[RADIOCARBON, VOL. 36, NO. 2, 1994, P. 247–250]

AMS ^{14}C AGE DETERMINATIONS OF TISSUE, BONE AND GRASS SAMPLES FROM THE ÖTZTAL ICE MAN

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ABSTRACT. ^{14}C ages of samples from the Ötztal Ice Man, found on the Hauslabjoch in the Tyrolean Alps in September 1991, were determined using accelerator mass spectrometry (AMS). Uncalibrated ^{14}C ages of 4555 ± 34 BP, 4560 ± 65 BP and 4535 ± 60 BP were measured on tissue (mean of four samples), bone and grass, respectively, from the Ice Man. The mean of all of our measurements is 4550 ± 27 BP.

INTRODUCTION

In September 1991, a mummified corpse was found in a small rock depression on the Hauslabjoch, in the Ötztaler Alps (part of the Simalaun Massiv), South Tyrol, Italy. Although the associated artifacts, including a copper axe, suggested a Late Stone Age origin, it was imperative to determine an absolute age for the Ice Man himself. Because the preciousness of the find dictated using the smallest possible amount of material for dating, ^{14}C dating by accelerator mass spectrometry (AMS) was the only option.

AMS measurements were performed independently at both the Zurich and Oxford AMS facilities (Bonani *et al.* 1992a). We report here in greater detail the specific procedures we used to prepare the Ice Man tissue and bone samples, as well as the small pieces of grass that were found in the bottle with the ice man tissue, and we supply the previously unreported grass age.

METHODS

We received samples from the Ice Man, stored in a glass jar, in November 1991. The sample material was first checked for contaminants by examination under a binocular microscope. At this time, the entrained pieces of grass were noted and separated from the skin and muscle tissue. For the Ice Man tissue sample, two pieces of what appeared to be skin were chosen from the bulk sample material. Because this material is similar to animal-skin parchment, which can easily dissolve (especially at high pH), we had to attenuate the normal acid-base-acid (ABA) treatment used to clean samples (*cf.* Bonani *et al.* 1992b). We reduced both the time for each step and the strength of the solutions with respect to those we usually use.

The samples were weighed, put into glass beakers with distilled water, and placed in an ultrasonic bath for 15 min. As a control, sample 8345.1a was left overnight in a 60°C oven to dry, without further treatment. The other samples were first rinsed and submerged in 0.2 M HCl (*ca.* 50% the strength we usually use) for 15–60 min, depending on the appearance of the material during treatment. After rinsing 3 times with distilled water, they were left for 15 min in water, which was then checked for pH=7. We used 0.05 M NaOH for the next pretreatment step, followed again by rinsing. The first acid step should remove carbonate contamination, whereas the base step leaches soluble humic substances. A final acid rinse ensures that no atmospheric CO_2 remains. The final soaking in distilled water and check of the pH preceded oven-drying the sample at 60°C. Weight loss due to pretreatment was often >50%. The grass sample was treated both ultrasonically and by the ABA

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procedure. Bone collagen was extracted from the bone samples by hydrolyzing the bone apatite with HCl (2 M). The remaining bone collagen was neutralized with water, oven-dried and then treated the same as the other samples.

After weighing, the samples were placed in pre-cooked (950°C) high-purity SiO_2 tubes with wire-form CuO and silver wire. The tubes were evacuated and sealed, then placed in a muffle furnace at 950°C where the sample material was oxidized to CO_2. The resulting CO_2 was reduced to graphite on cobalt in the presence of hydrogen (Vogel *et al.* 1984; Vogel, Southon and Nelson 1987) in a steel extraction line. For each sample, a volume equivalent to *ca.* 2 mg of carbon was reduced to graphite. The graphite-cobalt mixture from each sample was pressed onto disk-shaped copper targets for introduction to the ion source.

The graphite targets made from the tissue, bone collagen and grass samples were measured together with appropriate standards made from Oxalic Acid I and ANU Sucrose, and with blanks made from geological graphite and coal. $^{14}C/^{12}C$ and $^{13}C/^{12}C$ isotopic ratios for each target are measured quasi-simultaneously (Bonani *et al.* 1987). Details of the ^{14}C measurement procedures at the ETH/PSI accelerator facility can be found in Suter *et al.* (1984) and Wölfli (1987).

Results and discussion

Table 1 lists the conventional ^{14}C ages for the tissue, bone and grass samples. Each measurement was calculated using the procedures suggested by Stuiver and Polach (1977) with normalization to $\delta^{13}C = -25‰$, and are reported accordingly in years BP (years before 1950). The errors listed, which are at the 1 σ level, include the statistical (counting) error, the scatter of the standards and blanks, and the uncertainty in the $\delta^{13}C$ determination.

TABLE 1. Sample Information, Conventional ^{14}C Ages and $\delta^{13}C$ Values

Lab no.	Sample material	^{14}C age (yr BP)	$\delta^{13}C$ (‰)	Sample size (mg)
ETH-8345.1a	Tissue	4605 ± 65	−23.6 ± 1.0	16.5
ETH-8345.1b	Tissue	4500 ± 70	−25.6 ± 1.3	5.7
ETH-8345.1	Tissue	4585 ± 70	−22.9 ± 1.2	10.2
ETH-8345.2	Tissue	4515 ± 70	−23.9 ± 1.6	7.7
ETH-8342	Bone	4560 ± 65	−27.9 ± 1.0	13.5
ETH-8345.3	Grass	4535 ± 60	−25.4 ± 0.9	1.5

From the data in Table 1, we determined weighted averages (Table 2) for the measurements of the four tissue samples and for all of the measurements.

TABLE 2. Weighted Averages Calculated for the Conventional ^{14}C Ages

Lab no.	Sample(s) treated	^{14}C age (yr BP)	χ^2
ETH-8345.1–2	Tissue	4555 ± 34	0.57
ETH-8342, -8345	Tissue, bone, grass	4550 ± 27	0.36

Within the 1 σ error, no age differences could be discerned among grass, tissue and bone samples. Similarly, we observed no differences in ages between subsamples that received different cleaning methods. Specifically, from a single piece of tissue, one subsample was cleaned only ultrasonically (ETH-8345.1a), whereas two others were given complete precleaning treatment (ETH-8345.1 and -8345.1b). The second piece of tissue was treated with both ultrasonic and chemical procedures

(ETH-8345.2). The χ^2 values (Table 2) show that the weighted average can be used to determine the age of the Ice Man.

Figure 1 shows the results of calibrating (Niklaus *et al.* 1992) the conventional ^{14}C age for the Ice Man to the calendar age. The upper half of the figure shows the non-linear relation between the conventional ^{14}C age and the calendar age, which is based on high-precision ^{14}C measurements on tree rings (Pearson and Stuiver 1993; Stuiver and Pearson 1993). The three horizontal lines indicate the measured conventional ^{14}C age with a ± 1 σ error band. The histogram in the lower part of the figure illustrates the probability density function for the calibrated age ranges for the Ice Man tissue samples (in 10-yr intervals). The black-filled regions indicate the 1 σ area, which corresponds to the

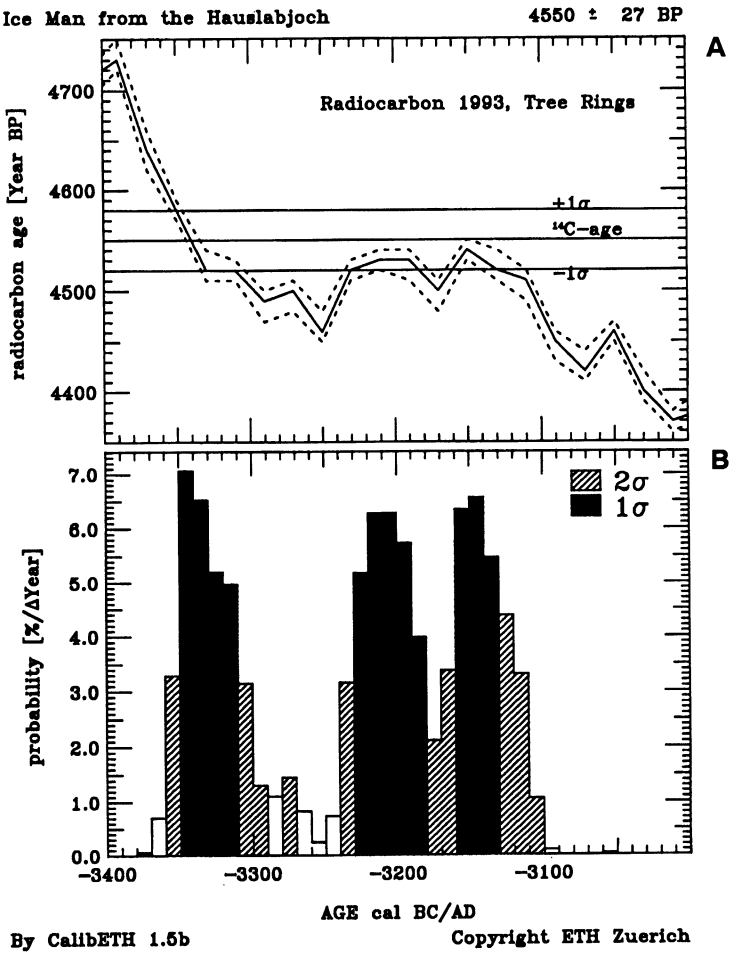

Fig. 1. A. The non-linear relation between the conventional ^{14}C age and the calendar age for the Ice Man. The three horizontal lines mark the conventional ^{14}C age with the corresponding 1 σ standard deviation (4550 ± 27 BP). - - - - = the ±1 σ error band of the calibration curve. B. The probability density function resulting from the calibration of the conventional ^{14}C age. Probability density is displayed as a histogram with a bar width of 10 yr. ■ = 1 σ area (68% probability that the true age lies within this region); ▨ = 2 σ region (95% probability). □ = area within which there is a 5% probability that the true age lies.

https://doi.org/10.1017/S0033822200040534 Published online by Cambridge University Press

intervals within which the calendar age lies in a 68% probability. Doubling the error from 1–2 σ extends the intervals of probable calendar ages, so that with a 95% probability, the actual age of the Ice Man lies somewhere within the solid black and hatched areas.

Because of the trend of the calibration curve (Fig. 1), calibrating the conventional ^{14}C age 4550 ± 27 BP, yields the following ranges and percentages of total area within the 2 σ confidence level:

3359–3294 BC	33%
3277–3268 BC	1%
3239–3105 BC	66%.

Thus, with 66% probability, the Ice Man died between 3239 and 3105 BC. A 33% probability exists that he died between 3359 and 3294 BC, and a 1% chance that he died between 3277 and 3268 BC. Thus, the Ice Man lived during the Late Neolithic, between 3359 and 3105 BC.

ACKNOWLEDGMENTS

We thank Nora Steiner for help in sample preparation, and our tandem crew. This project was partially supported by the Swiss National Science Foundation.

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