19. Antisera were raised against the high molecular weight toxin fraction before further separation by HPLC. A rabbit polyclonal antiserum was raised against the native toxin, and a monoclonal antibody (C5F2) was derived from mice immunized with heat-denatured toxin.
20. A plasmid DNA library was constructed in Bluescript KS$^+$ (Stratagene) from size-fractionated DNA partially digested with Sau 3a and transformed into competent library efficiency XL2-Blue MRF' *E. coli* (Stratagene). The library was screened with both the monoclonal and the polyclonal antitoxin antibodies. Immunoreactive clones were restriction mapped and sequenced with Sequenase 2.0 (United States Biochemical).
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32. For examination of gut pathology, we fed Tca toxin to neonate *M. sexta* for 3 hours and to first-instar larvae for 48 hours. Larvae were fixed in Bouin's fluid overnight at 4°C, dehydrated, and embedded in paraffin wax; 6-µm paraffin sections were stained with Weigert's hematoxylin followed by Cason's trichome stain; then they were examined by light microscopy.
33. Cells were spun down at 10,000*g* and then supernatants were concentrated in Millipore Centrifugal filtration units (Ultrafree, Biomax-100K) or diluted with growth medium.
34. Abbreviations for the amino acid residues are as follows: A, Ala; C, Cys; D, Asp; E, Glu; F, Phe; G, Gly; H, His; I, Ile; K, Lys; L, Leu; M, Met; N, Asn; P, Pro; Q, Gln; R, Arg; S, Ser; T, Thr; V, Val; W, Trp; and Y, Tyr.
35. We thank all at Dow AgroSciences Biotechnology for their encouragement and support of this project. Supported by Hatch funds, The Applied Research and Technology Fund, and The Industrial and Economic Development Fund, all administered by the University of Wisconsin-Madison and by DowAgrosciences.

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TECHNICAL COMMENTS

Ambiguities in Direct Dating of Rock Surfaces Using Radiocarbon Measurements

An attempt was made to date rock surfaces with accelerator mass spectrometry (AMS) radiocarbon measurements of rock varnishes or rock weathering rinds. In two case studies, samples pretreated in the laboratory of Dr. Ronald Dorn prior to AMS analysis have been found to contain significant quantities of carbon-rich materials of two distinct classes. Type I material resembles coal, whereas type II material resembles pyrolized wood charcoal fragments. In samples where these type I and type II materials were separated and AMS-radiocarbon dated, they were found to have widely differing radiocarbon ages. In these cases, the measurement of the radiocarbon age of the entire sample would yield results that are, at best, ambiguous. Neither type I nor type II materials were found in comparable samples that were independently prepared.

Since it was first developed in the early 1980s, direct dating of rock surfaces by accelerator mass spectrometry (AMS) radiocarbon analysis has become an integral tool in the fields of geomorphology and archaeology. This technique was pioneered principally by Dr. Ronald Dorn, now at Arizona State University. Results from several studies by Dorn and co-workers (*1*–*13*) have implied that organic material can generally be harvested from within or beneath the rock varnish layer that commonly encrusts rock surfaces in desert regions. This varnish is composed mainly of iron and manganese oxides but may also contain small amounts of organic material, thought to be composed of bacterial remains, plant detritus, or remains of lichen or algae. These studies indicated that AMS radiocarbon dates of this organic carbon could in many cases be used to provide minimum ages of the rock surface (*1*–*13*).

Recently, the AMS laboratory at the University of Arizona in Tucson became involved in a research project initiated by E. Malotki of Northern Arizona University aimed at trying to obtain radiocarbon dates of petroglyphs. Petroglyphs are pictures or images that have been carved, pecked, or scratched into a rock surface. These particular petroglyphs were probably created by archaic hunter-gatherer people who populated northeastern Arizona, possibly for several thousand years before about A.D. 1 (*14*). Malotki enlisted Dorn to help collect small samples of the rock and encrusting varnish from several petroglyphs located in a canyon in northeast Arizona. Dorn then took these samples to his laboratory, where the samples were chemically pretreated before they were sent to the AMS laboratory at the University of Arizona for radiocarbon analysis. This pretreatment (*8*) included treating the samples in concentrated hydrochloric acid and concentrated hydrofluoric acid. The five samples that Dorn subsequently submitted to the Arizona AMS facility were reportedly of subvarnish rock-matrix material from the weathering rind of these rocks and did not contain samples of the varnish itself. Four of these had been taken from petroglyphs and the fifth from a control rock surface that did not have a petroglyph carved into it.

When these five samples arrived at the University of Arizona AMS laboratory, visual examination of these samples revealed that two were greenish in color, whereas the other three samples had a whitish cast. This seemed unusual because all of these samples were from the Coconino Sandstone, which is a very homogeneous rock strata. Under a binocular microscope, all five of the samples were observed to contain large quantities of black particles, as much as $\sim$15% by volume (Fig. 1). There were two types of these particles. Type I particles are blocky, sub-angular particles with conchoidal fracture. They are jet-black in color and have glossy surfaces. These type I particles have a specific gravity greater than unity, and resemble fragments of either anthracite coal or vitrinite component of bituminous coal (Fig. 2). Many of these particles are large, 200 to 600 µm across. Analysis of this type I material showed that it contains approximately 50% carbon by mass. A specimen of this material, separated from a sample pretreated by Dorn, was forwarded to an expert on identification of coal, who identified the specimen as subbituminous coal from a vitrian layer (*15*) (Table 1).

The second type of black particles (type II) generally have a specific gravity of less than unity. They exhibit one or two pronounced lineations that resemble in size, structure, and arrangement, longitudinal tracheid cells and ray parenchyma or ray tracheid cells found in wood (Figs. 1 and 3). Many of these particles also are 200 to 600 µm in length, and a few are larger than 1000 µm in length. For comparison, a photomicrograph of bristlecone pine charcoal that we pyrolized is shown (Fig. 4). Thus, type II particles appear to be charred wood.

We separated some fragments of type I and type II carbon materials from one of these petroglyph samples in order to date each type using AMS radiocarbon measurements. These results were sufficient to show that type I grains are about 28,000 years old [conventional radiocarbon age in years before present (B.P.)], whereas type II grains are about 4000 years old (Table 2). Our failure to obtain an infinite (limiting) radiocarbon age on the coal-like material was

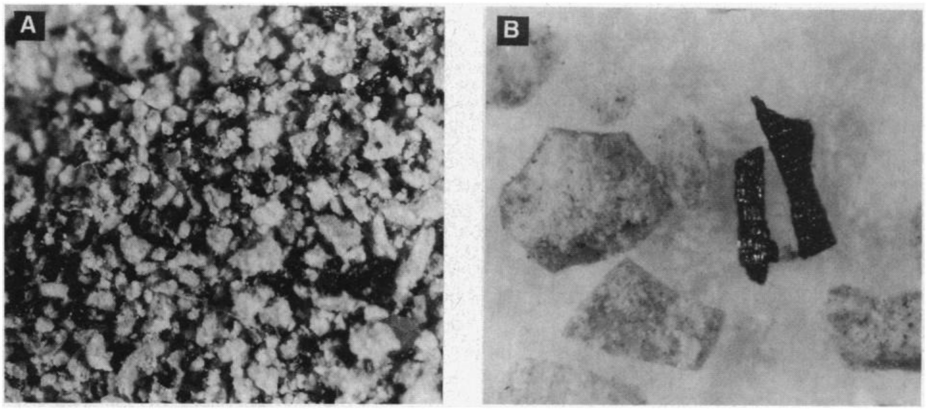

Fig. 1. (**A**) Photomicrograph of petroglyph sample EK95-8 submitted to the Arizona AMS laboratory by Dorn. This sample is composed of approximately 5 to 10% type I and type II material by volume. The field of view is approximately 7 by 6 mm. (**B**) Higher magnification view of petroglyph sample EK95-8. The field of view is approximately 1.5 by 1.3 mm. The large type II grains shown are approximately 0.5 mm in length. The rock mineral material consists mostly of loosely bound clasts of very fine grained (<0.01 mm) silt to clay-sized particles. Only a few mineral grains larger than this size are found in these samples.

Table 1. Results of a point-counting analysis by optical microscopy of specimen of type I material isolated from sample SL-4, which was prepared for radiocarbon analysis by Dorn. Specimen was mounted in epoxy, hand polished, and examined under plane polarized light at 400X magnification. 400 points were counted. Analysis was performed by MaryAnn Love Malinconico, Department of Earth and Environmental Sciences, Columbia University, Palisades, New York.

Material	Counts	Percent
Vitrinite	332	83.0
Pseudovitrinite	16	4.00
Sporinite	9	2.25
Cutinite	9	2.25
Resinite	10	2.50
Liptodetrinite	19	4.75
Fusinite	0	0.00
Semi-fusinite	2	0.50
Macrinite	1	0.25
Mineral matter	2	0.50
Total	400	100.00

presumably a result of incomplete separation of type I from type II materials. In any case, there are two distinct populations of grains with radically different appearances in these five samples submitted for radiocarbon analysis by Dorn. In the sample in which these two types were separately dated, these type I and type II grains have radically different radiocarbon ages as well.

It is unclear how these type I and type II materials could have been incorporated into these samples because they were supposedly derived from the rock weathering rind and not from the varnish layer encrusting the rock. The Coconino sandstone, which is the rock formation into which the petroglyphs were carved, is a very pure quartz sandstone, and to our knowledge, does not contain type I and type II organic carbon materials. Neither type I or II materials bear any resemblance to endolithic algal remains that one might expect to find in the weathering rind of the rock (*7*, *16*). The possibility that the ancient Anasazi artists who drafted these petroglyphs might have rubbed the surfaces of these glyphs with both coal and burnt wood seems small because the control sample (not a petroglyph surface) submitted by Dorn also contained these materials.

Nevertheless, because this possibility remained, Malotki and Beck revisited the same sites from which Malotki and Dorn had collected the earlier petroglyph samples. They resampled the same petroglyphs, in some cases to a proximity of 1 mm to the sample scars made earlier by Dorn. They also sampled seven other petroglyphs not sampled by Dorn, and also collected one large bulk sample of desert varnish and rock weathering rind approximately 10 cm^2 in size from an area near a petroglyph. The objective was to see if any of these surfaces might contain the type I or type II carbonaceous particles found in every one of

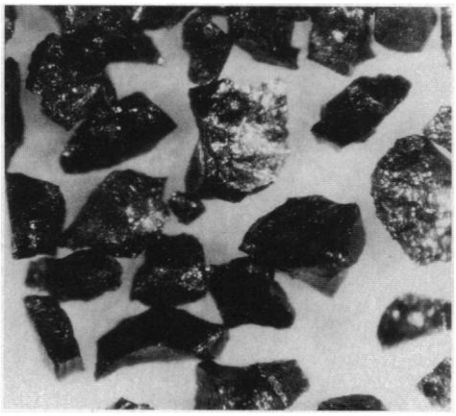

Fig. 2. Photomicrograph of typical type I particles separated from samples submitted to the Arizona AMS laboratory by Dorn. These particles were found in sample AA 2321. Note blocky, angular, surfaces with well-developed conchoidal (glassy) fracture. The typical size of grains shown in these photos is 0.2 to 0.6 mm.

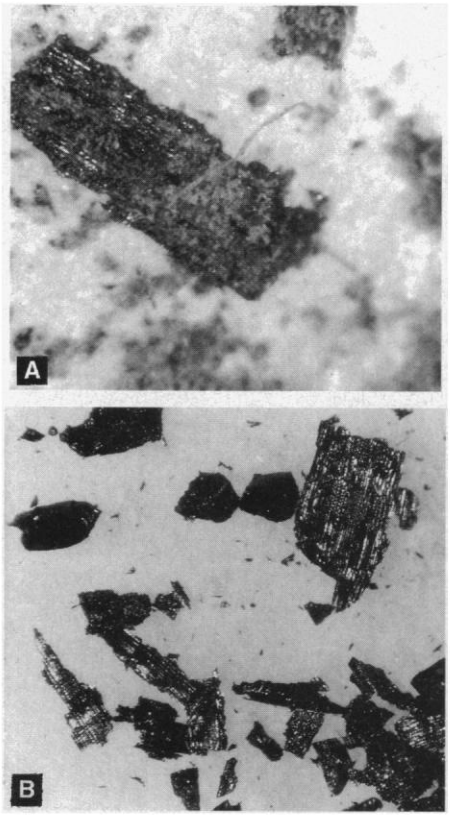

Fig. 3. Photomicrographs of particles found in samples submitted to the Arizona AMS laboratory by Dorn. (**A**) Type II grain found in a petroglyph sample (EK95-8). This grain is about 1 mm long. (**B**) Both type I and type II grains found in Dorn sample AA 2321. The typical size of type II grains shown is approximately 0.2 to 1.0 mm.

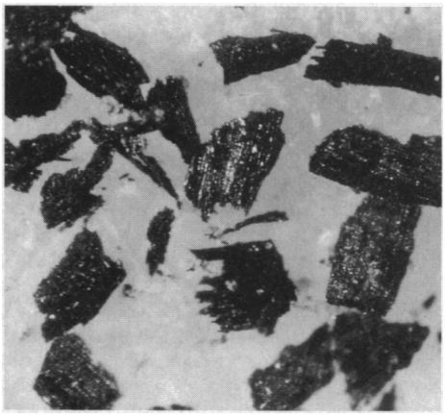

Fig. 4. Photomicrograph of pyrolized Bristlecone Pine wood. The material is shown for example and was not found in samples submitted by Dorn. This wood was pyrolized in the Arizona AMS laboratory for the sole purpose of comparison with type II particles, which are found in Dr. Dorn's samples (see Fig. 3). Typical grain size is 0.1 to 1.0 mm.

Dorn's five samples. None of the surfaces that we sampled revealed any trace of either type I or type II carbon-rich materials, even though our samples comprised both the weathering rind of the rock and the varnish layer (Fig. 5).

Similar findings have been made in a second study conducted by Broecker and Hajdas. In this study, it was found that samples pretreated by Dorn and submitted to the Zurich ETH-AMS facility for radiocarbon dating also contain what appears to be these same type I and II carbonaceous materials. These samples were from various geomorphic surfaces from the western United States. Of the 30 remainders still retained in Zurich of samples pretreated by Dorn, all but one contain type I or type II carbonaceous materials, or both, as described above. The Zurich Laboratory also has related samples that were pretreated by T. Liu of Columbia University (*17*), but none of these has been found to contain either type I or type II materials. Type I and type II fractions from two of the samples submitted to the Zurich laboratory that had been pretreated by Dorn were separated for analysis at Zurich (samples ETH 12816 and ETH 12815). As with the petroglyph sample, the coal-like particles yielded old radiocarbon ages, the wood-like component of the samples yielded young radiocarbon ages, and the bulk samples (containing a mixture of both of these materials) yielded intermediate ages (Table 2).

In August 1996, Liu and Broecker conducted an experiment to verify that their sample pretreatment methods were consistent with those of Dorn. In this experiment, Liu traveled to Arizona State University with four rock fragments he had collected from four different boulders from the Great Basin, in the western United States (*17*). Under Dorn's supervision, Liu extracted samples from these four rock fragments and then witnessed Dorn chemically pretreat these samples with HCl and HF. The four samples were left soaking in HF overnight in Dorn's laboratory. The following day Liu and Dorn returned to the laboratory to collect the acid-treated samples. Liu transported these to Columbia University, where he and Broecker observed that all four of the samples processed in Dorn's laboratory contained type I and type II materials. When Liu subsequently extracted and pretreated at Columbia University, using the same technique, additional samples from the same four rock fragments processed with Dorn, neither type I nor type II carbonaceous materials were found in any of the samples. Only the samples jointly processed by Dorn and Liu at Dorn's laboratory were found to contain type I and II materials.

Because of the unusual nature of these findings, we elected to examine the sample remainders available that Dorn had earlier submitted to the University of Arizona AMS facility. Ninety-nine such samples were in our possession in 1996. We recorded results of microscopic observations on 58 of these. Of those 58, five were too small to be evaluated with a binocular microscope. All the remaining 53 contained type I or type II materials, or both (Fig. 6). We isolated type I and/or type II fractions from several of these remainders and radiocarbon-dated the fractions (Table 2). As before, the coal-like particles yielded old radiocarbon ages, the wood-like component of the samples yielded young radiocarbon ages, and the bulk samples (containing a mixture of both of these materials) yielded intermediate ages.

Examples of these remainders of rock varnish or rock weathering rind material

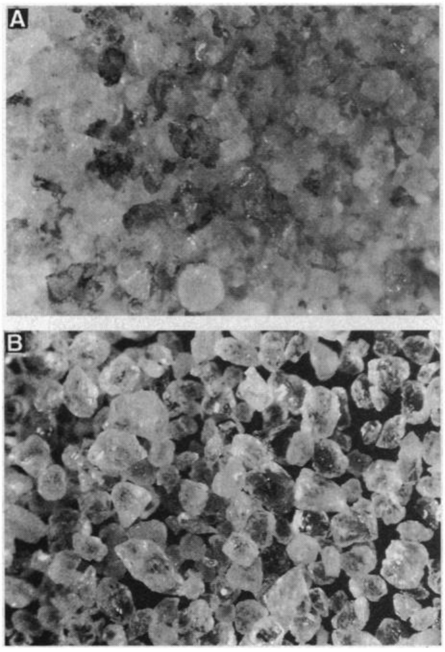

Fig. 5. (**A**) Photomicrograph of untreated sample of EM-96-1. This sample consists of both rock varnish and the underlying rock matrix material (weathering rind) collected from a petroglyph carved in the Coconino Sandstone. It was collected by Beck and Malotki from one of the same petroglyphs sampled by Dorn. Some grains are coated by desert varnish. Typical grain size is approximately 0.5 mm and is fairly restricted around this value. No obvious type I or type II particles can be seen. (**B**) Photomicrograph (on a black background) of EM-96-1 after soaking in sodium dithionate overnight. Sodium dithionate is a reducing agent that removes metal oxides from the surface of the mineral grains but leaves organic material unharmed. Dorn has stated that he frequently used sodium dithionate on varnish samples to remove these metal oxide materials (*3, 5, 6*). After this treatment, one filamentous structure (not shown), which may have been endolithic algae, was found in the sample. No particles of either type I or type II materials were found. The sample consists almost exclusively of angular quartz grains. According to Dorn (*6*), one of the steps in the pre-treatment of rock varnish or weathering rind material is to soak the sample in concentrated hydrofluoric acid (HF). This treatment will not dissolve organic carbon material. After soaking sample EM-96-1 overnight in concentrated HF at room temperature, nothing remained of this sample except the single filamentous structure mentioned above. No mineral grains survived this HF treatment. Compare this result to Fig. 1.

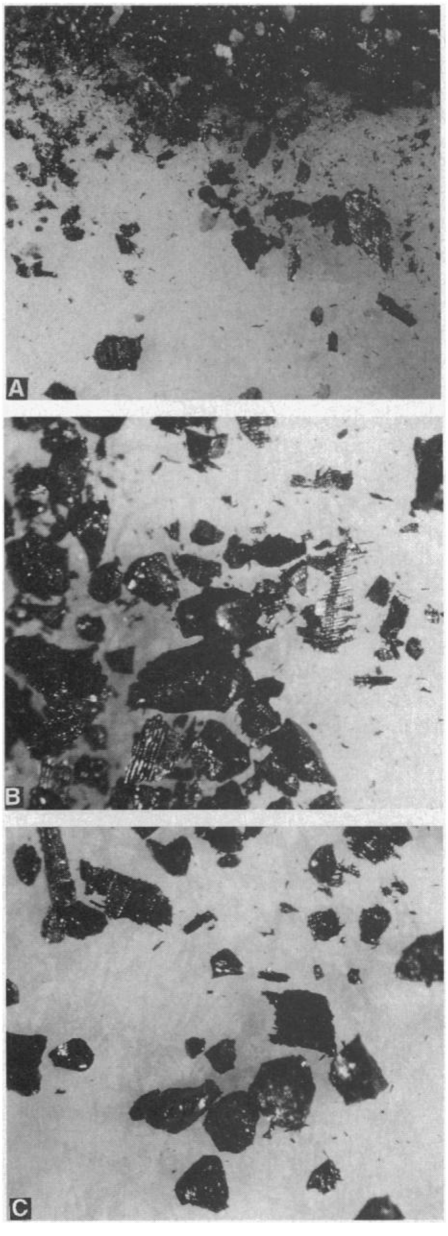

Fig. 6. (**A** to **C**) Photomicrographs of several samples previously submitted to the Arizona AMS laboratory by Dr. Dorn circa 1987. Both type I and type II particles can be seen in these photos. Typical grain size is approximately 0.1 to 0.6 mm in length in all photos.

submitted by Dorn to our laboratories include: (i) samples used in conjunction with calibration of the cation-ratio dating technique (*1*, *4*, *9*); (ii) samples from anthropogenic surfaces (*1*, *5*, *10–12*), some of which have been used to support the contention that there were pre-Clovis settlements in the Americas (*1*, *11*, *12*); (iii) samples used to date a variety of geomorphic surfaces found in the western United States (*1– 3*, *9*, *13*); (iv) samples submitted in connection with dating of ancient petroglyphs in Australia (*5*, *12*); and (v) rock varnish samples used for comparison of rock varnish AMS dating from Hawaiian lava flows (*2*, *4*) with conventional ^{14}C dating of plant charcoals collected from under the flows (*18*). This particular study (*4*) indicated that AMS ^{14}C dating of rock varnish material yielded ^{14}C dates nearly equivalent to those of plant charcoals collected from underneath the same lava flows.

Since we began work on this issue, Dorn has acknowledged that some of his samples contain "fibrous materials" and "dense shiny particles with a vitrinite-like" or "charcoal-like" appearance and that, in one instance, these two types of materials yielded substantially different radiocarbon ages (*19–21*). He has suggested that these materials may have been "inserted into rock material by older episodes of organic weathering" (*20*); or that they may have resulted from "ancient roots and microbial remains" that could "undergo diagenesis and can evolve into vitrinite" (*19*). Consequently, he warned recently, those interested in these data should be very cautious (*20*). It is unclear from these publications (*19–21*) in what percentage of his samples Dorn may have observed these particles.

In summary, of the remaining samples submitted to our facilities by Dorn that were large enough to inspect and that we have microscopically examined, all except one contain type I or type II carbonaceous materials, or both. We were unable to find either type I or type II materials in comparable samples that we independently prepared. Type I material resembles coal, whereas type II material resembles pyrolized wood charcoal. For several of the samples submitted by Dorn, we were able to separate type I or type II materials, or both, from the bulk sample, and radiocarbon-date the separated fractions (Table 2). In each case we have found large differences between the ages of type I, type II, and bulk sample material. Type I material is always older than the bulk age, and type II material is always younger than the bulk age of the sample. If a sample submitted for radiocarbon dating is found to contain two types of carbonaceous materials, each with a different radiocarbon age, then an analysis of the bulk mixture will not yield a reliable radiocarbon age. An apparent age can be determined, but this apparent age has no true age significance. Clearly, in these cases the bulk radiocarbon ages are ambiguous, and do not represent the true ages of the samples.

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Table 2. Results of AMS ^{14}C analyses of separated type I, type II, and Bulk material of several samples submitted for radiocarbon analysis by Dorn. This table includes measurements of five samples submitted to the Arizona AMS facility and two samples submitted to the Zurich AMS facility. In all cases, type I materials yielded ages significantly older than the bulk sample, whereas type II materials yielded ages significantly younger than the radiocarbon age of the bulk sample. Original results from CO_2 gas generated from two type II separates (AA 2320 and AA 2321) are not reported because they were inadvertently contaminated during processing at the Arizona AMS laboratory by another sample highly enriched in ^{14}C. No analyses presented in Table 2 were affected by this contamination. An additional remainder of one of these samples (AA 2321) was subsequently separated into type I and II materials and analyzed. The results of this repeated analyses are indicated with an (*). Ages for bulk analyses are from the indicated reference or are unpublished.

Lab number	Sample name	Type	Fraction modern	Radiocarbon age (years B.P.)
AA 21262	EK-95-8	I	0.0325 ± 0.0061	27,520 ± 1500
AA 21263	EK-95-8	II	0.6091 ± 0.0164	3,982 ± 216
AA 21724	ARV-52	I	0.0155 ± 0.0011	33,450 ± 560
AA 2321/T6541-I*	ARV-52	I	<0.0068	>40,030
AA 2321/T6541-II*	ARV-52	II	0.5943 ± 0.0658	4181 ± 890
AA 2321 (*22*)	ARV-52	Bulk	0.0435 ± 0.0022	25,190 ± 410
AA 21735	ARV-51	I	0.0306 ± 0.0054	28,010 ± 1305
AA 2320 (*2*)	ARV-51	Bulk	0.1209 ± 0.0023	16,970 ± 155
AA 21737	ARV-53	I	<0.0101	>36,900
AA 2322 (*2*)	ARV-53	Bulk	0.0800 ± 0.0022	20,290 ± 220
AA 6547/T6554	ARV-79	II	0.9052 ± 0.0094	800 ± 83
AA 6547 (*12*)	ARV-79	Bulk	0.1827 ± 0.0024	13,655 ± 105
ETH 12816	AC-8	I	0.01319 ± 0.0006	34,770 ± 370
ETH 12816	AC-8	II	0.7884 ± 0.0308	1910 ± 320
ETH 12816	AC-8	Bulk	0.1020 ± 0.0023	18,337 ± 183
ETH 12815	SL-1A	I	0.0104 ± 0.0005	36,660 ± 400
ETH 12815	SL-1A	Bulk	0.1450 ± 0.0023	15,510 ± 130

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22 January 1998; accepted 2 March 1998

Response: The association of rock varnish with heterogeneous organics, including carbonized woody tissues and vitrinite, has been documented in the literature for years, with inheritance and in-situ diagenesis suggested as hypotheses to explain this heterogeneity. When combined with independent replications of my results—a 1995 blind test in which I voluntarily participated—data they present on my samples, and their failure to duplicate my techniques, Beck *et al.*'s implicit claims are unequivocally disproven. Beck *et al.* (*1*) make three claims, one stated and two seemingly implied by earlier statements. The stated claim is that varnish radiocarbon dating yields ambiguous results. The implicit claims are that my results cannot be replicated and that my samples were manipulated to derive desired radiocarbon ages. This last assertion was made by N. A. Goodman (*2*), attorney for the University of Arizona (UA) scientists, and by E. Malotki (*3*). Drs. Beck and Jull (*4*) are reported as stating that my samples "could have been adulterated with finely ground bituminous coal and pulverized wood charcoal." These charges (*2–5*) are utterly false.

Here is the gist of their argument: they observed vitrinite and carbonized woody tissue (CWT) of different ages in my rock varnish samples. Beck *et al.* seem to imply that such combinations cannot occur naturally in the same sample, in part because of their failure to find them. In commenting on Beck *et al.*'s presentation at an Australian conference (*6*), A. Watchman is reported as stating "coal and charcoal do not occur together" and that "it took 'deliberate human action' to bring them together" (*4*). Thus, the critical issue in this controversy is whether vitrinite and CWT naturally co-occur with rock varnish.

In response, I discuss seven issues.

1) *My results have been fully replicated by others.* My findings were replicated in an independent study (*7*), where "Arrowsmith and Rice were trained by R. Dorn in sample collection and preparation procedures." Arrowsmith *et al.* write that "both the electron microprobe and the coal petrologic analyses indicate that carbon-rich granules are present in fractures associated with desert varnish in rocks from the White Tank Mountains western Piedmont (Arizona)." In particular "vitrinite and fusinite (an inertinite with well-developed cellular texture) macerals were dominant," and these particles occurred "in five of the 28 samples from 23 different cobbles." Their microphotographs show several vitrinite and CWT particles that exceed 0.27 mm in length. They stress that "there is no reasonable physical process by which the samples could become contaminated because the loci of observation were thoroughly fixed in rock." Furthermore, almost a decade ago my results were replicated by D. Tanner, who independently processed "split samples." Her samples yielded results equivalent to mine [(*8*), p. 1367].

This research confirms that vitrinite and CWT naturally co-occur under varnish in places where no natural deposits of coal are present in a region. These independent replications prove that the co-occurrence of these particles is not the result of "adulterated" (*4*) samples.

2) *The co-occurrence of different types of organics is well established.* More than a decade ago, using light microscopes, co-authors in Beck *et al.* and I looked at many of same samples discussed by Beck *et al.* We did not recognize vitrinite or CWT at that time because we assumed that the organic matter was "reworked by varnish-forming microorganisms" [(*8*), p. 1363]. We accordingly interpreted the resulting ages as "bulk" samples.

Before Beck *et al.* (*6*) "began work on this issue," I realized that there are heterogeneities in varnish organics and had submitted for publication the conclusion that heterogeneous ages for these materials mean that prior results are ambiguous (*9–17*). And prior to my publications, coal-like particles were identified under varnish by Karlov more than three decades ago (*18*). In 1986, both vitrinite and CWT were identified and concluded to be "abundant" in laminar calcrete [(*19*), p. 77)], a material found in desert soils and in fractures in weathering rinds (*20*). Charcoal encapsulation by rock coatings was recognized by Watchman in 1992 [(*21*), p. 64]. In 1993, I also published images of CWT in situ under varnish [(*12*), p. 28; (*22*), p. 722; (*10*), p. 35].

Beck *et al.* thus fail to cite other pertinent literature; this might seem to imply that the only possible source for the organics is intentional tampering (*2–5*). The research (*9–22*) shows that organic heterogeneity is the norm, rather than a cause for suspicion.

3) *Beck* et al. *failed to replicate my results because they apparently did not duplicate my techniques.* Replication is a cornerstone of science, but replication implies two related issues: duplication of methodological procedures and techniques, and replication of results. For obvious reasons, if an experiment or sampling procedure is not accurately duplicated, the failure of a replication is meaningless.

Beck *et al.*'s inability to replicate my results can be attributed to a series of significant oversights. First and foremost, Beck *et al.* refused my offers to train them at my expense in sample extraction and preparation. Woodward and Goodstein [(*23*), p. B11] explain that a key element of replication is to "work with someone from the original laboratory." I would not presume to walk into an accelerator laboratory and run a facility, even though I have read extensively on the topic. I have always maintained an open-door policy of sharing my techniques. I made this same offer to Arizona State, and the successful replication by Arrowsmith *et al.* is the result (*7*).

Second, Beck *et al.* state that their northern Arizona samples were taken next to mine. Beck *et al.* sampled petroglyph grooves. However, the material I sent to their lab came from petroglyph control surfaces. These are not petroglyph grooves, but from the same joint face as the petroglyphs. These were sampled to assess whether these joint faces were closed or open with respect to radiocarbon cycling. The samples I obtained from petroglyph grooves were submitted to Beta Analytic, Inc., not the UA lab. Different microenvironments, even several centimeters apart, commonly have different carbon concentrations (*9*, *10*, *13*). Contrary to their claims, Beck *et al.* failed to duplicate my sampling locations, and there is no reason to assume that their sampling of petroglyph grooves should yield results similar to those I obtained from different sampling locales.

Third, Beck *et al.* seem to make a false assumption about the abundance of subvarnish organics. They note that some of their samples had considerably less organic matter than the samples that I have submitted to labs, as if this somehow impugned the integrity of my samples. Many samples, however, lack organics. They may not be present even few millimeters from a location storing the organics. As a general rule, only about one in five samples I work with has extractable organics, but this average varies greatly depending on rock-face characteristics. For obvious reasons, I do not submit organically deficient samples for AMS dating. Comparisons of the organic contents of my best samples with their typical samples are not relevant.

A critical fourth methodological flaw is Beck *et al.*'s apparent use of an extraction technique that destroys the integrity of organic matter in their samples. Because extraction techniques are fundamental to the replication of my results, and given the omission of this methodological information in Beck *et al.*, I conducted an experiment on the effects of three different extraction techniques on a control sample (Fig. 1A) from the Northern Arizona site with observable in situ organics. On one subsample, I used a Dremel tool and a diamond abrasive wheel. On the second, I abraded the surface with a tungsten-carbide (dental drill bit) needle with a shearing motion typical of students' first efforts. The third was processed using the less destructive methods that I employ when I extract organics (*9*, *13*).

The sample processed with the Dremel grinder was mostly powdered: no intact organic matter remained; only a few fragments of rock varnish were left; and many of the harder quartz grains in the sandstone were fractured.

The sample abraded with a tungsten-carbide needle (Fig. 1B) is similar in appearance to Beck *et al.*'s Fig. 5A. In both images, there are large quartz grains, and occasional darker blocks of rock varnish that are sometimes attached to quartz grains. Critically, the abrasive scraping procedure crushed much the varnish and all of the weaker organics. Even in a quiet laboratory setting, the powdered varnish and pulverized organics are deflated from the sample surface by air turbulence generated in the abrasive scraping process. In the difficult field setting where Beck *et al.* collected petroglyph samples from nonhorizontal panels, finely pulverized organics would have been deflated away in less than a second.

In contrast, Fig. 1C shows an in situ fragment of vitrinite within the third subsample, extracted by approaches used to preserve organics (*9*, *13*). This sample is still physically embedded within the host rock material, just like the organics observed by Arrowsmith *et al.* (*7*).

Beck *et al.* may thus not have duplicated my techniques of petroglyph sampling. Instead, Beck *et al.* may have used a petroglyph sampling approach that both crushes and disperses organic remains.

Beck *et al.* also highlight T. Liu's success at ASU and subsequent failure to extract organics at Columbia as an indication that my results could not be replicated, seemingly suggesting the doctoring of Liu's ASU samples. Liu's successful extraction of organics likely occurred when I physically demonstrated different ways to mechanically extract organics while he worked in the ASU lab. Liu's lack of success at Columbia may have been because he attempted to extract organics from the same rock chips that had already been fully extracted at ASU or because these procedures cannot be learned in a few short hours (or both of the above reasons) (*24*).

Successful replication by Arrowsmith *et al.* (*7*), D. Tanner (*8*), the samples prepared at ASU by Liu while under my direction, all emphasize the need for training in the techniques used in this research. Similarly, the unsuccessful replication by Beck *et al.* and Liu at Columbia, and the fact that only two laboratories have published in this field (*13*, *25*) simply reinforce the point that these procedures are not simple and that they cannot be learned quickly. But Beck *et al.*'s failure is only partly attributable to these difficulties. Beck *et al.* also did not exactly duplicate my sampling locations, and may have used techniques that destroy the integrity of varnish-encapsulated organics.

4) *Tampering could not yield inferentially meaningful radiocarbon ages*. The nature of varnish organics and the way they are AMS dated make it effectively impossible to manipulate ages in order to obtain target dates. Before performing a dating analysis, accelerator laboratory personnel often take a subsample of what is submitted. The heterogeneous nature of the organic components, including their different sizes and densities, may cause the constituents of the subsamples to vary from split to split. For example, simply pouring a submitted carbon sample from its glass vial into a combustion vessel might result in changes in the relative mix of the organics, due to differential electrostatic attraction to the glass vial by organic fragments of different sizes and characteristics, and differential sorting of particles of varying masses and morphologies during the pouring process. This problem is exacerbated by small sample sizes.

Because one cannot know which combination and proportion of materials in a bulk sample will be analyzed by a radiocarbon lab, practically speaking, it is impossible to manipulate samples to obtain meaningful results—that is without grinding organics into a homogeneous dust. By their photographs, Beck *et al.* show that no such homogenizing of my samples occurred, further emphasizing the implausibility of any tampering.

5) *Beck* et al.*'s data on my samples show that tampering did not occur*. Table 2 in Beck *et al.* shows that no intentional manipulation of varnish radiocarbon ages could have occurred. For example, consider the age distribution of dated CWT and vitrinite from my samples. Purposeful manipulation would require both CWT and vitrinite ages, respectively, to group into tight statistical clusters. Yet Table 2 in Beck *et al.* shows the opposite. Four ages for CWT organics yield a mean of 2983 ± 1642 radiocarbon years B.P., with a range of 4500 radiocarbon years (150% of the mean). None of the CWT ages overlap at one standard deviation. The mean and standard deviation for the five finite vitrinite ages are 32,118 ± 3410 radiocarbon years B.P., with a range of 9100 radiocarbon years (28% of the mean). Only two of the ages for the vitrinite overlap at one standard deviation, and these are the two youngest ages at 27,520 ± 1500 and 28,190 ± 1305 radiocarbon years B.P. The vitrinite and CWT ages differ from one another statistically, but their own respective ages also differ immensely.

I could not possibly know the precise age of each of these different source organics, nor could I have mixed these different aged materials in exact enough proportions, for the small subsamples used in AMS dating, to obtain inferentially useful ages. I have no sources for the nine ancient organic samples, and I had no access to a radiocarbon lab to constrain accurately their ages in advance. But, if someone had access to such ancient organics of varying but known ages, and if cheating were the intention, it would be insane to mix different organic materials together instead of just using homogeneous materials with desired ages.

The AMS radiocarbon ages in Table 2 also refute Beck *et al.*'s interpretation that the vitrinite is "bituminous coal from a vitrian layer." Geologically ancient coal is radiocarbon infinite. For this reason, coal has been sometimes used to test newly constructed radiocarbon dating systems (*26*).

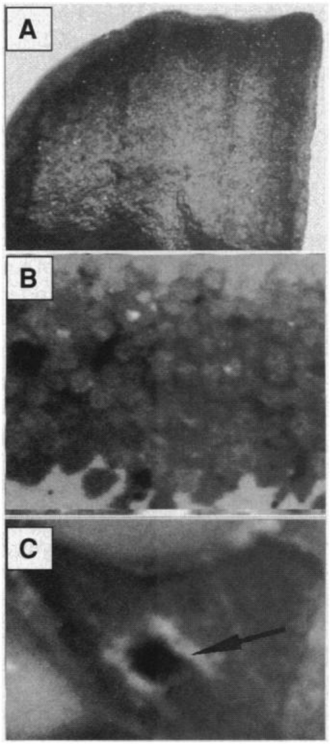

Fig. 1. (**A**) Optical view of a control sample, collected from Chevelon Canyon rock art site, northern Arizona. The darkened section (~ 5 mm thick) of the weathering rind contains subvarnish organic matter. (**B**) Photomicrograph of (A), prepared by abrading the sample with a tungsten-carbide needle with a motion roughly parallel to the surface of the sample. The resultant sample appears very similar to Fig. 5A in Beck *et al.*, and consists of sand grains and fragments of dark rock varnish. Typical grain size is approximately 0.5 mm. CWT and vitrinite were pulverized by the abrasive action, and then the dust-sized fragments were deflated by the subsequent air turbulence. (**C**) Photomicrograph of untreated sample of (A), prepared in such a fashion as to preserve organics in the weathering rind. An arrow points to a fragment of a dense, shiny organic particle (~0.2 mm wide) in a context similar to vitrinite particles reported by Arrowsmith *et al.* (*7*).

Yet five of the six vitrinite samples yielded finite AMS radiocarbon ages (*27*), and these range from 27,520 to 36,660 years B.P., with the only analytical overlap occurring with the youngest two samples.

Beck *et al.* state that finite ages for vitrinite are "presumably a result of incomplete separation of type I from type II materials." This explanation is implausible for several reasons. Cleaning CWT from the smooth, conchoidally fractured surface of vitrinite is substantially easier than cleaning typical archaeological and geological samples of roots and other heterogeneous contaminants. The smooth, glassy surface of vitrinite can simply be washed ultrasonically to remove the morphologically distinct CWT. Moreover, UA radiocarbon specialists developed a treatment to isolate vitrinite from charcoal (*28*) and, in one of the most long-lived debates in archaeology, admonished other scientists to follow such cleaning procedures (*29*). Recently, researchers have isolated different types of carbon in samples far more difficult to prepare than vitrinite and CWT (*30*). The existence of the one nonrepeat radiocarbon infinite age implies that Beck *et al.* did adequately clean this type I sample. And if one vitrinite sample was adequately cleaned, it would be hard to understand why procedures were not replicated, if indeed they were not. Given the above, it is far more likely that data derived from my samples mean just what Beck *et al.*'s Table 2 states: CWT and vitrinite in my samples have finite and widely varying ages, disproving the notion that they were introduced to manipulate radiocarbon ages.

6) *A blind test shows that tampering did not occur.* In 1995, I voluntarily participated in a blind test on the Coa, Portugal, petroglyphs. On the basis of other archaeological analyses, these were believed to have an age of about 18,000 radiocarbon years B.P. (*31*). These petroglyphs were threatened by dam construction, a circumstance that had resulted in widespread international news coverage, guaranteeing that my results would lead to close scrutiny. If my results are based on manipulated ages, I would never have voluntarily participated in a blind test, where such manipulation would stand a great chance of being exposed.

Watchman (*25*) and I (*17*) were taken to the petroglyphs separately by Portuguese authorities. "Each participant was asked to refrain from communicating with his colleagues as well as with the media for the duration of the experiment, to ensure that none of the dating scientists could in any way influence the findings of the others [(*32*), p. 878]." There was no communication between Watchman and myself prior to the submission of our independent reports. Watchman's (*25*) petroglyph ages had an average and standard deviation of 4600 ± 2100 radiocarbon years B.P. This overlaps with my results of 4100 ± 1100 years B.P. (*17*). This blind test showed that the "primary radiocarbon dates of Watchman and Dorn represented the same range [(*32*), p. 880]," an impossible result if I falsified my samples, especially given that the presumed age of the art was 18,000 years ago.

The Coa blind test is also the first time that I identified, separated, and dated vitrinite and CWT from the same sample. I received these AMS ages in 1995, and discussed the implications with a colleague at that time (*33*). I was given permission by the Portuguese funding agency to present my findings publicly, and did so at a professional meeting in May 1996 (*11*, *17*). This was before Beck *et al.* informed me that they had started working on this issue.

Not only did I voluntarily participate in a widely watched blind test of my technique, and not only did the results of this blind test demonstrate that tampering did not occur, but I also presented a paper in which I identified these problems before Beck *et al.* informed me of the "discovery" of this phenomenon. Such actions, of course, defy logic—at least, if tampering had occurred.

7) *Two scientific hypotheses have been offered to explain the co-occurrence of CWT and vitrinite in rock varnish.* Two hypotheses not discussed by Beck *et al.* explain the co-occurrence of vitrinite and CWT encapsulated by rock varnish: inheritance of different organics, and in situ diagenesis of the organics. These hypotheses are not mutually exclusive.

Inheritance of organics may occur because CWT may be transported by wind before encapsulation by rock coatings [(*21*), p. 64]. Whereas charcoal transport occurs over hundreds of kilometers (*34*), fire also comes into direct contact with rock varnish and influences its development (*35*). In archaeological contexts like petroglyphs, humans may apply charcoal to rock surfaces (*10–12*, *22*).

The in situ diagenesis of CWT influences sample age, because CWT is gradually replaced by Mn-Fe oxides (*10*, *12*, *22*). When samples are chemically digested in the lab, and in situ Mn-Fe dissolve, what were originally larger fragments of CWT are reduced greatly in size. Thus, the smallest CWT fragments should be the oldest. Analogous effects are seen in experiments on cellulose (*36*). If Beck *et al.* dated the larger CWT fragments that they photographed, they may have artificially created a bimodal age structure, when in reality there may be a continuum of ages for the vast majority of particles that are much smaller.

Vitrinite may be inherited from a variety of sources and processes, all prior to varnish formation. These include the host rock (*37*), for example, detrital grains in sandstones (*38*) or "black, brittle, solid bitumens anthraxolites, [which] occur directly inside hydrothermal veins" [(*39*), p. 754]. In addition, organisms leave remains in rock weathering pores (*40*), sometimes at great depths (*41*). Tree roots are seen 25 m deep in rock excavations; some of these fossil roots have ages from 30,000 years B.P. to "beyond ^{14}C limits (>50,000 years) (*42*)." Millions of years are, therefore, available for the diagenesis of organic tissues into vitrinite, especially in rocky landscapes with erosion rates of cm per 1000 years or less (*43*).

Vitrinite may also form in a subaerial setting under rock varnish, aided by several factors. Fe-Mn in varnish promotes diagenesis (*44*), as do temperatures (*38*) that reach 80°C [(*45*), p. 231]. The clay minerals in varnish (*46*) help promote the diagenesis of plant tissues into dense, solid organic matter (*47*), processes that are accelerated by high temperatures (*48*).

Another pathway of vitrinite diagenesis is in calcrete. "Abundant vitrinite" develops in laminar calcrete from the slow diagenesis of plant and fungal remains, and this vitrinite co-occurs with CWT (*19*). On many rock surfaces, calcrete precedes the formation of rock varnish in rock fractures (*20*); as rock fractures open and calcrete dissolves, vitrinite can be trapped by newly formed varnish.

Beck *et al.* present no alternative hypotheses for their failure to replicate my results, despite their refusal of my offer to train them in the field and at their laboratory. Yet, I present four lines of evidence, experimentation, and reasoning to show that the replication failures discussed by Beck *et al.* derive from a simple failure to duplicate my techniques. The unsuccessful replication attempts by Beck *et al.* and Liu at Columbia simply show that my extraction procedures are not simple and, in the hands of the inexperienced and untrained, yield few useful results. This is emphasized by Tanner's 1989 replication of my results and Arrowsmith *et al.*'s more recent independent confirmation, both of which occurred after training, and Liu's successful replication only while I was directing his efforts.

Consider just two ways that Beck *et al.* could have easily conducted falsification (*49*) tests of their preferred hypothesis with material at hand when they were conducting their investigation. First, although Beck *et al.* regularly make multiple measurements in their research (*30*, *31*, *50*), no replicate ages are reported on my samples. Replicate measurements would be an excellent test of my earlier point that the very nature of my samples makes it impossible to manipulate ages. Any inference of tampering would be proven false if split samples yielded different ages, as I predict.

Second, if Beck *et al.* had dated separate grain sizes in my samples, a continuum of ages may have resulted; this is a logical prediction (*14*, *16*) of Chitale's (*19*) hypothesis of diagenesis. Such a continuum would disprove the notion that two types of material with distinct ages were added to the samples to reach a target age.

Beck *et al.*'s commentary is based on analyses of samples that I prepared and submitted to them for the sole purpose of dating. Beck *et al.* did not and do not have my permission to publish data based on these samples. These samples are an important part of my ongoing research program to understand the heterogeneous nature and age of organic matter associated with rock varnish (*8*–*17*).

Beck *et al.* found vitrinite and CWT in my samples and seem to imply that these cannot co-occur naturally. Thirty years of publications and more recent studies by Chitale (*19*) and Arrowsmith *et al.* (*7*) disprove their argument. Tanner's [(*8*), p. 1367] 1989 processing of split samples, 1995 results of the only blind test in radiocarbon dating of petroglyphs (*32*), and Arrowsmith *et al.*'s 1998 SEM and petrographic replication (*7*) confirmed my techniques and results in three different ways. I have suffered a chain (*2*–*6*) of public gossip and innuendo for a year and a half—all in the face of their refusal of my offer to train them in field and laboratory techniques.

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