New Analytical Approaches to South Texas Cultural Assemblages

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South Texas contains thousands of prehistoric sites scattered across the inland regions. However, relatively few prehistoric inland sites were systematically excavated and reported prior to 1995 (cf. Black 1986; Collins et al. 1969; Hester 1969). The most intensive excavations were conducted at Choke Canyon Reservoir (Brown et al. 1982; Hall et al. 1986; Highley 1986; Scott 1982). Further west, excavations have also been conducted at Chaparrosa Ranch (Hester 1978; Montgomery 1978) and in the Tortugas Creek area (Hester and Hill 1972; Inman et al. 1998). Over the last five years, this situation has been gradually changing thanks in large part to the Texas Department of Transportation (TxDOT), Environmental Affairs Division, Archeological Studies Program's commitment to funding testing and excavation projects at buried sites along planned expansions of South Texas roadways (Mahoney et al. 2002; Quigg et al. 2000, 2002; Taylor and Highley 1995; Vierra 1998) as well as to other private companies initiating development projects and funded cultural resource investigations (Quigg and Cordova 1999, 2000; Miller at al. 2000).

As is the case elsewhere, most South Texas sites contain cultural assemblages dominated by lithic debitage, chipped stone tools, and burned rocks. Major classes of organic material, such as charcoal and bone, are absent or present in only limited numbers, with the notable exception of the Late Prehistoric Hinojosa site (Black 1986) and 41LK201 (Highley 1986). In the absence of direct evidence of subsistence resources, such as bones and burned seeds, other sources of information must be located. To extract more information from sites with restricted cultural assemblages, TRC Mariah Associates Inc. (TRC) has directed some old and some new analytical approaches towards these types of assemblages over the past few years. New analytical approaches have focused on burned rocks scattered across prehistoric occupation areas and from definable features, including examining burned rock size classes, analyzing their chemical composition, and even radiocarbon dating the burned rocks. Although long ignored, burned rocks are a major part of the cultural assemblage on many sites, they may be the most abundant artifact type in the assemblage, and they play a very important role in understanding specific human behavioral patterns as well as provide significant information concerning a variety of important issues.

Why focus on burned rocks? Nearly every archaeological site in South Texas has them, and they are not unique or restricted to any specific time period or culture. Most investigators assume that burned rocks were used primarily for cooking food. Natural rocks were collected and heated in fires, and the stored heat was used to cook food. If this general hypothesis is reasonable, then these rocks probably came into contact with food items and potentially absorbed some food residues while cooking the food (e.g., ovens, stone boiling). This notion—that burned rock matrices may contain residues from the foods they were used to cook—inspired us to focus on burned rocks as a source of information about what foods prehistoric peoples cooked and how they were prepared. It became necessary to locate existing, and to develop new, chemical procedures to extract food residues from the burned rocks and to learn how to interpret the results. When some initial chemical techniques had been identified, researched, and determined to have potential, we began to submit burned rock samples to various specialists to investigate what was in these burned rocks and what that might tell us. Time and space are not sufficient here to provide an in-depth discussion of the details concerning the analytical techniques, sample selection, handling procedures, extraction processes, and the various pitfalls of the techniques, nor to discuss all that has been learned from burned rock analyses; therefore, only selected results will be highlighted here. More detailed information on the techniques, samples, and results can be found in the reports of archaeological investigations upon which the following presentation is based (Quigg 1999; Quigg and Cordova 1999, 2000; Quigg et al. 2000, 2001, 2002).

This article focuses on three analytical techniques that have major implications to understanding the prehistory of South Texas and that are probably applicable to many other regions. The first technique targets visibly undetectable organic residues in the interiors of burned sandstone. Although rocks are thought of as impenetrable materials, minute pores and voids are often present in the rock matrix, specially in rocks like sandstone and limestone. These tiny pores expand when the rock is heated. During the cooking process, these tiny pores can become filled with minute food residues. Thus, the organic residues trapped in the tiny pores of the burned rocks can provide a broad picture of the foods that were cooked. The technique focuses on the fatty acid component of lipid residues, which must first be extracted and then identified. Second, stable carbon and nitrogen isotopes can also be extracted from these residues to aid in the identification of the classes of residues present. Third, we anticipated that the organic residues could be radiocarbon-dated to provide absolute ages for the heating and/or cooking event in which the rocks were used, thus providing a date for associated features, events, components, and other sites that also lack organic preservation.

LIPID RESIDUES

The fatty acid extraction was the easy part, although this is a very sensitive procedure. This analytical technique is quite complex and requires some sophisticated equipment, including a gas chromatograph machine, to obtain raw data about the chemical content of burned rocks that then must be interpreted by a knowledgeable specialist. Fatty acids are insoluble in water and relatively abundant. Unsaturated fatty acids, however, decompose more rapidly than saturated fatty acids; thus, the latter are the primary identifiable component. Interpretations are based on extensive knowledge of food chemistry and backed by experimental data. Understanding the decomposition of certain compounds is extremely important since these compounds will be under-represented in the analyses of old, decayed residues. This technique was originally designed for extracting residues from pottery sherds (Marchbanks 1989; Malainey 1997). Shifting the focus to burned rocks was troublesome and a learning process. The first attempts that targeted burned rocks were performed on samples from 41ZP176 and published in 1999 (Malainey 1999; Malainey et al. 1999a, b, c). This technique is still in the developmental stages, with new information and a greater understanding of the complexities constantly increasing and upgrading. That means that although we can obtain high-quality raw data during this chemical extraction process, there is a definite need to expand the reference/comparative collection to include South Texas samples, and the interpretation may change over time as the analyst gains more understanding and becomes more sensitive to minor variations in the numbers derived in the process. This is not an exact science and precise identifications to species level are not possible, but broad categories of foods can be recognized (e.g., plants, large herbivore, fish/corn) rather than individual species of plants and animals.

Of the 115 burned rock samples submitted for fatty acid analysis from all sites, we have had about an 86% success rate in obtaining interpretable residues. Of the 14% that could not be interpreted, most samples still yielded fatty acids but in such limited quantities as to make interpretation unwise. Burned rocks from four sites along the Rio Grande between Laredo and Falcon Reservoir constitute most of our sample on the recovered fatty acids and the results were summarized according to six broad food categories (Table 1). These broad food categories each include many different individual plants and animals, though these definitions may become more refined over time. Even though we cannot currently identify specific foods or plants, the interpreted results provide an intriguing glimpse into possible prehistoric subsistence patterns at these four sites.

For example, the Lino site (41WB436) reveals a great diversity of potential food-groups compared to the more focused use of fish/corn/mesquite beans at 41ZP176 and 41ZP364 (see Table 1). The variability, or in some cases the lack thereof, within one site and among sites is interesting. The diversity within one site may imply that no single resource supplied all of the prehistoric inhabitants' dietary needs. The burned rocks from the two Falcon Reservoir sites (41ZP176 and 41ZP364) did not yield any large herbivore residues, though the two sites near Laredo did so. This finding might reflect one or more possibilities, including, but not limited to, group preferences, age differences in the sites, or lack of the large herbivore resources (i.e., deer, antelope, or bison) in some regions. All four sites were occupied generally during the Archaic period, which covers a roughly 4000 year time span between about 6000 and 2000 years ago, though some minor use during recent times was also indicated.

The presence of certain fatty acids provide valuable clues as to whether the residue was of plant or animal origin. Confirmation may be possible in the future through the identification of sterols in the residue. Analysis of experimentally decomposed plant and animal cooking residues from the study area should enable the archaeologist to identify the most likely candidates.

STABLE CARBON AND NITROGEN ISOTOPES

In an attempt to help refine the broad fatty acid interpretations, we subjected the same burned rock samples to another chemical technique, stable carbon and nitrogen isotope analyses. The combination of these two chemical techniques targeting residues from inside burned rocks should increase our ability to identify certain food groups that the burned rocks were used to cook. Interpreting stable isotope data is as complex as analyzing fatty acids; however, isotope analyses are already established techniques (used mostly in biological disciplines). Stable carbon isotope signatures reflect how plants photosynthesize sunlight, and are generalized into three broad groups, C_4, C_3, and CAM. The chemical analysis of stable carbon isotopes yields a negative number that reflects one of these three broad plant groups or animals that fed on the various plant groups. The operating assumption is that the stable carbon value obtained would aid in distinguishing what plant groups were represented in the residues. The same is true of the nitrogen isotope value, although less is understood concerning nitrogen isotopes.

Table 1. Summary of lipid residue results on burned rocks from four prehistoric sites in South Texas.

Broad Food Categories	41ZP176 (n = 9)	41ZP364 (n = 10)	41WB437 (n = 43) Lino Site	41WB557 (n = 52) Boiler Site
Similar to Large herbivore			23%	46%
Similar to Large herbivore + plants			5%	20%
Similar to Plants or probable plants	22%	40%	28%	12%
Similar to Fish/corn/mesquite beans	56%	30%	7%	
Similar to beaver/Texas ebony seeds	11%	30%	7%	
Similar to Seeds/nuts/fruits			2%	10%
Not identifiable/insufficient residues	11%		26%	12%

The biological literature provides stable carbon and nitrogen isotope data for most plants, but the published values have been derived from the leaves rather than from the fruit or seeds. Clearly, the fruits and/or seeds, not the leaves, represent the commodity that was potentially consumed by prehistoric hunter-gatherers. Therefore, we began to amass a body of data on the isotopic values of modern seeds, fruits, and other representative food products (e.g., nuts) in the regions of interest. To date, about 100 samples of modern plant and animal remains have been analyzed to establish their stable carbon and nitrogen isotope values. Bones from animals that consumed various plant parts tend to yield isotopic values similar to the plants consumed by the animal. However, chemicals within animals' bodies alter the intake values (through fractionation), which add considerable complexity to the interpretation of results as the consumed plants passed upwards through the food chain. Consequently, this change of value is a complicating factor that must be carefully considered in the results and interpretations.

We completed 137 isotopic analyses on the same burned rocks that were analyzed for their fatty acid content. Although we have results from about 95% of the samples, it is difficult to summarize these data. Many factors influence the isotopic results, one of which is the amount of organic residue that is used in the detection process. In fact, many of the sample results have yielded numeric values that may indicate that the results are not to be trusted because the sample amount measured was to small to have confidence in the obtained value.

Incorporating the results from these two chemical techniques should help to narrow the interpretation of what broad food groups might be represented and/or to eliminate some potential food groups. For example, if the fatty acid interpretation indicates that large herbivore meat was prepared, and the isotopic analysis provides a less negative value (i.e., -13.3‰), the combination of the two results indicates that the large herbivore meat is probably bison. Bison have less negative values because they subsist extensively on C_4 grasses. If the carbon isotope value is more negative (i.e., -22‰), then the large herbivore would likely be a deer or antelope. In an instance where seeds/nuts/fruits is inferred based on the fatty acid results, a very low nitrogen isotope value (i.e., 1‰) may indicate that the fatty acid source was likely derived from a legume such as a mesquite bean. Thus, even though neither of the two chemical analyses provides precise identifications, combining the two techniques allows for more precision in isolating the food groups that were potentially cooked by the burned rocks.

These chemical approaches move toward the identification of the broad food groups that probably constituted the subsistence base at sites that lack direct faunal evidence for identifying subsistence. Even if animal bone and/or mussel shell remains are recovered from archaeological sites, the plant products consumed at these same sites is more often not preserved. One way to detect whether or not plants were part of the subsistence base would be through chemical analyses.

RADIOCARBON DATING

The other technique that was initiated at these four South Texas sites was the radiocarbon dating of the organic resides trapped inside the burned rocks. Specifically, the fatty acid residues discovered in the burned rocks were targeted for radiocarbon dating. Currently, we have not positively identified the dated organic material as fatty acids but, based on the work thus far, fatty acids are the most likely source of the dated organic matter. Once we learned that organic residues were retained inside burned rocks, dating these residues was the next logical step to pursue. Considerable time was spent with the technical experts at the radiocarbon facility, Beta Analytic, Inc., to ensure that we were on the right track with our assumptions. Here again, the dating of burned rocks is a brand new avenue and we are still learning about the pitfalls, although we have many positive results to go along with a few strange or unanticipated ages. We are, however, excited to state that it is a fact that organic residues are present in burned rocks and it is possible to date them through the accelerated mass spectrometry (AMS) radiocarbon process. Even though we receive an absolute age back from the laboratory, we must be very cautious in how we treat that date, since there may be more to what that number represents than first suspected. More controls and experiments are required to help resolve some of the potential problem areas and allow more confidence in using the acquired date as an indication of a targeted cultural event.

In trying to obtain absolute radiocarbon ages on burned rocks, we have achieved a 98% success rate from the 52 samples run through 2002. The standard deviation on the obtained assays ranged from 40 to 60 years, which is similar to that of most charcoal results. This brief article is not going to attempt to discuss each and every sample or discuss all that we have learned over the last six years; however, I would like to provide some examples, including some highlights as well as some "lowlights."

Ten burned rock samples were paired with charcoal samples in direct association with one another in archaeological context to investigate the correlation between the burned rock results and the charcoal radiocarbon results. Running dates on two different types of sample material from the same context allows for a general level of confidence to be established for the burned rock dates. Charcoal is considered the standard and most reliable means of establishing the absolute age of most cultural events. Of the 10 burned rock/charcoal sample pairs dated, five pairs were considered to exhibit good correlations between the two assays and five were considered to have poor correlations. An example of good correlation is from an excellent context in a shallow basin feature (Feature 10) at the Lino site (41WB437) in which one burned rock and one charcoal result yielded two radiocarbon assays that were only 10 radiocarbon years apart (Quigg et al. 2000). An example of a poor correlation is from a slightly scattered burned rock cluster (Feature 9d) at 41ZP364 in which the charcoal date was nearly 900 years older than the date obtained on a burned rock (Quigg and Cordova 2000). At the Boiler site (41WB557), we ran two charcoal samples and two burned rock samples from a single small, shallow basin feature (Feature 21). Three of the four assays clustered within 70 years of each other (between 170 and 240 years B.P.) with one outlier at 540 B.P. (Quigg et al. 2002). Also at the Boiler site, we ran two charcoal samples and three burned rock samples from a single burned rock cluster (Feature 22). Four of the five results were within 20 radiocarbon years of one another with one outlier only 240 years from the clustered dates (Quigg et al. 2002).

The Boiler site yielded very limited charcoal, and the charcoal that was present often was not directly associated with identified features. The exceptions were the two examples discussed above. The lack of charcoal directly associated with the burned rock clusters eliminated the possibility of cross-checking the ages of the burned rocks against charcoal dates. Instead, we dated multiple (two to four rocks) burned rock samples from selected features throughout the profile to determine if burned rocks within the same cluster would yield similar ages. As before, we got mixed results on the clustered samples. In Feature 14, a burned rock cluster that was interpreted as a small, dispersed dump, three separate burned rocks yielded three individual residue dates that were only 30 radiocarbon years apart (860, 880, and 890 B.P.) with standard deviations of less than 50 years (Quigg et al. 2002). In another loose cluster of burned rocks (Feature 23), three burned rock residue dates were only about 250 years apart.

In four instances, we dated two ends of the same burned rock to determine if the assays would be consistent with one another. Again, this approach provided mixed results. The four pairs of dates yielded radiocarbon results that were 80, 100, 390, and 1710 years apart. In general, three of the four pairs yielded what I believe to be acceptable ranges of ages and provided a relatively clear indication of the age of that particular feature.

The 50 radiocarbon dates on residues obtained from the sandstone burned rocks documented a broad range of time. The youngest burned rock assay obtained was 80 years B.P. The oldest date obtained was 7210 B.P. These results are exciting and there is no reason to believe that the oldest cultural events known in North America would be out of the range of this approach. These dates document that organic residues last at least 7000 years even in some environmental conditions such as those found in South Texas. This is especially important, as the older archaeological sites tend to yield less preserved charcoal or other organic materials with which to pursue radiocarbon dating. Thus, the burned rocks appear to be an alternative source for obtaining absolute radiocarbon dates in sites lacking other materials for dating.

OTHER ANALYSES

In an attempt to broaden our knowledge about the organic residues trapped inside burned rocks, we have subjected samples of burned rocks and natural rocks to various other analyses. Petrographic analysis was conducted on two natural sandstone pieces and compared to four pieces of burned sandstone from the Boiler site to investigate whether or not organic residues might be detected under the polarized light of the microscope. The petrographic results revealed nothing out of the ordinary and no materials were suspected to be organic residues (Hill 2002). The effects of firing were observed and differences in porosity and permeability were noted in two different types of sandstone (Hill 2002). It was suspected that the low power used in petrographic microscopes hindered the analyst's ability to detect minute residues.

Ground up burned sandstone and natural sandstone samples were submitted to a soils laboratory for chemical characterization, including such attributes as "percent organic matter," using two different techniques —percent carbonate by two different means—the carbon nitrogen ratios, percent carbon and nitrogen, soluble salts, available phosphate, and total phosphates (Quigg et al. 2002). Comparisons of the burned rock results to the natural rocks from the same context indicated the burned rocks yielded noticeably higher levels of carbonates, calcite, and dolomite, as well as high percentages of carbon. These chemical results were not unexpected and confirmed our earlier assumptions that burned rocks retain organic residues in their interiors.

We have encountered complicating factors that cause some concerns in interpreting the radiocarbon dates of the organic residues. Two pieces of what appeared to be non-burned sandstone from the lower part of the cultural deposits at the Boiler site were also submitted for radiocarbon dating. To my surprise, both apparently natural rocks yielded radiocarbon assays that dated to the Early Holocene (6610 ± 40 and 7220 ± 50 B.P. [Quigg et al. 2002:323]). The fact that these natural rocks yielded absolute radiocarbon ages presents potential problems in the interpretations of the ages obtained from cultural burned rocks. If these were truly natural rocks, then apparently natural soil organic residues may have penetrated the rocks and thus were dated. These natural organic residues might cause some background noise to the culturally derived residues that we are trying to date.

I would like to briefly address this apparent problem. If a natural rock contains natural organic residues prior to being used in a cultural event, it may not matter that much. Let me explain by quickly outlining the rock's potential use cycle to illustrate what I mean. The natural rock is collected (it potentially contains natural residues), then the rock is heated in a fire, presumably an open wood fire that would most likely reach temperatures above 600 degrees Celsius. Then the hot rock was used in one or more ways (boiling, roasting, or an earth oven) to transfer heat from the rock to the foods. When the rock comes into contact with the cultural food residues, it is my contention that the rock would absorb minute food residues into its interior. Once the rock cooled, it was either discarded or recycled in more use episodes, which again would begin with another heating episode. It is my contention that most, if not all, natural organic residues inside the unheated rock would most likely be burned off and destroyed in the initial cultural heating of the rock. Consequently, as the rock cools and absorbs food residues from its cultural use, it would be those cultural residues that filled the tiny pores and would be retained. Therefore, if natural, older organic residues were once present in an unheated rock that was subsequently selected for cultural use, those older residues most likely would be destroyed during the cultural heating event. The organic residues that are extracted, interpreted, and radiocarbon dated would represent cultural use episodes and not natural contaminants. When Dr. Malainey identifies soil lipids in the extraction process, which are fundamentally different in composition from food lipids and do not interfere with the identification of archaeological cooking residues (Malainey 1997), the soil lipids are cleared and ignored prior to interpreting the results.

In another attempt to learn more about the heating process—specifically the potential number of heating events and the temperatures to which rocks were heated—we have subjected at least 19 cultural burned rocks to demagnetization studies. Although this approach is still being developed, some differences in the number of heating events have been detected. Potential heating temperatures were identified and provide some understanding of the temperature ranges that the rocks were subjected to. A few burned rocks appear to have been used as boiling stones (Takac 2000).

SUMMARY

In summary, new analytical approaches and techniques directed at an often overlooked but ubiquitous artifact type—burned rocks—hold promise as means to gain greater insight into the subsistence practices of prehistoric hunter-gatherers at archaeological sites that lack direct evidence of such activities (e.g., faunal bones, carbonized plant remains). We have also pursued various analyses of the burned rocks to extract as much information as possible concerning their contents. Although the two chemical approaches applied to the organic residues extracted from inside burned rocks are complicated and can only be performed by specialists, they appear to have merit even though some results are questionable. These techniques have considerable potential to extract data that will contribute significant information to our understanding of the subsistence practices of prehistoric peoples in South Texas. As an outgrowth of learning that organic residues are preserved in burned rocks, it becomes important to investigate if these organic residues can be dated. Currently, there are some unknowns and some potential problem areas yet to be resolved, but it is quite apparent that burned rocks can be dated and, with care, they can be used as a means of establishing the age of cultural events. We believe that these new techniques, applied thoughtfully and carefully, will provide significant advances to our understanding of South Texas prehistory and, presumably, to other regions.

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