

NOTES AND COMMENTS

¹⁴C DATING OF LASER-OXIDIZED ORGANICS

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ABSTRACT. We used a continuous krypton ion laser to rapidly oxidize milligram-sized fragments of coniferous driftwood of known ages, and dated the resulting carbon dioxide by accelerator mass spectrometry (AMS). AMS ¹⁴C ages of non-pretreated young wood from different parts of two logs were within 10% of the ages of conventionally determined alkaline insoluble fractions. The age of the oldest whole wood measured after laser oxidation was within the error ranges of conventional values.

INTRODUCTION

Organic matter encapsulated in silica skins (Watchman 1990, 1992), oxalate crusts (Watchman 1991), rock varnish (Dorn *et al.* 1992) and other media (Loy *et al.* 1990) is generally scraped, dissolved or selectively oxidized (Russ *et al.* 1990; Russ, Hyman & Rowe 1992) from the underlying rock, and chemically processed into graphite for AMS ¹⁴C dating (Jull *et al.* 1986). Loss of microstratigraphic context and contamination are two significant problems that we hope to overcome in dating laminae in rock-surface accretions by oxidizing organic matter in cross-sections with a focused, low-energy, continuous laser.

METHODOLOGY

We mounted each sample on wire supports in a vacuum-tight, micro-combustion chamber (3 cm³) fitted with glass windows. Atmospheric CO₂ was evacuated from the chamber under high vacuum and pure oxygen was injected at 5 k Pa. Using a diffraction-reduced convergent lens (f=153 mm, 0.003 mm spot diameter), we focused onto the sample a continuous Kr-ion laser (Coherent Innova 2000, 2.5 W light power, 56.4 A tube current, 413 nm). CO₂ formed by laser oxidization was collected in a liquid nitrogen “trap” and δ¹³C was measured by mass spectrometry prior to making graphite (Jull *et al.* 1986).

We prepared an oxalate wafer from a precipitate of NBS oxalic acid (OX 1) and calcium chloride at room temperature because we required an oxalate standard for our future research goal of dating finely laminated whewellite (CaC₂O₄.H₂O) deposits on rock surfaces. We collected samples of *Larix* sp. (WB-182-66) and *Picea* sp. (WB-100-66) on Nordaustlandet, Svalbard (80°N 20°E) from driftwood logs embedded in raised shingle-beach deposits; we also collected from glacial till *Larix* sp. (WB-18-66) (Blake 1980, 1986; Lowdon & Blake 1980).

RESULTS

Our preliminary, uncalibrated AMS ¹⁴C age results (¹⁴C yr before AD 1950) from the Arizona AMS Lab (AA) are for laser-oxidized untreated whole wood (Table 1). The Geological Survey of Canada

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TABLE 1. Comparison between laser-AMS and conventional carbon isotopic and uncorrected ^{14}C age data for oxalate and coniferous driftwood

GSC-nos.	Sample	WT (mg)	Laser $\delta^{13}\text{C}\text{‰}$	GSC $\delta^{13}\text{C}\text{‰}$	GSC alk. insoluble (2 σ)	Laser-AMS untreated wood (1 σ)	AA-nos.
--	Oxalate	10.0	-18.1	-19.0	--	Modern	-8861
-3500	WB18-66	12.6	-24.2	-24.1	1120 \pm 60	1455 \pm 85	-8864
-2441	WB100-66	3.6	-26.3	-24.4	6150 \pm 60	5600 \pm 60	-8863
-2441	WB100-66	5.8	--	-24.4	6150 \pm 60	5540 \pm 75	-8045
-1728	WB182-66	9.8	--	-22.4	7940 \pm 140	7860 \pm 135	-8865

(GSC) ^{14}C measurements are for alkaline-insoluble fractions (cellulose and lignin) determined by conventional methods. We found that, by using the laser-AMS method, both the $\delta^{13}\text{C}$ ($= [^{13}\text{C}/^{12}\text{C}]_{\text{sample}} / [^{13}\text{C}/^{12}\text{C}]_{\text{standard}} - 1$) and the fraction of modern ^{14}C from a standard oxalate wafer were about 4.7% lower (equivalent to *ca.* 300 ^{14}C yr) than the accepted values for NBS OX I (-19.0‰ and 1.046%, respectively). We suspect the difference is caused by non-linear isotope exchange during formation of mono- and bi-hydrated oxalate salts rather than laser-induced processes, but further tests are needed to rule out laser fractionation. The $\delta^{13}\text{C}$ values for whole wood are higher in our laser-generated CO_2 than for the alkaline-insoluble fractions because different components were measured, and because slight isotopic fractionation may occur during incomplete laser oxidation (Powell & Kyser 1991).

The laser-oxidative method combined with ^{14}C AMS gives an age for the oldest wood within error ranges identical to the conventionally determined value (Table 1). Two laser-AMS ^{14}C ages for specimen WB100-66 only differ by about 1%, and indicate acceptable reproducibility of results. However, the ages for the young wood samples determined by laser-AMS are beyond the error ranges of their previously determined values, possibly because the two laser-AMS samples (WB-18-66, WB-100-66) were not sub-samples of fragments used in conventional dating. More important, the whole wood specimens are not homogeneous because they contain soluble fractions not in isotopic equilibrium with cellulose.

CONCLUSIONS

Our preliminary results demonstrate convincingly the feasibility of focusing a low-energy continuous laser to oxidize rapidly small samples of organic matter in geological, geomorphological and archaeological contexts for AMS ^{14}C dating. We are now collecting more data from our driftwood samples and developing the laser-oxidative method for routine dating of molluscan growth bands, subvarnish organic matter, siliceous coatings containing algal remains and oxalate-rich laminations.

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