ARCHAEOLOGIC SHERD DATING: COMPARISON OF THERMOLUMINESCENCE DATES WITH RADIOCARBON DATES BY BETA COUNTING AND ACCELERATOR TECHNIQUES

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ABSTRACT. Sherds can be dated by four independent methods: ¹⁴C beta counting on associated material, accelerator mass spectrometry on carbon traces on and within the sherd, thermoluminescence studies on minerals within the sherd, and stylistic form. Age analyses of materials and sherds from several sites are shown in this work. Each technique has its own frequently encountered non-laboratory sources of error. A combination of at least two independent techniques is indispensable for the highest level of confidence.

INTRODUCTION

Often, the most plentiful artifacts found in archaeologic excavations are ceramic sherds. Potsherds, with their often recognizable, distinctive styles, can form the basis of useful chronologic sequences used to trace the development of a region or culture. They are used as markers to correlate widespread sites and summarize the overall development of diverse civilizations or cultures.

Table 1 illustrates four principal methods of dating archaeologic sherds. ¹⁴C dating of associated charcoal was the first quantitative dating method for sherds and remains the most popular approach. Several studies have also been made by ¹⁴C dating carbonaceous remains extracted from large quantities of sherds (De Atley, 1980; Delibrias & Evin, 1979; Evin, 1983; Tauber, 1970; Taylor & Berger, 1968).

More recently, reliable techniques for thermoluminescence (TL) dating of sherds were developed (Aitken, Zimmerman & Fleming, 1968; Zimmerman, 1971; Fleming, 1979) which permitted quantitative age determinations on the actual marker artifact. Finally, the development of accelerator mass spectrometry (AMS) made possible ¹⁴C dating of very small amounts of food remains and other carbonaceous traces occasionally found on or within individual sherds (Bill *et al*, 1984).

The results of sherd dating from several archaeologic sites are presented here. Sites selected for sherd dating in south Florida were principally habitation mounds (Goggin, 1950; Doran, 1984; Carr & Beriault, 1984), all of Glades II period. Sherds from the Central Alpine region of Europe were from a variety of sites, stylistic descriptions of which were discussed previously (Bill *et al*, 1984). Comparisons of dates obtained with beta counting of associated charcoal, thermoluminescence of sherds, and AMS on included carbon are used to examine agreement among these methods.

Dating technique	Some advantages	Some disadvantages
Stylistic dating	 Dating of actual object of interest Done by archaeologist Least expensive 	 Usually uses information based on ¹⁴C dates, with the same errors of that method Subjective
¹⁴ C dating: beta counting of associated charcoal or other carbonaceous materials	 Extensive laboratory experience with method Moderate expense Objective 	 Does not date the actual object of interest Material is generally older, by an unknown amount, than the asso- ciated sherd Not always available in the site
¹⁴ C dating: AMS measure- ments on sherd soot, food remains, or included carbon	 Dating of actual object of interest Objective Food remains avoid the "old charcoal" problem 	 Soot comes from fire- wood that is older, by an unknown amount, than the sherd Sherds do not often con- tain soot or food remains Most expensive
TL dating	 Dating of actual object of interest Avoids "old charcoal" problem Moderate expense Objective 	 Sherd may have been ac- cidentally reheated after original firing Sherd might not have been completely zeroed in inefficient firing Some ceramics do not hold a TL signal

 TABLE 1

 Comparison of principal archaeologic sherd dating techniques

FLORIDA SITE DESCRIPTIONS

Addison Key

This represents the first attempt to excavate a deep stratigraphic black midden that could reveal an adequate sampling of the pottery sequence typical of the area in the Ten Thousand Island area of southwestern Florida. The only other ¹⁴C dates from this area were on Onion Key (also in this paper). The ¹⁴C and TL results correlate with the ceramic seriation sequence originally developed by Goggin (1950) and later expanded by Griffin (1984).

The mound is composed of shell with a black dirt midden (habitation mound) on top. The overall site consists of numerous mounds and ridges composed of shell which were dredged away in the early 1940s.

This site encompasses the Glades II period. There is a possibility of an earlier habitation period below the mound but this level is presently under water.

Rivermount Site

The site is composed of a black dirt midden on the New River in Broward Co, Florida. No ¹⁴C dating had been done on this river system which is the largest in SE Florida and represents an important component of this prehistoric settlement system.

This site was selected to obtain data on one of the few remaining sites along the river, the majority of sites having been destroyed by construction. The site itself is an elevated ridge along the river bank. The elevation is due to extensive cultural activity for several hundred years.

Panther Mound

This site is located on an everglades tree island in the southern everglades area. The mound sampled is a black dirt occupation midden which rises 1m above the surrounding island.

The site was selected as part of a National Park Service project involving a systematic sampling of all sites in the Big Cypress Preserve. This particular site held a wealth of small ceramic fragments from the Glades II period.

Onion Key

This site was also sampled as part of a National Park Service project. Previous ¹⁴C dates from this site (3) were anomalously old and indicated some type of contamination. A test pit was dug in the side of the mound from which charcoal and sherd samples were collected. The mound is composed of a shell base with a black dirt midden on top. This site and the Addison Key site are roughly contemporaneous.

EXPERIMENTAL

The benzene method was employed for the beta counting measurements (Polach & Stipp, 1967; Tamers, 1975). For AMS measurements, pretreated carbon samples were mixed with silver powder in a 1-to-5 ratio and pressed on copper targets (Bonani *et al*, 1984). General procedures of the ETH accelerator were described previously (Suter *et al*, 1984).

Thermoluminescence studies were made using the fine-grain technique (Zimmerman, 1971) with the 2 to 8μ size fraction. Radiation sensitivity was determined with calibrated ²⁴⁴Cm and ⁹⁰Sr plack sources. Uranium and thorium contents were obtained by alpha counting, and potassium was analyzed chemically. The quoted errors for TL are enlarged to include uncertainties in the environmental contribution to the observed signal where data was unavailable and best estimates were necessary.

Pretreatment of associated charcoal was done by standard techniques—crushing, hot acid and alkali solutions interspersed with rinsings with hot distilled water. Shell was strongly acid-etched to remove outer layers and checked by X-ray. Included carbon in sherds (for AMS) was treated somewhat differently. Each sherd was dried, crushed, and placed in deionized water. The minerals sank, leaving tiny pieces of carbonized organics (animal fats, plant fibers, or charcoal used in the tempering process) floating on the surface. Carbonized organics were isolated by centrifugation and given HCl to remove carbonates. The samples were then given a 0.5% NaOH heated bath for 1 hour and a subsequent 0.5% HCl rinse.

¹³ C/ ¹² C	-25.7	- 24.1 - 25.2	-20.8 -26.7	- 28.0	$^{-27.3}_{-25.7}$
AMS dating (yr BP) $\pm 1\sigma$	1110 ± 130	1090 ± 90 1520 ± 100	$1210 \pm 140 \\ 1320 \pm 140 \\ 1520 \pm 110 $	1550 ± 130	1080 ± 90 1650 ± 170
Sample no.	ETH-0285	ETH-0292 ETH-0284	ETH-0295 ETH-0221 ETH-0286	ETH-0283	ETH-0222 ETH-0291
$\begin{array}{c} TL\\ dating\\ (yr BP)\\ \pm 1\sigma \end{array}$	860 ± 70 900 ± 80 1100 ± 90	1200 ± 90 1450 ± 130 1510 ± 120 1410 ± 100	$\begin{array}{c} 960 \pm 110 \\ 930 \pm 100 \\ 970 \pm 100 \\ 1010 \pm 120 \\ 1040 \pm 120 \\ 1220 \pm 140 \end{array}$	1480 ± 100	1660 ± 140 1500 ± 100
Sample no.	UMTL-845 UMTL-846 UMTL-846	UMTL-848 UMTL-849 UMTL-850 UMTL-851	UMTL-852 UMTL-853 UMTL-853 UMTL-855 UMTL-855 UMTL-856	UMTL-842	UMTL-843 UMTL-844
1 ³ C/ ¹² C	-24.77 -25.13 -0.81 -26.11 -26.11 -0.57 -25.02	-25.62 -25.16 -25.21 -25.21 -1.10 -1.01 -24.20 -24.52 -24.52 -24.52 -24.52 -24.52 -24.52 -24.52 -24.52 -24.52 -24.52 -24.52 -24.52 -24.52 -24.52 -24.52 -24.52 -24.52 -24.52 -24.52 -24.52 -24.52 -24.52 -24.52 -24.52 -24.52 -24.52 -24.52 -24.52 -24.52 -24.52 -24.52 -24.52 -24.52 -24.52 -24.52 -24.52 -24.52 -24.52 -24.52 -24.52 -24.52 -24.52 -24.52 -24.52 -24.52 -24.52 -24.52 -24.52 -24.52 -24.52 -24.52 -24.52 -24.52 -24.52 -24.52 -24.52 -24.52 -24.52 -24.52 -24.52 -24.52 -24.52 -24.52 -24.52 -24.52 -24.52 -24.52 -24.52 -24.52 -24.52 -24.52 -24.52 -24.52 -24.52 -24.52 -24.5 -24.5		-25.09 -24.22 -24.48 -25.90	-25.57 -27.12 -24.69
Beta Counting (yr BP) $\pm 1\sigma$	$\begin{array}{c} 800 \pm 70 \\ 910 \pm 110 \\ 820 \pm 100 \\ 820 \pm 100 \\ 870 \pm 110 \\ 1080 \pm 140 \\ 1030 \pm 140 \end{array}$	$\begin{array}{c} 1010 \pm 150 \\ 1290 \pm 140 \\ 1330 \pm 130 \\ 1440 \pm 130 \\ 1530 \pm 110 \\ 1570 \pm 110 \\ 1370 \pm 100 \\ 1410 \pm 50 \end{array}$	$\begin{array}{c} 960 \pm 150 \\ 1000 \pm 150 \\ 2050 \pm 200 \\ 950 \pm 150 \\ 1220 \pm 140 \end{array}$	1480 ± 100 1570 ± 170 1400 ± 90 1280 ± 140 1530 + 110	1570 ± 170 1590 ± 170 1550 ± 120
Sample no.	1 UM-2532 UM-2531 UM-2530 UM-2530 UM-2528 UM-2527	UM-2509 UM-2523 UM-2522 UM-2519 UM-2516 UM-2515 UM-2515	UM-3091 UM-3092 UM-3093 UM-3094 UM-3094 UM-3095	UM-2405 UM-2404 UM-2403 UM-2401 UM-2401	UM-2399 UM-2402 UM-2400
Sample material	Key, Test Pit # Charcoal Charcoal Shell Charcoal Shell Charcoal	Charcoal Charcoal Charcoal Shell Shell Charcoal Charcoal	y Charcoal Charcoal Charcoal Charcoal Charcoal	<i>nt site</i> Charcoal Charcoal Charcoal Charcoal Charcoal	Charcoal Charcoal Charcoal
Level	Addision 33 55 5	9666001 1007	0nion Ke 1 2 3 4 5	Rivermou 3 5 6 7	8 9 Basal

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TABLE 2

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		Sherds	and charcoal fr	Pai om Shark Ri	nther Mound ver Slough, Everg	glades National	Park, Florida		
Level	Sample material	Sample no.	Beta counting ${}^{14}C$ dating (yr BP) $\pm 1\sigma$	1 ³ C/ ¹² C	Sample no.	$TL dating (yr BP) \\ \pm 1\sigma$	Sample no.	AMS dating (yr BP) $\pm 1\sigma$	¹³ C/ ¹² C %0
2 2	Shell	UM-3090	1170 ± 140	- 0.99	UMTL-858	490 ± 60	ETH-0290 ETH-0220	$\begin{array}{c} 1110 \pm 125 \\ 1240 \pm 240 \end{array}$	$^{-21.4}_{-27.7}$
					TARLE 4				
			Ζ.	Centr Veolithic to I	al Alpine region ron age sites in E	urope*			
Site		Sample no.	Beta counting ${}^{14}C$ dating (yr BP) $\pm 1\sigma$	¹³ C/ ¹² C	Sample no.	$\begin{array}{c} TL\\ dating\\ (yr BP)\\ \pm 1 \sigma \end{array}$	Sample no.	AMS dating (yr BP) ±1σ	¹³ C/ ¹² C
CH Egolzwi "Egolzwil 4'	ii (LU)	KN-1021 H-228/276 H-990/977	5880 ± 250 5940 ± 300 5750 ± 995		UMTL-861	4660 ± 930	ETH-0236	5470 ± 240	-21.0
CH Egolzwi	(ILU)	B-2727 B-2727 B-9798	5570 ± 200 5570 ± 200 5890 ± 900		UMTL-865	5850 ± 117	ETH-0133	5700 ± 150	-23.4
I Val di Pine	e 11-1	D-2120	007 I 1000		UMTL-866	2870 ± 570	ETH-0137	2970 ± 260	-23.1
FL Balzers	uua hal"	B-3910	2330 ± 190		UMTL-862 (hurned clav)	2500 ± 500	ETH-0138	2400 ± 180	-24.2
Valiate Dr					(Sherd)	2340 ± 470			
FL Balzers ''Areal Fose	ʻr"				UMTL-867	2410 ± 480	ETH-0139	2580 ± 120	-24.0
* The A	AMS and be	eta counting ¹⁴ C	dates have been r	reported previ	ously (Bill et al, 198	(4), except for ET	H-0236, which is	s a recent measure	nent.

TABLE 3

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After rinsing to neutral and drying, the samples were carbonized in ultrapure nitrogen to remove volatiles and adsorbed CO₂.

RESULTS AND DISCUSSION

Ages measured on contemporaneous materials from six archaeologic sites have been studied to compare the three independent dating techniques used to produce the dates. Results are shown in Tables 2–4. ¹⁴C dates have been corrected for isotopic fractionation in nature with ¹³C (Stuiver & Polach, 1977) and, in the case of marine shell samples, for a reservoir effect of 410 yr in Florida waters (Druffle & Linick, 1978). ¹⁴C dates are based on the 5730 yr half-life and have been corrected for the De Vries effect (Klein *et al*, 1982). This is necessary for the comparison of the ¹⁴C and TL results.

Results show that all three of the instrument dating approaches have produced at least one apparently anomalous date. In the Onion Key site, one beta counting ¹⁴C measurement (UM-3093) is too old. A possible explanation might be original use of old wood (Smiley, pers commun). The three AMS measurements here are all a little older than the TL dates, but are within 2σ statistics. AMS dating of charcoal or soot on or in the sherds could also be affected by old wood or pitch in the fire. Also, the charcoal could have been used as temper in manufacture.

Panther Mound shows a TL date (UMTL-851) that is apparently too young. Although the reason is uncertain, the sherd may have been subjected to accidental reheating after its manufacture. This could have occurred by a brush fire or fire that burned after the stratum had been partially buried.

The Rivermount site shows an AMS date (ETH-0222) that appears somewhat young, the reason for which is also not apparent. However, the sherd may have been intrusive, which, likewise, could also explain the anomalous TL result in Panther Mound. The possibility of sampling intrusive material is particularly serious in archaeologic sherd dating. Often, the best sherds are kept for collections and stylistic identification. Sherds without distinctive markings are likely to be those sacrificed in the destructive processes of the TL or AMS dating.

The Central Alpine region dates are in general agreement. TL measurements show large errors since the sherds did not have associated soil samples available for measurement. In these cases, as with samples from museum collections, TL error terms of 20% are assigned to cover all reasonable possibilities of true water content and environmental radioactivity contribution to the TL signal. Nevertheless, Egolzwin 4 (UMTL-861) shows the possibility of an inconsistency.

CONCLUSION

Although radiochemical and radiophysical dating methods are now highly developed and efficient, discrepancies between dates and apparently indisputable archaeologic evidence regularly appear. The problems are usually not in laboratory measurements, but, rather, in the field. For example, in a disturbed site, charcoal found close to a sherd cannot confidently be associated with the sherd. Wood collected by indigens for their camp fire could be lying on the ground dead for hundreds of years before being used. Also, sherds could have been reheated by brush fires 1000 yr after manufacture.

The existence of significant undetermined errors cannot be excluded from any age determination. No method is immune to processing grossly incorrect dates when unknown problems may exist with the sample at the collection site. Our results illustrate that this situation can occur frequently. A combination of at least two independent dating techniques is indispensable for the highest level of confidence.

REFERENCES

- Aitken, M J, Zimmerman, D W and Fleming, S J, 1968, Thermoluminescencent dating of ancient pottery: Nature, v 219, p 442-443.
- Bill, J, Keller, WA, Erne, R, Bonani, G and Wölfli, W, 1984, C14 dating of small archaeologic samples: Neolithic to Iron age in the Central Alpine region: Nuclear Instruments & Methods, v 233 (B5), no. 2, p 317–320.
- Bonani, G, Balzer, R, Hofmann, H J, Morenzoni, E, Nessi, M, Suter, M and Wölfli, W, 1984, Properties of milligram size samples prepared for AMS C-14 dating at ETH: Nuclear Instruments & Methods, v 233 (B5), no. 2, p 284-288.
- Carr, R and Beriault, J S, 1984, Prehistoric man in south Florida, in Gleason, P, ed, Environments of south Florida present and past, no. 2: Miami Geol Soc, p 1-14.
- De Atley, S. 1980, Radiocarbon dating of ceramic materials: progress and prospects, in Stuiver, M and Kra, RS, eds, Internatl ¹⁴C conf, 10th, Proc: Radiocarbon, v 22, no. 3, p 987 - 996.
- Delibrias, G and Evin, J, 1979, La datation par le radiocarbone des sites de Tintan et Chami, Mauritanie: Centre recherche anthropol prehist ethnog Mem, v 28, p 140-150.
- Druffle, E M and Linick, T W, 1978, Radiocarbon in annual coral rings of Florida: Geophysical Research Letters, v 5, p 913-916.
- Doran, G, 1984, Proton induced X-ray emission analysis of prehistoric Florida ceramics: Florida Anthropologist, v 37, no. 3, p 115-119.
- Evin, J. 1983, Materials of terrestrial origin used for radiocarbon dating, in Symposium on C-14 and archaeology, Groningen, Netherlands, Aug 1981: PACT, v 8-IV.1, p 235-275.
- Fleming, S, 1979, Thermoluminescence techniques in archaeology: Oxford, Clarendon Press.
- Goggin, J M, 1950, Stratigraphic test in the Everglades National Park: Am Antiquity, v 15, no. 3, p 228–246.
- Griffin, J W, 1984, Excavations at the Granada Site, vol 1: Archaeology and history of Granada: Tallahassee, Florida Dept of State.
- Klein, J. Lerman, J.C. Damon, P E and Ralph, E K, 1982, Calibration of radiocarbon dates: tables based on the consensus data of the Workshop on Calibration of the Radiocarbon Time Scale: Radiocarbon, v 24, p 103–150.
- Polach, H A and Stipp, J J, 1967, Improved synthesis techniques for methane and benzene radiocarbon dating: Jour Applied Radiation Isotopes, v 18, p 359–364. Stuiver, M and Polach, H A, 1977, Discussion: Reporting of ¹⁴C data: Radiocarbon, v 19 p
- 355 363
- Suter, M R, Balzer, R, Bonani, G, Hofmann, H, Morenzoni, E, Nessi, M, Wolfli, W, Andrée, M, Beer, J and Oeschger, H, 1984, Precision measurements of C-14 in AMS-some results and prospects: Nuclear Instruments & Methods, v 233 (B5), no. 2, p 117-122.
- Tamers, M A, 1975, Chemical yield optimization of the benzene synthesis for radiocarbon dating: Jour Applied Radiation Isotopes, v 26, p 676-682.
- Tauber, H, 1970, Radiocarbon dating of potsherds from Tell Shimshara, in Mortensen, P, ed, Tell Shimshara: Copenhagen.
- Taylor, R E and Berger, R, 1968, Radiocarbon dating of the organic portion of ceramic and wattle-and-daub house construction materials of low carbon content: Am Antiquity, v 33, p 363-366.
- Zimmerman, D W, 1971, Thermoluminescent dating using fine grains from pottery: Archaeometry, v 13, p 29.