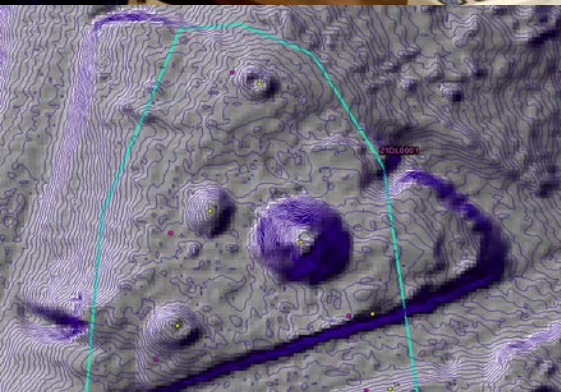
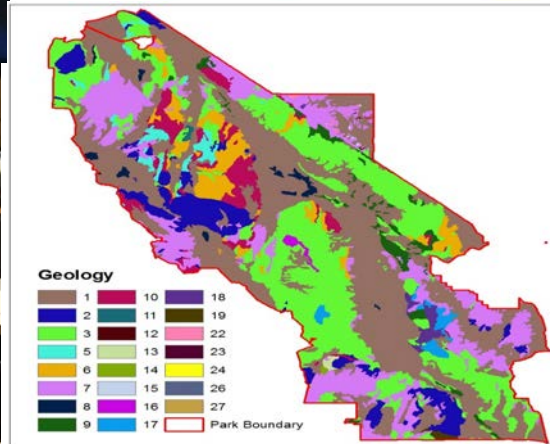
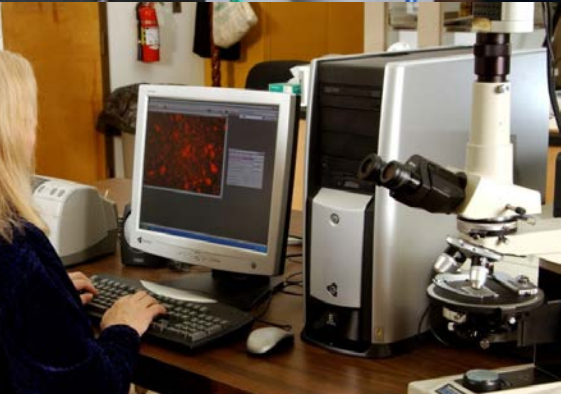
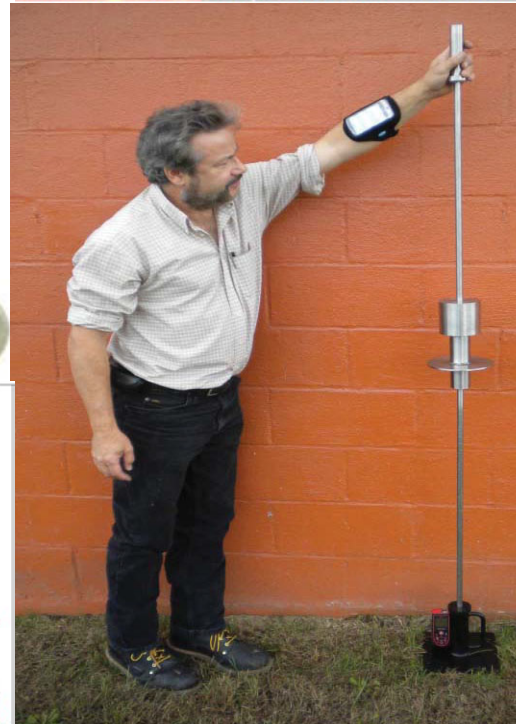
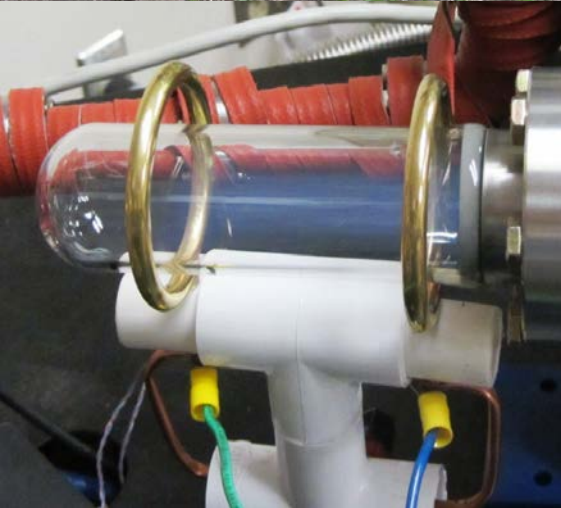
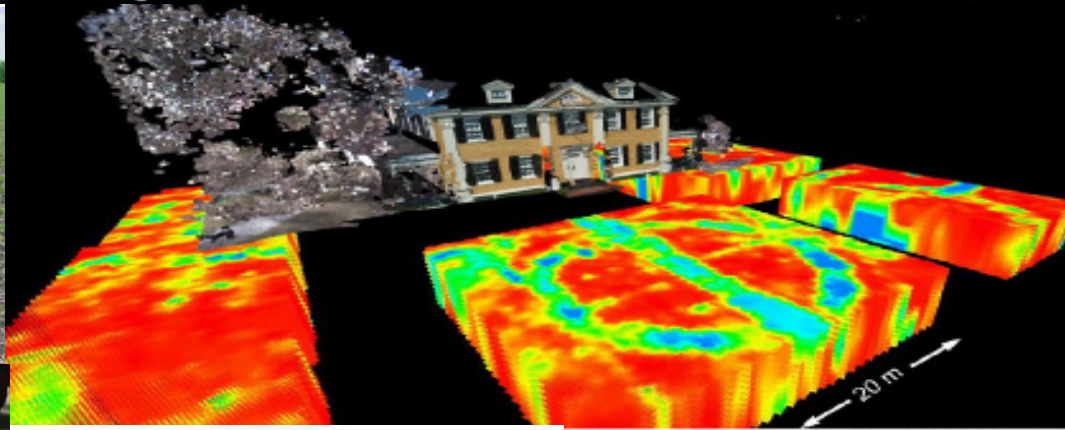


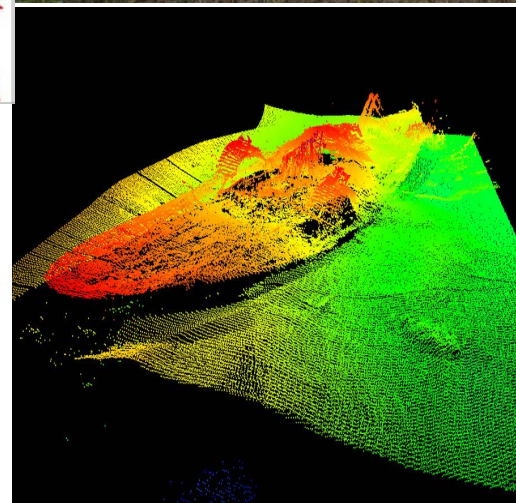


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2015



Twenty-Year Retrospective of National Center for Preservation  
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**Proceedings of the  
Twenty-Year Retrospective of  
National Center for Preservation Technology  
and Training  
Sponsored Archeology**



San Francisco  
April 18, 2015

Edited by: Tad Britt, NCPTT

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# The National Center for Preservation Technology and Training: A Twenty-year Retrospective of Sponsored Research for Archeological Innovation

Since 1994, the National Center for Preservation Technology and Training, a program of the National Park Service ([ncptt.nps.gov](http://ncptt.nps.gov)), has engaged in state-of-the-art research in archeological treatments and technologies. The Center provides grants, education, research, and training opportunities in the areas of archeology and collections, architecture and engineering, materials conservation, and historic landscapes. To date, over \$10 million dollars have been spent on sponsored research via our grants program. This symposium is a 20 year retrospective and is focused on the innovative contributions of the award recipients to the archaeological sciences, methods, and technologies.

Specifically, the authors were asked to re-examine their original work and address the impact of their research on their respective fields; how their work has influenced their research; and progress in their study areas since their initial award. These proceedings include topics ranging from the development and fielding of magnetic susceptibility, archaeogeophysics, and a friction cone-penetrometer, to plasma extraction 14C analysis, site location probability models, ceramic thin-section analysis, freshwater shell artifact and temper sourcing, and Native American consultation protocols.

The papers and presentations included here reflect the depth and breadth of the types of studies funded by NCPTT. The materials herein are presented in their entirety in their original format with minor editing. We hope you find the proceedings interesting and informative, and look forward to the next twenty years. Special thanks to Tad Britt, Chief of NCPTT's Archeology & Collections program, who conceived and organized this retrospective. Thanks also to both the authors and readers for your contribution and patience in getting the publication ready.

Kirk A. Cordell  
Executive Director  
National Center for Preservation Technology and Training  
April 2015

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# Cold Plasma Oxidation and “Nondestructive” Radiocarbon Sampling

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Mark MacKenzie (Conservation Unit, Museums of New Mexico)

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The development of radiocarbon dating in the mid-twentieth century has revolutionized archaeological chronology (Libby 1955; Wood 2015). The combination of relatively simple theory and the ability to estimate relative isotope abundances resulted in the generation of age estimates for samples of organic carbon from archaeological contexts. In the ensuing decades, the power of radiocarbon dating has increased dramatically. Archaeologists are now much more sophisticated in understanding underlying principles, leveraging that knowledge into a far more reliable selection of samples and interpretation of results. The calibration of atmospheric variation in cosmogenic radiocarbon levels has resulted in much improved calendric date range interpretations, and isotope measurement with accelerator mass spectrometry (AMS) has reduced the size of samples, allowing dating based on annual plant parts.

Even with all of these advancements, a basic limitation has persisted: the perception of radiocarbon dating as a destructive technique. Pretreatments and the transformation of organic compounds into forms of carbon that can be either counted or measured require the destruction of either 10ths of grams or at best milligrams of sample material. Acid-base-acid pretreatment effectively digests sample material during the removal of potential carbonate, oxalate, and humic acid contamination, while graphitization is a common transformative step in preparation of samples for isotope measurement (Aitken 1990; Taylor 1987). Under the conventional approaches available today, the decision to date an object is a decision to sacrifice a tangible part of it to a destructive process.

## Plasma Extraction

In the late 1980s, Marvin Rowe was challenged by colleagues in rock art studies with the problem and potential of dating small amounts of ancient pictograph pigments. The thin-layer applications of organic pigments (charcoal) and the potential use of

organic binders in mineral paint layers were outside the realm of the normal approaches to radiocarbon sampling. Amounts of organic carbon in the pigments were extremely small, contamination from carbonates was a risk in many samples, and the amounts of datable carbon that would survive pretreatment were problematic for dating.

In 1989 Rowe was inspired to explore how low-pressure, low-temperature, oxygen plasmas could be used to extract organic carbon from pictograph samples for dating. Jon Russ, Marian Hyman, and Rowe (Figure 1) assembled the first plasma sampling apparatus at the Chemistry Department at Texas A&M University (TAMU), and the first dating sample was produced in 1990 (Russ et al. 1990, 1991). The experimental nature of the venture was emphasized as the radio frequency (RF) generator caught fire and self-destructed at the end of the first sampling run, but the potential of the technique was also confirmed. Over the next several years, three more generations of plasma systems were built (Russ et al. 1993; Chaffee et al. 1993a, b; Ilger et al. 1994b), and additional rock art dates were produced (Hyman and Rowe 1992; Russ et al. 1992a, b; Chaffee et al. 1994a, b, c; Ilger et al. 1994a, b, 1995, 1996).



Figure 1. Marian Hyman, Marvin Rowe, and Jon Russ at the Chemistry Department, Texas A&M University.

A significant advantage of the plasma technique is that the inorganic rock substrate (often including carbonates) does not decompose

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during exposure to low energy oxygen plasmas. This eliminates the need to use extensive acid pretreatments because the plasma temperatures used ( $< 150^{\circ}\text{C}$ ) are below the decomposition temperatures of both carbonates and oxalate minerals, and only organic carbon is isolated for radiocarbon measurement (Russ et al. 1992b; Chaffee et al. 1993a). Later research added the argument that plasma oxidation is preferable to conventional acid pretreatments because acid washes may not completely remove oxalate minerals, which are commonly associated with rock surfaces and which would contaminate conventional radiocarbon dates (Hedges et al. 1998; Armitage et al. 2001).

In 1996, Rowe received a grant from the National Center for Preservation Training and Technology (NCPTT). That funding allowed Rowe and his TAMU students to date further pictograph samples and continue to refine the plasma technique for radiocarbon sampling (Armitage et al. 1997, 1998, 2000b; Hyman and Rowe 1997a, b; David et al. 1999a, 2001; Hyman et al. 1999; Pace et al. 2000; Diaz-Granados et al. 2001; Steelman et al. 2001). By this time the technique was established as arguably the most reliable method for dating pictographs drawn with inorganic pigments: red, yellow, brown, purple and black. The organic matter being dated was presumably due to the addition of binders or vehicles when the paints were made and applied. The NCPTT goal of training new generations of scientists was achieved as TAMU graduate students carried plasma sampling devices with them to their subsequent teaching positions. Professor Karen Steelman has continued this work in her laboratory at the University of Central Arkansas, as has Professor Ruth Ann Armitage at Eastern Michigan University.

Plasma oxidation has successfully dealt with many issues of rock art dating although some concerns still remain (Rowe 2007, 2009; Rowe and Steelman 2003b; Steelman and Rowe 2012). Since its inception, plasma-chemical carbon extraction has been used to date rock paintings from all around the world (Rowe 2001, 2004, 2005a). At least one pictograph has been dated by the plasma oxidation technique in Arizona (Armitage et al. 2000b; Steelman et al. 2004a), California (Armitage et al. 1997, 2005), Colorado (Rowe 2004), Idaho (Stelman

et al. 2002b), Missouri (Diaz-Granados et al. 2001, 2015; Duncan et al. 2015), Montana (Chaffee et al. 1994d; Scott et al. 2005), South Dakota (Armitage and Tratebas, unpublished date), Texas (Bates et al. 2015; Boyd et al. 2014; Brock et al. 2006; Chaffee et al. 1993a, b; Hyman and Rowe 1992, 1997 a, b; Hyman et al. 1999; Ilger et al. 1994b, 1996; Jensen et al. 2004; Pace et al. 2000; Rowe 2003, 2005b; Russ et al. 1990, 1993), Utah (Chaffee et al. 1993a, b, 1994a, b, c) and Wisconsin (Stelman et al. 2001). The following countries also have pictographs dated by the plasma oxidation technique: Angola (Ilger et al. 1995), Australia (Armitage et al. 1998, 2000a; David et al. 1994, 1997, 1998a, b, 1999a, b, 2000, 2001); Belize (Rowe et al. 2001), Brazil (Rowe and Steelman 2003a; Steelman and Rowe 2005; Steelman et al. 2000, 2002a), France (Ilger et al. 1994a), Guam (Hunter-Anderson et al. 2013), Guatemala (Armitage et al. 2001; Miller et al. 2002; Robinson et al. 2007; Rowe and Steelman 2004, 2007), Mexico (Ilger et al. 1995), Nicaragua (Baker and Armitage 2013), Russia (Stelman et al. 2002c), and Spain (Stelman et al. 2005a).

### Nondestructive Applications

Although Rowe and colleagues were aware of the potential of the plasma sampling technique for non-destructive dating of more than just rock art (Ilger et al. 1996; Hyman and Rowe 1997a, b, 1998), that aspect of the technique was not pursued in earnest until 2002 (Rowe 2005c; Steelman and Rowe 2002, 2004; Steelman et al. 2005b; Terry et al. 2006). They examined the effect of multiple oxygen plasmas on a shirt tag. The black ‘u’ shown in Figure 2 faded slightly, but only after many plasma runs (all at the relatively high plasma temperature of circa  $150^{\circ}\text{C}$ ). Additional work ensued on the radiocarbon standard TIRI wood sample of Belfast pine as shown in Figure 3. Minor changes can be seen on the thin, almost transparent, right hand side of the sample after the sample was subjected to enough plasma runs to collect more than 20 radiocarbon dates. Dating results for a series of plasma-derived TIRI Belfast pine samples analyzed by Steelman (2004) for her dissertation is presented in Figure 4. Steelman used the early and late results in this series as impetus to explore potential sources of younger and older carbon contamination that could have been introduced as part of the plasma extraction procedure, but the



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range of these results is perfectly consistent with the range of both TIRI and FIRI interlaboratory comparison results reported by Scott (2003).

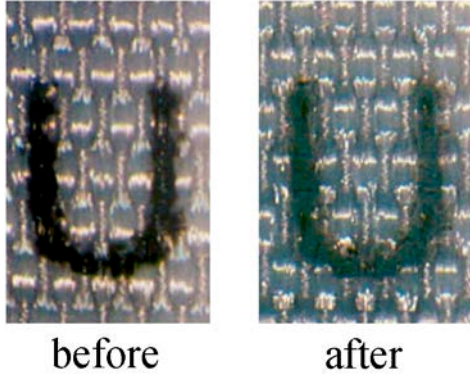


Figure 2. Images of a printed “shirt tag” before sampling and after exposure to many oxygen plasma sampling runs (adapted from Steelman and Rowe 2002).

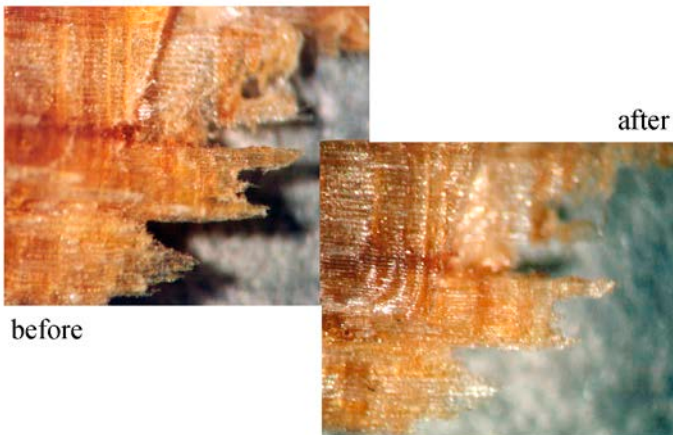


Figure 3. Images of a TIRI wood sample before and after the collection of sufficient carbon for 20 radiocarbon dates (adapted from Steelman and Rowe 2002).

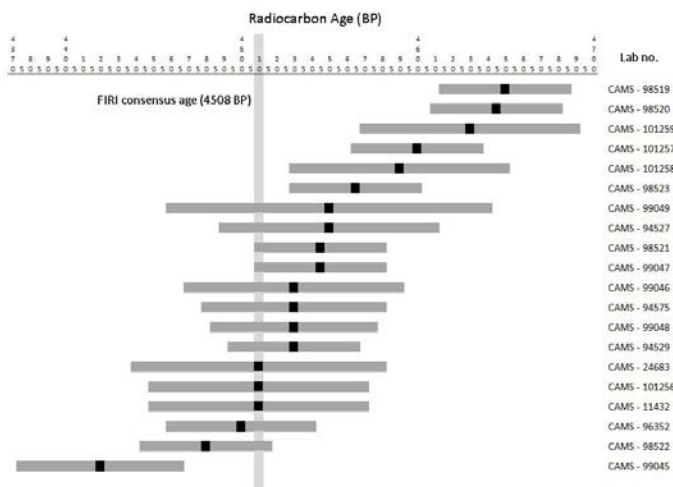


Figure 4. Oxygen plasma derived dates for the TIRI

**Belfast pine standard based on samples collected by Karen Steelman for her dissertation research (2004). Radiocarbon assays were by AMS measurement after graphite conversion, and the FIRI consensus age for Belfast pine (4,508 BP) is included for comparison.**

A dating project was undertaken on an infant burial recovered from the Lower Pecos River region of Texas (Stelman et al. 2004b). The partially mummified infant had been placed in a grass “nest,” with textile wrappings and associated wooden funerary objects. Samples were submitted for three conventional radiocarbon dates, including acid-base-acid pretreatments, and ten plasma CO<sub>2</sub> samples were collected. CO<sub>2</sub> samples were collected from a <1 inch square piece of bone and tissue, a blade of grass, a piece of a mat, a piece of twine, and a sotol stalk. An average of the plasma-derived CO<sub>2</sub> dates was as 2137±13 years BP compared with the average of 2128±20 years BP for the conventional dates. Agreement between dates based on the two sampling approaches is excellent, while visual comparison of the sotol stalk (as an example) before and after three plasma treatments showed no visible change (Figure 5).



Figure 5. Images of a sotol flower stalk before and after radiocarbon sampling (adapted from Steelman et al. 2004b).

### Plasma Extraction

Rowe and colleagues had focused their attention on developing reliable methods of oxygen plasma-extraction of carbon for dating until 2004. The low temperature of the plasma had effectively eliminated concerns about carbonate and oxalate contaminants, however, potential humic acid contaminants remained a concern. To achieve “nondestructive” radiocarbon dating, humic contaminants must be removed (or confirmed absent) without significantly altering the artifact. Conventional practice is to pretreat samples for carbonate and humic acid contamination simultaneously by washing in strong

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acid, then in strong base (alkali), and finally again in strong acid, often at elevated temperatures (usually 50°C). That approach to pretreatment works if the sample is intended for destruction, but the treatment is inconsistent with the potential non-destructive advantages of plasma extraction. Three approaches to humic acid removal have begun to be explored: applications of plasma oxidation itself, pH 8 solution washes, and super critical fluid (SCF) extraction.

Plasma oxidation has been explored successfully as a destructive pretreatment technique (Bird et al. 2010), but there is potential for investigation as a non-destructive pretreatment as well. Destructive pretreatment with oxygen plasmas selectively removed contaminants from crushed charcoal samples at RF power levels of 2 to 100 watts and temperatures up to 150°C. At these elevated temperatures, preliminary acid washes are necessary if carbonate contamination needs to be removed. Organic compounds within the samples are then oxidized differentially, and radiocarbon dating tests of standards revealed that significant proportions of contaminants could be successfully removed. Although low for plasmas in general, the energies and temperatures involved also reduce the total sample volume significantly, ashing up to half the initial sample weight in the process of lowering contaminant concentrations to an acceptable level. Since this oxygen plasma pretreatment is a surface active technique, crushing the sample to maximize particle surface area is necessary in advance of carbon isotope measurement.

The lower temperature plasma conditions used in non-destructive sample collection (described completely below) are much gentler but also appear to have the potential to selectively remove sample components. We have yet to determine how effectively different procedures can be manipulated to remove contaminants, but we have unintentionally been able to distill different fractions of TIRI Belfast pine wood samples at different power and pressure levels of plasma treatment. Experimentation in the potential for differential oxidation of contaminants at low plasma temperatures is an appropriate focus for future research.

Phosphate buffer solutions are another

approach to pretreatment for humic acids that work well with plasma radiocarbon sampling. Mary Ellen Ellis (2008), one of Armitage’s students at Eastern Michigan University, used a phosphate buffer solution as a solvent wash for humic acid contaminants. The pH 8.0 buffer solution can remove humic acid contaminants at temperatures of 50°C or less, using repeated ultrasonicated washes and rinses. This pretreatment does require that the object or material be robust enough to soak in the aqueous solution for hours or days at a time, but in combination with the low energy of oxygen plasma sampling, harsh acid-base-acid treatment is not necessary.

In 2004 Rowe received a second grant from NCPTT to support the investigation of SCF extraction as a pretreatment technique. With the NCPTT support, Rowe was able to take sabbatical leave to work with Dr. Jerry King at Los Alamos National Laboratory (LANL). LANL had an SCF group who was willing to collaborate in Rowe’s study and explore his idea for removing humic acids by means of SCF dissolution. Unfortunately, LANL access was abruptly closed due to unrelated security breaches, and the proposed study was delayed until the last two weeks of the sabbatical period. The early results were very promising, and King suggested following up this brief initial study. Many factors again delayed the collaboration, and King moved from LANL to become a Professor at the University of Arkansas-Fayetteville. Studies there were first published in 2012 (Lay et al. 2012) followed by a summary by Rowe et al. (2013). SCF has tremendous potential for non-destructive humic acid removal, especially under conditions where the target material could be damaged by exposure to in aqueous solutions.

### The New Mexico Plasma Laboratory

Rowe and the New Mexico coauthors of this paper are currently building a low energy oxygen plasma radiocarbon sampling laboratory at the Center for New Mexico Archaeology (CNMA) in Santa Fe, New Mexico. As a joint venture of the archaeology and conservation divisions of the New Mexico Department of Cultural Affairs, the goal is both to continue research on plasma extraction as a non-

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destructive radiocarbon sampling technique and to provide sampling services to the archaeological and museum communities.

The basic architecture of the apparatus is a high vacuum system that is capable of achieving and maintaining vacuums of  $10^{-6}$  torr (Figure 6). Glass sample chambers of various sizes are attached to the vacuum systems and to manifolds for the introduction of both high purity argon and oxygen gasses for cleaning and sampling steps. Low energy plasmas are generated at gas pressures of 1-3 torr using an RF generator that can maintain plasmas at power levels as low as 5 watts and chamber temperatures of  $35^{\circ}\text{C}$  or less. After gas samples are generated by plasma oxidation, water vapor is separated with a dry ice-acetone trap, and the  $\text{CO}_2$  for radiocarbon dating is condensed within a 4 mm outside diameter glass tube using a liquid nitrogen bath. The glass tube is flame-sealed, retaining the  $\text{CO}_2$ , and the ampoule is separated from the apparatus for shipment to the ETH Zurich AMS laboratory under the direction of Lukas Wacker. ETH Zurich is capable of the direct AMS dating of  $\text{CO}_2$  samples of 40-100 micrograms, bypassing the need for graphite conversion.



**Figure 6. Overview of the plasma sampling apparatus at the Center for New Mexico Archaeology.**

While the architecture and theory of sampling are relatively straight forward, the steps in the sampling process are complex and contingent on the characteristics of the materials being sampled. Pretreatment, if necessary, is carried out prior to

initiating plasma sampling. After evacuating the empty sampling chamber to a vacuum of at least  $10^{-6}$  torr, research purity oxygen is introduced at a low pressure (1-3 torr). An initial oxygen plasma cleansing of the chamber is then carried out to eliminate any extraneous contaminating carbon from the previous run or from handling of the chamber between runs. Oxygen plasmas are repeated until less than 0.5 micrograms of carbon as carbon dioxide is detected, and then the sample to be processed is placed into the chamber. After the introduction of the sample into the sampling chamber, contamination from modern atmospheric  $\text{CO}_2$  must be minimized, both as ambient gas in the system (removed with the high vacuum) and as  $\text{CO}_2$  that may be adhering to the surfaces of the sample or the chamber. After evacuating the sample chamber, research purity argon is introduced at low pressure (1-3 torr). The sample is bathed in a low-energy argon plasma (as low as 5 watts and  $30^{\circ}\text{C}$ ). Argon is close to  $\text{CO}_2$  in molecular size, and the plasma scours the sample and the surfaces of the apparatus, dislodging adhered  $\text{CO}_2$ . The sample can off-gas at this stage, releasing water vapor, absorbed  $\text{CO}_2$  and other gases, and compounds that become volatile under warm low vacuum conditions. Chamber pressure is monitored after each argon plasma run and after the application of liquid nitrogen and dry ice acetone traps, characterizing the amount of evolved or liberated gases. Gases are pumped out of the system, or if there is any reason to retain these gases, those with high enough boiling points can be captured from either of the traps. The volume of gas evolved during cleaning is monitored, and the argon plasma cleaning step is repeated as often as necessary to eliminate the possibility of any significant remaining contamination potential. When less than 0.5 micrograms of carbon as carbon dioxide is captured, the sample is now ready to be processed using the plasma oxidation technique. Since the argon cleaning stage is not chemically reactive, little if any carbon is being removed from the material other than as absorbed or adsorbed  $\text{CO}_2$ . The exception may be rare samples whose composition includes hydrocarbon compounds that can be volatilized at the low operating temperature and pressure of the plasma.

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Following the argon cleaning stage, low pressure (1-3 torr) research purity oxygen is introduced. A low energy oxygen plasma is initiated in the chamber (as low as 5 watts and 30°C), and the plasma is maintained for as long as is necessary to produce at least 40-100 micrograms of carbon in the form of CO<sub>2</sub>. The time necessary varies with the nature of the material being dated, both in composition and in surface area that is exposed to the plasma. Depending on the sample characteristics, as little as 10 or 15 minutes may be all that is needed to collect enough gas. Chamber size can also affect the amount of carbon that is oxidized irrespective of the amount of time that the plasma is running. Water vapor and traces of other gases are produced in addition to CO<sub>2</sub>. When sufficient CO<sub>2</sub> has been created, the plasma is turned off, and the accumulated gas is subjected first to a liquid nitrogen trap to capture whatever gases have been created in the chamber (primarily CO<sub>2</sub>). After 10-15 minutes the liquid nitrogen bath is removed and a dry ice acetone trap is initiated in order to separate water vapor and other temperature specific contaminants while releasing the accumulated CO<sub>2</sub> into the closed system. After determining that sufficient gas has been captured, the gas is subjected to another liquid nitrogen trap to concentrate the CO<sub>2</sub> within a 4 mm outside diameter glass tube. Pressures are monitored to ensure that adequate carbon has been produced, and then the tube is sealed into an ampoule and separated from the apparatus. Multiple samples can be collected as vouchers or for other analyses.

### Research Vignettes

Multiple materials and samples have been subjected to argon and oxygen plasmas during the design and refinement of the CNMA apparatus and sampling procedures. The overall goal has been to build a device and develop protocols that can generate reliable radiocarbon samples with little or no risk of damage to the artifact or material being sampled. This has meant considerable experimentation with electrode design, RF power levels, gas pressures, sampling temperatures, and plasma exposure times, all with a variety of different target materials. The vignettes below represent observations and potential

future research directions for the CNMA laboratory and for plasma radiocarbon sampling in general.

### Plasma Characteristics

Argon and oxygen plasmas have characteristic colors (Figure 7). Those colors change subtly during cleaning and sampling as the gas composition within the sample chamber changes. Color change may provide useful information on the types and compositions of gas mixtures that are created. This may be especially valuable in the oxidation stage of sampling, improving the ability to determine when oxidation has proceeded sufficiently to produce the target volume of CO<sub>2</sub> for dating. More precise determination of this threshold may lower the risk of destructive consequences of sampling on different types of artifacts or materials.



**Figure 7. Simultaneous running of two argon plasmas with ring RF electrodes (left) and bar electrodes (back right).**

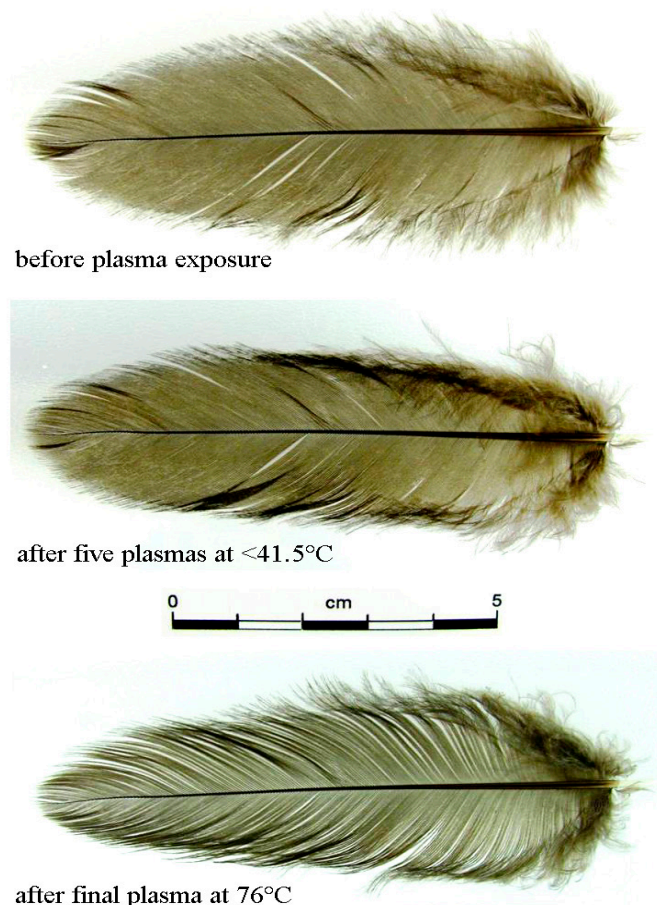
### Feather Extraction

A modern turkey feather was subjected to sample collection steps in an effort to explore the effects of the plasma sampling protocol on delicate organic materials. A ‘before-plasma’ picture was taken of the feather (Figure 8) so that we could assess the extent of physical change due to subsequent plasma exposures. The feather was then inserted into a plasma chamber and subjected to a 1 hour, 5 watt argon plasma. There was no significant release of carbon dioxide, and we began the first of a series of oxygen plasma sampling runs, evacuating the chamber and providing new oxygen at 3 torr for each run. Sampling runs were conducted at 8 watts of RF power for 1 hour; two runs at 10 watts for 1 hour; and a final run at 5 watts for 1 hour. In total, the feather was subjected to 5 plasmas at 5-10 watts of RF power for a total of 5 hours. The feather was removed from the system, examined under 20X for damage, and re-photographed (see Figure 8). No damage was apparent, even to the fine downy

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feather components. During the plasma runs, the temperature ranged from 33°C to a maximum of 41.5°C (91.4°F - 107°F).

The feather was again placed into the plasma chamber. It was then subjected to a stronger oxygen plasma of 20 watts RF power for an hour. The maximum temperature on that run was 76°C (169°F). Once again the feather was removed from the system and re-photographed (see Figure 8). At the higher RF power and higher temperature, the feather exhibited visible structural change. The damage was not in the form of erosion of downy components but in an increasing fragility of the structure of the feather and susceptibility to mechanical damage during insertion and withdrawal from the chamber. Even though we did not retain the carbon dioxide from the plasma oxidations, four of the five runs produced more than the 50 to 100 micrograms of carbon (even at 5 watts) required for an accelerator mass spectrometer (AMS) radiocarbon date.



**Figure 8. Effects of repeated plasma oxidation sampling on a modern turkey feather. No apparent effect was noted after five sampling runs at RF power of from 5-10 watts and oxygen plasma temperatures of between 33 and 41.5°C (91.4°F - 107°F). A marked fragility is apparent after a single sampling run at 20 watts of RF power and a temperature 76°C (169°F), although fine downy elements appear unaffected.**

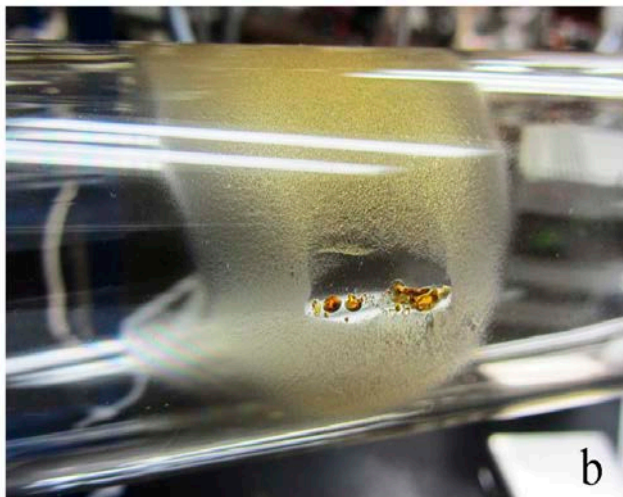
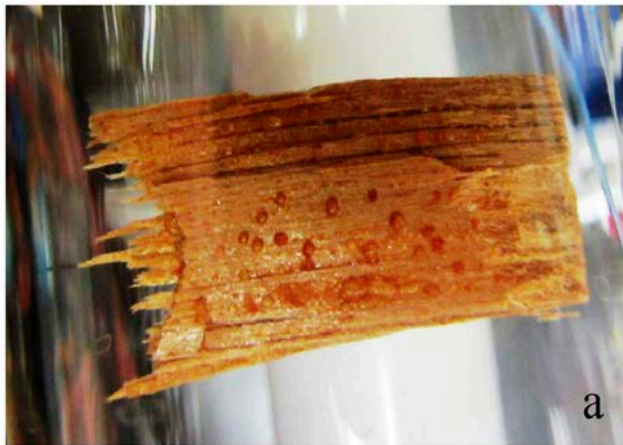
Our conclusion is that at the lower RF powers, and particularly at 5 watts, with an associated temperature of only about 29°C (84°F) for the argon plasma and 34°C (93°F) for the oxygen plasma, the feather was virtually unchanged visually.

### Distillation

A variety of RF power settings were used during the collection of calibration radiocarbon samples from a piece of TIRI wood (Belfast pine). Rowe's previous experiences in plasma extraction of TIRI wood radiocarbon samples, extracted at much higher power levels than we currently use, had been unremarkable. However, one combination of settings in the CNMA series resulted in the volatilization

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of a resinous component of the TIRI wood sample (Figure 9). Resin bubbled to the surface of the wood, and a volatile component evolved from the wood and condensed on the interior surface of the sample chamber. The date derived from  $\text{CO}_2$  from this run (ETH61251.1) was perfectly consistent with dates on  $\text{CO}_2$  developed from runs that did not fractionate the sample. However, the phenomenon suggests that manipulation of plasma energy and sampling temperature may be used to collect  $\text{CO}_2$  from discrete compositional components of some sample types. This is similar, in a low power sense, to the use of oxygen plasmas as a pretreatment ashing protocol to remove more easily oxidized contaminants prior to collecting  $\text{CO}_2$  from more stable components of a sample (Bird et al. 2010).



**Figure 9.** TIRI Belfast pine wood with resinous exudate on the wood surface and condensate on the interior of the sampling chamber tube.

This aspect of the plasma extraction process may also have potential for isolating samples of what actually is contributing to the  $\text{CO}_2$  being dated. Gases produced from the material during the initial argon cleaning step can be captured and analyzed by other techniques to provide information on volatile components that might be contributing to the later oxidation step, such as binders in rock art pigments.

### Masking

Composite materials pose challenges to radiocarbon dating, both within and outside the context of non-destructive approaches to sampling. A feature of plasma oxidation is the expectation that only carbon-containing compounds that are directly exposed to energized oxygen species of the plasma will be released from the object being sampled. Exposure to non-energized oxygen molecules should not result in oxidation, and those carbon components should not be included in the radiocarbon sample. This expectation raises the possibility of masking objects to be sampled, allowing only a pre-selected portion to be dated.

A potential application in the Southwestern United States is the radiocarbon dating of the organic paint constituents of potsherds. The organic (carbon) paint is created by preparing and then applying a plant extract to the surface of the unfired vessel, either as a binder for a mineral pigment or by itself within pottery traditions that create designs solely in carbon. During firing, molecules of the plant extract are carbonized within the surface of the vessel, and they should be accessible for plasma oxidation. However, some of the pottery traditions use Cretaceous era carbonaceous clays for vessel construction, and plasma exposure needs to eliminate or minimize contributions of carbon from this source. Two masking approaches will be tried initially, one simply using aluminum foil and the other using a painted-on suspension of inert material (such as aluminum oxide powder). Oxygen species penetrating beneath the foil should lose energy and become non-reactive with carbon compounds that have been protected by the mask. Similarly, oxygen species that diffuse through the porous powder coating should be non-reactive by the time they reach the underlying surface. Both masks

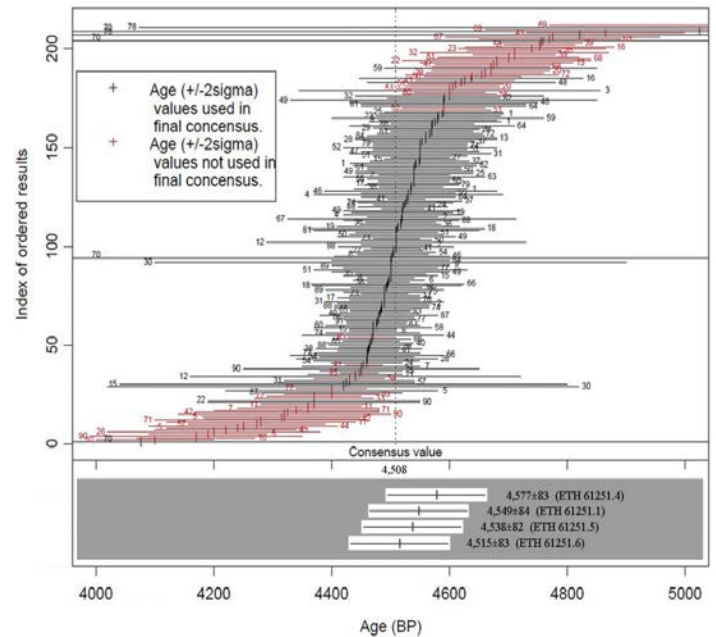
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can be removed or reset to allow the collection of radiocarbon samples from different areas of complex artifacts.

If either of these masking approaches is effective, masking will increase the potential applications of non-destructive sampling. For composite artifacts, such as darts, it is conceivable that complementary radiocarbon samples could be submitted from the foreshaft, from sinew attaching the point to the foreshaft, and from any residue adhering to the point.

### Calibration of the New Mexico Apparatus

In May of 2015, a series of calibration samples was submitted to the AMS laboratory at ETH Zurich for radiocarbon dating using a gas ion source for direct insertion of carbon dioxide (Fahrni et al 2013; Ruff et al 2007; Wacker et al 2013). These included CO<sub>2</sub> from the TIRI/FIRI Belfast wood standard (including a sample collected as part of the plasma run with the distillation effect described above, ETH61251.1). Results are presented in figure 10. The FIRI consensus date is 4,508 BP for all measurement methods, while the consensus date for ages estimated by AMS measurement is 4,519 BP (Scott 2003:Table 7.1). The mean of the four New Mexico dates is 4,545 BP, in agreement with the FIRI interlaboratory comparison results. The New Mexico results are also consistent with dates produced from other plasma-collected samples from other laboratories (see Figure 4), including the tendency for mean dates to be slightly older than dates for samples collected and processed by other techniques.



**Figure 10.** AMS dating results for four New Mexico plasma collections from the TIRI Belfast pine standard (lower panel). The results are compared with the interlab comparison results for the same standard from the FIRI study (adapted from Scott 2003, Figure 7.3).

Risks of contamination in plasma oxidation sampling are ever present due to potential failures of vacuum seals and of argon or oxygen gas contamination. Routine re-sampling of standards, including yet-to-be-sampled dead carbon sources, will be necessary to confirm the reliability of the CNMA sampling technique and the accuracy of the associated radiocarbon dates.

### Conclusions

Plasma oxidation as a radiocarbon sampling technique began as a novel but relatively narrowly focused idea to deal with the challenges of dating rock art. It has evolved into a technique that has the potential to solve not just one but a series of problems in archaeology, museum studies, and conservation. Support from NCPTT was essential in the nurturing and development of the original technique, initiating a research trajectory that continues to expand.

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