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Cosmogenic Chlorine-36 Chronology for Glacial Deposits at Bloody Canyon, Eastern Sierra Nevada

FRED M. PHILLIPS, MAREK G. ZREDA, STEWART S. SMITH, DAVID ELMORE, PETER W. KUBIK, PANKAJ SHARMA

Deposits from mountain glaciers provide an important record of Quaternary climatic fluctuations but have proved difficult to date directly. A chronology has been obtained for glacial deposits at Bloody Canyon, California, by measurement of the accumulation of chlorine-36 produced by cosmic rays in boulders exposed on moraine crests. The accumulation of chlorine-36 indicates that episodes of glaciation occurred at about 21, 24, 65, 115, 145, and 200 ka (thousand years ago). Although the timing of the glaciations correlates well with peaks of global ice volume inferred from the marine oxygen isotope record, the relative magnitudes differ markedly. The lengths of the moraines dating from 115 ka and 65 ka show that the early glacial episodes were more extensive than those during the later Wisconsin and indicate that the transition from interglacial to full glacial conditions was rapid.

The lateral and terminal glacial moraines that extend from canyon mouths along the eastern front of the Sierra Nevada have been a focus of geological interest since the earliest investigations in the area [1]. The relatively arid climate of the region has resulted in slow rates of moraine weathering, and the nesting of younger moraines within older ones has permitted inferences concerning relative age. Blackwelder [2] originally distinguished four glaciations, which he termed (from youngest to oldest) the Tioga, Tahoe, Sherwin, and McGee. Two additional glaciations have since been proposed [3]: the Tenaya (between the Tioga and Tahoe) and the Mono Basin (between the Tahoe and Sherwin).

On the basis of qualitative estimates of weathering rates, Blackwelder [2] correlated the Tioga and Tahoe glaciations with the late and early Wisconsin glaciations in the midwestern United States [now dated at 12 to 24 ka and 59 to 74 ka, respectively, based on correlation with ocean sediment cores [4]]. Sharp and Birman [3] later correlated the Tenaya and Mono Basin glaciations with the mid-Wisconsin and Illinoian midwestern glaciations [the Illinoian can be correlated with marine isotope stage 6, 130 to 190 ka [4]]. Carbon-14 dates on organic materials in sediments above or below Tioga-age glaciated sediments and on organic material in basal rock varnish [5] on moraine boulders have demonstrated that the Tioga glaciation occurred between 25 ka and 11 ka and is thus correlated with the late Wisconsin continental glaciation [5, 6].

The suggested correlations of the older deposits have remained controversial because of inadequate absolute age control. Limits have been placed with K-Ar or 40Ar-39Ar dates on interbedded lava flows [7, 8], but the paucity of minerals suitable for dating, conflicting dates, and the inherent uncertainties of having to rely on limiting ages have not allowed a closely constrained chronology to be established [9, 10]. Additionally, relative dating methods must be used in order to correlate glacial deposits from sites with interbedded volcanic materials suitable for dating to deposits at sites lacking such materials, and these methods have frequently yielded ambiguous results [10]. Understanding the relation of moun-

tain glaciations to continental ice caps is necessary for interpretation of the climate dynamics, and this requires accurate dates.

Direct dating of glacial landforms is intrinsically difficult because they are constructed out of older rocks, and most dating techniques measure the formation age of minerals rather than the age of geomorphic redistribution. Rock varnish 36Cl and cation ratio methods [6] can provide at least limiting minimum ages. However, the climate is not arid enough to develop or to preserve reliably rock varnish at most Sierra Nevada moraines. Therefore, we have developed and applied a different method for dating glacial moraines and other landforms, accumulation of cosmogenic 36Cl [11, 12].

Production rates of 36Cl in the subsurface are small because of the low underground radiogenic neutron flux [13]. When buried rocks are exposed at the land surface (for example, because of glacial excavation) 36Cl begins to accumulate because of exposure to cosmic-ray activation. The most important cosmic-ray reactions are cosmogenic thermal neutron activation of 36Cl, spallation reactions on 7K and 46Ca, and muon capture by 46Ca. The exposure age of a rock that meets the model assumptions of complete shielding followed by rapid exposure that continues without interruption until the time of measurement and negligible rock surface erosion is given by

$$t = \frac{-1}{\lambda} \ln \left( 1 - \frac{\lambda N(R - R_0)}{P} \right)$$  \hspace{1cm} (1)

where $t$ is the exposure age, $\lambda$ is the decay constant for 36Cl (2.30 x 10^{-6} yr^{-1}), $N$ is the chlorine content (atoms per kilogram of rock), $P$ is the total cosmogenic production rate of 36Cl per unit mass of rock (corrected for the effects on the cosmic-ray flux of geomagnetic latitude, elevation, and depth below the land surface), $R$ is the measured 36Cl/Cl ratio, and $R_0$ is the 36Cl/Cl ratio supported by radiogenic (from U and Th) neutrons [14].

Bloody Canyon (Fig. 1) in the Mono Basin...
Basin was selected for the initial attempt at dating the eastern Sierra Nevada glacial sequence because it has been well studied (1–3, 15, 16), a large number of morphologically distinct moraines are preserved there, and the relative age sequence can be clearly inferred from topographic relations among many of the moraines. Although Blackwelder (2) did not map the Bloody Canyon glacial deposits, he did identify the Tahoe and Tioga moraines on a photograph. Bloody Canyon is the type area for the Mono Basin glacial deposits and can also be considered to be the type area for the Tenaya designation in the eastern Sierra Nevada (3).

The samples for dating were collected from the top 5 cm of the largest boulders along the crest of each of the moraines (Fig. 1). The moraine crests were sampled because boulders there were deemed least likely to have rolled or shifted during erosion. The largest (that is, tallest) boulders were used because they were most likely to have been exposed at the surface before any erosion of the moraine crest, and because their upper surfaces were more likely to have projected above the average snow depth; snow can reduce cosmogenic ¹⁰B production by attenuating the cosmic rays reaching the rock beneath. The height of the boulders sampled ranged from 0.5 to 5.4 m. The ³⁶Cl/Cl ratios (Table 1) were measured by accelerator mass spectrometry (17).

The calculated ³⁶Cl exposure ages (Fig. 2) from the Tioga moraine are fairly tightly clustered whereas samples from the older moraines show progressively more scatter, and a few of the samples are markedly younger than the oldest values. Several factors may produce scatter or systematic errors, including (i) analytical errors, (ii) ³⁶Cl inherited from an earlier exposure episode, (iii) preferential leaching of ³⁶Cl compared to stable Cl, (iv) erosion of the rock surface by spalling, shattering, or granular disintegration, (v) gradual exposure of the rock as a result of erosion of till matrix from the moraine crest, and (vi) snow cover. Of these, the first may be expected to cause an unbiased (possibly normal) distribution of boulder ages from a single moraine, the second to produce a bias toward older ages, and all of the rest to produce a bias toward younger ages. Examination of the age distribution from the Tioga moraine, which can be inferred from independent ¹⁴C dates to have an age near 20 ka and for which the effects of processes (iii) through (vi) are probably relatively small, shows no anomalously old outliers. On this basis inherited ³⁶Cl is assumed not to be a major problem for the older moraines. In summary, we hypothesize that boulder ages from young moraines should exhibit an unbiased distribution, but as the moraines grow older, this distribution will become skewed toward ages younger than the age of the landform. For the older moraines, then, a value other than the mean is needed to give the best estimate of the age of the landform. The maximum age is a reasonable choice on the grounds that at the older ages, the bias components swamp the analytical and sampling errors, and the maximum value is the one least affected by such errors.

The mean of the three oldest boulder ages from the Tioga moraine, 21.4 ka, is in excellent agreement with the independent ¹⁴C age estimates (5, 6). The Tenaya moraine was hypothesized by Sharp and Birman (3) to result from a glaciation intermediate in age between the Tioga and Tahoe. Burke and Birkeland (16), however, were not able to distinguish between the Tioga and Tenaya moraines on the basis of relative dating methods. The mean of the four oldest boulder ages from the Tenaya moraine, 24.3 ± 0.9 ka, is within the independent radiocarbon age range of the Tioga glaciation (5) and indicates that the Tenaya moraines at Bloody Canyon represent an early (and extensive) stage of the same glacial advance as the Tioga glaciation.

On the basis of the ³⁶Cl age estimates, we have subdivided the Tahoe moraines, as mapped by Sharp and Birman (4), into an older and younger Tahoe. This subdivision closely resembles that proposed by Gillespie (7) on the basis of relative-dating parameters. The boulder ages for the younger Tahoe show a close maximum grouping near 60 ka, whereas the older Tahoe ages group close to 140 and 200 ka. There is no overlap between the maximum of the younger Tahoe samples and the minimum of the older Tahoe samples. Although the Tahoe deposits can be interpreted as a single moraine ridge (3), they can also be interpreted as an older moraine that was mantled by a younger one except at its distal end (7). The distal

<table>
<thead>
<tr>
<th>Map number</th>
<th>Sample</th>
<th>K₂O (%)</th>
<th>CaO (%)</th>
<th>Cl (ppm)</th>
<th>α* (cm² kg⁻¹)</th>
<th>³⁶Cl (¹⁰B/¹⁰Cl)</th>
<th>Boul dar age† (ka)</th>
<th>Moraine age (ka)</th>
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<td>1</td>
<td>BC86-1TI</td>
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<td>2.3</td>
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<td>400 ± 20</td>
<td>12.2</td>
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<td>BC86-3TI</td>
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<td>2.1</td>
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<td>536 ± 39</td>
<td>20.4</td>
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<td>2.9</td>
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<td>5.2</td>
<td>455 ± 20</td>
<td>20.8</td>
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<td>2.1</td>
<td>24</td>
<td>4.7</td>
<td>1350 ± 100</td>
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<td>1.7</td>
<td>3.7</td>
<td>40</td>
<td>3.4</td>
<td>244 ± 31</td>
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<td>1.5</td>
<td>67</td>
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<td>1.5</td>
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<td>1700 ± 162</td>
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<td>2.2</td>
<td>75</td>
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<td>4310 ± 250</td>
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<td>115</td>
<td>6.0</td>
<td>2790 ± 200</td>
<td>149</td>
<td>218</td>
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</table>

*α* is the macroscopic thermal neutron absorption cross section per unit mass of rock calculated from chemical data reported in (14). †Calculated using Eq. 1. ‡Samples not used in calculation of the mean age, but which are plotted in Fig. 2.
end exhibits multiple, subdued moraine crests. The wide scatter in the individual boulder ages from the older Tahoe moraine may be due to weathering and erosion effects. However, whereas boulder 19 was from the innmost older Tahoe crest and 23 was a 5-m-high boulder close to the terminal portion of the crest, boulders 20 through 22 were located close together on the northern flank of the middle morainal ridge. These three boulders all yielded ages older than the other samples and may therefore be recording an early pulse of glaciation (~200 ka), and 19 and 23 may date another separate pulse close to the culmination of isotope stage 6 (at ~140 ka).

The Mono Basin boulder ages cluster between 80 and 119 ka. The boulders on the Mono Basin moraine crests generally show evidence of weathering and spalling, which would result in minimum ages. We therefore interpret the most likely age for the Mono Basin moraines to lie in the range 110 to 120 ka (18).

The Bloody Canyon moraine chronology based on 36Cl is in apparent conflict with that determined by Dorn et al. at Pine Creek (6). They obtained varnish-cation-ratio dates ranging from 143 to 156 ka for boulders on the main Tahoe moraine at that site. None of the moraines at Pine Creek were reported to fall within the range 60 to 70 ka that we determined at Bloody Canyon. Recent reevaluation of the cation leaching curve used at Pine Creek may resolve this discrepancy (6).

On the basis of the 36Cl buildup chronology for the Bloody Canyon moraines we propose that the classical Sierra Nevada glacial nomenclature be revised. Although the term “Tahoe” is a useful geomorphic designation for moraines of early Illinoian to mid-Wisconsin age, there were probably at least four (and possibly more) distinct glacializations during this period. We agree with Gillespie (19) that if the term is to be retained it should be limited to the early Wisconsin glacial pulse, in conformity with Blackwelder’s original intention.

The new Bloody Canyon chronology supports synchronicity of glacial timing between Sierra Nevada mountain glaciation and the averaged global ice volume history indicated by the marine oxygen isotope record (Fig. 3). The intervals 120 to 110 ka and 70 to 60 ka correspond to important ice volume peaks during the buildup of Wisconsin ice, and 24 to 20 ka to the interval of maximum global ice (4, 20). The 36Cl age estimates for the Bloody Canyon glacial maxima also correspond closely to episodes of minimum Northern Hemisphere summer insolation (21). They thus offer additional support for the Milankovitch theory of global glacial timing.

Although there is good agreement between the timing of glaciations at Bloody Canyon and those recorded by the marine 14O signal, there is strong disagreement with regard to glacial magnitude. The marine record (20) shows a fairly regular progressive buildup of ice from the penultimate interglacial through the last glacial maximum at about 20 ka. In contrast, at Bloody Canyon the longest moraines were deposited during the earlier glaciations (Mono Basin and younger Tahoe), and progressively smaller moraines (Tenaya and Tioga) were formed during the Wisconsin maximum. This inverse relation between the glacial magnitudes shows that the response of mountain glaciers and continental ice sheets to climatic forcing differed markedly. A rapid transition from full interglacial to full glacial conditions is indicated by the 114 and 119 ka dates on the extensive Mono Basin moraines. One interpretation of the discrepancy between the sudden transition to full glaciation indicated at Bloody Canyon and the much slower change in the marine oxygen isotope record is that the global climate may have suddenly switched to a glacial mode (21). Small mountain glaciers might respond rapidly to this impulse, but continental ice sheets might take much longer to arrive at equilibrium with the global glacial climate.

REFERENCES AND NOTES
used in calculating the total cosmogenic $^{36}$Cl production rate were 3560 ± 465 atoms of $^{36}$Cl per mole K per year, 2745 ± 245 atoms of $^{36}$Cl per mole Ca per year and (2.64 ± 0.42) × 10⁷ thermal neutrons stopped per kilogram of rock per year. These values are at the rock surface, at sea level, and at 90° geologic latitude.


17. The samples were ground to a particle size smaller than the mean grain diameter and leached with deionized water to remove meteoric or anthropogenic $^{36}$Cl. They were then dissolved in hot HF and HNO₃, and the CI was extracted as HCl by air stripping, followed by trapping in a AgNO₃ solution (14). Major and minor elements were measured by x-ray fluorescence and rare earth elements by inductively coupled plasma atomic-emission spectrometry. The Cl content was measured in a teflon diffusion cell using an ion-specific electrode technique. P. S. Arscavage and E. L. Campbell, Terra Nova 30, 745 (1983); H. N. Elshelwein, Geostand. News. 11, 115 (1987). Values given are the mean of three to five replicate analyses. A value of $30 \times 10^{-13}$ [typical for granitic rocks (12)] was assumed for the radiogenic $^{36}$ClCl ratio ($R_0$). This radiogenic $^{36}$Cl background ranges from about 5% to less than 1% in relation to the cosmogenic $^{36}$Cl used for dating.

18. We have considered two alternative hypotheses: (i) The boulder age distribution from the Mono Basin moraines may indicate more than one glaciation between 80 and 120 ka. Some evidence from moraine morphology supports the notion that there were multiple glaciations (6), although the evidence is not as strong as for the older Taboe moraine. (ii) Even the oldest boulder dates on the Mono Basin may only be minimum and the Mono Basin glaciation could have an age of ~140 ka. Although this assumption would bring the Bloody Canyon deposits into better agreement with the marine $^{14}C$ record regarding the relative glacial volumes during isotope stages 6 and 5d, in the absence of positive results to support this assumption, we prefer the interpretation indicated by the maximum measured ages.


23. This research was supported by National Science Foundation grants EAR-8603440, SES-8901347, PHY-8515908, and PHY-8818281. We thank A. R. Campbell, C. V. Kruger, and D. Elliott-Fisk for help in sample collection. L. Brandvold and the New Mexico Bureau of Mines and Mineral Resources for use of laboratory facilities. R. Dorn and A. Gillespie for helpful comments, and R. Tong and S. Tullia-Fitzpatrick for help in the $^{36}$Cl analyses.

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Biomimetic Total Synthesis of Proto-Daphniphylline

SERGE PIETTRE AND CLAYTON H. HEATHCOCK*

Proto-daphniphylline, the imputed biogenic parent of the Daphniphyllum alkaloids, has been assembled in a biogenetically styled laboratory synthesis in which a pentacyclization process is the fundamental synthetic stratagem. This extraordinary transformation involves the formation of six $\sigma$-bonds under the influence of three elementary reagents—potassium hydroxide, ammonia, and acetic acid. The facility of the process adds credence to the previous speculation that a similar process is an important step in the biosynthesis of the Daphniphyllum alkaloids.

The oriental deciduous tree Yu-zurilha (Daphniphyllum macrodum) Miquel contains a family of squa-lene-derived alkaloids, of which daphniphylline (1) and secodaphniphylline (2) are representative members (1). It has been suggested that these complex natural products are biosynthesized from squa-lene by way of the embryonic precursor 3, proto-daphniphylline (2). In this report, we describe a remarkably simple synthesis of 3 wherein the five rings are formed in a series of three straightforward chemical transformations of the acyclic dialdehydes (E)-4 or (Z)-4.

As shown in Scheme 1, the lithium enolate of tert-buty1 (t-Bu) acetate was alkylated with homogenyl iodide (5) (3) to give ester 6, which was deprotonated with lithiu-m diisopropylamide (LDA). The resulting enolate was alkylated with the dimethyl acetal of 4-bromobutanal to provide ester 7. Mild acidic hydrolysis of the acetal function afforded aldehyde 8, which was allowed to react with another mole-equivalent of the lithium enolate derived from ester 6 to provide hydroxy-dieste r 9. Dehydration of this $\beta$-hydroxy ester was accomplished by successive treatment with methanesulfo-nyl chloride in the presence of triethylamine and 1,8-diazabicyclo[5.4.0]jundec-7-ene (DBU); diester 10 was obtained as a 10:1 mixture of E and Z isomers at the newly created double bond. The isomers were separated by chromatography on silica gel, and the major isomer was reduced by treatment with disoubutylaluminum hydride (DIBAL). The resulting diol was oxidized by the method of Swern (4) to obtain (6E,14E,18E)-10,11-dihydroxy-17,18-dialdehyde (4).

Scheme 2: Alkylation of the lithium enolate of tert-buty1 trimethylsilylacetate with halide 5 gave ester 11, which was deprotonated with LDA and treated with aldehyde 8. The diester produced in this manner (Peterson olefination reaction (5)) is a 7:3 mixture of Z and E double-bond isomers. The isomers were separated by chromatography and the major isomer converted in two steps to (Z)-4.

Both (E)- and (Z)-4 were converted into proto-daphniphylline (3) by the following simple procedures (Scheme 3). Gaseous ammonia was added to a dichloromethane solution of the dialdehyde, ammonium acetate, and triethylamine hydrochloride. After 16 hours at room temperature the solvent was removed under vacuum and the residue was taken up in glacial acetic acid. After 2 hours at 80°C, compound 3 was obtained in 15 ± 2% yield. The yield of 3 was improved to 50% by the following modified three-step procedure. A benzene solution of the dialdehyde was added to a vigorously stirring solution of 50% aqueous potassium hydrox-ide containing 5% by mole of tetra-n-buty-

*To whom correspondence should be addressed.