with a few tresses of long dark hair protruding from under its cap. The facial portion of the skull has been crushed and broken into fragments, probably by the weight of the overlying rocks and accumulated grave fill. Some body parts are identifiable even without removing the robe, most notably two small skin-encased feet that peek from under the covers.

A framework of crossed sticks was driven into the cave fill to form X-shaped braces. A circular nest of grass was placed on the braces to cradle the bundled body. Once the bundle had been placed in the grass nest, atop the supports, the entire assemblage was covered with three simple one-over-one plaited mats. Plaiting is one of the oldest techniques in the basketry tradition of northern

Mexico and the Lower Pecos region and undecorated checkerweaves such as these are one of the more common fiber artifacts retrieved from the dry rock shelter deposits (Andrews and Adovasio 1980:333, 359, 366-367; McGregor 1992:47). The first and finest mat was made of elements averaging about 3 mm in width. Selvages on both sides permit measurement of the original width—39 cm—but both ends are frayed. The middle mat is intact and measures 53 by 37 cms. The elements average 5 mm wide and are tightly woven, with 90 degree turns at the selvages. The top mat is one end of a much larger piece, folded over to form a double layer. One end and both corners are intact but the other edge is raveling and irregular. The elements in this mat were unstripped sotol leaves and ranged from 0.8 to 1.5 mm in width, following the natural taper of the leaf. According to the finder, the entire feature was covered with flat rocks—either metates or locally available limestone slabs.

EXPERIMENTAL METHODS

Six materials from a naturally mummified infant burial from Hinds Cave, southwest Texas, were radiocarbon dated using plasma oxidation and AMS analysis: bone with mummified flesh; grass; woven mat; wood stick (desert ash); sotol stalk; and twine. Sub-samples were taken from each material for various chemical pretreatments (see Tables 1 and 2). Chemical pretreatment was damaging to materials, but for those sub-samples that received no pretreatment

the plasma oxidation process is virtually non-destructive. Photographs

demonstrating the non-destructive nature of plasma oxidation are taken of

samples that received no chemical pretreatment. Sub-samples from four of the six

materials were also sent to Lawrence Livermore National Laboratory's Center for

Accelerator Mass Spectrometry (CAMS) for traditional acid-base-acid

pretreatment, combustion, and AMS radiocarbon dating for comparison and

inclusion with the plasma oxidation results.

We produce a plasma glow with radio frequency capacitive coupling using

two external copper electrodes on the ends of a 9.9-cm diameter glass sample

chamber (Rowe and Steelman 2003:14). Free electrons are accelerated to

sufficient energies to cause ionization in a small fraction of gas molecules and

atoms. Low-temperature plasmas produce excited neutral and ionic species with

high chemical energy. The reactive species allow reactions that normally occur

only at high temperatures to proceed at low temperatures (50 to <150ºC) and

gentle conditions. Electrons gain kinetic energy from the oscillating electric field,

while the temperature of the gas components is increased by elastic collisions

between electrons and the gas. Electrons are thermally isolated from the gas

components by the very large mass difference. Temperatures of the plasma gas

can thus remain near ambient temperatures at the same time the electrons are

sufficiently energetic to break molecular bonds. The plasma converts organic

carbon to carbon dioxide, which is collected in a glass tube cooled with liquid

nitrogen. This carbon dioxide is used to make a graphite target for radiocarbon measurement on the AMS at CAMS. Radiocarbon dates of secondary radiocarbon standards have demonstrated the general validity and accuracy of the method (Steelman and Rowe 2002, 2003).

RESULTS AND DISCUSSION

Radiocarbon Dates

All 19 of our radiocarbon results from the Hinds Cave baby bundle are listed in Tables 1 and 2 and shown in Figure 2. All radiocarbon ages have been corrected for measured $_{-}^{13}C$, except the bone with flesh and the sotol stalk. Using plasma oxidation, ten radiocarbon dates combined to give a weighted average of 2137 ± 13 B.P. for five of the six materials from the baby bundle (excluding the desert ash wood stick). All ten plasma oxidation results for these materials pass a χ^2-test and are coeval. CAMS's combustion results have a weighted average of 2128 ± 20 B.P. for the grass, woven mat, and twine. A null hypothesis was performed and statistically there is no significant difference between the two methods at 95% confidence. Thus, an overall weighted average of the 13 radiocarbon dates is 2135 ± 11 B.P. for the infant burial.

The wood stick of desert ash is much more recent than the other burial materials and is consequently not part of the original assemblage. All five plasma oxidation radiocarbon results pass a χ^2-test and are coeval with one another with a

weighted average of 946 ± 15 B.P. Using combustion, CAMS obtained a single
result of 905 ± 35 B.P., which overlaps at 1_ with the plasma oxidation weighted
average. The overall weighted average of the six radiocarbon dates is 939 ± 14
B.P. All the dates are illustrated in Figure 2 as calibrated probability curves,
which were generated using the calibration program OxCal (Ramsey 2000;
Stuiver et al. 1998).

The wood stick and sotol stalk had been previously radiocarbon dated: 1310 ±
97 B.P. (TX-5897; corrected for $\delta^{13}C$) and 2710 ± 50 B.P. (TX-5987),
respectively. The discrepancy between these two previous dates indicated that one
was in error, possibly because extraneous sticks or stalks may have been picked
up with the bundle when it was collected. The older date – 2710 B.P. – was
originally considered suspect because preservation of the perishable material
seemed more consistent with the later date (Turpin 1991). Our radiocarbon results
for the infant bundle disagree markedly with these two previous radiocarbon
dates.   For the wood stick, a date of 1310 B.P. differs from our multiple
determinations averaging 939 ± 14 B.P. For the sotol stalk, a date of 2710 B.P.
also disagrees with our average of 2135 ± 11 B.P. for the burial. When the same
material was submitted for dating, agreement between plasma oxidation and
combustion radiocarbon dates occurred. The previous two dates for the wood
stick and sotol stalk are in error and should be disregarded.

Virtually Non-destructive Plasma Exposure

Plasma oxidation is non-destructive in the sense that there is no observable physical or chemical change in the artifact; however, microscopic amounts of material are removed by gentle surface oxidation and desiccation may be experienced. The 37 cm-long sotol stalk was inserted whole into the plasma chamber for oxidation, with no chemical pretreatment. Figure 3 shows photographs of the sotol stalk from the infant burial bundle before and after plasma extraction. Even thin fragile portions of the artifact are unaffected. However, in some cases, simply placing an artifact under vacuum may cause serious deterioration in the structural integrity of the material through desiccation. These artifacts from southwest Texas were preserved since antiquity primarily because they were naturally desiccated and exposure to a vacuum during the plasma oxidation process did not affect them. Other archaeological materials that would benefit from plasma oxidation are also most likely preserved because of desiccation and would not be harmed during the plasma oxidation process. However, during previous studies on modern samples, plasma oxidation and exposure to a vacuum also did not appear to damage non-desiccated materials such as a modern textile and paper. Plasma oxidation will in almost all cases be less damaging than the removal of samples for conventional pretreatment and combustion. However, any decision by archaeologists, curators, or conservators to

apply the technique should be made in consultation with scientists who specialize in this type of measurement.

Chemical Pretreatment

Traditionally, when an archaeological sample is radiocarbon dated, acid-base-acid pretreatments remove contamination from buried archaeological materials before combustion and ^{14}C measurement. The first acid treatment is used to dissolve carbonate minerals that would adversely affect a radiocarbon age determination. The base wash removes soil organic contamination, called humic and fulvic acids. The final acid wash removes adsorbed carbon dioxide from the base solution.

For this particular case study, there was no shift in radiocarbon ages due to various chemical pretreatments (see Figure 1), indicating that contamination from soil organic matter (humic and fulvic acids) was negligible. If contamination is present in a sample, chemical pretreatment must be accomplished before plasma oxidation to obtain accurate radiocarbon dates. The mild conditions of the plasma, however, allow acid treatment to be excluded from traditional acid-base-acid pretreatments for radiocarbon dating. Since the plasma does not decompose carbonate minerals, they do not have to be removed by acid treatment before plasma oxidation. The elimination of acid pretreatment makes non-destructive radiocarbon dating a possibility. However, for radiocarbon dating using plasma oxidation to become a viable means for non-destructive analysis, a non-

destructive method for removal of humic and fulvic contamination must be realized.

CONCLUSIONS

Plasma oxidation is non-destructive, with no observable alteration of organic materials after plasma exposure. There is no statistical difference between combustion and plasma oxidation derived results. Radiocarbon ages with good precision were obtained for various materials from a Hinds Cave infant burial bundle, a *single event,* with plasma-extracted carbon dioxide samples as small as 80 µg carbon. For these samples, chemical pretreatment traditionally used to remove carbonates and humic acid contamination was not necessary. Other samples may require some type of chemical pretreatment before employing plasma oxidation for radiocarbon dating. Removal of carbonate contamination with acid is not necessary with plasma oxidation because the plasma does not decompose carbon-containing minerals, but we do not know of any non-destructive method to remove contamination such as humic and fulvic acids. We are exploring the use of supercritical carbon dioxide cleaning as a non-destructive alternative to traditional acid-base-acid treatment. Even though agreement was observed for this case study, more work is needed for plasma oxidation to be applicable on a routine basis to a wide selection of samples from a "typical" archaeological context.

This infant burial, only the second one in the Lower Pecos River region to be radiocarbon dated, is dated accurately to 2135 ± 11 B.P., the most exhaustively dated event in that region. The calibrated age range (2σ) for the burial is 340–320 cal B.C. (2.8%) and 210–110 cal B.C. (92.6%). This is 135 years B.P. more recent than the other dated infant burial from an unknown site in the same Lower Pecos River region, but both are in the same subperiod, the Flanders-Blue Hills (2300-1300 B.P.).

Although a sample of two is far too small to support any generalizations about infant mortuary practices during this or any other time period, the consensus that most of the bundled burials in the Lower Pecos region belong to the Late Archaic or Flander-Blue Hills period is bolstered by the results of this pilot project. The ability to systematically date many of the objects from bundled burials without risk to the specimens provides a means of further testing that hypothesis as well as determining if changes in funerary behavior can be coordinated with shifts in settlement patterns, subsistence strategies, art forms and tool kits.

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Figure 1. Photograph of the bundled infant burial removed from Hinds Cave, SW Texas.

Figure 2. Probability curves, generated using the calibration program OxCal, show the calibrated age ranges for six materials from the infant burial bundle (Ramsey 2000; Stuiver et al. 1998). The wood stick is younger and not part of the original burial.

Figure 3. Photographs of the sotol stalk before and after its exposure to the plasma oxidation process.

Table 1. Radiocarbon Dates on Hinds Cave Burial Materials.

Sample	Treatment	μg C	$\delta^{13}C$ (‰)	CAMS #	Years B.P.[a]	Calibrated Age (2_)
Plasma Oxidation						
Bone/skin	None	180	-25	95670	*2125 ± 35*	360 – 300 cal B.C. (8.9)
						240 – 40 cal B.C. (86.5)
Grass B2	Water	145	-14.65	88194	2125 ± 40	360 – 290 cal B.C. (11.8)
						240 – 40 cal B.C. (83.6)
Grass C3	0.1 M NaOH	90	-14.65	86525	2210 ± 45	390 – 160 cal B.C. (95.4)
Grass D3	1 M NaOH	210	-14.65	91410	2095 ± 40	350 – 320 cal B.C. (2.2)
						210 cal B.C. – 10 cal A.D. (93.2)
Mat A3	None	340	-23.91	85491	2095 ± 50	360 – 300 cal B.C. (5.0)
						240 cal B.C. – 30 cal A.D. (90.4)
Mat F2	A-B-A	145	-23.91	92188	2155 ± 40	360 – 270 cal B.C. (30.4)
						260 – 50 cal B.C. (65.0)
Twine D5	1 M NaOH	100	-16.30	93678	2170 ± 45	380 – 90 cal B.C. (95.4)
Twine F3	A-B-A	80	-16.30	93679	2155 ± 45	360 – 50 cal B.C. (95.4)
Sotol A2	None	205	-25	95671	*2120 ± 40*	360 – 290 cal B.C. (9.5)
						240 – 40 cal B.C. (85.9)
Sotol A3	None	150	-25	94532	*2135 ± 40*	360 – 280 cal B.C. (17.1)
						260 – 40 cal B.C. (78.3)
Average					2137 ± 13	350 – 320 cal B.C. (6.2)
						210 – 110 cal B.C. (89.2)
Combustion						
Grass	A-B-A		-14.65	96371	2115 ± 35	350 – 320 cal B.C. (5.0)
						210 – 40 cal B.C. (90.4)

Mat	A-B-A	-23.91	96372	2140 ± 40	360 – 290 cal B.C. (18.9) 240 – 50 cal B.C. (76.5)
Twine	A-B-A	-16.30	96374	2120 ± 35	350 – 310 cal B.C. (6.6) 230 – 220 cal B.C. (1.1) 210 – 40 cal B.C. (87.7)
Average				2128 ± 20	350 – 320 cal B.C. (4.1) 210 – 50 cal B.C. (91.3)
Average all				**2135 ± 11**	340 – 320 cal B.C. (2.8) 210 – 110 cal B.C. (92.6)

[a]All dates in Table 1 are corrected for $\delta^{13}C$ except those in italics for which a $\delta^{13}C$ of -25‰ was assumed.

Table 2. Radiocarbon Dates for the Wooden Stick of Desert Ash.

Sample	Treatment	µg C	$\delta^{13}C$ (‰)	CAMS #	Years B.P.	Calibrated Age (2_)
Plasma Oxidation						
Ash A2	None	340	-24.91	88193	940 ± 40	1010 – 1190 cal A.D. (95.4)
Ash A3	None	360	-24.91	85492	920 ± 35	1020 – 1210 cal A.D. (95.4)
Ash D1	1 M NaOH	785	-24.91	89606	955 ± 30	1010 – 1160 cal A.D. (95.4)
Ash D3	1 M NaOH	410	-24.91	91407	940 ± 35	1020 – 1190 cal A.D. (95.4)
Ash D4	1 M NaOH	770	-24.91	93683	970 ± 35	1000 – 1160 cal A.D. (95.4)
Average					946 ± 15	1020 – 1070 cal A.D. (30.9)
						1080 – 1160 cal A.D. (64.5)
Combustion						
Ash	A-B-A		-24.91	96373	905 ± 35	1030 – 1220 cal A.D. (95.4)
Average all					**939 ± 14**	1020 – 1160 cal A.D. (95.4)