Reports

Cation-Ratio and Accelerator Radiocarbon Dating of Rock Varnish on Mojave Artifacts and Landforms

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The first accelerator radiocarbon dates of rock varnishes are reported along with potassium/argon ages of lava flows and conventional radiocarbon dates of pluvial lake shorelines, in an empirical calibration of rock varnish $K^+ + Ca^{2+}/Ti^{4+}$ ratios with age in the Mojave Desert, eastern California. This calibration was used to determine the cation-ratio dates of 167 artifacts. Although cation-ratio dating is an experimental method, some dates suggest human occupation of the Mojave Desert in the late Pleistocene.

SURFACE SITES CONSTITUTE A LARGE proportion of the archeological record in the southwestern United States. These sites contain important information about prehistoric settlement-subsistence systems (*1*). However, artifacts amenable to dating, such as obsidian for hydration dating or chronologically diagnostic artifacts, are not always present, leaving much of the archeological record at these sites effectively undatable. Similarly, precise ages of desert landforms can be established only rarely. It was demonstrated recently that rock varnish, a ubiquitous coating on stable natural and cultural rock surfaces in arid lands (*2*), can be dated by cation-ratio (CR) analysis (*3*). In this report we present the first ^{14}C dates of rock varnish by tandem accelerator mass spectrometry (TAMS) and we expand preliminary efforts establishing CR dating as a chronometric method (*3*).

Organic matter is present in rock varnish in only small amounts, typically less than 1 percent by weight (*4*). Because varnish coatings are usually less than 100 μm thick, a large area of varnish is required to extract enough carbon for a TAMS analysis. Depending on the thickness of the varnish, 1,500 to 20,000 cm^2 of varnish is needed.

Organic matter in rock varnish is concentrated for TAMS radiocarbon analysis by first removing all but the lowest layer of varnish from the rock surface with a tungsten-carbide needle under $10 \times 45 \times$ magnification. Then the basal layer of varnish is scraped off. The organic matter in this lowest layer is concentrated by removing the carbonates with HCl, silicates with HF, iron oxides with sodium dithionite, and manganese oxides with acidified hydroxylamine hydrochloride (*5*). Details on the TAMS method are given in (*6*). TAMS radiocarbon analyses have been made of organic matter extracted from the lowest layer of varnish from six different sites (Table 1).

There are uncertainties concerning these TAMS dates beyond analytical errors of the TAMS method. Organic method from more youthful (overlying) layers of varnish may be incorporated, but contamination from the underlying rock is unlikely because control samples had no measurable organic matter. Possible fractionation of the organic matter in varnish by the extraction procedure (*5*) was evaluated by subjecting to this treatment a sample that was previously radiocarbon dated by gas proportional counting at the University of California, Los Angeles. The radiocarbon age of the sample after treatment differed from that before treatment by 4.8 percent. That certain cations are leached from cation-exchange complexes in varnish and at least some of the organic matter is not suggests that organic matter may be found in components of varnish that are relatively immobile (*2, 3, 7*), perhaps in birnessite or goethite (*8*). Also, the nature of the organic matter in varnish is uncertain, except that it reflects $\delta^{13}C$ values of surrounding plants (*4*).

Analysis by TAMS allows the age of rock varnishes on landforms to be determined. However, varnish on most archeological surfaces cannot be dated in this fashion because of the large surface area required. Furthermore, ^{14}C TAMS cannot be applied to landforms beyond the range of the radiocarbon method. It is possible, in these cases, to use CR dating.

Cation-ratio dating is based on differences in the rates at which minor chemical elements are leached out of rock varnish. The cations used are K^+, Ca^{2+}, and Ti^{4+} because they are readily analyzed by x-ray emission techniques. Since K^+ and Ca^{2+} are more soluble than Ti^{4+}, they are removed more

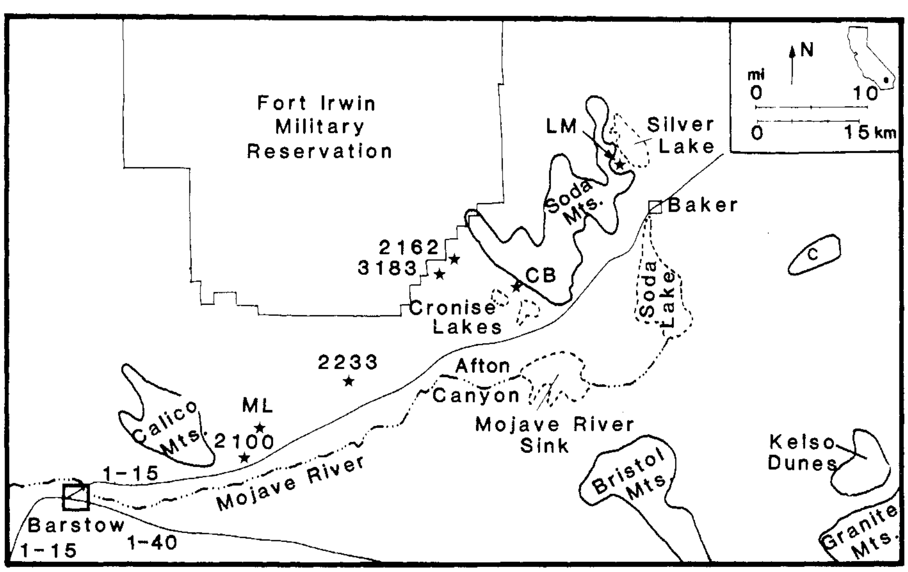

Fig. 1. Locations of calibration and archeological sites. CA-SBr-541 (Baker site) is near Baker. CA-SBr-2100, -2162, -2223, and -3183 are noted by site numbers and are adjacent to a powerline road. The Silver Lake artifacts and the radiocarbon-dated varnish sample from the eastern Soda Mountain piedmont were collected near the Lake Mohave (LM) calibration locale on the west side of Silver Lake. The TAMS calibration points are identified in Table 1. The Cima basalt flows are at C.

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Table 1. Tandem accelerator mass spectrometry (*6*) ^{14}C dates and CR's for varnishes on landforms in the Mojave Desert. Radiocarbon ages are given in conventional ^{14}C years B.P. (half-life of ^{14}C, 5568 years). Abbreviations in parentheses correspond to those in Figs. 1 and 2.

Sampling sites	^{14}C years B.P.	$K^+ + Ca^{2+}/Ti^{4+}$
Cima volcanic field basalt flow (a-1) (*10*)	14,600 ± 800 (AA-356)	3.93 ± 0.05
Alluvial fan near Silver Lake (SLP) (*11*)	13,100 ± 500 (AA-671)	4.19 ± 0.07
Shoreline of Manix Lake (ML) (543 m)	16,800 ± 700 (AA-670)	3.88 ± 0.06
Alluvial fan, East Cronise basin (CB)	16,100 ± 800 (AA-714)	4.01 ± 0.06
Alluvial fan, East Cronise basin (CB)	9,700 ± 430 (AA-937)	4.67 ± 0.21
Alluvial fan, East Cronise basin (CB)	1,370 ± 360 (AA-938)	6.15 ± 0.16

rapidly over time than Ti^{4+} from varnish cation-exchange complexes. The CR used here, $K^+ + Ca^{2+}/Ti^{4+}$, is determined by proton-induced x-ray emission (PIXE) analysis (*9*). The CR's of varnish correlate well with the K/Ar ages of basalt flows and rhyolite domes in the Coso Range of eastern California (*3*), allowing construction of a cation-leaching curve to date varnish in that region chronometrically.

A cation-leaching curve is presented here for the Mojave River basin, eastern California. It is based on the correlation of varnish $K^+ + Ca^{2+}/Ti^{4+}$ ratios with K/Ar ages of basalt flows in the Cima volcanic field (*10*), TAMS ^{14}C dates for rock varnish (Table 1), ^{14}C dates for *Anadonta* shells and tufa on the high shoreline of Lake Mohave at Silver Lake (*11*), and an estimate of the initial CR of the material incorporated into varnish (*12–15*). These locations are shown in Fig. 1. At least five PIXE analyses were made of varnish from each calibration site. Each PIXE analysis is a separate composite of varnish collected from several rocks.

A semilogarithmic least-squares regression forms the cation-leaching curve for the Mojave River basin (Fig. 2). The line is described by the equation $y = 12.71 - 2.07 \log x$, where y is the $K^+ + Ca^{2+}/Ti^{4+}$ ratio of rock varnish and x is years before the present (B.P.). Uncertainties in this regression line are caused by limited precision in the youthful K/Ar dates, the age range of the Lake Mohave high shoreline (*11*), the limited number of calibration points, and difficulties associated with TAMS ^{14}C dating of varnish. Possible systematic errors include the lag between the age of a surface and onset of varnishing on that surface, uncertainty associated with preparation of varnish for CR dating (*3*), the necessity of thin targets for PIXE analyses (*9*), and the inherent problem with a semilogarithmic plot that only a small change in a CR produces a relatively large change in age, especially for older samples.

The cation-leaching curve for the Mojave River basin differs from an earlier curve for the Coso Range (*3*), in that it has a more rapid leaching rate, a lower initial ratio, and no clear break at the Pleistocene-Holocene boundary. These differences emphasize that the reliability of each cation-leaching curve is limited to the region where it is established.

Figure 2 has been extended to cover the archeological sites in the Mojave River basin (Fig. 1). Support for this extension is threefold. First, the chemistry of airborne fallout at the archeological sites is similar to the calibration locales (*15*). This is consistent with other work indicating that thousands of square kilometers can receive dustfall with a similar composition over time (*16*). Second, the microenvironmental factors affecting varnish leaching (varnish micromorphology, lichen development, seasonality and amount of precipitation, vegetation, and so on) are similar at these sites. Third, several TAMS calibration points are derived near these archeological sites.

The CR ages of varnishes on 167 artifacts from six surface sites in the Mojave River basin are summarized in Table 2. A CR date for varnish on an artifact is derived from the average CR and standard error of PIXE analyses of multiple samples. For example, PIXE analyses of three separate samples of varnish on artifact UCLA 657-1 (Silver Lake site) yield an age estimate of 4800 years B.P. in Fig. 2 for the mean CR of 5.08. The upper standard error of the PIXE analyses (5.11) yields an age of 4600 years B.P. and the lower standard error (5.05) an age of 5000 years B.P. The date reads 4800 ± 200 years ($n = 3$). If there is only enough varnish on an artifact for one PIXE analysis, the age uncertainty is based on the experimental analysis code (*9*). Uncertainties about the regression in Fig. 2, as well as the minimal scatter ($r = -0.99$), provide little meaning to the use of a confidence band about the least-squares line to generate error margins of CR age determinations.

Most of the artifacts consist of chert flakes and cores collected in well-defined workshop areas (*17*). These types of artifacts were chosen because they could be reconstructed or refitted, thereby providing assurance of human origin and an internal check on the CR dates of individual artifacts. The small number of dates before the late Holocene in Table 1 is likely from sampling only refittable sequences, as the probability of finding refitted sequences should decrease with age. Increasing population during the Holocene would have had an additive effect.

Nineteen dates (11 percent) are between 7000 and 4500 years B.P., the approximate time of the hypothesized "altithermal" climatic period in the western United States (*18*). Eight of these are from sites CA-SBr-2162 and -2223 and are wind-abraded under the varnish coating; therefore, these date only the end of aeolian abrasion. The remaining 11 CR dates are not from ventifacted artifacts and are distributed evenly from 7000 to 4500 years B.P. These data suggest that the Mojave River basin was occupied by humans during the hypothesized altithermal period.

Also of interest are the CR dates older than 10,000 years. They are listed by site in the footnotes to Table 2. Almost all of these dates are for varnish on primary chert waste flakes. These late Pleistocene CR measurements are the oldest dates on surface arti-

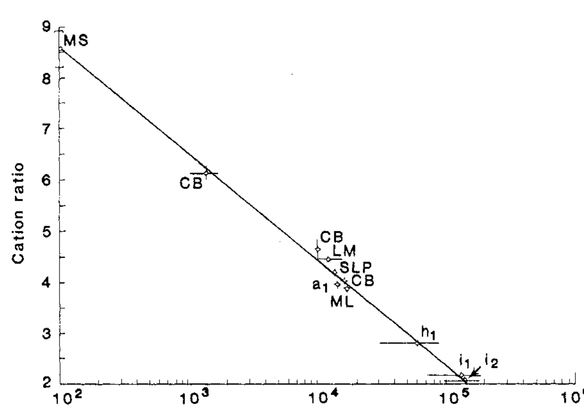

Fig. 2. Varnish cation–leaching curve for the Mojave River basin, eastern California. The cation ratio used is $K^+ + Ca^{2+}/Ti^{4+}$. Each ratio represents the average of at least five PIXE of varnish from that site; h_1, i_1, and i_2 represent K/Ar-dated basalt flows in the Cima volcanic field (*10*); LM represents the high shoreline of Lake Mohave at Silver Lake bracketed by radiocarbon dates between 10,000 and 15,500 years B.P. (*11*). Correlations of CR's with TAMS ^{14}C dates of varnish are listed in Table 1. MS is the <2-μm fraction of Mojave basin aeolian deposits (*15*), representing the initial varnish CR. Brackets on the left indicate the standard deviation for MS. The horizontal and vertical bars represent age uncertainties and the standard deviations of the varnish CR's. The line is a semilogarithmic least-squares regression that indicates the probable rate of varnish cation leaching in the Mojave River basin.

Table 2. Cation-ratio dates of 167 surface artifacts from six sites in the Mojave Desert (locations noted in Fig. 1).

Sites	Number of artifacts in age class (years B.P.)								
	≤1,000	1,050 to 3,000	3,100 to 5,000	5,100 to 7,000	7,100 to 9,000	9,100 to 11,000	11,100 to 13,000	13,100 to 15,000	15,100+
SBr-2100*	21	8	9	5	0	4	2	1	3
SBr-2162†	0	1	0	4	3	0	0	0	0
SBr-2223†	0	2	0	4	1	0	0	0	0
SBr-3183‡	33	32	18	1	2	0	3	1	2
SBr-541	1	1	3	0	0	0	0	0	0
Silver Lake	0	1	1	0	0	0	0	0	0
Total	55	45	31	14	6	4	5	2	5

*Individual artifact dates (in years B.P.) older than 10,000 years at this site: 10,200 ± 1,400, 11,500 ± 250, 12,600 ± 1,750, 14,100 ± 1,500, 15,500 ± 1,250, 20,900 ± 10,900, and 21,900 ± 4,600. †Aeolian abrasion of artifacts is evident at these sites; therefore, these determinations of varnish age are useful only as minimum estimates of the age of artifact manufacture. ‡Individual artifact dates (in years B.P.) older than 10,000 years at this site: 11,500 ± 1,600, 11,500 ± 1,750, 12,600 ± 1,900, 13,800 ± 1,950, 15,800 ± 1,650, and 17,000 ± 4,600.

facts from North America. Keeping in mind the sensitivity of a semilogarithmic plot to register large age variations with only small CR changes and the large error margins associated with a few of these dates, there are several reasons that suggest some degree of confidence for these early dates:

1) The CR dates are in five refitted flake-core sequences. Each sequence is given an age that represents an average (and standard error) of the mean CR dates of the artifacts in the sequence. The sequence ages are 21,400 ± 700 ($n = 2$), 14,200 ± 1,500 ($n = 3$), and 11,850 ± 900 years B.P. ($n = 2$) for site CA-SBr-2100 and 16,400 ± 840 ($n = 2$) and 12,400 ± 1,100 years B.P. ($n = 4$) for site CA-SBr-3183. The standard deviation within these five sequences averages 7.1 percent—much smaller than the errors associated with the individual artifacts. This indicates that older CR dates are mutually consistent.

2) A varnish micromorphological stratigraphy of lamellate over botryoid, diagnostic of a late Pleistocene age in varnishes in the study area (*19*), occurs in all examined artifacts dated as Pleistocene by the CR method.

3) Confidence is provided by four varnish-radiocarbon calibration points in the late Pleistocene.

4) Varnish was sampled from the unworked surfaces of many of the artifacts. In all the artifacts this varnish had substantially older (more leached) CR's than the varnish on the adjacent worked surfaces.

These CR dates suggest a tentative pre-Clovis human occupation of southeastern California, as only four CR dates are older than 12,000 years B.P. when two standard errors of the PIXE analyses are used to calculate the lower error margin of the CR dates. We realize that this is controversial, particularly because of the scarcity of well-dated and clearly human pre-Clovis material elsewhere in the New World and because of problems with claims for a late Pleistocene human occupation in the California Desert (*20*).

The pre-Clovis occupation in the Mojave Desert is thought to be represented by the Manix Lake Lithic Industry (*21*). The CR dates from sites CA-SBr-2100 and -3183 and the nature of the archeological material support the idea that such Manix Lake sites represent quarries used for lengthy time spans, rather than concentrations of debris specific to relatively ancient periods of prehistory in this region (*21*). Artifacts from CA-SBr-541 (Baker site) were previously thought to be older than about 9000 years B.P. (*22*) on the basis of typological considerations, but the best varnished artifacts from the Baker site are no older than about 4000 years B.P. In addition, a previous collection of varnish on five Manix Lake tools from the surface of the Calico site (*3*) gives a CR date of 2100 ± 1550 years B.P. ($n = 5$). Thus, sites thought to represent the Pre-Projectile Point Horizon on the basis of typological considerations appear to be in part middle to late Holocene in age.

The precision of CR dating varies with the amount of varnish present on an artifact and with the structure and hardness of the underlying surface. On artifacts with sufficient varnish for multiple PIXE analyses, error margins average 10.3 percent. However, on artifacts with enough varnish for only one PIXE analysis, uncertainty was based on the experimental analysis code, and the error margin of the dates averages 28.6 percent.

Another method of evaluating the precision of CR dating is to examine variations within 50 refitted sequences of flakes and cores. The standard errors of the CR dates within these sequences average 9.8 percent. Assuming that all the members of a refitted sequence were worked at the same time, the true CR date for any artifact from this region is likely to be within approximately 10 percent of the date obtained from the CR value.

There are, however, several potential limitations on the interpretation and accuracy of CR dates for artifacts. These difficulties, combined with the uncertainties represented in Fig. 2, suggest that a precision of 10 percent is only a minimum estimate. CR dating only estimates the chronometric ages of subaerial rock varnish, thus inferring a minimum age of the underlying surface. Manganese oxide deposits (*23*) and coatings of amorphous silica (*24*) may occur along with varnish on some artifacts. While these are distinguishable from varnish under 45× magnification, special precautions must be taken to avoid contamination of the varnish sample. In a few cases of youthful CR dates, artifacts from the same refitted sequence have significantly different percentages of varnish surface cover, even though they have similar CR ages, because of microenvironmental disparities. This implies that unvarnished artifacts are not necessarily younger than varnished artifacts. Finally, these CR dates are tied to the part of the calibration that is based on radiocarbon years.

In conclusion, it is possible to obtain radiocarbon dates on rock varnishes by TAMS analysis. This has important implications for the study of desert geomorphology, as the minimum ages of landforms can now be determined radiometrically to the limit of the radiocarbon method. Older landforms can be CR-dated, and accelerator dating of ^{10}Be and ^{36}Cl in varnish may be feasible. While TAMS radiocarbon dating of varnish on surface artifacts is not now possible, varnishes on 167 surface artifacts in the Mojave Desert have been CR-dated. Although the CR method is still experimental, these dates suggest that the Mojave Desert may have been occupied by humans during the late Pleistocene.

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12. Recent research established that the fraction of airborne fallout containing particles smaller than 2 μm is the basic source of varnish constituents (*2, 3, 13*). Scanning electron microscopy of historical, human-made surfaces in the Mojave Desert show no varnish growth after 50 to 70 years but the beginnings of varnish colonization in about 100 years (*14*). Thus 100 years B.P. is estimated to be the time required for the initiation of varnish growth and 100 years is assigned in Fig. 2 as the age of the <2-μm fraction of aeolian deposits collected at the calibration and archeological sites (*15*).
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15 March 1985; accepted 6 September 1985

Periodic Extinction of Families and Genera

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Eight major episodes of biological extinction of marine families over the past 250 million years stand significantly above local background ($P < 0.05$). These events are more pronounced when analyzed at the level of genus, and generic data exhibit additional apparent extinction events in the Aptian (Cretaceous) and Pliocene (Tertiary) Stages. Time-series analysis of these records strongly suggests a 26-million-year periodicity. This conclusion is robust even when adjusted for simultaneous testing of many trial periods. When the time series is limited to the four best-dated events (Cenomanian, Maestrichtian, upper Eocene, and middle Miocene), the hypothesis of randomness is also rejected for the 26-million-year period ($P < 0.0002$).

SEVERAL INVESTIGATORS HAVE PROposed that major biological extinctions exhibit a stationary periodicity through geologic time (*1–3*), with estimated period lengths ranging from 26 to 32 million years. Each proposal has included the strong implication that the periodicity itself indicates a single driving mechanism, be it earthbound (*1*) or extraterrestrial (*2, 3*). We present new data and additional analyses to support our conclusion that extinctions during the last 250 million years follow a 26-million-year periodicity (*2*).

The claims of periodicity have produced considerable controversy (*4*). Many of the negative criticisms can be summarized by two fundamental arguments: first, periodicity is just an artifact of uncertainties in the geologic time scale or in the identification of extinction events (*5*); second, periodicity is the natural consequence of many complex causes of extinction operating independently (*5, 6*).

The first argument says that inclusion of random noise in the form of spurious data could create the appearance of periodicity where none actually exists. The analyses in question (*2, 3*) start by asking whether extinction events are randomly distributed in time. This is the fundamental null hypothesis for formal statistical testing as well as the conventional wisdom in paleontology. Only if this hypothesis of randomness can be rejected with high confidence can a search for a nonrandom pattern begin. Inaccurate geologic dates or nonexistent extinction events will degrade the sample in a direction toward randomness and away from any regular signal. Thus, to include uncertain data is to make statistical testing more conservative. To argue that uncertainty in the data explains the observed periodicity is illogical.

The second argument is based on a misconception of randomness. If extinction events (as opposed to individual species extinctions) are caused by a complex of time-independent processes, they should exhibit a random (Poisson) distribution in time, typified by irregular clusters of closely spaced events separated by gaps of widely varying length. Even if the individual events in a cluster cannot be distinguished because of poor time resolution, the clusters themselves will be irregularly spaced. The surprisingly uniform spacing of extinction events in Mesozoic-Cenozoic time is thus distinctly atypical of phenomena driven by complexes of independent processes.

Considerable confusion surrounds the definition of "mass extinction" and "extinction event." At one extreme, about five large events may be singled out as the mass extinctions and all others relegated to background. At the other extreme, a continuous range of extinction intensities may be treated, with the five largest events being analogous to the 100-year flood of hydrology and other,

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