

MEASUREMENTS OF COSMOGENIC ^{14}C PRODUCED BY SPALLATION IN HIGH-ALTITUDE ROCKS

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ABSTRACT. The production of radioisotopes at the Earth's surface by cosmic-ray effects has been discussed for many years. Only in the past few years, with the higher sensitivity provided by accelerator mass spectrometry (AMS) in detecting ^{10}Be , ^{26}Al and ^{36}Cl , have the radioisotopes produced in this way been measured. We report here our measurements of cosmogenic ^{14}C in terrestrial rocks at high altitude, and comparisons to other exposure-dating methods.

INTRODUCTION

Radionuclides are produced at the surface of the Earth by the interaction of cosmic-ray particles with matter, in a manner similar to the production in meteorites in space, or in the upper atmosphere. Lal and Peters (1967) discussed the production of cosmogenic isotopes at the surface of the Earth, as a function of atmospheric depth. At depths greater than about 100 g cm^{-2} , the flux attenuates with a mean free path of about 150 g cm^{-2} . The production rate is >1000 times less at sea level than, *e.g.*, on the surface of the Moon (Jull, Donahue & Reedy 1991).

Before the advent of accelerator mass spectrometry (AMS), it was not possible to measure the concentrations of these cosmogenic radioisotopes, which typically are of the order of 10^6 atoms g^{-1} in a rock. Processing very large samples of rock by counting a long-lived radioisotope was impractical. Only a few measurements on shorter-lived isotopes were possible, such as that of Lal, Arnold and Honda (1960), who studied ^7Be in a mountain lake, and Yokoyama, Reyss and Guichard (1977), who studied ^{24}Na in objects exposed on the top of Mont Blanc. These authors recognized that measurements of cosmogenic radioisotopes in terrestrial samples would one day be feasible.

The last five years of AMS studies demonstrate this feasibility, in studies on ^{10}Be , ^{26}Al (Lal & Arnold 1985; Nishiizumi *et al.* 1986; Klein *et al.* 1986) and ^{36}Cl (Phillips *et al.* 1986) in rocks. Similarly, improvements in rare-gas mass spectrometry enabled studies of cosmogenic ^3He (Craig & Poreda 1986; Kurz 1986). Further advances have been made in these isotopic systems since these initial reports. We reported on cosmogenic ^{14}C in rocks at the Thirteenth International Radiocarbon Conference in Dubrovnik (Jull *et al.* 1989), and in ice (Lal *et al.* 1990). Others have advanced the fields of cosmogenic ^3He and ^{21}Ne by rare-gas mass spectrometry (Poths & Goff 1990; Staudacher & Allegre, 1990; Cerling 1990; Kurz *et al.* 1990).

We describe here our efforts to improve our measurements of cosmogenic ^{14}C . Our interest in this subject grew out of the original observation that the ^{14}C level in Antarctic meteorites of very old terrestrial age was not zero (Brown *et al.* 1984; Jull *et al.* 1984; Jull *et al.* 1989). The detection of the small cosmogenic signal requires careful separation from any atmospheric ^{14}C , and interpretation must take into account the several factors that can influence the production rate. To that end, we have concentrated on *in-situ* ^{14}C in samples from three areas where measurements on other radioisotopes are already available: volcanic rocks, glacial till and lava flows from Mauna Kea, Hawaii, and Meteor Crater.

PRODUCTION OF COSMOGENIC ^{14}C IN ROCKS

The number of ^{14}C atoms per gram of rock (N_{14}) produced by cosmic-ray spallation in a surface rock is given by

$$N_{14} = \frac{P_o}{(\lambda + \rho a/L)} (1 - e^{-(\lambda + \rho a/L)t}) \quad (1)$$

where P_o is the production rate at the rock surface per gram of rock, λ is the decay constant in yr^{-1} , ρ is the density of the rock in g cm^{-3} , L is the mean free path of the cosmic-ray neutrons in g cm^{-2} , a is an erosion rate (cm yr^{-1}) and t is the time of exposure in years. Englert *et al.* (1988) and Sisterson *et al.* (1991) showed that the production of ^{14}C in SiO_2 is predominantly (95–97%) from oxygen. For a production at some depth in the rock, d , one can define

$$P_d = P_o e^{-\rho d/L} \quad (2)$$

where d is the depth in cm. The production rate, P_o , can also be calculated for a given altitude, if the sea-level rate, P_s , is known. Thus,

$$P_o = P_s e^{(1033-x)/L} \quad (3)$$

where x is the atmospheric depth in g cm^{-2} . The production rate for different geomagnetic latitudes will also vary. The number of particles that are not deflected by the Earth's magnetic field depends on the cut-off magnetic rigidity (Shea, Smart & Gentile 1987). Thus, more cosmic-ray particles are incident at higher latitudes.

EXPERIMENTAL METHODS

Samples of 3–10 g of rock were crushed in a steel mortar and pestle. To remove weathering carbonates, the resultant powder was treated with 3 N HCl until no further reaction was observed. The sample was then rinsed with distilled water, and dried. Up to 4 g of the dried rock was mixed with 6–8 g of iron chips, which act as a combustion accelerator and susceptor for radio-frequency (RF) induction. The mix was placed in an alumina crucible and heated in air to 500°C for 1 h. After this time, the crucible was placed in the RF furnace. The system was evacuated, and then filled with oxygen. The sample was heated up to 1700°C in the RF furnace for 3–4 min in a flow of oxygen. The gases evolved flow through an MnO_2 trap to remove sulfur compounds, and Pt/CuO at 450°C to convert all carbon compounds to CO_2 . The CO_2 was collected along with water and liquid oxygen in a -196°C trap. After heating was completed, the sample was allowed to cool. After cooling, the oxygen was pumped away, and the CO_2 was separated from the water by warming the trap to -78°C (dry ice/alcohol mixture). The sample CO_2 was collected and its volume measured in a known volume, using a capacitance manometer. The sample gas was then diluted to *ca.* 1 cm^3 , with ^{14}C -free CO_2 . The gas was reduced to graphite in a small reaction system, similar to that used by Slota *et al.* (1987), but the Ni foil is no longer used (Donahue 1991). The reaction follows the Boudouard (1902) disproportionation reaction



The CO_2 reduced to CO over Zn at 450°C ; then the CO reacted further with Fe at 625°C to produce graphite. Any CO_2 produced over the Fe was converted back to CO by the Zn.

The graphite powder was pressed into an accelerator target holder (see Slota *et al.* 1987), and backed with a 1-mm Al plug. The sample graphite was loaded into the accelerator with 7 other

samples and 2 standards. Linick *et al.* (1986) and Donahue, Jull and Toolin (1990) have described the AMS measurement procedures in detail.

RESULTS AND DISCUSSION

On the basis of our earlier work (Donahue, Jull & Toolin 1990), it was clear that some samples from Mauna Kea, Hawaii had lower than the expected saturated value of ^{14}C . One sample, which was estimated to be 23 ka old by *in-situ* ^{36}Cl , had only the equivalent of 3–4 ka exposure to ^{14}C , assuming no erosion. We therefore discuss comparisons of ^{14}C in rocks from three areas, where data from other radioisotopes are available.

In-Situ ^{14}C in Volcanic Rocks

The first study was a series of measurements on *in-situ*-produced ^{14}C in volcanic rock samples, from the eastern Sierra Nevada, California, and southern New Mexico, which had previously been studied by Leavy (1987) for ^{36}Cl content. The results are reported in Table 1, and plotted in Figure 1, compared to the expected saturated content of ^{14}C as a function of altitude. Figure 1 clearly shows that a number of the samples reported in Table 1 are below the saturated amount of ^{14}C , whereas five other samples plot at or close to saturation. In Column 3 of this table, we list previously measured ages of a series of volcanic rocks, based on K-Ar or obsidian hydration dating (Leavy 1987). It should be mentioned that the very young K-Ar ages quoted may have large errors. Column 4 gives the number of ^{14}C atoms per gram of rock determined in our measurements. Column 5 is the “apparent” saturated production rate, P_a , determined from the age (t , Column 3) and number of ^{14}C atoms g^{-1} (N_{14} , Column 4) using the equation

$$N_{14} = \frac{P_a}{\lambda}(1 - e^{-\lambda t}) \quad (5)$$

Column 6 is the P_a corrected to an apparent sea-level production rate, P_{as} . Physically, P_{as} is the saturated activity that a rock would achieve at sea level in the absence of erosion. It is related to P_s (see Eq. 1 and 3) by the expression

$$P_{as} = \frac{P_s \lambda}{\lambda + \rho a/L} \quad (6)$$

From this equation, using the results in Column 6 of Table 1, and the previously determined P_s of 19 ± 2 atoms $\text{g}^{-1} \text{yr}^{-1}$ (Donahue, Jull & Toolin 1990), we can deduce a value for a , the erosion rate in cm yr^{-1}

$$a = \frac{L\lambda}{\rho} \left(\frac{1 - P_{as}/P_s}{P_{as}/P_s} \right) \quad (7)$$

Values of the erosion rate calculated from Equation 7 are listed in the last column of Table 1. The results indicate differences in erosion rate among rhyolite, basalt and obsidian. The rhyolites have the fastest erosion rate, as one would expect from observation of the friability of the samples. Erosion rates of basalt and obsidian were uniformly low. Sample SLR-6 is a well-cemented rhyolite, and from our data, has a lower erosion rate than other rhyolite. We conclude from these results that the ^{36}Cl content cannot be used as an estimate of age without correcting for the effects of erosion, which are significant even over the mean life of ^{14}C , and thus, must dominate the interpretation of *in-situ* production of longer-lived and stable isotopes.

TABLE 1. *In-Situ* ^{14}C In Rocks and Estimates Of Erosion Rate

| Sample | Atm. depth (g cm ⁻²) | Age (yr) | ^{14}C atoms g ⁻¹ × 10 ⁵ | ^{14}C atoms g ⁻¹ (saturated) × 10 ⁵ | P _a | P _s (atoms g ⁻¹ yr ⁻¹) | Erosion (cm yr ⁻¹) |
|--|-------------------------------------|--------------------------|--|---|----------------|---|-----------------------------------|
| <i>Punch Bowl Crater, Mono Craters Volcanic Field, California (rhyolite)</i> | | | | | | | |
| SLR-2 | 778 | 10,450 | 2.24 ± 0.75 | 3.1 ± 1.2 | 37.5 | 6.9 ± 2.7 | 0.011 ± 0.004 |
| MC-1 | 778 | 10,450 | 8.44 ± 0.38 | 11.8 ± 4.4 | 143 | 26 ± 10 | <0.001 |
| MC-3 | 765 | 8700 | 1.18 ± 0.37 | 2.2 ± 0.8 | 26.0 | 4.4 ± 1.6 | 0.020 ± 0.007 |
| MC-4 | 726 | 8700 | 1.71 ± 0.43 | 2.6 ± 0.8 | 31.8 | 4.1 ± 1.3 | 0.022 ± 0.007 |
| SLR-3 | 763 | 6450 (assume >20,000) | 12.6 ± 0.7 | 23.3 ± 0.4 | (281) | (46) | 0 |
| MC-2 | 763 | 6450 | 1.77 ± 0.55 | 3.3 ± 1.3 | 152 | 25 ± 1 | 0.012 ± 0.004 |
| <i>Wilson Butte, Mono Craters Volcanic Field, California (rhyolite)</i> | | | | | | | |
| SLR-6 | 759 | 2500(6) | 2.11 ± 0.34 | 8.1 ± 1.3 | 98 | 15.7 ± 2.5 | 0.0009 ± 0.0001 |
| <i>Panum Crater, Mono Craters Volcanic Field, California (obsidian)</i> | | | | | | | |
| SLR-4 | 796 | 1300(6) | 1.50 ± 0.40 | 10.3 ± 2.7 | 125 | 26 ± 7 | 0 |
| <i>Long Valley Caldera, California (rhyolite)</i> | | | | | | | |
| SLR-7 | 742 | 106,000 | 3.34 ± 0.35 | | | | |
| SLR-7B | 742 | 106,000 | 3.59 ± 0.35 | 3.5 ± 0.3 | 42 | 6.0 ± 0.5 | 0.013 ± 0.001 |
| MM-3 | 742 | 106,000 | 3.56 ± 0.36 | 3.6 ± 0.4 | 43 | 6.2 ± 0.6 | 0.012 ± 0.001 |
| <i>Long Valley Caldera, California (basalt)</i> | | | | | | | |
| MM-2 | 777 | 145,000 | 8.66 | 8.7 | 105 | 19.1 | 0 |
| <i>Cat Hills, New Mexico (basalt)</i> | | | | | | | |
| DA-1 | 849 | 140,000 | 4.33 ± 0.40 | 4.30 ± 0.4 | 52 | 15.4 ± 1.4 | 0.0010 ± 0.0001 |
| SG-1B | 850 | 320,000 | 4.35 ± 0.38 | 4.35 ± 0.38 | 53 | 15.5 ± 1.4 | 0.0010 ± 0.0001 |

*P_a is the apparent production rate at the sample altitude, in atoms g⁻¹ yr⁻¹.**P_s is the apparent sea-level production rate, in atoms g⁻¹ yr⁻¹.

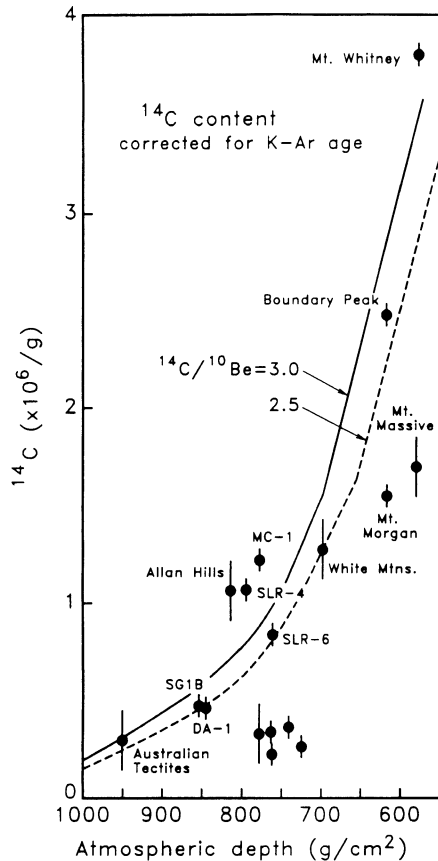


Fig. 1. Cosmogenic ^{14}C (atoms g^{-1}) for samples in Table 1, plotted against atmospheric depth (g cm^{-2}). The labeled points are from Donahue, Jull and Toolin (1990). The numbered points are saturated samples listed in Table 1.

^{14}C in Glacial Till and Lava Flows from Mauna Kea, Hawaii

We investigated the *in-situ* buildup of ^{14}C in glacial deposits and eroded lava flows on Mauna Kea. The high altitude of these samples compensate, to some extent, for the lower production rate at 18°N latitude. Results from several samples are presented in Table 2. Column 3 lists the ages of the rocks as determined by *in-situ* ^{36}Cl and desert-varnish ^{14}C studies (Dorn *et al.* 1991). Column 4 lists our measurements on these rock samples. From our results and the previously determined P_s of 13 ± 1 atoms $\text{g}^{-1} \text{yr}^{-1}$ at this location (Donahue, Jull & Toolin 1990), and from Equations 1 and 3, assuming no erosion, we calculate exposure ages in Column 5. Alternatively, in order for our results to be consistent with the longer estimates of age (Dorn *et al.* 1991), we have determined erosion rates as listed in Table 2.

Periodic episodes of erosion may be the explanation for the apparent ages determined by ^{36}Cl , because of the difference in the time integrated by the accumulation of this isotope (*ca.* 500 ka), as opposed to ^{14}C . Differences with varnish ^{14}C ages may be explained by incorporation of old carbon into the varnish. The current level of ^{36}Cl cannot be explained by concurrent production with the ^{14}C . One possibility is that the samples were periodically moved or rotated. This could certainly explain the Sample 5 boulder (Table 2). For Sample 2, no *in-situ* ^{36}Cl is available. If the *in-situ* ^{14}C age (or erosion rate) is correct, the carbon in the desert varnish (which gives a conventional ^{14}C age of 16.6 ka) must reflect the age of some different event, or the material contains older carbon, which was deposited on the rock surface.

TABLE 2. *In-Situ* ^{14}C in Rocks from Mauna Kea, Hawaii

| Sample | Atm. depth (g cm ⁻²) | Age estimate (ka) | ^{14}C atoms g ⁻¹ × 10 ⁵ | ^{14}C age estimate (ka) | Erosion rate (cm yr ⁻¹) |
|--|----------------------------------|-------------------|---|-----------------------------------|-------------------------------------|
| 1. Glacially eroded lava flow, 4054 m | 625 | 14.7 ± 0.5* | 14.8 ± 0.5 | 14–20 | --- |
| 2. Glacially eroded lava flow, NW side, 3962 m | 621 | 16.6 ± 0.2** | 7.3 ± 0.3 | 4.4–6.5 | 0.009 |
| 5. Older Makanaka glacial till, 3500 m | 671 | 20.3 ± 2.3* | 4.1 ± 0.5 | 2.6–4.6 | 0.010 |
| 7. Glacially eroded bedrock, 3169 m | 709 | --- | 9.9 ± 0.7 | >20 | --- |
| 8. Waihu end moraine 3109 m | 714 | 63 ± 2* | 8.3 ± 0.8 | >18 | |

* ^{36}Cl age (Dorn *et al.* 1991)**Desert varnish (Dorn *et al.* 1991)TABLE 3. *In-Situ* ^{14}C in Quartz from Meteor Crater, ArizonaAltitude: 1680 m; Atmospheric depth: 804 g cm⁻²

| Sample | Age estimate (yr) | ^{14}C atoms g ⁻¹ × 10 ⁵ | <i>In-situ</i> ^{14}C age (yr) | Equivalent erosion rate |
|--------|-------------------|---|---|-------------------------|
| M-1 | 19,900 ± 1100* | 7.8 ± 0.9 | >13,000 | |
| M-2 | 19,200 ± 1300* | 4.9 ± 1.0 | 5000–11,000 | 0.004 ± 0.001 |
| M-3 | 12,500 ± 1100* | 0.5 ± 0.7 | <1500 | 0.09 ± 0.13 |
| M-4 | 11,700 ± 800* | 4.8 ± 1.2 | 4000–14,000 | 0.004 ± 0.001 |
| M-5 | 39,800 ± 1800* | 17.8 ± 0.9 | >20,000 [†] | |
| M-7 | >37,000** | 15.1 ± 1.4 | >20,000 [†] | |

*Age estimate from *in-situ* ^{10}Be and ^{26}Al (Nishiizumi *et al.* 1989)**Age estimate from Dorn, unpublished ^{14}C age on rock varnish[†]Contains more ^{14}C than expected from production rate, 97 ± 9 atoms g⁻¹ yr⁻¹

Meteor Crater

We have also studied *in-situ* cosmogenic ^{14}C in some quartz samples from Meteor Crater, Arizona; 5 quartz samples extracted from sandstone by Nishiizumi *et al.* (1989) and 1 massive quartz (no. 7), showed some desert varnish accumulation. Results of measurements of these samples are listed in Table 3. Recent results of measurements on *in-situ* ^{36}Cl in Meteor Crater samples indicated that this crater formed about 50 ka ago (Phillips *et al.* 1991; Nishiizumi *et al.* 1991). Our samples M1, M5 and M7 are consistent with this result. Our interpretation of M2, M3 and M4 is, most simply, that these rocks had a recent exposure to cosmic rays shorter than those of the other three samples. These results illustrate the importance of good sample selection in *in-situ* exposure age studies.

CONCLUSIONS

We have studied the levels of ^{14}C produced by cosmic-ray spallation in samples from locations in the eastern Sierra Nevada, Mauna Kea and Meteor Crater. Many of these samples show lower than the number of ^{14}C atoms per gram of rock expected from other measurements of the rock-surface

age. We take this as evidence for the importance of both erosion and possible complicated exposure histories of these samples. Measurement of more than one cosmogenic isotope is probably necessary to define a real "exposure age."

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