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Radiocarbon

An International Journal of Cosmogenic Isotope Research

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A J T JULL

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J WARREN BECK
GEORGE BURR

Managing Editor
DAVID R SEWELL

Associate Managing Editor
KIMBERLEY TANNER ELLIOTT

Department of Geosciences
The University of Arizona
717 East Ft. Lowell Road
ucson, Arizona 85712-1201 USA

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FROM THE EDITOR

As I write my first comments here as editor of *RADIOCARBON*, I am reminded that this journal is an important part of the culture of the scientists who consider themselves "radiocarbon-dating specialists" and "cosmogenic isotope experts", as well as those who use these measurements to interpret their own studies, whether in archaeological, geological or other sciences.

The last few years have seen major changes in the personnel at the journal, the most recent being the departure of David Sewell as managing editor. David has moved to the University of Virginia, where he has accepted an editorial position. Hence, the editor finds himself in the midst of further changes. I am lucky to have found that my two colleagues, George Burr and Warren Beck, were agreeable to take on the new roles of "associate editors". We are also happy to announce that Kim Elliott will take over as managing editor. We have also made some new appointments to our editorial board to better reflect a balance of conventional radiocarbon, AMS, and new applications amongst the board. The new board will meet at the 17th International Radiocarbon Conference in Jerusalem in June 2000.

Various events have prompted us to re-evaluate the journal's financial position, and I can now report more comfortably than I could earlier this month that *RADIOCARBON*'s financial position is on the mend. With both the generous support of some of our most reliable "Friends of *RADIOCARBON*" and the support of the university, we are in a better position to face the new millennium with a viable as well as highly respected scientific journal. However, we still need the continued financial and moral support of our readers, as well as your support in keeping *RADIOCARBON* as a quality scientific journal. This can only be done by publishing some of the best aspects of our science in this valuable journal.

The past, is therefore, but prologue. What of *RADIOCARBON* in the next millennium? We find ourselves facing many issues. Some encourage electronic publication as the wave of the future. This is an avenue we are currently exploring, although we are also committed to continuing a hard-copy version of the journal. We also hope to encourage the submission of more diverse papers to *RADIOCARBON*. In particular, submissions on other cosmogenic isotopes, new applications of radiocarbon, and discussion of interesting controversies in the field are all welcome. Small date lists are not excluded at this time, but we eventually hope to divert date lists of any size into the electronic version of the journal. I have looked over recent papers that have been submitted to *RADIOCARBON*, and I am delighted to tell you that we have some fascinating papers to appear in forthcoming issues.

The year 2000 will also bring two special issues, the first is dedicated to our dear friend and former managing editor, Renee Kra. Renee ran this journal with a particular spirit and energy from 1968 to 1996. We hope the issue will be a suitable tribute to her. The issue should be available in advance of the 2000 Radiocarbon Conference. Our second special issue, edited by Hans van der Plicht, will focus on possible new calibration records, such as varved sediments and speleothems. A third project in 2000–2001 will be the Proceedings of the 17th International Radiocarbon Conference. I look forward to meeting new and old friends alike at that meeting.

In closing, I would like to end on a personal note. For a successful journal, it is not sufficient to just stay the course. We need new ideas, suggestions, and lots of advice. In order to facilitate that, both the associate editors and I will listen to any suggestions, comments, advice, and complaints from you, the reader. Please feel free to send us your comments by e-mail, fax, or the postal service. We will try to give a thoughtful reply to all of them. Above all, please continue to consider *RADIOCARBON* as *your* place to publish original work on the applications of ^{14}C and cosmogenic isotopes, to problems of chronology, and the development of natural systems.

A J T Jull

FROM THE MANAGING EDITOR

After six years with *RADIOCARBON*, I am leaving to take an editorial position at the University Press of Virginia in Charlottesville, my wife's home town (where she has also found a good job in her field). Kimberley Elliott, who has been with *RADIOCARBON* since the week after I started, will take over as managing editor.

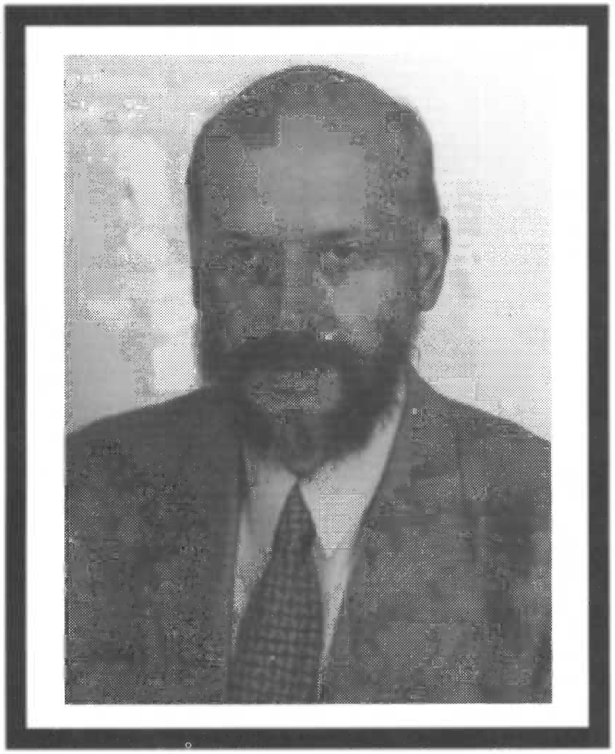
So for the second issue in a row one of our editors is saying farewell to readers, authors, and the radiocarbon community. The latter was a completely new world to me when I began: my previous editorial work and academic training had mostly been in the humanities and social sciences other than archaeology. But Renee Kra was a careful mentor, and the challenge she offered her junior editors to carry on her standards of excellence is one of her many legacies to the journal.

When I joined the staff in 1993, I had just begun to figure out what HTML was all about, and thought it might be fun to see if I could get a World Wide Web page running for *RADIOCARBON*. (In fact, we had a gopher server before we had a Web server. If you remember gopher servers, consider yourself an Internet old-timer.) I had no idea that within six years the Internet would become not only the university's but the world's dominant vehicle for communication and sales, or that we would be shipping entire issues of the journal to our printing company on a few floppy disks (in PDF format) instead of sending a large box full of camera-ready pages and photographs. But despite the computer revolution, I expect the familiar red-covered paper *RADIOCARBON* to be our medium for years to come—in fact, we've been gratified by how many orders we've had in recent months for full sets of back issues, which take up several meters of physical space on the shelf.

I leave the journal having come to understand and appreciate what I can only call the poetry of radiocarbon dating. In a famous passage of James Joyce's *Ulysses,* Stephen Daedalus walks along the seashore, noting evidence of shipwreck in bits of debris and of the moon's pull in the nearing tide, and echoes the German theosophist Jakob Boehme in musing, "Signatures of all things I am here to read." What for Boehme was a mystic exercise in seeing the pattern of the Divine running through the physical world is for the isotope researcher a literal truth. From the isotopic signatures in bones of Viking burials we infer what Eric the Red ate in the 10th century; from those in ice extracted from hundreds of meters below the Greenland ice cap we refine our estimates of climate change in the late Pleistocene. In publishing articles that report and analyze such data, *RADIOCARBON* adds to the story of where we and our planet have been, and to educated guesses about where we may be headed. (Or where our thermometers and sea levels are headed, at least.)

As anyone will understand who has visited Tucson, it is not easy to leave an office that looks north to the Santa Catalina mountains. I will miss the Sonoran Desert and the radiocarbon community alike, and look forward to occasions for revisiting both when I can.

David Sewell



EDE HERTELENDI (1950–1999)

Ede Hertelendi was born in Szeged, Hungary in 1950. He studied in his home town and graduated as a physicist in 1974. He began work at the Institute of Nuclear Research of the Hungarian Academy of Sciences in 1975, and he worked for the institution until his tragic death in 1999.

During the 1970s, research institutes in Hungary faced grave difficulties in procuring up-to-date equipment. Ede, an experimental physicist blessed with excellent technical abilities, was able to design and build his own hardware. He established the Laboratory of Environmental Studies, which to date is one of the best-equipped laboratories in our Institute. He played a decisive role in developing the only low-level β-counting facility in Hungary (see references 1–4), which could be used in archaeological, hydrological and geological research alike. He equipped this radiocarbon measuring system with his own combustion and purification system for CO_2 preparation and a converter for methane synthesis. The determination of $\delta^{13}C$ correction is carried out by an automatic, computer-controlled mass spectrometer used for measuring isotope ratios (5–6), another construction by Ede. Based on worldwide intercalibration measurements, this system has been reckoned with as one of the ten most precise measuring units in the world.

Ede applied the radiocarbon method in many varied areas of science. His interests ranged from archaeology and hydrology, all the way to the hottest issues in environmental research. He identified the absolute age of numerous archaeological samples (e.g. 7–11). His measurements were fundamental in determining the chronological sequence of Neolithic archaeological sites in Eastern Hungary (12–16). He also took part in Bronze Age research in Hungary (17–19). His measurements of ^{14}C as well as isotope analytical studies were of great help in defining the palaeoenvironments influencing the distribution of prehistoric multilayered tell settlements in the Carpathian Basin (8000–35,000 BP) (20–21). He also carried out dating projects on bogs and sediments from Lake Balaton (23). Ede's extensive ^{14}C and isotope analytical measurements contributed to the palaeoecological reconstruction of loess formation in numerous sample squares in the Great Hungarian Plain (24–28).

Ede also studied the origins of karstic water (29–30), the vulnerability of aquifers (31) as well as the application of ^{14}C and other natural isotopes in hydrological and geological research (32–37). He wrote the first description of the radiocarbon method in Hungarian (38). Moreover, he studied the effect of human activity on the concentration of ^{14}C in the atmosphere (39–44).

Ede was a renowned and widely acknowledged public personality, a member of the Committees for Radiation Protection and Environmental Physics, Radoanalytical as well as Environmental Geochemistry of the Hungarian Academy of Sciences. He also sat on the Subcommittee for Nuclear Energetics. He was a member in the governing bodies of the COST-65, COST-67 and COST-621 Actions of the European Union.

His achievements were acknowledged when he was granted the "Interdisciplinary Prize" by the Institute of Nuclear Research of the Hungarian Academy of Sciences in 1986. He also received the "Elemér Szádeczky-Kardoss Prize" from the Hungarian Academy of Sciences in 1988. In addition, he was awarded the "Institute Prize" from the Institute of Nuclear Research of the Hungarian Academy of Sciences in 1992, and the "Dénes Gábor Prize" of the NOVOFER Foundation in 1998.

Ede was not only an outstanding scholar, but excelled as a teacher as well. His university lectures, motivated by enthusiasm for his research, were extremely popular among the students. Over the years, he was chief adviser on 12 graduate theses and two currently on-going PhD dissertations. His knowledge and special skills, transferred to his students, will remain with us and his students will follow in his footsteps.

Éva Svingor

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REPORTING ^{14}C ACTIVITIES AND CONCENTRATIONS

Willem G Mook • Johannes van der Plicht

Centre for Isotope Research, Groningen University, Nijenborgh 4, 9747 AG Groningen, the Netherlands

ABSTRACT. Three modes of reporting ^{14}C activities are in use, in part analogous to the internationally accepted (IAEA) conventions for stable isotopes: (1) *absolute activity,* the specific activity of ^{14}C or the activity per gram of carbon; (2) *activity ratio*, the ratio between the absolute activities of a sample and the standard; and (3) *relative activity,* the difference between the absolute activities of a sample and standard material, relative to the absolute standard activity. The basic definitions originate from decisions made by the radiocarbon community at its past conferences. Stuiver and Polach (1977) reviewed and sought to specify the definitions and conventions. Several colleagues, however, have experienced inadequacies and pitfalls in the definitions and use of symbols. Furthermore, the latter have to be slightly amended because of the use of modern measuring techniques.

This paper is intended to provide a consistent set of reporting symbols and definitions, illustrated by some practical examples.

1. INTRODUCTION

This paper is meant to update the conventions and symbols recommended for the reporting of radiocarbon data in Stuiver and Polach (1977), henceforth abbreviated as S&P77.

The basic definitions are straightforward and will be repeated here. However, the use of symbols will be made more transparent by limiting their number and clarifying their meaning with the addition of a limited number of super- and subscripts.

The *absolute (specific) ^{14}C activity*, that is, the ^{14}C radioactivity (in Bq or, conventionally, in disintegrations per minute [dpm] per gram of carbon) is given the symbol

$$^{14}\mathrm{A}\ \text{(in dpm/gC)}\ . \tag{1}$$

The vast majority of ^{14}C laboratories are unable to make such absolute measurements: the measuring efficiency is unknown. Also, in general, the absolute ^{14}C content of a sample is not relevant. Therefore, the sample activities are compared with the activity of a reference material, the international standard. In practice, the number of ^{14}C registrations (= β counts from ^{14}C decay in radiometric detectors such as proportional counters and liquid scintillation counters; registrations of ^{14}C concentration in AMS systems) is related to the number of registrations from the reference sample under equal conditions. This results in the introduction of a *^{14}C activity ratio* or *^{14}C concentration ratio:*

$$\begin{aligned} ^{14}\mathrm{a} &= \frac{\text{measuring efficiency} \times {}^{14}\mathrm{A}_{\text{sample}}}{\text{measuring efficiency} \times {}^{14}\mathrm{A}_{\text{reference}}} = \frac{^{14}\mathrm{A}_{\text{sample}}}{^{14}\mathrm{A}_{\text{reference}}} \\ &= \frac{^{14}\text{C decay rate in the sample}}{^{14}\text{C decay rate in the ref. material}} = \frac{^{14}\text{C concentration in the sample}}{^{14}\text{C concentration in the ref. material}}\ . \end{aligned} \tag{2}$$

Because in the numerator and denominator of the last two fractions, the detection efficiencies are equal for sample and standard, **the use of the ratio ^{14}a is adequate for any type of measuring technique.** Henceforth, **we will use the symbol ^{14}A for the *^{14}C content*** (radioactivity as well as concentration) **of a sample,** whether the analytical technique applied is radiometric or mass spectrometric (AMS).

We have chosen to have symbols refer to ^{14}C by prefixing the superscript "14", as in ^{14}A, ^{14}a, and $^{14}\delta$ (likewise $^{13}\delta$), because it leaves space for other superscripts and it makes the symbols less complicated; compare, for instance, $^{14}\delta^0$ with $\delta^{14}C^0$. Moreover, these symbols are unambiguous.

Under natural circumstances the values of ^{14}a are between 0 and 1. In order to avoid a large number of decimals, it is general practice to report these values in percent (%). However, it should be noted that this practice does not change the fact that ^{14}a is a number generally between 0 and 1. The consequence is that the factor 10^2 should *not* enter into equations (as $^{14}a/10^2$).

In some cases the differences in ^{14}C content between samples are small. Therefore, the use of relative abundances has been adopted from the stable-isotope field, in this case the *relative ^{14}C content (activity or concentration),* $^{14}\delta$, defined as the difference between sample and standard ^{14}C content as a fraction of the standard value:

$$^{14}\delta = \frac{^{14}A - {}^{14}A_{Ref}}{^{14}A_{Ref}} = \frac{^{14}A}{^{14}A_{Ref}} - 1 = {}^{14}a - 1 \quad . \tag{3}$$

The values of δ are small numbers and therefore generally given in per mill (‰). However, we want to stress that ‰ is equivalent to the factor 10^{-3}; δ is thus dimensionless and not a unit. As with ^{14}a defined above, there is no need (it is actually incorrect) to add 10^{-3} to δ in mathematical equations.

A ^{14}C *reference material* or *standard* was chosen to represent as closely as possible the ^{14}C content of carbon in naturally growing plants. The ^{14}C content of the standard material itself does not need to be, in fact is not, equal to the *standard ^{14}C content*. The definition of the standard ^{14}C activity is based on 95% of the specific activity of the original NBS oxalic acid (Ox1) in the year AD 1950 (Karlén et al. 1966), as will be discussed in more detail later.

Before the definition of standards can be completed, two factors have to be discussed that complicate the standardization of ^{14}C results and the respective symbols.

1.1. The Question of Isotope Fractionation

During the transition of carbon from one compound to another—for example, the assimilation of CO_2 by plants or the exchange of CO_2 between the air and surface water—isotope fractionation occurs for ^{13}C as well as ^{14}C. The laboratory treatment of sample materials may also introduce an isotopic change, for instance by an incomplete chemical reaction. If this fractionation were neglected, samples of different chemical composition (carbonate, plants) but made of carbon of the same age (determined by atmospheric CO_2) would seem to have different ages.

Therefore, in order to make ^{14}C ages comparable, a correction has to be applied for this fractionation effect. The theoretical relation between ^{14}C and ^{13}C fractionations is written as

$$\frac{^{14}A_N}{^{14}A} = \left(\frac{^{13}R_N}{^{13}R}\right)^{\theta} \quad , \tag{4}$$

where ^{14}A and ^{13}R refer to the measured activity, $^{14}A_N$ to the fractionation-corrected or *normalized* ^{14}C activity, and $^{13}R_N$ (or rather $^{13}\delta_N$) is an internationally adopted standard value. This value is $^{13}\delta_N$

= −25‰ with respect to VPDB (the PDB standard is discontinued and replaced by Vienna PDB with NBS19 as the calibration standard for $^{13}\delta$ and $^{18}\delta$; Gonfiantini 1984), being a representative average of the majority of organic samples in nature. Also the standard activity has to be normalized in the same way. The only exception is that, for historical reasons, the old Oxalic Acid standard (Ox1) is normalized to its own $^{13}\delta$ value of −19‰.

For natural processes the value of θ is approximately 2 (Craig 1954). Since we have no reliable experimental evidence for the true value of θ (in cases where the relation between the fractionations could be measured [Meijer and Li 1998] or calculated [Mook 2000], the θ value is rather about 1.9), and because this uncertainty is irrelevant in light of the analytical precision of ^{14}C measurements, we will use $\theta = 2$ as a sufficient approximation.

1.2. The Question of Radioactive Decay

For radioactive samples a measured activity depends on the time of measurement, t_m:

$$^{14}A(t_m) = {}^{14}A(t_0)e^{-\lambda\Delta t} \qquad \text{or} \qquad {}^{14}A = {}^{14}A^0 e^{\lambda(t_m - t_0)} \quad , \tag{5}$$

which is also valid for the standard material. Therefore, when reporting an absolute ^{14}C content, the year for which the value is valid must be specified. The same is true for the standard. The year of reference was chosen to be AD 1950; the superscript ^{0} refers to this "year 0". The standard activity thus is valid for the year 1950. As the ^{14}C content of samples reduces in time simultaneously with the standard material, any comparison between the two results in a ^{14}C content valid for the year 1950 (= t_0).

The consequence is that an absolute activity, resulting from a ^{14}C analysis that is based on a comparison with the Oxalic Acid standard, is valid for the year 1950, irrespective of the time of measurement:

$$^{14}a \times 0.226 = {}^{14}A^0$$

The original value of the decay constant λ is 1/8033 a^{-1} (Libby 1952), based on a half-life of 5568 a; $T_{1/2}$ of ^{14}C was later corrected to 5730 a (Godwin 1962), resulting in a decay constant of 1/8267 a^{-1} (see our later discussion with the examples in section 3).

1.3. Definition of the ^{14}C Standard Activity

We can now define the *standard activity* as 95% of the activity of the specific batch of Oxalic Acid nr 1 in AD 1950 (in S&P77 defined as A_{ABS}):

$$^{14}A^0_{RN} = 0.95 \; {}^{14}A^0_{Ox1N} = 13.56 \pm 0.07 \; \text{dpm/gC} = 0.226 \pm 0.001 \; \text{Bq/gC} \quad , \tag{6}$$

where R stands for "Reference", N for "Normalized" for isotope fractionation (to $^{13}\delta$ = −25‰, except in the case of Ox1 to $^{13}\delta$ = −19‰) and dpm/gC means disintegrations per minute per gram of carbon, while the superscript ^{0} refers to the fact that the definition is valid for the year 1950 only.

The definition presented by Equation 6 is related to time by

$$^{14}A_{RN} = {}^{14}A^0_{RN} \; e^{-\lambda(t_i - t_0)} = 0.95 \; {}^{14}A^0_{Ox1N} e^{-\lambda(t_i - t_0)} = 0.95 \; {}^{14}A_{Ox1N} \quad , \tag{7}$$

where t_0 and t_i refer to the year 1950 and to the moment of the origin of the sample, respectively; in other words, $t_i - t_0$ is the true age of the sample. Here S&P77 use the symbol A_{ON}.

Because the original supply of oxalic acid has been exhausted, a new batch of oxalic acid (Ox2) is available for distribution by the NIST (formerly US-NBS).

Through careful measurement by a number of laboratories (Mann 1983), the ^{14}C activity was related to that of the original Ox1 by

$$^{14}A^0_{\mathrm{Ox2N}} = (1.2736 \pm 0.0004)\ ^{14}A^0_{\mathrm{Ox1N}} \quad . \tag{8}$$

Contrary to the Old Oxalic acid (with a true $^{13}\delta$ value of −19.2 ‰, H Craig, personal communication; Mann 1983), the New Oxalic acid (with $^{13}\delta_{\mathrm{Ox2}} = -17.6$‰) is to be normalized (= corrected for isotope fractionation) to $^{13}\delta = -25$‰, while both activities refer to AD 1950.

All $^{13}\delta$ values are with respect to the VPDB standard (Gonfiantini 1984).

Consequently the standard activity is

$$^{14}A^0_{\mathrm{RN}} = \frac{0.95}{1.2736} = 0.7459\ ^{14}A^0_{\mathrm{Ox2N}} \quad \text{or} \quad ^{14}A_{\mathrm{RN}} = 0.7459\ ^{14}A_{\mathrm{Ox2N}} \quad , \tag{9}$$

where the $^{14}A^0$ values for Ox1 and Ox2 refer to the activity of the material in 1950, irrespective of the time of measurement.

2. FINAL DEFINITIONS

We can now more carefully specify the ^{14}C content of a sample in terms of the *activity ratio* or *concentration ratio* as the ratio between the measured ^{14}C content and the value of the standard (which always has to be normalized for $^{13}\delta$), with both terms referring to the time of measurement, as mentioned in Equation 2:

$$^{14}a = \frac{^{14}A}{^{14}A_{\mathrm{RN}}} = \frac{^{14}A^0 e^{-\lambda(t_m - t_0)}}{^{14}A^0_{\mathrm{RN}} e^{-\lambda(t_m - t_0)}} = \frac{^{14}A^0}{^{14}A^0_{\mathrm{RN}}} = {}^{14}a^0 \quad , \tag{10}$$

where t_0 refers to the year 1950, ^{14}A is the activity of the sample measured at time t_m, and $^{14}A_{\mathrm{RN}}$ (= $0.95\,^{14}A_{\mathrm{Ox1}}$ with $^{13}\delta_N = -19$‰ or $0.746\,^{14}A_{\mathrm{Ox2}}$ with $^{13}\delta_N = -25$‰) is the value of the standard determined with the same detection efficiency, at about the same time t_m and corrected for isotope fractionation. In this way ^{14}a results from different laboratories become comparable. The decay of sample and standard ^{14}C content from t_0 to t_m is described by Equation 5. S&P77 do not use a specific symbol for ^{14}a and refer to this as A_S/A_{ON}.

Again it should be emphasized that the consequence of the relative measurement is that the resulting value of ^{14}a is independent of the year of measurement (see §1.2).

As was anticipated above, certain studies of natural systems are concerned with only small differences in ^{14}C content. In those cases it is conventional to report ^{14}C data as the relative difference between the measured sample activity and that of the reference:

$$^{14}\delta = \frac{^{14}A}{^{14}A_R} - 1 = \frac{^{14}A^0}{^{14}A^0_R} - 1 = {}^{14}a - 1 \quad . \tag{11}$$

Here, as usual, the δ value is a small number and is therefore reported in ‰ ($\equiv 10^{-3}$). (See the related remarks following Equation 3.)

The $^{14}\delta$ values are quoted by S&P77 as $d^{14}C$.

As mentioned, it has become common practice to normalize ^{14}C results for deviations of the measured $^{13}\delta$ from −25‰. Now the discussion of the normalization procedure can be completed. Since conventionally $^{13}C/^{12}C$ values are related to the international standard for stable isotopes, Vienna PDB, Equation 4 can be rewritten as

$$\frac{^{14}A_N\,/\,^{14}A_{RN}}{^{14}A\,/\,^{14}A_{RN}} = \left[\frac{^{13}R_N\,/\,^{13}R_{VPDB}}{^{13}R\,/\,^{13}R_{VPDB}}\right]^2 ,$$

or using Equation 10, with $^{13}\delta_N = -25‰$, and consequently $1 + {}^{13}\delta_N = 0.975$:

$$^{14}a_N = {}^{14}a\left[\frac{1+{}^{13}\delta_N}{1+{}^{13}\delta}\right]^2 = {}^{14}a\left[\frac{0.975}{1+{}^{13}\delta}\right]^2 , \tag{12}$$

(in S&P77 referred to as A_{SN}/A_{ON}), and likewise

$$^{14}A_N = {}^{14}A\left[\frac{0.975}{1+{}^{13}\delta}\right]^2 \tag{13}$$

(in S&P77 referred to as A_{SN}), while Equation 11 transforms into

$$^{14}\delta_N = {}^{14}a_N - 1 , \tag{14}$$

in S&P77 indicated by $D^{14}C$.

3. EXAMPLES

In general, the presentation of ^{14}C results depends on the type of application. After each formal treatment we will present some realistic examples to illustrate the applicable definitions and equations.

3.1. Enhanced ^{14}C Radioactivity (nuclear industry, biomedical research)

In studies on the extent of radioactive contamination, for instance by ^{14}C, the absolute radioactivity of the sample is required. Consequently the result of the routine ^{14}C measurement, in this case the ^{14}a value, has to be converted back to the absolute value by

$$^{14}A = {}^{14}a \times 13.56\ (\text{dpm/gC}) = {}^{14}a \times 0.226\ (\text{Bq/gC}) . \tag{15}$$

The absolute activity obtained by multiplying ^{14}a by 0.226 is valid for the year 1950 (see Equation 10). In fact, it has to be corrected for radioactive decay of the standard from 1950 (t_0) to the year of sampling (t_s). Moreover, the activity in this case is not to be normalized. If the laboratory provides only normalized values, $^{14}a_N$, these have to be "de-normalized" as shown in Equation 16.

We can now meet our commitment to give the equation for the radioactivity at the time of sampling ($^{14}A^S$) and for de-normalization by applying Equations 6, 10, 12 and 15:

$$
\begin{aligned}
{}^{14}A^{S} &= {}^{14}a_{N}^{0}\left(\frac{1+{}^{13}\delta}{0.975}\right)^{2} e^{-\lambda(t_s-t_0)}\ {}^{14}A_{RN}^{0} \\
&= {}^{14}a_{N}\left(\frac{1+{}^{13}\delta}{0.975}\right)^{2} e^{-(t_s-1950)/8267}\times 0.226\,(\mathrm{Bq/gC})\ ,
\end{aligned}
\tag{16}
$$

where t_0 and ${}^{14}a^0$ both refer to AD 1950, and t_s and A^S to the year of sampling; λ is based on the true ${}^{14}C$ half-life of 5730 yr; ${}^{14}a_N$ is the routinely acquired (normalized) ${}^{14}C$ content of the sample.

Example: Insect from Chernobyl

The AMS-measured ${}^{14}C$ concentration ratio is

$$ {}^{14}a_N = 1.7172 \text{ or } = 171.72\ \% \ . $$

The ${}^{13}\delta$ analysis obtained in the AMS system resulted in

$$ {}^{13}\delta = -34.8\ ‰ \ . $$

This unusual value is due to the fact that the original sample—because of its expectedly high ${}^{14}C$ content—was not processed in the routine combustion system, instead being carbonized to provide an amorphous carbon sample. In the accelerator this behaves differently from the routine samples, resulting in a relatively weak ${}^{13}C$ ion beam.

The ${}^{14}a_N$ value routinely obtained now has to be de-normalized according to

$$ {}^{14}a = {}^{14}a_N\,[(1 + {}^{13}\delta)/0.975]^2 = 1.6829 \text{ or } = 168.29\ \% \ . \qquad \text{[cf. Eq 12]} $$

The absolute activity in the year 1950 would have been

$$ {}^{14}A^0 = {}^{14}a^0 \times 0.226 = {}^{14}a \times 0.226 = 0.380 \text{ Bq/gC} \ . \qquad \text{[cf. Eq 15]} $$

This value has to be corrected for radioactive decay of the standard to the year of sampling $t_s = 1986$:

$$ {}^{14}A^S = {}^{14}A^0 \exp[-(1986 - 1950)/8267] = 0.379 \text{ Bq/gC} \ . \qquad \text{[cf. Eq 16]} $$

3.2. Hydrology

From a geochemical point of view, the use of ${}^{14}a^S$, the non-normalized ${}^{14}C$ content at the time of sampling, is more meaningful than a δ value. Instead of applying a normalization correction, the initial ${}^{14}C$ content of groundwater is approximated based on specific geochemical inference of the origin of the inorganic carbon content. Furthermore, if we are dealing with groundwater ages, it is irrelevant—from a hydrological point of view—whether ages count back in time from the year of sampling (calculation based on ${}^{14}a^S$) or from 1950 (calculation based on ${}^{14}a = {}^{14}a^0$). Moreover, the precision of routine ${}^{14}C$ dating is ±50 yr or more in any case. Consequently, we can equally well use the simplest ${}^{14}a$ value in % as is obtained in basic laboratory procedures.

Without ${}^{13}C$ normalization, the activity ratio in the year of sampling is

$$ {}^{14}a^{S} = {}^{14}a_{N}\left(\frac{1+{}^{13}\delta}{0.975}\right)^{2} e^{-(t_S-1950)/8267} \ . \tag{17} $$

in S&P77 indicated by A_S/A_{ABS}.

Often these values are given in percent of modern carbon (pMC or pmc) or percent modern (pM). In addition, pM/100 (= $^{14}a^s$) is sometimes called "fraction modern". However, the symbol pM is used by water chemists and oceanographers to mean picoMole. Therefore, pmc, pMC, pM, and similar variants should not be used: **% is adequate in combination with a well-defined symbol.**

Example: Groundwater

The measured and normalized activity or concentration ratio is

$$^{14}a_N = 0.537 = 53.7\ \% \ .$$

The measured $^{13}\delta$ value of the total carbon content (as obtained by the extraction procedure) is

$$^{13}\delta = -13.82\ ‰ \ .$$

In groundwater hydrology we are interested in the ^{14}C content of the water sample at the time of sample collection. Therefore, the ^{14}a value has to be de-normalized:

$$^{14}a = {}^{14}a_N\ [(1 + {}^{13}\delta) / 0.975]^2 = 0.549 = 54.9\ \% \ . \qquad \text{[cf. Eq 12]}$$

The ^{14}C content in the year of sampling (1998) then is

$$^{14}a^S = {}^{14}a_N\ [(1 + {}^{13}\delta) / 0.975]^2\ \exp[-(1998 - 1950) / 8267] = 0.546 = 54.6\ \%$$
$$(\equiv 54.6 \text{ pmc (or pMC)} = \% \text{ of modern carbon}) \ . \qquad \text{[cf. Eq 17]}$$

Using more or less sophisticated models, the ^{14}C and the ^{13}C data, together with information on the chemical composition of the water sample, can be used to estimate the sample age (i.e. the period of time since the infiltration of the water). A straightforward "water age" as obtained by simply applying Equation 24 is not possible.

3.3. Oceanography and Atmospheric Research

The same equation holds for the oceanographic applications. However, as the spread of the data generally is quite small, it is common practice to report the ^{14}C data as relative numbers, in other words, as decay-corrected $^{14}\delta$ values:

$$^{14}\delta^S = {}^{14}a^S - 1 = \frac{^{14}A}{^{14}A^0_{RN}} - 1 \ . \qquad 18$$

In general, results are also corrected for isotope fractionation (= normalized):

$$^{14}\delta^S_N = {}^{14}a^S_N - 1 = {}^{14}a \cdot e^{-(t_s - t_0)/8267} \left(\frac{0.975}{1 + {}^{13}\delta}\right)^2 - 1 \ . \qquad (19)$$

This symbol is equivalent to $\Delta^{14}C$ as defined by S&P77.

These equations are used to express the ^{14}C content of samples of ocean water as well as of atmospheric CO_2.

Again we must stress that a δ value is generally written in terms of ‰ and is therefore a small number. In equations it should thus **not** be written as $^{14}\delta/10^3$.

Example: Oceanic DIC and Atmospheric CO_2

The ^{14}C content resulting from a routine measurement includes normalization to $^{13}\delta = -25‰$, as follows.

1. Deep-ocean bottom water DIC (=dissolved inorganic carbon)

$$\text{with } {}^{14}a_N = {}^{14}a_N{}^0 = 0.872 = 87.2\% \ . \qquad \text{[cf. Eq 10]}$$

$$\text{and: } {}^{14}\delta_N = {}^{14}a_N - 1 = -0.128 = -128‰ \ , \qquad \text{[cf. Eq 14]}$$

$$\text{with } {}^{13}\delta = +1.55\ ‰ \ .$$

When corrected for the fact that the resulting ^{14}C content is valid for the year 1950 instead of the year of sampling (1990):

$$\begin{aligned} {}^{14}\delta_N^S &= {}^{14}a_N \ \ \exp[-(1990-1950)/8267]-1 = -0.132 = -132\% \\ &\equiv \Delta^{14}\text{C} \quad (\text{S\&P77}) \ . \end{aligned} \qquad \text{[cf. Eq 19]}$$

2. Atmospheric CO_2

$$\text{with } {}^{14}\delta_N = {}^{14}a_N - 1 = 0.253 = +253\ ‰ \ , \qquad \text{[cf. Eq 14]}$$

$$\text{with } {}^{13}\delta = -7.96\ ‰ \ .$$

When corrected for the fact that the resulting ^{14}C content is valid for the year 1950 instead of the year of sampling (1985):

$$\begin{aligned} {}^{14}\delta_N^S &= {}^{14}a_N \ \ \exp[-(1985-1950)/8267]-1 = 0.247 = +247 \\ &\equiv \Delta^{14}\text{C} \quad (\text{S\&P77}) \ . \end{aligned} \qquad \text{[cf. Eq 19]}$$

With atmospheric CO_2 samples it remains problematic whether the ^{13}C correction applied (according to Equation 12) is correct. The measured $^{13}\delta$ value may very well be affected by the admixture of biospheric or fossil-fuel CO_2, instead of by isotope fractionation alone, as is the presumption—not necessarily valid—of the correction procedure (Mook 1980).

3.4. Geochemistry

In geochemical studies it is often necessary to know the original ^{14}C content of a sample in the year of the sample origin t_i (such as the year a tree ring was formed), instead of the activity in 1950, as is routinely obtained. As the measured ^{14}C content (activity or concentration) is valid for the year 1950, it has to be corrected for radioactive decay from the year of origin (t_i) to 1950,

$$^{14}A^i = {}^{14}A^0 \ e^{-(t_i - 1950)/8267} \ , \tag{20}$$

and equally for the normalized values:

$$^{14}A_N^i = {}^{14}A_N^0 \ e^{-(t_i - 1950)/8267} \ . \tag{21}$$

For this correction again the "correct" half-life of ^{14}C (5730 a) must be used with $\lambda = 1/8267\ a^{-1}$. Furthermore, $^{14}A^0$ and $^{14}a^0$ refer to 1950 and $^{14}A^i$ and $^{14}a^i$ to the year of the origin of the carbon containing sample material.

Finally the relations for ^{14}a and $^{14}\delta$ can be established. For the normalized and age corrected values we can now write

$$\begin{aligned} {}^{14}\delta^i &= {}^{14}a^i - 1 = \frac{{}^{14}A^i}{{}^{14}A^0_{RN}} - 1 \\ &= \frac{{}^{14}A^0\ e^{-(t_i - 1950)/8267}}{{}^{14}A^0_{RN}} - 1 = {}^{14}a \cdot e^{-(t_i - 1950)/8267} - 1 \ , \end{aligned} \tag{22}$$

equivalent to $\delta^{14}C$ in S&P77, and for the normalized values,

$$^{14}\delta^i_N = {}^{14}a^i_N - 1 = {}^{14}a \cdot e^{-(t_i - 1950)/8267} \left(\frac{0.975}{1 + {}^{13}\delta} \right)^2 - 1 \ , \tag{23}$$

equivalent to Δ in S&P77.

Example: Atmospheric ^{14}C Content from a Known-Age Sample: Wood from a Tree Ring

The measured activity ratio, that is, the activity with respect to 0.95 times the measured and normalized oxalic acid activity, is

$$^{14}a = {}^{14}a^0 = ({}^{14}A \,/\, {}^{14}A_{RN}) = 0.4235 \quad \text{or} \quad = 42.35\ \% \ . \qquad \text{[cf. Eq 10]}$$

The measured $^{13}\delta$ of the sample:

$$^{13}\delta = -22.5\ ‰ \ .$$

The normalized activity ratio of the sample is

$$^{14}a_N = {}^{14}a\ [0.975 \,/\, (1 - 0.0225)]^2 = 0.4213 \quad \text{or} \quad = 42.13\ \% \ . \qquad \text{[cf. Eq 12]}$$

Using the normalized, age-corrected ^{14}a value calculated above, the conventional ^{14}C age is

$$T = -\,8033\ \ln {}^{14}a_N = 6943\ \text{BP} \ . \qquad \text{[cf. Eq 24]}$$

The normalized relative activity is

$$^{14}\delta_N = {}^{14}a_N - 1 = -\,0.5787 \quad \text{or} \quad = -578.7\ ‰ \ . \qquad \text{[cf. Eq 14]}$$

Suppose the tree ring is dated dendrochronologically to 5735 cal BC, i.e. $t_i = -\,5735$ a.

The age-corrected, i.e. the original, ^{14}C content of the sample is then

$$\begin{aligned} {}^{14}\delta_N{}^i &= {}^{14}a_N{}^i - 1 = {}^{14}a_N \exp[-(-5735 - 1950)/8267] - 1 \\ &= 0.0674(5) \quad \text{or} \quad = +67.4(5) \qquad (\equiv \Delta^{14}C \text{ defined by S\&P77}) \ . \end{aligned} \qquad \text{[cf. Eq 23]}$$

3.5. ^{14}C Ages

In geological and archeological dating, ages are based on the normalized $^{14}a_N$ (to $^{13}\delta = -25‰$) and on a half-life of 5568 yr, and are calculated by applying

$$\text{Conventional age} = -8033 \ln {}^{14}a_N \; . \qquad (24)$$

This defines the ^{14}C time scale in years BP (Before Present, i.e. AD 1950). This time scale needs to be calibrated in order to obtain historical ages (cal AD, cal BC, cal BP). For the calibration procedures and conventions we refer to the special Calibration Issues published by *Radiocarbon* (the most recent is Stuiver and Van der Plicht 1998).

4. SUMMARY

The interrelation between the various definitions and symbols, and their validity over the time scale, is illustrated by Figure 1. In Table 1 all symbols used are classified and compared to the symbols used by S&P77.

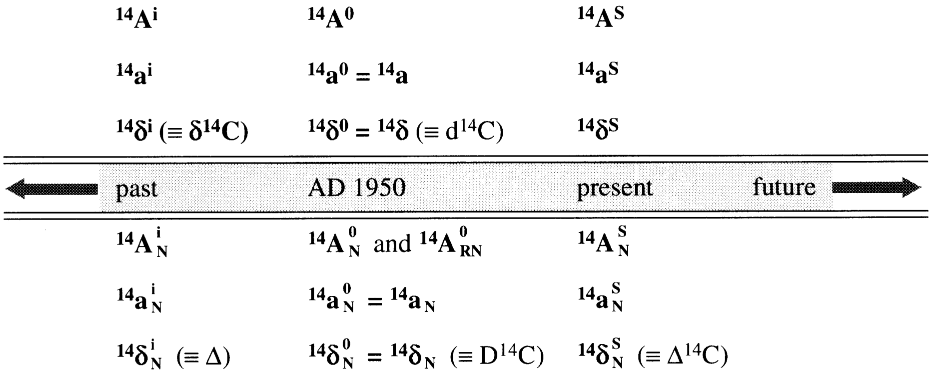

Figure 1 Illustration of the definition of symbols for reporting ^{14}C data along the time scale from the past (for the data to be corrected for age/decay), via the year 1950 to the time of sample collection(s) and the future. Above the line are the non-normalized data (not corrected for isotope fractionation); below are the the normalized values. The bold symbols are defined in this paper, the non-bold (in parentheses) were defined in S&P77.

Symbols stand for:
A = absolute activity or concentration
a = activity/concentration ratio to standard
δ = relative ^{14}C content (i.e. deviation of activity or concentration from standard)

Super- and subscripts stand for:
N = normalized
0 = time zero ≡ AD 1950
i = initial ≡ time of growth/formation
s = time of sampling

5. CONCLUDING REMARKS

In this paper we try to clarify definitions and symbols used in presenting results from ^{14}C analyses on a variety of materials, obtained by a variety of analytical instrumentation. In the past, basic agreements were reached at the International Radiocarbon Conferences, in particular concerning the use of reference materials (standards), the adoption of a standard activity, the application of the "wrong" and the "right" ^{14}C half-life, and the correction for isotope fractionation. The rapid spread of ^{14}C applications throughout a variety of disciplines has sometimes led to the introduction of personal or ad hoc definitions, resulting in considerable confusion. Because a few practical symbols were lack-

Table 1 Review of the symbols used in reporting ^{14}C activities. The upper symbol is defined in this paper. The symbols in the shaded areas refer to S&P77 (Stuiver and Polach 1977). In the 3rd column the symbols apply to the decay-corrected values, i.e. corrected for decay of the sample activity from 1950 to the year of sampling (Equations 17–18). The symbols in the 6th column refer to Equations 21–23. The superscript 0 assigns the value to the year 1950.

	Measured at t_0 (1950); a and δ at any time	Normalized	Decay-corrected to sampling at t_S	Decay-corrected and normalized	Age-corrected from origin at t_i	Age-corrected and normalized
Absolute ^{14}C content of sample	$^{14}A^0$	$^{14}A_N$	$^{14}A^S$	$^{14}A^S_N$	$^{14}A^i$	$^{14}A^i_N$
	A_S	A_{SN}				
Absolute ^{14}C content of standard	$^{14}A^0_R$	$^{14}A_{RN}$		$^{14}A^0_{RN}$		
		A_{ON}		A_{ABS}		
^{14}C abundance ratio	$^{14}a = {}^{14}a^0$	$^{14}a_N$	$^{14}a^S$	$^{14}a^S_N$	$^{14}a^i$	$^{14}a^i_N$
	A_S/A_{ON}	A_{SN}/A_{ON}	A_S/A_{ABS}	A_{SN}/A_{ABS}		
Relative ^{14}C content	$^{14}\delta = {}^{14}\delta^0$	$^{14}\delta_N$	$^{14}\delta^S$	$^{14}\delta^S_N$	$^{14}\delta^i$	$^{14}\delta^i_N$
	$d^{14}C$	$D^{14}C$	$\delta^{14}C$ (1)	$\Delta^{14}C$ (2)	$\delta^{14}C$ (3)	Δ (4)

The upper symbols in each block are defined by their sub- and superscripts. The S&P77 symbols are shown in the shaded areas. They refer to the following fields of study:

(1) in hydrology the use of the ^{14}a value (or rather $^{14}a^S$) is more common (no specific symbol proposed by S&P77)
(2) in oceanography and atmospheric studies
(3) in geochemical studies if age correction is possible
(4) idem, such as past ^{14}C variations from tree rings

ing, the strange habit arose of having physical quantities defined or recognized by the "units" that accompany numerical results. Examples of this are the introduction of pM, pmc, or pMC.

We have introduced several new "rational" symbols, based on A (radioactivity), a (activity ratio or concentration ratio), and δ (relative difference in activity or concentration), provided with a limited number of super- and subscripts. These are proposed to replace symbols such as $d^{14}C$, $D^{14}C$, $\delta^{14}C$, $\Delta^{14}C$, and Δ. For practical reasons, reference in the symbols to ^{14}C is made by prefixing the superscript "^{14}" as in "^{14}C" itself. Rather than indulging ourselves with the expectation that the ^{14}C community will now immediately adopt our propositions, we hope that this paper will at least contribute to a better understanding of the complicated matter of reporting ^{14}C results, and perhaps form the basis for a new, uniform notational system in the various disciplines that rely on ^{14}C data.

As confusion still exists concerning "relative" and "absolute" pM and pmc or pMC, we note that:

1. Their definition is not unique;
2. They interfere with the definition of pM as picoMole (= 10^{-12} mole) used in water chemistry and oceanography as the unit for tracer concentrations (in picoMole/liter);
3. Their use is not needed.

Accordingly, we recommend that the use of pM (and preferably also of pmc or pMC) be abandoned completely, and that we restrict ourselves to the use of % (equivalent to 10^{-2}, Equation 2) and ‰ (equivalent to 10^{-3}, Equation 3), in combination with the proper symbols.

Finally, we summarize the most relevant equations to clarify the systematics of our presentation.

Measurement:

$$\frac{\text{standardized net counting rate sample}}{\text{standardized net counting rate Ox1 or Ox2}} = {}^{14}a$$

	Sample	**Reference/Standard**
Absolute activity at time of measurement t_m:	^{14}A	$^{14}A_R$
Normalization for fractionation:	$^{14}A_N = {}^{14}A\left[\frac{0.975}{1+{}^{13}\delta}\right]^2$	$^{14}A_{Ox1N} = {}^{14}A_{Ox1}\left(\frac{0.981}{1+{}^{13}\delta}\right)^2$ $^{14}A_{Ox2N} = {}^{14}A_{Ox2}\left(\frac{0.975}{1+{}^{13}\delta}\right)^2$
Correction to 1950 = t_0:	$^{14}A^0 = {}^{14}Ae^{\lambda(t_m-1950)}$ $^{14}A^0_N = {}^{14}A_N e^{\lambda(t_m-1950)}$	$^{14}A^0_R = {}^{14}A_R e^{\lambda(t_m-1950)}$ $^{14}A^0_{RN} = {}^{14}A_{RN} e^{\lambda(t_m-1950)}$

Standard activity:

$$^{14}A^0_{RN} = 0.95\ {}^{14}A^0_{Ox1N} = 0.7459\ {}^{14}A^0_{Ox2N} = 13.56 \text{ dpm / gC}$$

Measurement (result independent of time of measurement):

$$\frac{\text{net standardized CR of sample}}{\text{net standardized CR of Ox1}} = {}^{14}a = {}^{14}a^0$$

automatically valid for 1950: $^{14}a \times 13.56$ dpm/gC $= {}^{14}A^0$

	Not normalized	**Normalized**
Corrected to time of sampling t_s:	$^{14}A^S = {}^{14}Ae^{-\lambda(t_s-1950)}$	$^{14}A^S_N = {}^{14}A_N e^{-\lambda(t_s-1950)}$
Corrected to time of sample origin t_i:	$^{14}A^i = {}^{14}Ae^{-\lambda(t_i-1950)}$	$^{14}A^i_N = {}^{14}A_N e^{-\lambda(t_i-1950)}$

Activity/concentration ratio:

$$^{14}a = \frac{^{14}A}{^{14}A_{RN}} = \frac{^{14}A^{0}}{^{14}A^{0}_{RN}} = {}^{14}a^{0} \qquad {}^{14}a_{N} = \frac{^{14}A_{N}}{^{14}A_{RN}} = \frac{^{14}A^{0}_{N}}{^{14}A^{0}_{RN}} = {}^{14}a^{0}_{N}$$

Corrected to t_s and t_i:

$$^{14}a^{S} = {}^{14}a \; e^{-\lambda(t_S-1950)} \qquad {}^{14}a^{S}_{N} = {}^{14}a\left(\frac{0.975}{1+{}^{13}\delta}\right)^{2} e^{-\lambda(t_S-1950)}$$

$$^{14}a^{i} = {}^{14}a \; e^{-\lambda(t_i-1950)} \qquad {}^{14}a^{i}_{N} = {}^{14}a\left(\frac{0.975}{1+{}^{13}\delta}\right)^{2} e^{-\lambda(t_i-1950)}$$

Absolute activity at time of sampling to be obtained from:

$$^{14}A^{S} = {}^{14}a^{S} \times {}^{14}A^{0}_{RN} = {}^{14}a^{S} \times 13.56 \text{ dpm/gC}$$

Relative ^{14}C Content (i.e. deviation from standard):

$$^{14}\delta = {}^{14}a - 1 \qquad {}^{14}\delta_{N} = {}^{14}a_{N} - 1$$

As measured valid for 1950:

$$^{14}\delta^{0} = {}^{14}\delta$$

Corrected to t_s and t_i:

$$^{14}\delta^{S} = {}^{14}a^{S} - 1 = {}^{14}a\, e^{-\lambda(t_S-1950)} - 1$$

$$^{14}\delta^{i} = \quad = {}^{14}a\, e^{-\lambda(t_i-1950)} - 1$$

$$^{14}\delta^{S}_{N} = \quad = {}^{14}a^{S}\, e^{-\lambda(t_S-1950)} - 1 = {}^{14}a\left(\frac{0.975}{1+{}^{13}\delta}\right)^{2} e^{-\lambda(t_S-1950)} - 1$$

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RADIOCARBON ANALYSIS OF *PINUS LAGUNAE* TREE RINGS: IMPLICATIONS FOR TROPICAL DENDROCHRONOLOGY

Franco Biondi

Scripps Institution of Oceanography, Geosciences Research Division, University of California–San Diego,
La Jolla, California 92093-0244, USA. Email: fbiondi@ucsd.edu

Julianna E Fessenden

Department of Biology, University of Utah, 257 S 1400 E, Salt Lake City, Utah 84112-0840, USA

ABSTRACT. A promising species for tropical dendrochronology is *Pinus lagunae*, a pine tree found in Baja California Sur (Mexico) around lat 23.5°N. In 1995, we sampled a total of 27 wood cores from 13 *Pinus lagunae* trees in Sierra La Victoria (23°36′N, 109°56′W), just north of Sierra La Laguna, at an elevation of 1500–1600 m. Selected trees were locally dominant, but their ring-width patterns could not be crossdated. To test the hypothesis that visible growth layers in *Pinus lagunae* are formed annually, we measured radiocarbon amounts in individual rings by means of accelerator mass spectrometry (AMS). Twenty-three ^{14}C measurements were used to trace the location of the 1963–64 "bomb spike" in 3 wood increment cores. By comparing the location of that $\Delta^{14}C$ extreme with the number of visible radial wood increments, it was possible to conclude that 2 cores had a number of locally absent rings, while the 3rd one included a few years with more than one growth layer. Therefore, ring-width patterns of sampled *Pinus lagunae* were not consistent from one tree to another, most likely because of climatic regime in combination with microsite features. While the possibility of generating *Pinus lagunae* tree-ring chronologies cannot entirely be ruled out, the development of dendrochronological proxy records of climate from coniferous species in tropical North America should focus on species and sites that experience a more pronounced seasonality.

INTRODUCTION

Current widespread concern over future changes in climate stresses the need for long-term yet annually resolved climatic histories. Tropical regions are among the areas of the globe where long instrumental records of climate are scant, even though the tropics are the epicenter of the global hydrological cycle (Berner and Berner 1987). At the Western North American interface between tropical and subtropical regions, precipitation variability is greatly dependent on tropical Pacific forcing. In the winter, El Niño conditions are associated with increased rainfall in a region stretching longitudinally from Southern California and Baja California to Western Texas and the southeastern United States (Diaz and Kiladis 1992; Stahle and Cleaveland 1993). In the summer, monsoonal activity brings heavy rainfall northward from the tropical eastern Pacific over the Gulf of California into Arizona and New Mexico, with largest monthly rainfall amounts occurring along the western slopes of the Sierra Madre Occidental (Douglas et al. 1993; Adams and Comrie 1997). Because of those two processes, the tropical portion of the North American Cordillera is characterized by a pronounced latitudinal transition in precipitation regime: going southward from the United States into Mexico, climate changes from a winter-wet summer-dry pattern in subtropical Southern California, to a winter-dry summer-wet pattern in tropical Nueva Galicia—the Mexican coastal states of Jalisco, Colima, and Nayarit (Rzedowski and McVaugh 1966; Mosino and Garcia 1974; Metcalfe 1987).

At interannual to interdecadal scales, proxy records of climate in mid-latitudes can be derived from exactly dated, annually resolved tree-ring chronologies (Fritts 1976). Teleconnections with tropical phenomena, such as ENSO, can also be used for dendroclimatic reconstruction (Diaz and Markgraf 1992; Stahle et al. 1998). To date, dendrochronological studies of tropical trees have been limited by the fact that xylem growth is either uninterrupted or that growth layers are not visually identifiable (Bormann and Berlyn 1981). Some success has been obtained using selected species, mostly from mountainous areas (D'Arrigo et al. 1994; Buckley et al. 1995; Biondi et al. 1999), but paleoclimatic information from tropical terrestrial environments is still extremely scarce, particularly in North America.

A promising species for tropical dendrochronology is *Pinus lagunae*, a pine tree found in Baja California Sur around lat 23.5°N (Perry 1991). The natural range of the species occupies an area that is intermediate, both latitudinally and climatologically, between Southern California and Nueva Galicia. Rain falls mostly from July to October, but a second wet period occurs in December–January (Díaz 1995). Preliminary research has suggested that the species can be used for dendrochronological studies (Díaz et al. 1994), even though the establishment of a reliable master tree-ring chronology has been problematic (personal communication, TH Swetnam, Laboratory of Tree-Ring Research, University of Arizona, Tucson). To test the hypothesis that visible growth layers in *Pinus lagunae* are annual rings, we applied radiocarbon dating by means of accelerator mass spectrometry (AMS ^{14}C; Bowman 1990). For this purpose, the Δ^{14}C record from 1950 to the present is remarkably consistent in the atmosphere as well as in organic matter (including terrestrial vegetation and tree rings), showing a spike in 1963–64 (the "nuclear bomb effect"), and an exponentially decreasing pattern ever since (Stuiver et al. 1981; Bowen 1990; Schell and Ziemann 1989). Our objective was to determine if such a Δ^{14}C signature existed in tree rings that should have formed in the early 1960s based on their placement along the radial sequence of wood increment layers.

MATERIALS AND METHODS

Tree-ring samples were collected in cooperation with S Díaz Castro (CIBNOR, La Paz, Baja California Sur, Mexico) and D Stahle (Tree-Ring Laboratory, University of Arkansas) in November 1995. The field site (23°36′N, 109°56′W) is in Sierra La Victoria, just north of Sierra La Laguna, at an elevation of 1500–1600 m. A total of 27 wood cores from 13 trees were obtained using 40–50 cm long increment borers with a 4.3–5.2 mm internal diameter. All samples were taken from the main stem, about 1 m above the ground. Selected trees were locally dominant, with heights between 10 and 15 m, and diameters between 30 and 50 cm at coring height. The outside of several trees had been scarred either by low-intensity fire or by local people for extracting resin. In the laboratory, cores were vertically aligned, then glued to grooved wooden mounts. Cores were surfaced using a belt sander and progressively finer sandpaper, until tracheid walls were clearly visible under a stereo-zoom binocular microscope. A large number (>200) of growth layers could be counted on a single core, but no visual or numerical crossdating of tree-ring features could be established among samples. Tree rings, albeit visible, often have faint boundaries (Figure 1), and their width patterns do not match from one core to another, even when cores are from the same tree.

Three cores were selected for ^{14}C analysis (Figure 2). Each core was taken from a different tree. Two cores (SLV02A and SLV06B) belonged to young-looking trees on gentle terrain, while the 3rd core (VIC05B) belonged to an old-looking tree from a steep ravine where pines showed signs of stunted growth. Tentative dates were assigned to growth layers by assuming that the outermost ring (next to the bark) was formed in 1995, and that visible rings were annual. We manually picked 3 or 4 rings next to the estimated location of the mid 1960s, plus one more ring about 10 to 15 rings on either side of the main group (Figure 2). Leavitt and Danzer's (1993) method was used for homogenizing the wood and extracting the holocellulose. The holocellulose was then mixed with CuO in a 10:1 ratio by mass, and combusted in Vycor® tubing at 850 °C for 3–4 h. The CO_2 produced was cryogenically isolated, sealed in Pyrex® tubing, and shipped to the National Ocean Sciences AMS Facility at Woods Hole Oceanographic Institution for ^{14}C analyses. Given the small size of visible growth layers in *P. lagunae* wood samples, the AMS process was preferred to the conventional β-counting method. CO_2 was reacted with a catalyst to form graphite, which was then pressed into targets and analyzed on the accelerator along with standards and process blanks.

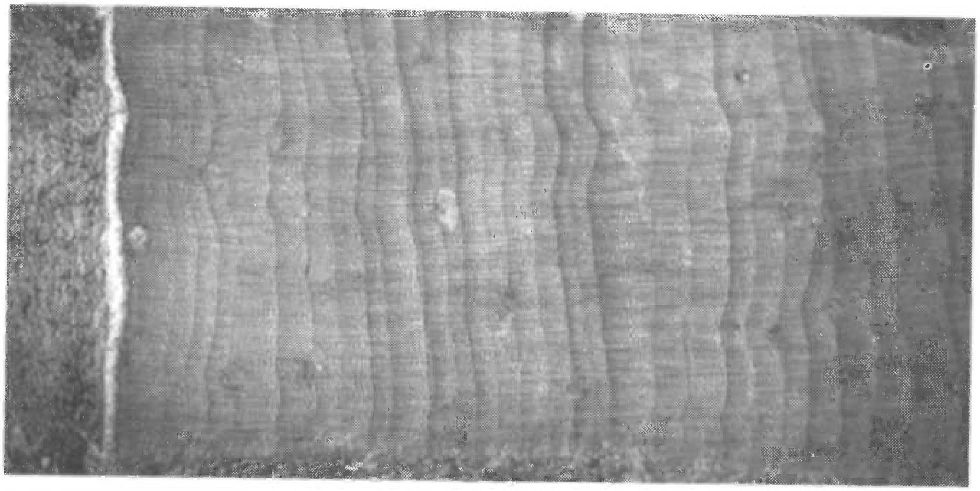

Figure 1 Growth layers of *Pinus lagunae* as seen on the monitor screen of our image analysis system. Outside bark is to the left.

RESULTS

Radiocarbon analyses are summarized in Table 1 and plotted in Figure 3. Initially, measured $\Delta^{14}C$ was compared with its expected values from 1950 to 1992 (Stuiver and Polach 1977; personal communication, JR Southon, Lawrence Livermore National Laboratory, Center for AMS). It was evident that $\Delta^{14}C$ measurements were not consistent with the preliminary dates assigned to the growth rings (Figure 3). More specifically, $\Delta^{14}C$ values of SLV06B and VIC05B peak about 10 yr later than expected. Therefore, the dates assigned to sampled rings must be shifted at least 10 yr back in time to match expected $\Delta^{14}C$ values. This shows that 10 or more extra rings had to be present besides those that were counted backwards from the bark on cores SLV06B and VIC05B, and the absence of those rings indicates that tree growth was severely limited by unfavorable conditions. Conversely, $\Delta^{14}C$ values of SLV02A followed the reference curve more closely, but peaked a few years earlier than expected (Figure 3). Therefore, the dates assigned to sampled rings need to be shifted a few years forward in time. This demonstrates that a few extra rings were counted going from the bark toward the pith on core SLV02A, and the presence of those additional growth layers reveals that more than one growing season may occur within the same year.

To test these initial results, we sampled 2 more rings from each core (Figures 2 and 4). The rings were chosen to fill gaps in the $\Delta^{14}C$ time series, and to validate the temporal shifts identified from the first set of sampled rings. As shown in Figure 4, the 2 additional $\Delta^{14}C$ values for SLV06B were quite high, and clearly defined an 8–10 yr difference from the reference curve. The 2 additional $\Delta^{14}C$ values for VIC05B uncovered a slow rise in ^{14}C concentration, pointing to at least a 17-yr divergence from the expected curve (Figure 4). Therefore, the number of locally absent rings in VIC05B was about twice as large as that in SLV06B, which agrees well with the much slower mean growth rate in VIC05B than in SLV06B. Measured $\Delta^{14}C$ of the additional rings sampled on SLV02A fell almost exactly on the initial curve (Figures 3 and 4). This finding confirmed that the ring sequence of SLV02A was off by about 5 yr because of intra-annual growth bands.

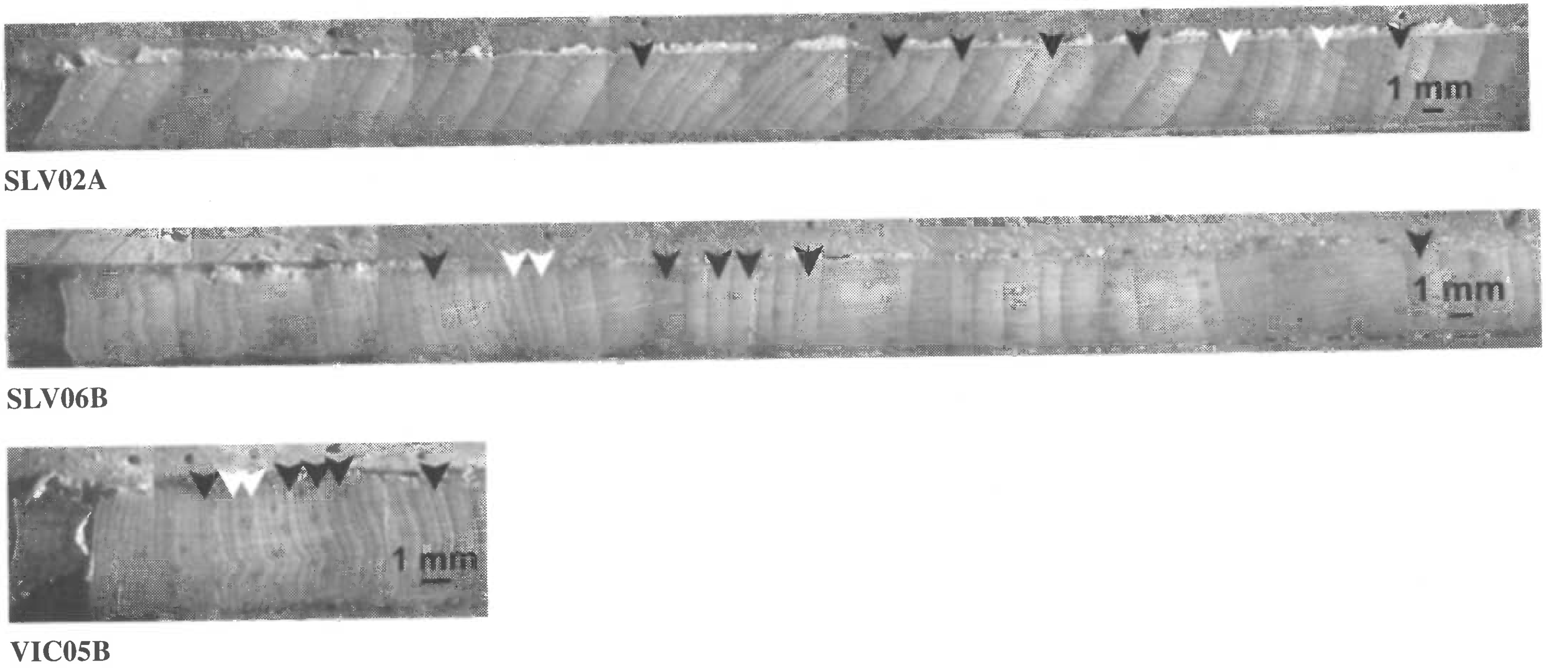

Figure 2 The 3 *Pinus lagunae* wood increment cores used for AMS ^{14}C measurement of selected rings. Both initially (black arrows) and additionally (white arrows) sampled rings are marked. Cores are shown with the outside bark on the left (next to the figure border).

Table 1 AMS ^{14}C measurements on *Pinus lagunae* wood increment cores, including initially and additionally (in italics) sampled rings (Figure 2)

Core	Estimated year[a]	CO_2 (mL)	NOSAMS number	$\delta^{13}C$ (‰)	$\Delta^{14}C$ (‰)
SLV02A	1980	0.82	19299	−24.12	189
SLV02A	1967	1.80	19300	−21.60	514
SLV02A	1965	2.23	19301	−22.28	522
SLV02A	1963	2.75	19302	−22.03	558
SLV02A	1960	2.74	19303	−22.47	675
SLV02A	1950	2.60	19304	−21.51	70
SLV02A	*1954*	*1.09*	*21302*	*−22.88*	*157*
SLV02A	*1958*	*1.00*	*21303*	*−23.53*	*587*
SLV06B	1980	2.42	19305	−21.68	356
SLV06B	1967	2.75	19306	−20.01	184
SLV06B	1965	2.73	19307	−21.63	9
SLV06B	1963	2.75	19308	−21.42	−38
SLV06B	1960	2.65	19309	−21.33	−54
SLV06B	1950	2.71	19310	(−21.00)[b]	−27
SLV06B	*1971*	*0.74*	*21304*	*−22.42*	*624*
SLV06B	*1973*	*1.40*	*21305*	*−22.22*	*662*
VIC05B	1980	1.43	19311	−21.31	347
VIC05B	1967	0.46[c]	19312	−20.82	−78
VIC05B	1963	2.61	19313	−20.61	−28
VIC05B	1961	2.02	19314	−20.72	−43
VIC05B	1950	1.50	19315	−19.59	−49
VIC05B	*1974*	*1.45*	*21306*	*−21.14*	*6*
VIC05B	*1977*	*0.64*	*21307*	*(−25.00)*[b]	*255*

[a] Estimated year of formation, based on the number of identified rings from the outside bark.
[b] Estimated.
[c] Diluted.

DISCUSSION

The absence of crossdating among ring patterns had already shown that *Pinus lagunae* xylem layers at the study area could not be accurately assigned to calendar years. It was also likely that trees showing signs of old age and stunted growth (such as VIC05) would not form growth layers at the base of the stem during unfavorable years (Fritts 1976). It is puzzling, however, that trees with apparently similar mean growth rates would either include extra rings (SLV02) or skip growth years (SLV06).

Climate regime at the study area is likely to be the main cause for the asynchronous wood growth among *P. lagunae* individuals. Based on data from 1944 to 1983 for the city of La Paz (Figure 5), about 70 km to the north, mean annual temperature is 23.5 °C, with mean monthly temperature ranging from 17.4 °C in January to 29.7 °C in August. Total precipitation averages 159 mm annually; monthly it ranges from 1 mm in April to 48 mm in September. While the warm temperatures should not limit tree growth at any time of the year, precipitation is scarce, and tends to have 2 separate

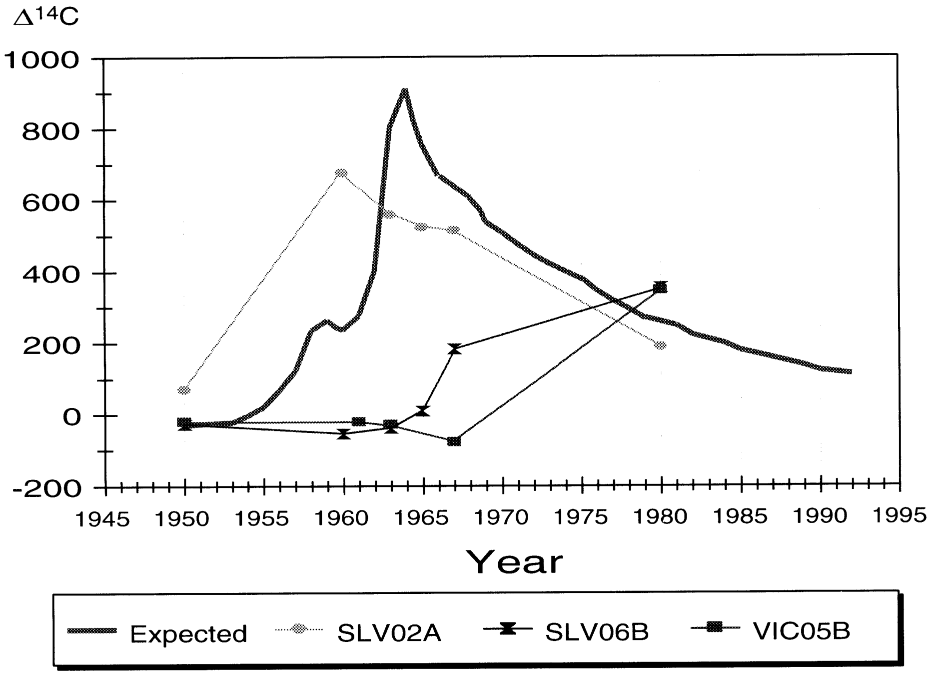

Figure 3 Plot of $\Delta^{14}C$ (‰) measurements on initially sampled rings of wood increment cores (Table 1) compared to expected values provided by John R. Southon (Lawrence Livermore National Laboratory, Center for AMS, personal communication)

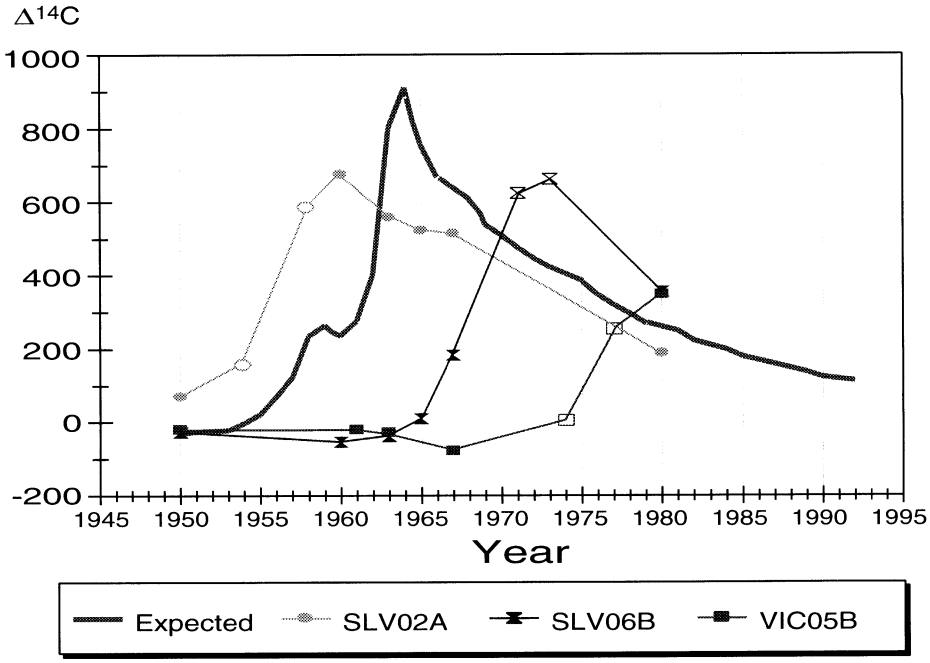

Figure 4 Same as Figure 3, with both initially (filled symbols) and additionally (empty symbols) sampled rings (Table 1)

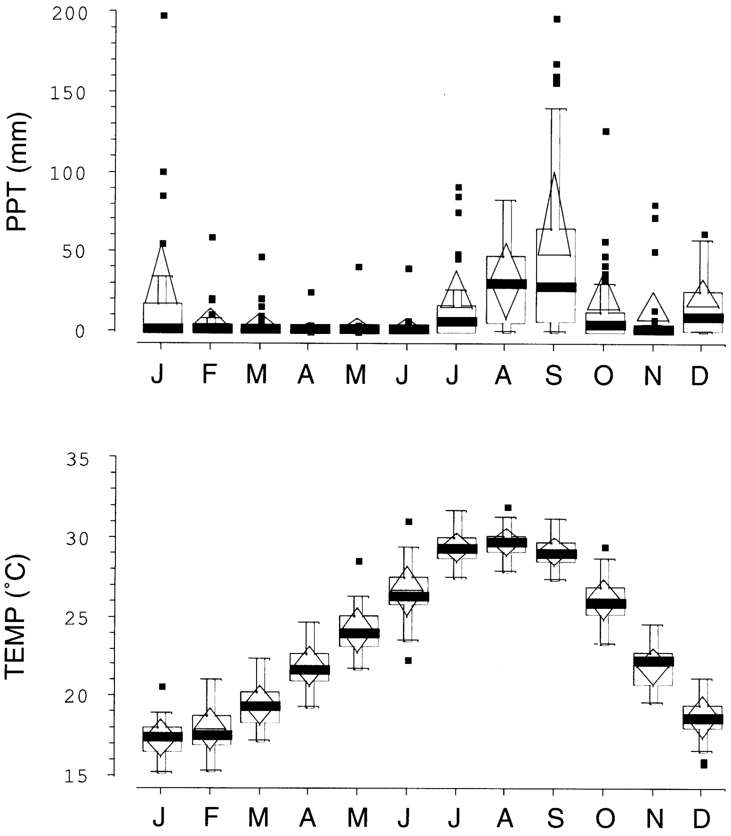

Figure 5. Climatic diagram for La Paz (24°10′N, 110°17′W, 32 m elevation) from 1944 to 1983 (source: PACLIM data set, Cayan et al. 1991). Box-whisker symbols (SAS Institute 1990) for each monthly variable show the median (heavy horizontal line), the 25th and 75th percentiles (bottom and top edges of the box, respectively), the mean ±1 standard deviation (base and vertex of triangle, respectively), the last point within 1.5 interquartile ranges from the 25th and 75th percentiles (vertical bars below and above the box, respectively), and the extreme values (solid squares).

peaks, a major one in late summer/early fall, and a minor one in winter (Figure 5). Assuming a water-year spanning the previous November to the current October, 28% of total precipitation falls during the winter (November–February) and 69% during the summer/fall (June–October). Such a climate regime is intermediate between that of San Diego (Southern California, USA), where November to February precipitation accounts for 65% of the total, and the climate regime of Colima (Colima, Mexico), where 91% of precipitation falls from June to October. It is therefore possible that seasonal drought is responsible for the absence of visible growth layers at the base of some pine stems, especially when combined with limited soil buffering because of a steep and/or rocky slope. At the same time, depending on individual features of the trees and on microsite conditions, separate wet periods could generate distinct growth layers within the same year in other trees.

Note that, despite such dating problems at the annual level, multiannual ring-width patterns could still correspond from core to core. For instance, there is a series of about 10 consecutive small rings in both SLV02A (between the first 2 sampled rings nearest to the bark; Figure 2) and in SLV06B (between the first initial and the first additional sampled ring nearest to the bark; Figure 2) that are likely to represent the same time period. This loosely defined pattern matching, which is not uncommon when dating ocean laminated sediments (personal communication, JC Herguera, CICESE), is perhaps feasible here as well, but it must not be confused with dendrochronological crossdating, which assigns an exact calendar year to every wood increment (Douglass 1941; Stokes and Smiley 1968; Baillie and Pilcher 1973; Holmes 1983).

The present study provides a clear example of how AMS ^{14}C measurements can be used to test for the presence of annual rings when crossdating fails. We demonstrated that ring-width patterns of sampled *Pinus lagunae* were not consistent from one tree to another, and we accurately uncovered their divergence from expected values. Most likely, microsite conditions are more important than regional climate as limiting factors for tree growth. Earlier dendrochronological studies (Díaz et al. 1994; Díaz 1995) were based on vigorous trees with no scars and regular shape growing on gently sloping terrain at higher elevations. Ongoing research at the Laboratory of Tree-Ring Research in Tucson, Arizona, indicates that a great deal of time and effort from highly experienced personnel is required to analyze *P. lagunae* samples using standard dendrochronological techniques. For the purpose of developing dendrochronological proxy records of climate from coniferous species in tropical North America, we thus recommend focusing on species and sites that experience a more pronounced seasonality, such as those found at timberline on the high peaks of Central America (Perry 1991; Arno and Hammerly 1984).

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CALCULATION OF PAST DEAD CARBON PROPORTION AND VARIABILITY BY THE COMPARISON OF AMS ^{14}C AND TIMS U/TH AGES ON TWO HOLOCENE STALAGMITES

Dominique Genty[1] • Marc Massault[1] • Mabs Gilmour[2] • Andy Baker[3] • Sophie Verheyden[4] • Eddy Kepens[4]

ABSTRACT. Twenty-two radiocarbon activity measurements were made by accelerator mass spectrometry (AMS) on 2 Holocene stalagmites from Belgium (Han-stm1b) and from southwest France (Vil-stm1b). Sixteen thermal ionization mass spectrometric (TIMS) U/Th measurements were performed parallel to AMS analyses. The past dead carbon proportion (dcp) due to limestone dissolution and old soil organic matter (SOM) degradation is calculated with U/Th ages, measured calcite ^{14}C activity and atmospheric ^{14}C activity from the dendrochronological calibration curves. Results show that the dcp is different for the 2 stalagmites: between 10,800 and 4780 yr from present dcp=17.5% (σ=2.4; n=10) for Han-stm1b and dcp=9.4% (σ=1.6 ; n=6) between 3070 and 520 yr for Vil-stm1b. Despite a broad stability of the dcp during the time ranges covered by each sample, a slight dcp increase of about 5.0% is observed in the Han-stm1b sample between 8500 and 5200 yr. This change is synchronous with a calcite δ^{13}C increase, which could be due to variation in limestone dissolution processes possibly linked with a vegetation change. The dcp and δ^{13}C of the 2 studied samples are compared with 5 other modern stalagmites from Europe. Results show that several factors intervene, among them: the vegetation type, and the soil saturation leading to variable dissolution process systems (open/closed). The good correlation (R^2=0.98) between the U/Th ages and the calibrated ^{14}C ages corrected with a constant dcp validates the ^{14}C method. However, the dcp error leads to large ^{14}C age errors (i.e. 250–500 yr for the period studied), which is an obstacle for both a high-resolution chronology and the improvement of the ^{14}C calibration curves, at least for the Holocene.

INTRODUCTION

Several studies have demonstrated the great interest in speleothems (stalagmites, flowstones) for the study of paleoenvironments (Gascoyne 1992; Lauritzen 1995; Bar-Mattews et al. 1996). More recently, the study of annual-growth laminae in stalagmites has shown the great chronological and paleoclimatological potential of such deposits: annual growth laminae can be visible (Genty and Quinif 1996) or luminescent under UV light (Shopov and Dermendjiev 1990; Baker et al. 1993). However, 2 major problems still motivate research work: 1) finding a good paleoclimatic signal that can be transformed into a transfer function; and 2) checking the chronology by independent methods, especially for recent deposits where laminae can be counted. Paleoclimatic signals have already been detected, such as laminae thicknesses as indicators of paleoprecipitation (Railsback et al. 1994; Genty and Quinif 1996; Liu et al. 1997; Tan et al. 1997). Luminescence emission is also linked to rainfall (Baker et al. 1997), but such high-resolution studies require an extremely accurate chronology to calibrate the laminae signal with the instrumental climate records or historical data. For that, we can use:

1. Annual laminae counting. Generally, this cannot go very far back in time because of hiatuses or discontinuities in laminae series. The longest known laminae series is from China, where more than 1100 luminescent laminae have been counted continuously, but that is exceptional and linked with very regular climate variations such as monsoons (Tan et al. 1997).
2. Detection of the increase in ^{14}C activity due to bomb activity. However, this only gives the position of the pre-bomb period (around 1950–1955; Genty et al. 1998);
3. Excess ^{210}Pb for the last 100 yr. This method has been applied successfully to only one stalactite and needs more testing (Baskaran and Iliffe 1993).

[1] Université de Paris-Sud, Laboratoire d'Hydrologie et de Géochimie Isotopique, EP 1748, CNRS, bât. 504, F-91405 Orsay Cedex, France
[2] The Open University, Department of Earth Sciences, Milton Keynes, MK7 6AA, England
[3] University of Exeter, Department of Geography, Amory Building, Rennes Drive, EX4 4RJ Exeter, England
[4] Vrije Universiteit Brussel, WE-GISO, Pleinlaan 2, 1050 Brussel, Belgium

4. U/Th TIMS. This is problematic for young samples (i.e. a few hundred years old) because of the low ^{230}Th content, which is made worse by low uranium content samples.
5. ^{14}C AMS. This has great potential for high-resolution chronology for recent deposits, provided that the dead carbon proportion (dcp), due to the dissolution of the limestone and to the oxidation of soil organic matter (SOM), is known (its value, error and variation over time).

The limestone dissolution equation shows that half of the carbon should come from the limestone and the other half from soil CO_2. Then, the expected dead carbon proportion should be theoretically 50%. This is not what we observe. As explained in a former study (Genty and Massault 1997), dcp, up to recently, has been calculated in speleothems in different ways: 1) by measurement of the ^{14}C activity on modern stalagmites (Vogel 1983; Gewelt 1986); 2) by age-distance interpolation up to the top of the stalagmite (linear least-square fit; Broecker and Olson 1960; Gey and Hennig 1986; Railsback et al. 1994; Talma and Vogel 1992); 3) by comparison with pollen extracted from speleothems, which is an indirect way to find the age of the calcite deposit (Bastin and Gewelt 1986); and 4) by comparison with U/Th ages (Vogel 1983; Holmgren et al. 1994). From these earlier studies, the average dcp (or dilution factor q, which is its complement, and more often used by hydrologists) is 15% ± 5 ($q=0.85 \pm 0.05$) (details in Genty and Massault 1997). More recently, using annually laminated stalagmites and the AMS technique, we have demonstrated that dcp due to limestone dissolution can be calculated with pre-bomb values obtained on ^{14}C activity time series of modern stalagmites (Genty et al. 1998, 1999). On the 3 sites already studied, the pre-bomb dcp (mid 1950s) varies from 9.0% to 12.2% ± 1.5. This shows that despite differences in climate and vegetation conditions, the pre-bomb dcp is relatively homogeneous. This study's aim is to calculate past dcp to see if it has changed during the Holocene. The study has at least 3 interests:

1. The study of hydrological and paleohydrological processes. Variation in dcp is the consequence of variation in dissolution processes in the unsaturated zone. These processes are controlled by the vegetation and climate.
2. Speleothem dating. The variation range of dcp during a specific period (i.e. Holocene) will control the error of speleothem ^{14}C ages. If dcp is stable, then AMS ^{14}C dating will be very useful for dating speleothems mainly because it is cheaper, requires less material (10 mg of calcite is sufficient), and is more convenient (laboratory preparation is much faster and simpler) than the U/Th method.
3. Construction of calibration curves. If we demonstrate that the past dcp is relatively constant and that it can be calculated with pre-bomb ^{14}C activity, as we have done on modern stalagmites, then past atmospheric ^{14}C activity could be calculated using U/Th ages and measured speleothem ^{14}C activity.

SITE AND SAMPLE DESCRIPTIONS

The 2 stalagmites chosen for investigation in this study are Vil-stm1b from the Villars cave (Dordogne, France; L = 45°30′N, l = 0°50′E, Z = 175 m), and Han-stm1b from the Han-sur-Lesse cave (Belgium; L = 50°08′N, l = 5°10′E, Z = 180 m). The Villars cave developed in Jurassic limestone, whereas the Han-sur-Lesse cave developed in Devonian limestone. Forest covers most of both sites, but the area just above Vil-stm1b is composed of grassland. Han-stm1b is 138 cm high and dark brown calcite. Vil-stm1b is composed of white calcite and is 109 cm high. Both samples are composed of an alternation of porous/compact calcite every few tens of centimeters. Growth laminae are visible on most of the upper half of Vil-stm1b.

METHODS

Mass Spectrometric Measurements

The main advantage of using AMS for ^{14}C, and TIMS for U/Th, is that very little sample is needed: 12–77 mg for AMS and 600–3000 mg for U/Th. This quantity of calcite corresponds to 2–10 yr of deposits for AMS and 5–50 yr for TIMS, depending on the sample's average vertical growth rate.

AMS ^{14}C Measurements

Calcite powders were reacted with H_3PO_4 to obtain CO_2. The gas was graphitized on iron with hydrogen at 650 °C for 100 min. Residual gas was used for stable isotope measurements on a SIRA spectrometer. Carbon atoms were counted with an accelerator mass spectrometer (Tandetron, Gif-sur-Yvette, France). Analytical errors, including laboratory errors, are ± 0.1‰ for $\delta^{13}C$ and between 0.4 and 1.0 pMC for ^{14}C activity. The blank correction is 0.4 pMC. Errors on ^{14}C ages include the dcp errors, which increase the final error by about 3 times (see below).

TIMS U/Th Measurements

TIMS U/Th measurements were done at 2 different laboratories: the Open University (England) for Vil-stm1b, and the GEOTOP Laboratory at the University of Quebec at Montreal (Canada) for Han-stm1b. Analytical procedures are summarized for both samples.

Samples were dissolved with nitric acid and spiked with a mixed ^{229}Th-^{236}U spikes (Vil-stm1b) and ^{229}Th-^{236}U-^{233}U (Han-stm1b). Uranium and thorium fractions were separated on anion exchange columns using standard techniques (Edwards et al. 1987). Both uranium and thorium were loaded onto graphite coated Re filaments and analyses carried out using Finnigan MAT262 (Vil-stm1b) and VG Sector (Han-stm1b) mass spectrometers. The former is equipped with a potential quadrupole and a secondary electron multiplier, the latter with an electrostatic analyzer and an ion-counting Daly detector. Errors were propagated from the in-run statistics and the uncertainties on the spike isotopic composition. The relatively high error on $^{230}Th/^{229}Th$ results for Vil-stm1b reflects the low count rates obtained for these small young, low uranium content samples. The detrital calculation used corrects for both uranium and thorium detrital contribution to the sample. It is assumed that the detrital component has a $^{232}Th/^{238}U$ molar ratio of 5, that the detrital uranium is in secular equilibrium, and that all the ^{232}Th is of detrital origin. Since $^{230}Th/^{232}Th$ activity ratios for all samples were >50, they can be viewed essentially as having negligible detrital input (except for U/Th-G sample at the base of Han-stm1b), hence the detrital age corrections are typically very small. Ages were calculated using the standard equation and the decay constants used for ^{234}U, ^{238}U, ^{230}Th and ^{232}Th were 2.835×10^{-6}, 1.55125×10^{-10}, 9.1952×10^{-6} and 4.9475×10^{-11} yr^{-1}, respectively.

Dead Carbon Proportion Calculations

The past dcp (dcp_{past}) of the 2 stalagmites studied was calculated as the following:

$$dcp_{past} = \left[1 - \left(\frac{a^{14}C_{int.}}{a^{14}C_{atm.\ init.}}\right)\right] \cdot 100\% \tag{1}$$

where $a^{14}C_{init.}$ is the initial ^{14}C activity of the calcite and $a^{14}C_{atm.\ init.}$ is the atmospheric ^{14}C activity of the time of deposition, they are defined by:

$$a^{14}C_{\text{int.}} = \frac{a^{14}C_{\text{mes.}}}{\exp(\lambda \cdot t)} \quad , \tag{2}$$

with t=U/Th age in years and λ is the decay constant of ^{14}C, using 5730-yr half life where $a^{14}C_{\text{mes.}}$ is the measured calcite activity and $a^{14}C_{\text{atm. init.}}$ is found with the U/Th age on the calibration curves (bidecadal $\Delta^{14}C$ values of the Calib 3.0 data set; Stuiver and Kra 1986; Bronk Ramsey 1994).

Another method has been used to estimate the dcp on modern stalagmites (dcp_{modern}) (Genty et al. 1998, 1999). We briefly describe this method here as we compare and discuss modern dcp and past dcp. The calcite ^{14}C activity time series was reconstructed using modern stalagmites that possess annual growth laminae. Laminae counting permits to obtain a high resolution chronology. The ^{14}C activity curves obtained have shown that the ^{14}C peak due to nuclear weapon tests is time delayed in the stalagmites by up to 20 yr and that the decrease in the ^{14}C activity is highly variable from one site to another. The dcp_{modern} is given by the following:

$$dcp_{\text{modern}} = \left[1 - \left(\frac{a^{14}C_{\text{mes.}}}{a^{14}C_{\text{atm.}}}\right)\right] \cdot 100\% \tag{3}$$

where $a^{14}C_{\text{mes.}}$ is the pre-bomb calcite ^{14}C activity and $a^{14}C_{\text{atm.}}$ is the pre-bomb atmospheric ^{14}C activity.

Dead Carbon Proportion Errors

For equation (3) it can be reasonably assumed that the average error on dcp is 1.5% because average error on AMS measurements is 0.7%, and we suppose the same for the atmospheric measurement (Genty and Massault 1997). For equation (1), the error has been calculated using: 1) error on $a^{14}C_{\text{mes.}}$; 2) error on U/Th age; and 3) error on past atmospheric ^{14}C activity read on the dendrochronological calibration curve (Bidecadal $\Delta^{14}C$ values of the Calib 3.0 data set; Stuiver and Kra 1986; Bronk Ramsey 1994). Results show that dcp error is relatively high: 2.7% for Han-stm1b and 4.0% for Vil-stm1b (Tables 1 and 2).

RESULTS AND DISCUSSION

Dead Carbon Proportion and Calcite $\delta^{13}C$ Variations

The past dcp calculated on Vil-stm1b and Han-stm1b stalagmites gives these results (Tables 1–6):

1. Average dcp is higher for Han-stm1b (17.5%; 1σ=2.4; n=10) than for Vil-stm1b (9.4%; 1σ=1.6; n=6) (Figure 1).
2. In the Han-stm1b stalagmite, the dcp goes through a broad maximum between 8500 and 5200 yr (19.6%; 1σ=0.8; n=5) while it is about 5% lower elsewhere.
3. Vil-stm1b average dcp are within error margins.

Table 1 ^{14}C and dead carbon proportion results on Vil-stm1b stalagmite[a]

Sample name	Lab nr (PA-)	Position (cm/base)	Error (cm)	Weight (mg)	$a^{14}Cm$ (pMC)	Error (pMC)	Conv. ^{14}C age (yr BP)	Error (yr)	dcp1 (%)	Error	dcp1 corrected $a^{14}C$ (pMC)	Error (pMC)	Conv. dcp corrected ^{14}C age (yr BP)
14C-C	234	108.7	0.2	22.5	96.6	1.0	270	80	9.4	1.5	106.7	2.5	
14C-H	276	106.2	0.2	12.2	86.5	0.9	1160	80	9.4	1.5	95.5	2.4	368
14C-L	290	101.3	0.2	43.1	86.4	0.7	1170	60	9.4	1.5	95.4	2.2	380
14C-G	275	91	0.2	28.7	87.4	0.7	1080	60	9.4	1.5	96.5	2.2	
14C-K	289	80.4	0.2	34.9	83.6	0.6	1440	60	9.4	1.5	92.3	2.1	650
14C-B	233	71	0.2	35.6	76.3	1.0	2170	110	9.4	1.5	84.2	2.5	1380
14C-J	288	59.5	0.2	23.9	74.9	0.6	2320	60	9.4	1.5	82.7	2.1	1530
14C-I	287	42.5	0.2	15.9	68.6	0.6	3030	70	9.4	1.5	75.7	2.1	2240
14C-F	274	24.6	0.2	25	65.1	0.6	3450	60	9.4	1.5	71.8	2.1	2660
14C-E	273	5	0.2	27	63.4	0.6	3660	70	9.4	1.5	70.0	2.1	2860
14C-A	232	1.5	0.2	77.6	62.3	0.6	3800	70	9.4	1.5	68.7	2.1	3010

Sample name	Error (yr)	Calib. cor. ^{14}C age yr AD/(−)BC	Error + yr AD/(−)BC	Error − yr AD/(−)BC	U/Th ages yr AD/(−)BC	$\Delta^{14}C_{atm.}$ (‰)	Error (‰)	$a^{14}C_{atm.}$ (pMC)	Error (pMC)	$a^{14}C_{init.}$ (pMC)	Error (pMC)	dcp (%)	Error (%)
14C-C		1990	1989	1991									
14C-H	200	1940	1937	1943									
14C-L	180	1935	1800	1993									
14C-G					1480	8.8	2	100.88	0.2	93.1	3.2	7.7	3.4
14C-K	180	1310	1160	1460	1320	−12.8	1.6	98.72	0.16	90.8	4.9	8.1	5.1
14C-B	240	675	400	950	620	−18.7	2.1	98.13	0.21	90.1	4.4	8.2	4.6
14C-J	200	510	320	700	760	−16.4	1.3	98.36	0.13	87.0	3.0	11.5	3.1
14C-I	220	−250	−550	50									
14C-F	230	−725	−1050	−400	−590	−2.7	1.7	99.73	0.17	89.0	3.7	10.8	3.9
14C-E	240	−1100	−1400	−800									
14C-A	240	−1225	−1550	−900	−1070	3.8	1.8	100.38	0.18	90.3	3.5	10.1	3.7

[a] $a^{14}{}_{m}$ = measured ^{14}C activity; dcp1 = averaged past dead carbon proportion (see text); Calibration of ^{14}C ages have been done with OxCal v2.8 program (Stuiver and Kra 1986; Bronk Ramsey 1994); $\Delta^{14}C_{atm}$. and $a^{14}{}_{atm.}$ = atmospheric ^{14}C activity of the known age deposit (grace to U/Th age) from calibration curves; dcp = calculated dead carbon proportion using U/Th ages, $a^{14}{}_{m}$ and $a^{14}{}_{atm.}$ (past dcp).

Table 2 ^{14}C and dead carbon proportion results on Han-stmlb stalagmite[a]

Sample name (^{14}C-)	Lab nr (PA-)	Position (cm/base)	Error (cm)	Weight (mg)	a^{14}Cm (pMC)	Error (pMC)	Conv. ^{14}C age (yr BP)	Error (yr)	dcp1 (%)	Error	dcp1 corrected a^{14}C (pMC)	Error (pMC)	Conv. dcp corrected ^{14}C age (yr BP)
G	376	3	0.1	25.1	19.8	0.4	13,020	160	17.5	1.5	24.0	1.9	11,480
A	291	12.3	0.1	14.3	25.5	0.5	10,990	160	17.5	1.5	30.9	2.0	9440
E	302	33	0.1	15.5	27.7	0.4	10,300	110	17.5	1.5	33.6	1.9	8760
B2	293	43.5	0.1	11.4	28.2	0.7	10,180	200	17.5	1.5	34.1	2.2	8630
B1	292	54.3	0.1	23.2	30.6	0.5	9510	130	17.5	1.5	37.1	2.0	7970
H	378	67	0.1	26.5	30.9	0.5	9430	110	17.5	1.5	37.5	2.0	7880
C	294	93.7	0.1	24.5	36.5	0.4	8090	90	17.5	1.5	44.3	1.9	6540
I	379	101	0.1	23.6	37.2	0.5	7940	100	17.5	1.5	45.1	2.0	6390
F	303	108.9	0.1	11.6	39.8	0.4	7410	90	17.5	1.5	48.2	1.9	5870
D	301	129.4	0.1	21.3	45.5	0.4	6330	70	17.5	1.5	55.1	1.9	4790
J	380	137.7	0.1	29.6	50.7	0.4	5460	60	17.5	1.5	61.4	1.9	3910

Sample name (^{14}C-)	Error (yr)	Calibrated cor. ^{14}C age (BC)	Error (+ BC)	Error (– BC)	U/Th ages (BC)	$\Delta^{14}C_{atm.}$ (‰)	Error (‰)	a$^{14}C_{atm.}$ (pMC)	Error (pMC)	a$^{14}C_{init.}$ (pMC)	Error (pMC)	dcp (%)	Error (%)
G	640	11,550	12,300	10,800									
A	520	8750	9600	7900	8690	127.4	2.9	112.74	0.29	92.7	2.7	17.8	3.0
E	450	7750	8400	7100	8060	109.7	2.2	110.97	0.22	93.7	2.2	15.6	2.5
B2	520	7700	8400	7000	8010	101.9	1.9	110.19	0.19	94.5	3.2	14.2	3.4
B1	430	6925	7450	6400	7080	89.9	4.9	108.99	0.49	91.7	2.6	15.8	3.1
H	420	6750	7300	6200	6520	63.6	1.8	106.36	0.18	86.6	2.0	18.6	2.2
C	340	5400	5750	5050	5140	89.9	1.7	108.99	0.17	86.6	2.0	20.5	2.2
I	360	5275	5650	4900	5210	97.6	2.7	109.76	0.27	89.0	2.5	18.9	2.8
F	320	4775	5200	4350	4390	75.4	2.3	107.54	0.23	86.1	3.2	19.9	3.4
D	280	3525	3950	3100	3220	67.5	1	106.75	0.1	85.5	2.1	20.0	2.2
J	250	2350	2700	2000	2780	53.1	1.4	105.31	0.14	90.3	1.4	14.2	1.5

[a] a$^{14}_{m}$ = measured ^{14}C activity; dcp1 = averaged past dead carbon proportion (see text); Calibration of ^{14}C ages have been done with OxCal v2.8 program (Stuiver and Kra 1986; Bronk Ramsey 1994); $\Delta^{14}C_{atm.}$ and a$^{14}_{atm.}$ = atmospheric ^{14}C activity of the known-age deposit (grace to U/Th age) from calibration curves; dcp = calculated dead carbon proportion using U/Th ages, a$^{14}_{m}$ and a$^{14}_{atm.}$ (past dcp).

Table 3 U/Th results of Vil-stm1b stalagmite[a]

Sample name	Position (cm/base)	Error (cm)	^{238}U (ppm)	Error (ppm)	^{234}U/^{238}U act.	Error act.	^{234}U (ppm)	Error (ppm)	^{230}Th (ppb)	Error (ppb)	^{232}Th (ppb)	Error (ppb)	^{230}Th/^{232}Th act.	Error act.	^{230}Th/^{234}U act.	Error act.	Row age (yr/present)	Error (yr)	Cor. age (yr/present)	Error	% error (1σ)
U/Th-G	96	0.3	0.13040	0.00054	0.99385	0.00718	6.97E-06	5.05E-08	7.88E-06	3.58E-07	0.02706	0.00016	71.53	3.28	0.00373	0.00017	410	20	400	20	5.2
U/Th-C	91	0.3	0.13802	0.00013	1.0025	0.01888	7.44E-06	1.86E-08	1.10E-05	1.74E-07	0.0812	0.0001	50.58	0.80	0.00488	0.00013	530	15	520	15	2.6
U/Th-H	79.7	0.3	0.13547	0.0003	0.99671	0.00481	7.26E-06	3.55E-08	1.40E-05	5.69E-07	0.06748	0.0003	64.96	2.66	0.00636	0.00026	690	30	680	30	4.6
U/Th-F	70.7	0.3	0.15365	0.00064	0.94893	0.01278	7.84E-06	1.09E-07	3.03E-05	7.32E-07	0.15442	0.00025	62.89	1.52	0.01277	0.00036	1400	40	1370	40	3.6
U/Th-B	59.5	0.3	0.09428	0.00007	1.0052	0.0189	5.10E-06	1.25E-08	1.76E-05	2.85E-07	0.0197	0.0001	236.41	4.01	0.01137	0.0003	1240	35	1240	35	2.7
U/Th-E2	24.6	0.3	0.14071	0.00021	1.00498	0.01930	7.61E-06	2.49E-08	5.43E-05	1.33E-06	0.0383	0.00034	306.13	7.99	0.02354	0.00077	2590	85	2590	85	3.3
U/Th-A	1.5	0.3	0.12185	0.00043	1.0762	0.0276	7.05E-06	1.24E-07	5.99E-05	5.88E-07	0.1457	0.0002	89.71	0.89	0.02804	0.00082	3090	90	3070	90	3

[a] Act. = activity; Cor. Age = detrital corrected U/Th age. Error margins are 1σ.

Table 4 U/Th results of Han-stmlb stalagmite[a]

Sample name (U/Th-)	Position cm/base	Error (cm)	^{238}U (ppm)	Error (ppm)	^{232}Th (ppb)	Error (ppb)	^{234}U/^{238}U act.	Error act.	^{230}Th/^{234}U act.	Error act.	Cor. age (yr/present)	Error
G	1.75	0.90	1.34340	0.00477	281.460	6.60110	1.87040	0.0095	—	—	—	—
A	12.10	0.90	1.5877	0.00588	7.6749	0.0287	1.8487	0.01	0.0942	0.0009	10,680	110
E	32.90	0.60	1.7961	0.0082158	1.7748	0.0077535	1.8227	0.0112	0.0889	0.0008	10,060	95
B2	43.50	0.60	1.7778	0.0057904	1.7456	0.0069017	1.8212	0.0105	0.0885	0.0008	10,010	95
B1	54.30	0.70	1.3398	0.00594	15.235	0.05296	1.7538	0.0102	0.0805	0.0009	9070	105
H	66.95	0.95	1.2331	0.0046701	3.0343	0.01142	1.7247	0.0088	0.0757	0.0006	8520	70
C	93.80	0.4	0.83868	0.0035	14.84	0.059	1.7396	0.0121	0.0638	0.0008	7140	90
I	101.00	1.00	0.90814	0.0033586	12.828	0.07443	1.7982	0.0132	0.0644	0.0009	7210	105
F	108.80	0.6	0.93735	0.0049	10.7	0.071	1.7574	0.0161	0.0573	0.0015	6392	172
D	129.45	0.55	0.90229	0.00312	4.3076	0.013419	1.7457	0.0094	0.047	0.0007	5220	80
J	136.10	1.60	1.0657	0.00397	1.4631	0.0051206	1.7113	0.0102	0.0431	0.0003	4780	35

[a] Act. = activity; Cor. Age = detrital corrected U/Th age. Error margins are 1 σ.

Dcp Difference between Han-stm1b and Vil-stm1b Stalagmites

Differences between averaged dcp in Han-stmlb and Vil-stmlb can be due to numerous causes:

1. Differences in limestone dissolution processes (i.e. difference in the relative importance of open/closed systems; Hendy 1971; Dulinski and Rozanski 1990) due to differences in the soil and host rock characteristics (porosity, thickness) and/or in climate and vegetation conditions.
2. A different time residence of the seepage water. The longer the water stays in the micro-fissure network, the more limestone is dissolved and the dead-carbon proportion is greater. The residence time of the water can be controlled by the limestone thickness above the studied site. Because limestone thickness above Han-stmlb is 50 m while it is 10 m above Vil-stmlb, it is possible that this thickness variation explains the higher dcp for the Han-stmlb sample.

Table 5 $\delta^{13}C$ of Vil-stmlb stalagmite

Sample name	Position (cm/base)	U/Th age	Estimated error (–yr)	$\delta^{13}C$ (‰ PDB, ±0.1)
Vil-stm1-0	0.00	−1100 BC	90	−10.80
Vil-stm1-2	2.00	−1060 BC	90	−10.77
Vil-stm1-4	4.00	−1020 BC	90	−10.45
Vil-stm1-10	10.00	−900 BC	90	−10.65
Vil-stm1-20	20.00	−680 BC	85	−10.42
Vil-stm1-30	30.00	−380 BC	80	−11.09
Vil-stm1-40	40.00	10 AD	70	−10.77
Vil-stm1-50	50.00	400 AD	50	−11.32
Vil-stm1-60	60.00	750 AD	30	−9.88
Vil-stm1-74	74.00	860 AD	50	−8.39
Vil-stm1-80	80.00	1315 AD	30	−8.72
Vil-stm1-90	90.00	1470 AD	15	−10.01
Vil-stm1-100	100.00	1870 AD	15	−9.74
Vil-stm1-106	106.00	1940 AD	10	−8.84

Except for the high dcp (30–35%) found in a stalagmite from Castelguard cave (Canada) where no soil develops (Gascoyne and Nelson 1983), the past dcp found in Han-stmlb and Vil-stmlb is in agreement with the already published dcp found in modern speleothems from temperate countries (15% ± 5; see above for references). The dead carbon proportion (or the dilution factor q, which is its correlant, often used by hydrologists [q=(100–dcp)/100)], is the consequence of limestone dissolution processes in the unsaturated zone. Limestone dissolution can occur, theoretically, under 2 processes called open and closed systems (Hendy 1971), or coincident and sequential systems (Drake 1983, 1984). In an open system, the seepage water is in contact with soil CO_2 during limestone dissolution, while in a closed system, dissolution occurs when the seepage water is isolated from soil CO_2. In the first case, dissolved inorganic carbon (DIC) (HCO_3^-), which comes from limestone dissolution, is changed by dissolved CO_2 which can be degassed; consequently, old C can be removed from the seepage water leading toward a low dcp. In the second case (closed system), limestone dissolution, which brings the dead carbon in the water, is limited by the quantity of dissolved CO_2 which is itself controlled by the soil pCO_2. After complete dissolution, theoretical dcp goes up to 50%.

Soils in karst areas are generally very thin (5–20 cm thick) and carbonate is quickly reached by the infiltrated water, so consequently the 3 phases (CO_2, water, and limestone) are gathered, and an open system is more likely to occur, at least during the first stage of seepage. During water infiltration, the

Table 6 $\delta^{13}C$ of Han-stmlb stalagmite

Position (cm/base)	Interpolated U/Th ages (yr present)	$\delta^{13}C$ ‰ PDB (± 0.1)	Position (cm/base)	Interpolated U/Th ages (yr present)	$\delta^{13}C$ ‰ PDB (± 0.1)	Position (cm/base)	Interpolated U/Th ages (yr present)	$\delta^{13}C$ ‰PDB (± 0.1)	Position (cm/base)	Interpolated U/Th ages (yr present)	$\delta^{13}C$ ‰PDB (± 0.1)
3	10,949	−6.80	37	10,039	−7.23	71	8312	−6.95	105	6798	−6.31
4	10,920	−7.20	38	10,035	−6.89	72	8261	−6.45	106	6696	−6.34
5	10,890	−7.18	39	10,030	−7.24	73	8210	−5.98	107	6594	−6.07
6	10,860	−7.49	40	10,025	−6.94	74	8159	−6.18	108	6492	−6.16
7	10,831	−7.10	41	10,020	−7.08	75	8108	−6.63	109	6390	−6.35
8	10,801	−7.44	42	10,016	−6.94	76	8057	−6.34	110	6331	−6.37
9	10,771	−7.12	43	10,011	−6.98	77	8006	−6.37	111	6273	−6.40
10	10,741	−7.39	44	9926	−7.14	78	7955	−6.70	112	6214	−6.43
11	10,712	−7.15	45	9841	−7.16	79	7904	−6.59	113	6156	−6.18
12	10,682	−7.27	46	9756	−6.80	80	7853	−6.56	114	6097	−6.44
13	10,652	−6.57	47	9671	−6.62	81	7803	−6.37	115	6039	−5.85
14	10,623	−7.09	48	9586	−6.67	82	7752	−6.31	116	5980	−5.77
15	10,593	−7.89	49	9500	−6.83	83	7701	−6.95	117	5922	−6.26
16	10,563	−7.69	50	9415	−6.92	84	7650	−6.75	118	5863	−5.88
17	10,533	−8.18	51	9330	−6.54	85	7599	−6.70	119	5805	−6.12
18	10,504	−7.67	52	9245	−6.47	86	7548	−6.73	120	5746	−6.22
19	10,474	−7.35	53	9160	−6.50	87	7497	−5.86	121	5687	−6.13
20	10,444	−7.49	54	9075	−6.34	88	7446	−5.82	122	5629	−6.11
21	10,415	−7.42	55	9032	−6.77	89	7395	−6.42	123	5570	−6.51
22	10,385	−6.86	56	8989	−6.15	90	7344	−6.45	124	5512	−6.73
23	10,355	−6.99	57	8946	−6.86	91	7293	−6.42	125	5453	−6.84
24	10,325	−7.38	58	8903	−6.21	92	7242	−6.44	126	5395	−6.81
25	10,296	−7.45	59	8860	−7.05	93	7191	−6.49	127	5336	−6.78
26	10,266	−7.45	60	8817	−6.54	94	7140	−6.39	128	5278	−6.80
27	10,236	−7.56	61	8774	−6.70	95	7149	−6.39	129	5219	−6.72
28	10,207	−7.19	62	8731	−6.61	96	7159	−6.62	130	5164	−6.59
29	10,177	−7.23	63	8688	−6.50	97	7168	−6.77	131	5109	−6.62
30	10,147	−7.27	64	8645	−6.88	98	7178	−6.82	132	5054	−6.77
31	10,117	−6.85	65	8602	−7.01	99	7187	−7.06	133	4999	−6.43
32	10,088	−7.35	66	8559	−7.00	100	7197	−7.00	134	4943	−6.26
33	10,058	−7.17	67	8516	−6.98	101	7206	−5.91	135	4888	−6.81
34	10,053	−7.55	68	8465	−6.91	102	7104	−6.12	136	4833	−6.60
35	10,049	−7.82	69	8414	−6.83	103	7002	−6.09	137	4778	−6.46
36	10,044	−7.79	70	8363	−6.67	104	6900	−6.16			

CO_2 gas volume in contact with water and limestone decreases and dissolution conditions become closer to the closed system conditions. As explained by earlier studies (Hendy 1971; Drake 1984), the real situation in the karst unsaturated zone is an intermediate between these 2 dissolution processes for 3 reasons: 1) vegetation roots penetrate the rock formation up to several meters and by their respiration they produce CO_2, which can play an important role in the pCO_2 balance and then in the dissolution process; 2) it has been demonstrated that in most karst areas the micro-fissures are filled by both water and gas, the main components of a 2-phase seepage (Mangin 1975; Fleyfel 1979; Fleyfel and Bakalowicz 1979), and this might increase the possibility of open system conditions at different depths in the karst; and 3) depending on the meteorological conditions (i.e. rainfall intensity, evapotranspiration), the seepage will be variable and exchanges between the different phases will also vary. The dead carbon difference observed between the 2 samples studied could be the consequence of a different proportion of open and closed regimes during the dissolution process.

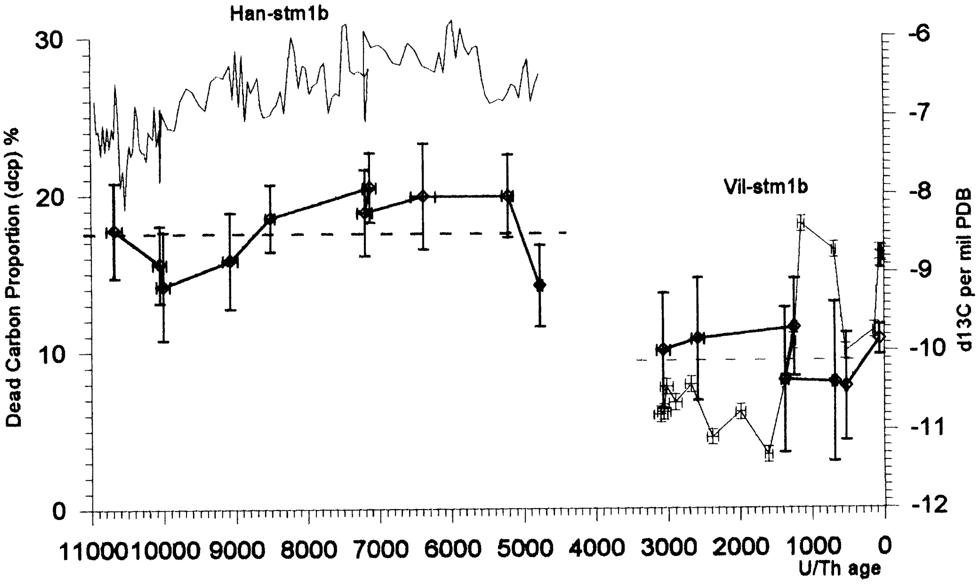

Figure 1 Dead carbon proportion (dcp) and $\delta^{13}C$ (thin lines) vs. U/Th ages of the 2 studied stalagmites. Note that for each stalagmite, the dcp stays relatively stable during the growth: average dcp is 17.5% (σ=2.4%; n=10) for Han-stm1b and is 9.4% (σ=1.6% ; n=6) for Vil-stm1b. However, we note a slight increase of the dcp and of the $\delta^{13}C$ between 8500 and 5200 yr for Han-stm1b. This could be the consequence of a vegetation change (see text).

$\delta^{13}C$ Differences between Han-stm1b and Vil-stm1b Stalagmites

Because limestone $\delta^{13}C$ is much higher than soil $\delta^{13}C$ (between –2‰ and +2‰, and between –20‰ and –24‰ for soil CO_2 under C3 plants), $\delta^{13}C$ measurements in the precipitated calcite give useful information about the sources of the DIC species, and consequently on the dissolution processes. The ^{13}C content of the precipitated calcite in speleothems is controlled by the following:

1. Soil CO_2 $\delta^{13}C$, which depends on the photosynthetic pathway and hence on the vegetation type: under C3 plants $\delta^{13}C$ is about –22‰, whereas it is about –12‰ for C4 plants (Dever et al. 1982; Dörr and Münnich 1986; Fleyfel 1979; Hendy 1971).
2. The temperature, which controls the isotope fractionation between soil CO_2 and DIC (Mook et al. 1974), and between DIC and precipitated $CaCO_3$ (Mook 1980).
3. The quantity of dissolved limestone and its $\delta^{13}C$ (–1.2‰ for Han-sur-Lesse site, –1.9‰ for Villars site), controlled by the dissolution process (open/closed system proportions).

Table 7 Calculation of the theoretical calcite $\delta^{13}C$ under different environmental conditions using a mixing model (dcp) and isotopic fractionation factors (see text for details)[a]

	Temp. (°C)	Soil $\delta^{13}C$	$\delta^{13}C$ <diss.	dcp (%)	Limestone $\delta^{13}C$	$\delta^{13}C$ >diss.	$CaCO_2$ $\delta^{13}C$
A							
Villars site	10	−20.30	−10.70	9.40	−1.90	−9.87	−9.72
	11	−20.30	−10.82	9.40	−1.90	−9.98	−9.77
	12	−20.30	−10.93	9.40	−1.90	−10.08	−9.83
	13	**−20.30**	**−11.05**	**9.40**	**−1.90**	**−10.19**	**−9.88**
	14	−20.30	−11.17	9.40	−1.90	−10.29	−9.93
	15	−20.30	−11.28	9.40	−1.90	−10.40	−9.99
	16	−20.30	−11.39	9.40	−1.90	−10.50	−10.04
Han-sur-Lesse site	6	−17.80	−7.72	17.50	−1.20	−6.58	−6.64
	7	−17.80	−7.84	17.50	−1.20	−6.68	−6.68
	8	−17.80	−7.96	17.50	−1.20	−6.78	−6.73
	9	**−17.80**	**−8.08**	**17.50**	**−1.20**	**−6.88**	**−6.78**
	10	−17.80	−8.20	17.50	−1.20	−6.97	−6.82
	11	−17.80	−8.32	17.50	−1.20	−7.07	−6.86
	12	−17.80	−8.43	17.50	−1.20	−7.17	−6.91
B							
Villars site	13	−17.00	−7.75	9.40	−1.90	−7.20	−6.89
	13	−18.00	−8.75	9.40	−1.90	−8.11	−7.80
	13	−19.00	−9.75	9.40	−1.90	−9.01	−8.70
	13	**−20.00**	**−10.75**	**9.40**	**−1.90**	**−9.92**	**−9.61**
	13	**−21.00**	**−11.75**	**9.40**	**−1.90**	**−10.82**	**−10.51**
	13	−22.00	−12.75	9.40	−1.90	−11.73	−11.42
	13	−23.00	−13.75	9.40	−1.90	−12.64	−12.33
Han-sur-Lesse site	9	−15.00	−5.28	17.50	−1.20	−4.57	−4.47
	9	−16.00	−6.28	17.50	−1.20	−5.39	−5.29
	9	**−17.00**	**−7.28**	**17.50**	**−1.20**	**−6.22**	**−6.12**
	9	**−18.00**	**−8.28**	**17.50**	**−1.20**	**−7.04**	**−6.94**
	9	−19.00	−9.28	17.50	−1.20	−7.87	−7.77
	9	−20.00	−10.28	17.50	−1.20	−8.69	−8.59
	9	−21.00	−11.28	17.50	−1.20	−9.52	−9.42
C							
Villars site	13	−20.30	−11.05	5.00	−1.90	−10.59	−10.28
	13	−20.30	−11.05	7.00	−1.90	−10.41	−10.10
	13	**−20.30**	**−11.05**	**9.00**	**−1.90**	**−10.23**	**−9.92**
	13	−20.30	−11.05	11.00	−1.90	−10.04	−9.73
	13	−20.30	−11.05	13.00	−1.90	−9.86	−9.55
	13	−20.30	−11.05	15.00	−1.90	−9.68	−9.37
	13	−20.30	−11.05	17.00	−1.90	−9.49	−9.18
Han-sur-Lesse site	9	−17.80	−8.08	12.00	−1.20	−7.25	−7.15
	9	−17.80	−8.08	14.00	−1.20	−7.12	−7.02
	9	−17.80	−8.08	16.00	−1.20	−6.98	−6.88
	9	**−17.80**	**−8.08**	**18.00**	**−1.20**	**−6.84**	**−6.74**
	9	−17.80	−8.08	20.00	−1.20	−6.70	−6.60
	9	−17.80	−8.08	22.00	−1.20	−6.57	−6.47
	9	−17.80	−8.08	24.00	−1.20	−6.43	−6.33
D							
Han-sur-Lesse site	6	**−18.40**	**−8.32**	14.20	**−1.20**	**−7.31**	**−7.37**
	10	**−17.40**	**−7.80**	**20.50**	**−1.20**	**−6.45**	**−6.29**

[a] **A**: T changes, soil $\delta^{13}C$ and dcp are constant. **B**: soil $\delta^{13}C$ changes, T and dcp are constant. **C**: dcp changes, T and soil $\delta^{13}C$ are constant. **D**: temperature, soil $\delta^{13}C$ and dcp changes to take into account the observed calcite $\delta^{13}C$ variation in the Han-stm1b stalagmite (see text). Numbers in bold correspond to our measurements.

As Figure 1 shows, similar to the dcp, the average $\delta^{13}C$ is higher in Han-stm1b (–6.7‰; σ=0.5; n=135) than in Vil-stm1b (–9.8‰; σ=1.1; n=17). We have calculated the theoretical precipitated calcite $\delta^{13}C$ using a simple mixing model (dcp for limestone dissolution; see Genty et al. 1999 for details) and hypotheses have been made for temperature, soil CO_2 $\delta^{13}C$ and dcp variations (Table 7). Apparently, the main factor that controls the calcite $\delta^{13}C$ is soil $\delta^{13}C$, and reasonable changes in temperature (±3 °C, which is much higher than the Holocene temperature variation) and in dcp do not significantly change the calcite $\delta^{13}C$. For Vil-stm1b, the theoretical calcite $\delta^{13}C$ agrees well with measured $\delta^{13}C$ for a mean temperature of 13 °C and a soil CO_2 $\delta^{13}C$ of –20.5‰, which is in the range of accepted values –20‰ to –22‰ for soil under C3 vegetation (Fritz et al. 1978; Dörr and Münnich 1986 etc.). However, for the Han-stm1b stalagmite, the calculated calcite $\delta^{13}C$ can fit with the measured values only if we use a very high soil CO_2 $\delta^{13}C$ (–17.8‰). Here, reasonable variations in dcp or in temperature do not change the calculated calcite $\delta^{13}C$ sufficiently. This suggests 2 explanations. The first, a different vegetation with a higher proportion of C4 vegetation (which produces a higher soil CO_2 $\delta^{13}C$) above the Han-sur-Lesse cave, can be rejected because C4 plants characterize semi-arid climate, which did not occur during this period in Belgium (Bastin 1990; Bastin and Gewelt 1986; Lamb 1995). The second explanation is that some process enriched the Han-stm1b in ^{13}C. These processes could include 1) evaporation, unlikely because of the high humidity in the cave (close to 100%) and also because other stable isotopes ($\delta^{18}O$) satisfy the Hendy equilibrium criteria (Hendy 1971); 2) increases in closed/open system ratio or decreases in soil pCO_2 as suggested by a recent semi-dynamic model (Dulinski and Rozanski 1990); or 3) degassing and $CaCO_3$ precipitation during seepage in the unsaturated zone (Baker et al. 1997).

Table 8 Examples of dcp and $\delta^{13}C$ variations in Holocene and modern stalagmites. On modern samples, the dcp has been calculated with the ^{14}C activity curve on modern and laminated stalagmites[a]

Sample	Location	dcp (%)	Error	$\delta^{13}C$ ‰ PDB	Error	Nr of analyses	Method	Stalagmite age	Ref[b]
Fau-stm14	La Faurie (Dordogne, SW France)	9	1.5	−10.3	0.1	21	Bomb-^{14}C curve	Modern	1
Han-stm5	Han-sur-Lesse (Belgium)	12.2	1.5	−9.4	0.1	14	Bomb-^{14}C curve	Modern	2
Pos-stm4	Postojna (Slovenia)	11.6	1.5	−9.6	0.1	11	Bomb-^{14}C curve	Modern	3
Vil-stm1	Villars (SW France)	9.4	1.5	−9.9	0.1	11	U/Th-^{14}C age comparison	3.07 ka to 0 ka	3
Han-stm1	Han-sur-Lesse (Belgium)	17.5	1.5	−6.8	0.1	135	U/Th-^{14}C age comparison	11 ka to 4.8 ka	3
BFM-Boss	Brown's Folly Mine (Great Britain)	17.5	1.5	−10.0	0.1	5	Bomb-^{14}C curve	Modern	4
SU	Sutherland (Scotland)	35.5	1.5	−10.7	0.1	5	Bomb-^{14}C curve	Modern	4

[a] Genty et al. (1998) and Genty and Massault (1999)

[b] 1=Genty and Massault (1999); 2=Genty et al. (1998); 3=This study; 4=Baker and Genty, unpublished.

Comparison with Other Published Stalagmite dcp and $\delta^{13}C$

To better understand the causes of the dcp and $\delta^{13}C$ variations, we compared our data with 5 more data points from stalagmites already studied and where the dcp has been calculated (Table 8 and Figure 2; Genty et al. 1998; Genty and Massault 1999; Baker and Genty, unpublished). For these new examples, the dcp was calculated with the ^{14}C activity curve reconstructed over the last 50 years and, when possible, with annual growth laminae (visible or luminescent). The dead carbon proportion is calculated at the pre-bomb level, as explained earlier, by comparing the atmosphere and the calcite activity around 1950 AD. Figure 2 shows the following:

- 5 samples are grouped in a $\delta^{13}C$ range between –10.3‰ and –9.3‰ PDB and a dcp range between 9% and 18%;
- 1 sample has a high $\delta^{13}C$ and a relatively low dcp (Han-stm1);
- 1 sample has a low $\delta^{13}C$ and a high dcp (SU-96-7).

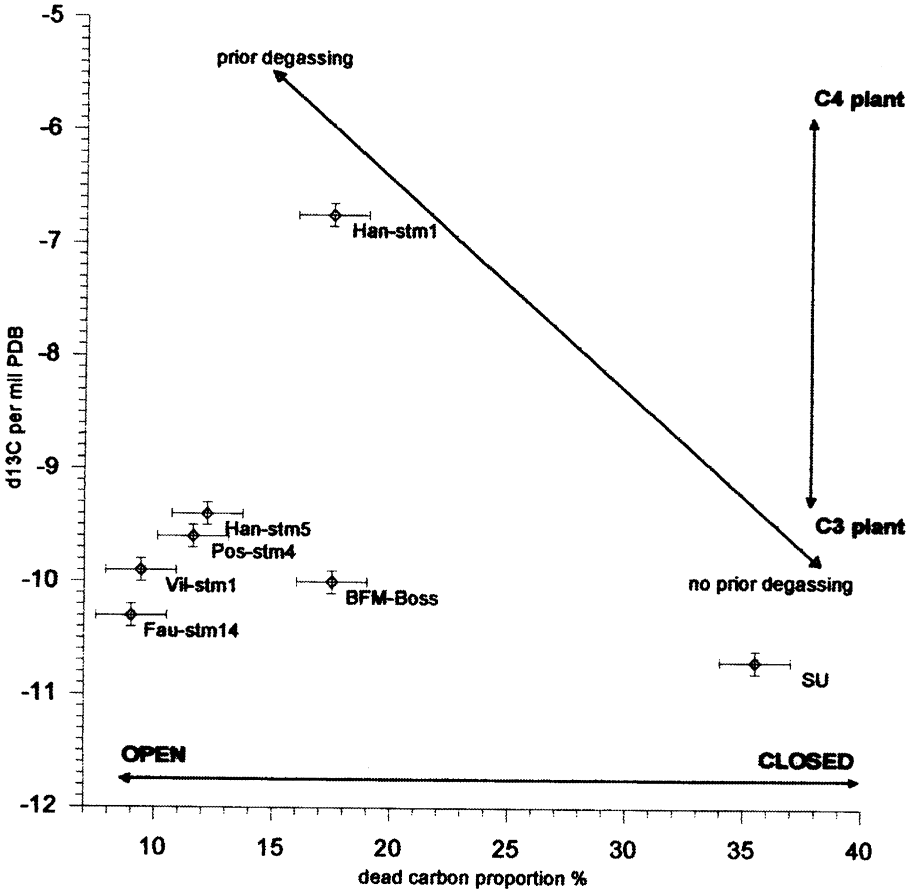

Figure 2 $\delta^{13}C$ vs. dcp; for Vil-stm1 and Han-stm1, dcp is the average of calculated past dcp (this study), for Fau-stm14, Pos-stm4, Han-stm5, BFM-Boss and SU the dcp has been calculated on modern stalagmites with the pre-bomb ^{14}C activity curve (see text, Table 8 and Genty et al. 1998; Genty and Massault 1999).

Several factors can explain the $\delta^{13}C$ and dcp values of the different samples: 1) change in the open/closed dissolution system proportion; 2) change in the type of vegetation (C3/C4) leading to a change in the soil CO_2 $\delta^{13}C$; or 3) possible degassing of the seepage water (and $CaCO_3$ precipitation) prior to entering the cave (Baker et al. 1997). The set of 5 stalagmites (Fau-stm14, BFM-Boss, Vil-stm1b, Pos-stm1 and Han-stm5) corresponds to caves that develop in limestone (Jurassic and Paleozoic). All are modern (i.e. <150 yr) except Vil-stm1b, whose mean dcp has been calculated on the last 3 ka. Soil that develops above these caves is thin (<30 cm thick), vegetation is varied and of

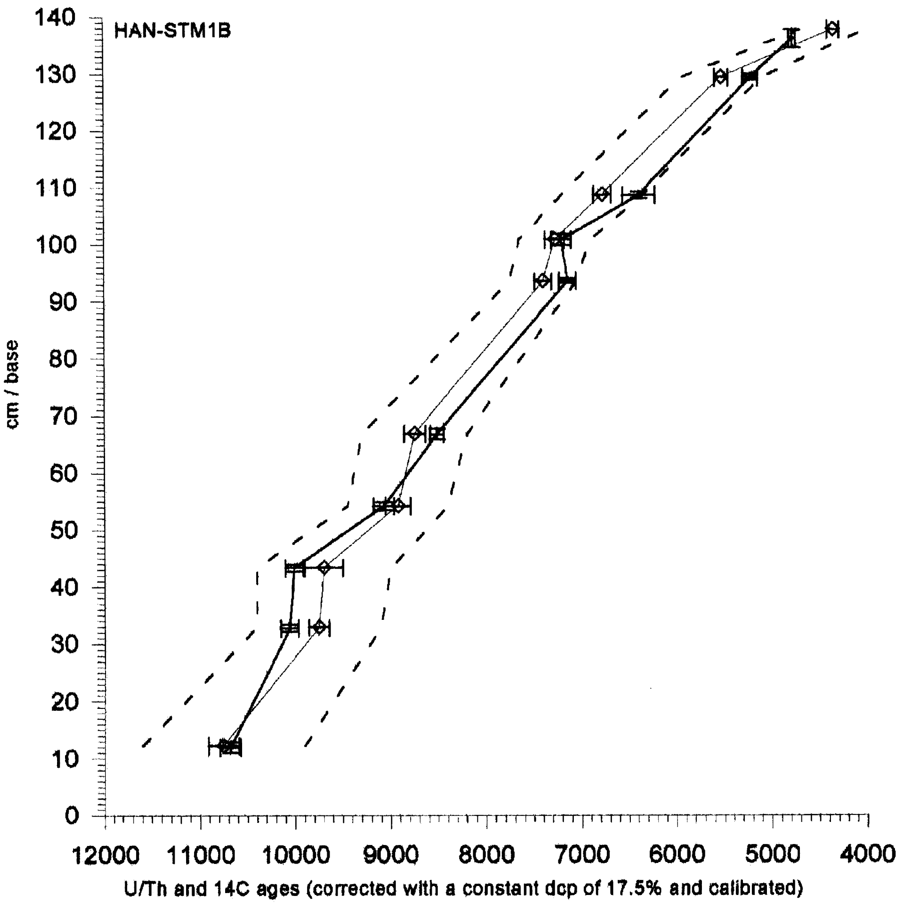

Figure 3 Growth curves of Han-stm1b stalagmite (Han-sur-Lesse Cave, Belgium). Note the very good correlation between ^{14}C (dcp corrected and calibrated ; diamonds and thin continuous line) and U/Th ages (thick continuous line). Dashed lines are error limits for ^{14}C ages, and have been calculated using an average dcp error of 1.5% and analytical errors (horizontal bars on diamonds).

the C3 type (deciduous woodlands and grasslands) and, despite slight variations in rainfall and temperature, climate is temperate and humid. Such environmental conditions seem to have favored an open-system dissolution process. The Han-stm1b sample comes from the same cave as one of the earlier samples (Han-sur-Lesse cave), but its average dcp and δ^{13}C were calculated on the first half of the Holocene. As explained earlier, the high δ^{13}C is difficult to interpret, but its dcp is in the range of the previous set. The SU-96-7 sample comes from a Scottish cave overlain by peat that develops in a dolomitic rock formation (Baker et al. 1993). The high SU-96-7 dcp value suggests that the dissolution system is almost closed. Its low δ^{13}C (–10.7‰ ±0.1) is likely the consequence of closed-system conditions and the very low soil CO_2 δ^{13}C (–29‰; Baker et al. 1999). These particular conditions are the consequence of a peat layer overlying the cave that is 60–100 cm thick and always saturated at the base; this prevents the occurrence of open-system conditions.

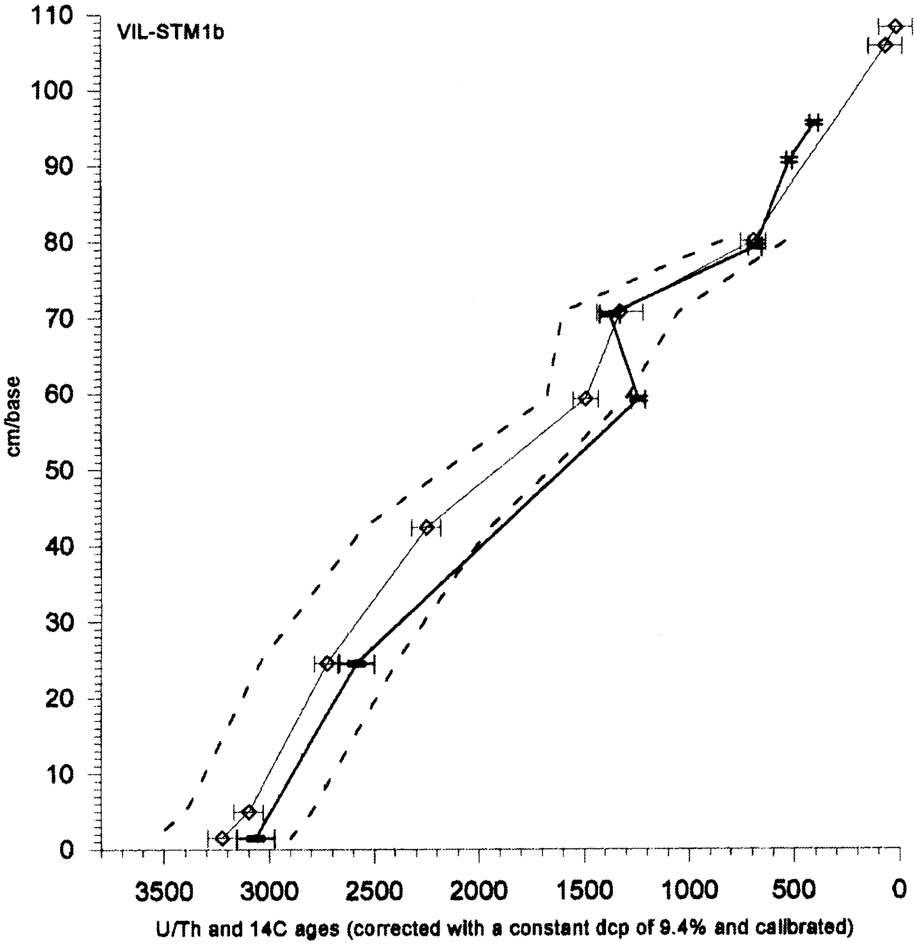

Figure 4 Growth curves of Vil-stm1b stalagmite (Villars Cave, SW France). Except 1 sample, the correlation between ^{14}C (dcp corrected and calibrated; diamonds and thin continuous line) and U/Th ages (thick continuous line) is very good. Dashed lines are error limits for ^{14}C ages, and have been calculated using an average dcp error of 1.5% and analytical errors (horizontal bars on diamonds).

Dead Carbon Proportion Time Variation

Despite the high dcp errors (Figure 1), one notes a broad parallel time variation of dcp and $\delta^{13}C$ on the Han-stm1b stalagmite. This is particularly visible between 10 ka and 7.2 ka, when dcp increases from 14.2% to 20.5% (6.3%), and $\delta^{13}C$ increases from –7.4‰ to –6.3‰ (0.9‰). These dcp and $\delta^{13}C$ variations might be the consequence of a vegetation change that occurred during this period in Belgium (Bastin 1990; Bastin and Gewelt 1986; Blanchon and Shaw 1995; Dansgaard et al. 1989; Lamb 1995), but because errors are large and because we have only one example, such a hypothesis needs confirmation.

The Vil-stm1b stalagmite shows a significant increase of $\delta^{13}C$ (3.3‰) between 760 AD and 1315 AD. This increase coincides with a slight dcp decrease that might not be significant, as it is within the error margin. We plan further measurements to try to interpret this $\delta^{13}C$ shift.

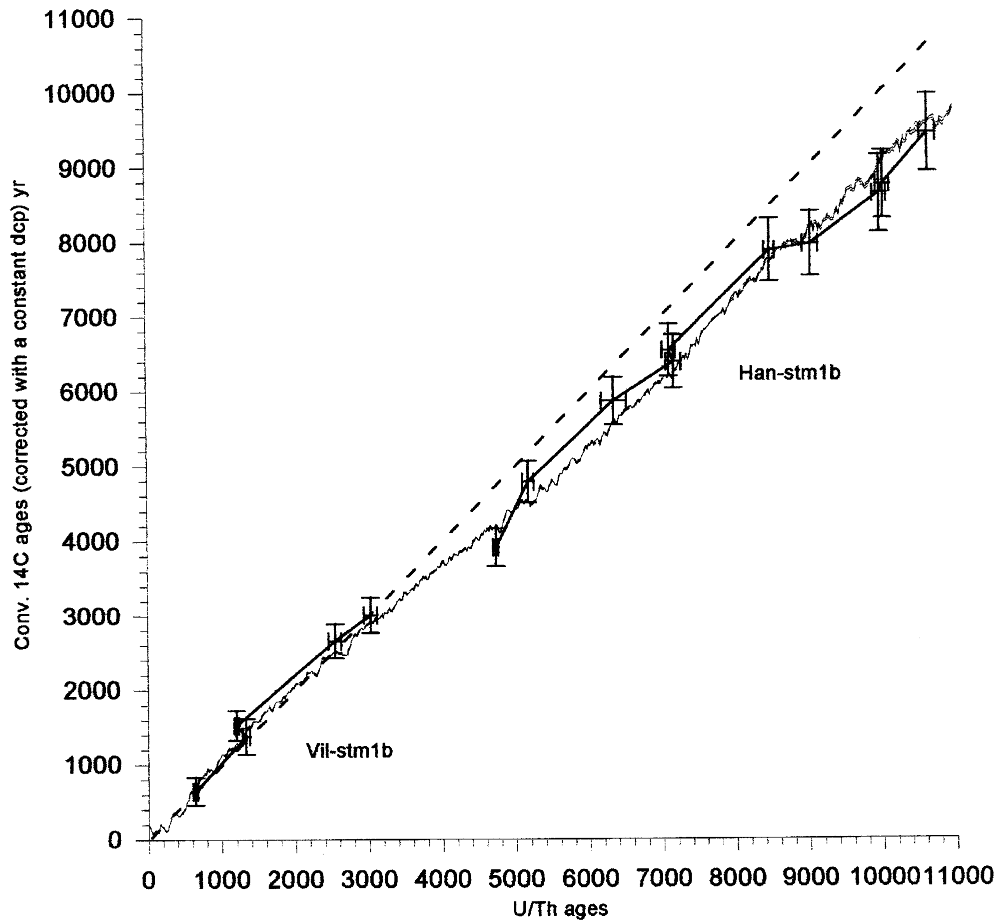

Figure 5 Comparison of conventional ^{14}C ages (dcp corrected), U/Th ages (of Han-stm1b and Vil-stm1b stalagmites), and calibration curve from dendrochronology (Stuiver and Kra 1986). Note the good fit between our measurements and the calibration curve.

Is ^{14}C AMS Suitable for Dating Speleothems?

Comparison between ^{14}C and U/Th ages on speleothems has not always produced similar results, even after dcp correction and calibration. In a Late Pleistocene stalagmite from Botswana (LII4, Lobatse II Cave), ^{14}C ages are younger than U/Th ages by 5–10 ka. This was explained by post-depositional introduction of younger ^{14}C and by increased atmospheric ^{14}C concentration (Holmgren et al. 1994). Conversely, a stalagmite from the Cracow-Wielun Upland area (Poland) showed a much older ^{14}C age (23 ka ± 0.2) than the U/Th age (18 ka ± 0.8); this discrepancy has not found a satisfactory explanation, but may be due to some unproven diagenetic processes (Pazdur et al. 1995).

We have compared here TIMS U/Th and AMS ^{14}C ages corrected by a constant dcp (average of the dcp_{past}: 17.5% for Han-stm1b, and 9.4% for Vil-stm1b). Results obtained show that except for the U/Th-F sample of the Vil-stm1b stalagmite, all TIMS U/Th ages are within the ^{14}C error margins (Figures 3 and 4). The correlation between the U/Th ages and the ^{14}C ages (dcp corrected and calibrated) is particularly good for the Han-stm1b stalagmite ($R^2 = 0.99$; $n = 10$; Figure 3). Consequently, it is tempting to say that ^{14}C AMS is a good method for dating stalagmites. However, the following problems still need to be considered:

1. To assume a constant dcp, this must be estimated by another means on a modern speleothem which can be a recent stalagmite or the top of an active one. Thus, the dcp can be calculated with the pre-bomb ^{14}C activity value;
2. If we consider a constant dcp, as above, the error on the ^{14}C age will be controlled by the error made on the modern dcp, which is about ±1.5% (Genty and Massault 1997). This leads to an age error between 250 and 500 yr for the Holocene (Tables 1 and 2). However, as we have demonstrated here, the dcp can vary by more than 6% (Figure 1), and then the real uncertainty is much higher and is dependent upon the unknown dcp_{past} variability.

Are Stalagmites Suitable for the Construction of Calibration Curves?

One of the first comparisons between these 2 dating methods (U/Th and ^{14}C) was made on a stalagmite from the Cango Cave (South Africa) and this suggested that between 30 and 40 ka BP, the level of ^{14}C in the atmosphere was higher (Vogel 1983). More recently on a stalagmite from the same site, a calibration curve (conventional ^{14}C ages vs. Uranium Series age) was established between 20 ka and 50 ka BP, which demonstrated the potential of speleothems for the reconstruction of past atmospheric ^{14}C activity (Vogel and Kronfeld 1997).

Our results show that AMS ^{14}C (dcp corrected with mean dcp_{past}) and TIMS U/Th ages fit relatively well with the calibration curve; data points are parallel to the curve within the error margin and we note that well pronounced variations of the calibration curve are followed by our data (for example the small "plateau" between 8200 and 9000 yr; Figure 5). However, for the Holocene, it appears that speleothems will not improve the accuracy of calibration curves, for at least 2 main reasons: analytical dcp error, and dcp variability.

For Holocene samples, analytical error is typically ±0.7 pMC for AMS ^{14}C, which is equivalent to an age error of ±60 yr. As explained above, the ^{14}C age error is between 250 and 500 yr (for the Holocene) if we suppose a constant dcp with an error of ±1.5%. But, we have shown here that despite the large errors made on dcp_{past} (averages are ±3.9% and ±2.8% for Vil-stm1b and Han-stm1b, respectively), it is likely that dcp has varied over time by several percent (6% in Han-stl1b stalagmite; Figure 1). Consequently, the total error must be much higher. Compared to errors made on existing calibration curves (between ±10 yr and ±60 yr for the Holocene), the error on speleothem samples is much too high for the improvement of the accuracy of such curves.

For the Glacial and Late Glacial periods, however, ^{14}C analytical errors increase (for example, between 50 yr and 490 yr in the V3 Cango stalagmite for ages <40 ka; Vogel and Kronfeld 1997) and consequently, relative dcp error decreases. For these times beyond the range of dendrochronological calibration, errors made on speleothems are comparable to errors made on other materials used for calibration curves (corals, lake sediment macrofossils; Bard et al. 1990; Kitagawa and Van der Plicht 1998). Thus, if the dcp did not change more than ±3% as observed in Han-stm1b, speleothems would be good tools for the construction of calibration curves. However, we must keep in mind that 2 important problems remain: 1) finding speleothems that grew during glacial periods, which probably means low latitude areas (Lauritzen et al. 1990, 1995); and 2) ensuring that dcp did not change significantly (i.e. more than the amount observed in Han-stm1b stalagmite), which might not be likely during climatic transitions as we know that between glacial and Holocene periods, vegetation and climatic conditions changed significantly.

CONCLUSION

This study demonstrates that:

1. Calibrated AMS ^{14}C ages, which have been corrected assuming a constant dead carbon proportion, show good agreement with TIMS U/Th ages. However, when taking into account the error estimated on the dcp, this will increase the final error on the ^{14}C age by about a factor of 3;
2. In the 2 stalagmites studied, the dead carbon proportion did not change significantly during the Holocene; we estimate 9.4% (σ=1.6; n= 6) for Vil-stm1b stalagmite and 17.5% (σ=2.4; n=10) for Han-stm1b stalagmite. The difference between the 2 stalagmites is due to differences in the limestone dissolution process, which is controlled by vegetation dynamics, climatic conditions and geological settings as demonstrated by the $\delta^{13}C$ data. Comparison with other stalagmites where dcp and $\delta^{13}C$ have been calculated demonstrate the variability and the complexity of the dissolution processes from open to closed system conditions.
3. In the Han-stm1b stalagmite, despite the broad stability observed (17.5% ±2.4%), dcp has increased by more than 6% between 10 ka and 7.2 ka and remained high until 5.2 ka. This increase is accompanied by an increase in the calcite $\delta^{13}C$ of 0.9‰. This could be the consequence of a more intense dissolution process likely due to a change in the vegetation cover; but more information is needed to confirm this hypothesis.
4. Calibrated AMS ^{14}C ages, corrected with a constant dcp, show an excellent correlation with the TIMS U/Th ages (R^2=0.99); consequently, provided that the dcp can be calculated on a modern part of a stalagmite (with the pre-bomb calcite ^{14}C activity value) and that the past dcp remains in the ±3% range variability observed in our samples, the ^{14}C AMS technique can be a good tool for the dating of the stalagmites, and despite the fact that dcp error greatly increases the error made on the age, this technique still has the advantages of simplicity, requiring very little matter, and a low price;
5. Using speleothems as a tool for establishing calibration curves is confronted by 2 problems: 1) the ^{14}C age error, which is much higher, at least for the Holocene, than the error found in the already published curves; and 2) the hypothesis of a constant dcp, which is needed to reconstruct past atmospheric ^{14}C activity, and which is unlikely, especially during climatic transitions.

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COMPARING CARBONATE AND ORGANIC AMS-^{14}C AGES IN LAKE ABIYATA SEDIMENTS (ETHIOPIA): HYDROCHEMISTRY AND PALEOENVIRONMENTAL IMPLICATIONS

Elisabeth Gibert[1] • Yves Travi[2] • Marc Massault[1] • Tesfaye Chernet[2] • Florent Barbecot[1] •
Fatima Laggoun-Defarge[3]

ABSTRACT. We studied a 12.6-m-long sequence from Lake Abiyata (Central Ethiopia) to establish a reliable and accurate chronology for use in global paleoclimatic reconstructions. The 26 accelerator mass spectrometry radiocarbon (AMS ^{14}C) ages, performed on carbonates and organic matter, define 2 parallel chronologies, representing the complete Holocene period. However, these chronologies show a significant discrepancy from 500 to 900 BP in depth; ages obtained on carbonates were always older than those on organic matter. The hydrogeological and geochemical behavior of the Lake Abiyata basin has shed light on this discrepancy. We found that the carbonate crystallization is due mainly to the mixing of lake waters with groundwaters from the multi-layered aquifer contained in the 600-m-thick basement of the lake. The ^{14}C activity of total dissolved inorganic carbon (TDIC) measured by AMS from bottom and surface lake waters (111.4 and 111.8 pMC, respectively) confirms that the mixing occurs at the water-sediment interface. This evidence of groundwater participation in the carbonate crystallization calls into question the current paleoclimatic reconstructions based on inorganic carbonates in lakes. Specific attention should thus be given to the respective proportions of each end-member in the mixing for the quantitative estimation of the groundwater input. This will help to validate the paleoenvironmental reconstructions and to highlight an eventual diagenetical evolution of inorganic carbonates during burial, via the study of pore waters.

INTRODUCTION

For several decades, efforts have concentrated on the multidisciplinary study of climatic archives in the tropics because they represent key regions in the present-day monsoonal circulation, as well as its reconstruction during the Quaternary. Despite specific and complex meteorological features such as air masses stopped eastward by the Rift Valley and the Turkana Jet, which enhances aridity (Nicholson 1996), it has been shown that long-term rainfall variations in eastern Africa are linked to quasi-global climate fluctuations, with major changes in atmospheric circulation and/or moisture balance since the Last Glacial Maximum at 18,000 BP (uncalibrated time scale; Webb et al. 1993; Rozanski et al. 1996).

Lacustrine cored sequences are some of the most useful materials for continental paleoclimate studies because they often present sensitive, continuous records of past environments with high sedimentary rates. Despite the fact that studies previously conducted on the Ziway-Shala basin (Lakes region, central Ethiopia) have focused on outcropping sections along the Bulbula River (Figure 1; Gasse and Street 1978; Street 1981; Bonnefille et al. 1986), several lacustrine transgressive/regressive phases have been registered in the basin during the Late Pleistocene-Holocene period. The first one is the Ziway-Shala IV (Z-S IV), occurring at 11,500 BP. After 10,000 BP, evidence was found for 3 major lake highstands: 1) Z-S V from 9950 to 8500 BP; 2) Z-S VI between 6500 and 4800 BP; and 3) Z-S VII, starting shortly after 2500 BP.

All these Late Pleistocene-Holocene highstands caused the 4 lakes to be connected, producing a large, unique lake (Street 1981; Figure 1). Therefore, the Ziway-Shala basin represents a unique, important basin that is highly sensitive to hydrological fluctuations, and so, is a key site for an accu-

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[1] CNRS-UMR 8616, Equipe «Hydrologie, Paléohydrologie et Paléoenvironnement», Université Paris-Sud, Bâtiment 504, 91405 Orsay cedex, France. Email: egibert@geol.u-psud.fr
[2] Laboratoire d'Hydrogéologie, Département de Géologie, Faculté des Sciences d'Avignon, 33 rue Louis Pasteur, 84000 Avignon, France
[3] Laboratoire de Géologie de la Matière Organique, Université d'Orléans, 45000 Orléans, France

rate reconstruction of Late Quaternary paleoenvironmental changes in east Africa (Gasse and Street 1978; Street 1981; Gillespie et al. 1983; Bonnefille et al. 1986; Johnson 1996).

The main problem encountered for such paleoclimatic reconstructions remains the establishment of reliable ^{14}C time scales (e.g. PAGES Report 1995; Johnson 1996; Gasse et al. 1990; Talbot and Johannessen 1993; Fontes et al. 1992, 1993, 1996; Talbot and Kelts 1996; Colman et al. 1996), especially in sites subjected to intense volcano-tectonic activity (mainly along the Wonji Fault belt; Le Turdu et al. 1999; Figure 1), and for which lacustrine deposits may have registered climatic and tectonic events simultaneously during specific phases.

Solving this problem requires the calculation of the initial ^{14}C activity of water from which authigenic materials have been formed, with at least 2 essential goals:

1. To determine a reliable, accurate chronological framework of the cored lacustrine sequence based on authigenic samples. This also requires that there be no evidence of post-sedimentary geochemical evolution. Many processes can modify, partly or completely, the original isotopic signature of such materials: (i) the disequilibrium in ^{14}C activity between atmospheric CO_2 and the total dissolved inorganic carbon (TDIC) of surface water (so-called "reservoir effect"); (ii) ^{14}C-free CO_2 inputs either along faults, or geothermal springs (sometimes dominant process in tectonically active areas) or via large aquifers connected to the lake and where radioactive decay has occurred (the so-called "hard water effect" *sensu stricto*); (iii) percolation of humic compounds or chemical exchanges with pore water after deposition; (iv) mixing between authigenic and reworked materials; and (v) coring artifacts as well as contamination during core transportation and/or analytical treatments. The first 3 propositions are related to the origin of the TDIC in lake waters (Fontes and Gasse 1991; Fontes et al. 1996). Such evolutions may influence both authigenic carbonates and organic matter of aquatic origin, and in most cases, ^{14}C dates will only represent apparent ages (e.g. Durand et al. 1984; Fontes et al. 1996). Furthermore, as described in Geyh, Schotterer and Grosjean (1998), the month-to-month evolution of the TDIC within a year of inorganic carbonate crystallization and/or CO_2 consumption during organic matter development may also induce a *temporal* discrepancy in the ^{14}C datings of such materials in $CaCO_3$-type lakes.
2. To both calibrate and ensure the validity of past environmental scenarios reconstructed via the stable isotope contents of authigenic materials such as the ^{13}C and ^{18}O contents of inorganic carbonates, or the ^{13}C and ^{15}N contents of organic matter (Gasse et al. 1991; Fontes et al. 1996; Talbot and Kelts 1996).

In this study, we present 26 AMS-^{14}C dates which were obtained on a 12.5-m-long sedimentary sequence cored in Lake Abiyata in 1994 performed on either inorganic carbonates or organic matter. The comparison of the carbonate and organic dates allows us to better understand the discrepancy observed between these 2 series in relation to the hydrological and hydrochemical processes and background that exist in Lake Abiyata.

DESCRIPTIVE BACKGROUND

Lake Abiyata (7°40′N, 38°40′E; 1500 m asl; Figure 1) is part of the Ziway-Shala basin, located in the 100-km-long tectonic closed structure related to the NNE–SSW caldera system. It is in the axial zone of the Main Ethiopian Rift. The Ziway-Shala basin is bordered to the east by the Arussi-Bale Massif and to the west by the Shewan plateau.

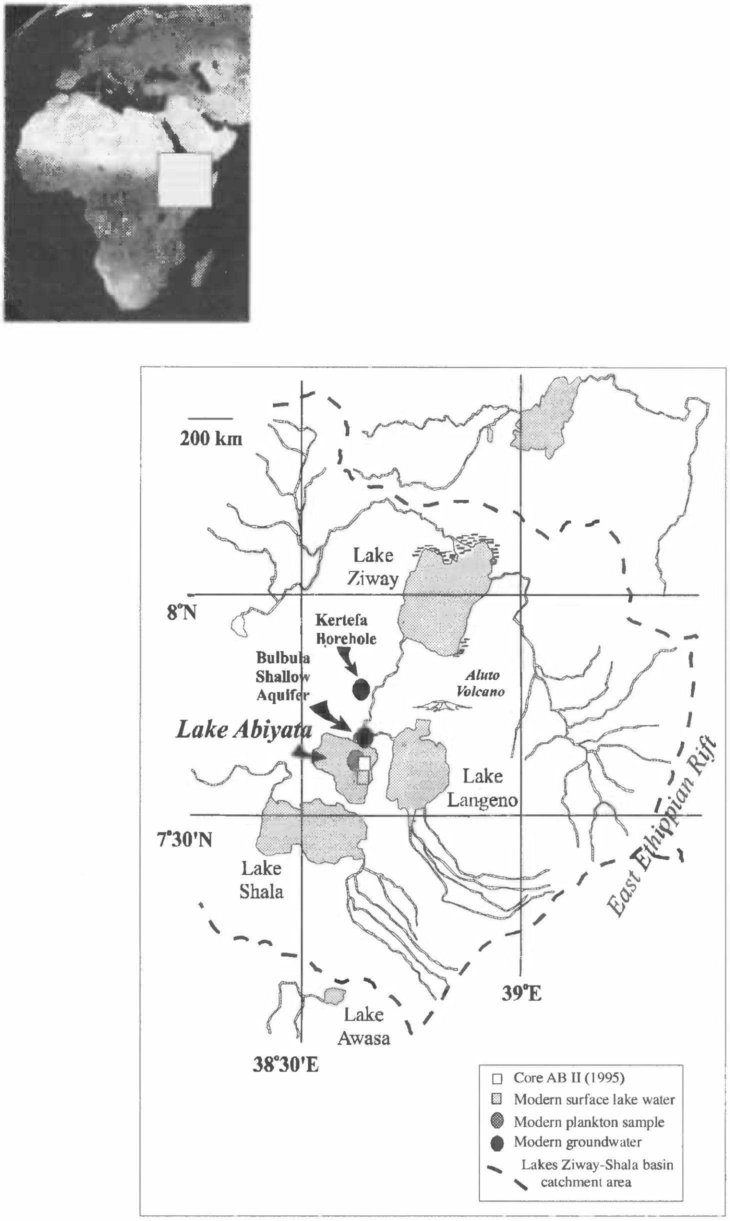

Figure 1 Lake Abiyata, East Ethiopian Rift

Lake Abiyata, which has been considered the final stage of sedimentary infilling of the basin, corresponds to a half, deep graben. Its catchment area is limited to the east by the NNE–SSW faults of the Katlo horst, and to the west by the volcanic systems linked to the Shala volcano. Its northern shore opens on the Bulbula alluvial plain where the thickness of lacustrine and alluvial deposits can reach 600 m (Le Turdu et al. 1999). The western and southwestern margins of Lake Abiyata have shown volcanic activities at 0.18 Ma. Since then, only very slight tectonic movements from the NNE fault system during the Holocene are suspected (Le Turdu et al. 1999), in agreement with the eastward migration of tectonic activity of the East African Rift. Despite the fact that intense geothermal activity is observed at the Corbetti and Aluto volcanic centers (Le Turdu et al. 1999; Figure 1), geothermal springs do not influence the Lake Abiyata basin.

Modern Hydrological System

The region has a tropical–monsoonal climate, with a mean annual precipitation of 1180 mm and a mean annual temperature of 16.3 °C (Addis-Ababa station; Figure 1; IAEA/WMO Network; Rozanski et al. 1996). The pluviometric regime is driven by the yearly oscillation of the intertropical convergence zone (ITCZ), which determines a warm-wet summer (maximum rainfall from June to September) and a dry, cold and windy winter.

A simple surface network, mainly developed upstream (around Lake Ziway), connects the 3 northern lakes of the basin via the Horakelo River from Lake Ziway to Lake Abiyata, and the Bulbula River from Lake Ziway to Lake Langeno (Figure 1). Lake Abiyata is therefore the terminal lake of the Ziway-Langeno-Abiyata subsystem, since no present-day surface connection exists between Lake Abiyata and Lake Shala.

Although Lake Ziway receives the most important input of surface waters from the whole basin, chemically speaking, it remains a highly stable reservoir (Figure 1; Chernet 1998). The majority of this inflowing water returns to the atmosphere by evaporation, while only 10% comes out as evaporated surface flows towards Lake Abiyata.

The lacustrine sedimentary basement of the Lake Abiyata basin is characterized by a multi-layered aquifer in which 2 main levels are exploited: (1) the alluvial shallow aquifer of the Bulbula Plain connected to the lake and supporting most of the local village water-supply wells; and (2) a deeper system exploited by the Soda Ash Plant. Despite some clayey lens, which can cause the deeper aquifer to be locally confined, these 2 producing levels can be considered hydraulically connected at the basin scale. Groundwater flowpaths move towards Lake Shala, which has the lowest elevation (1550 m; Chernet 1998). In that context, modern surface water of Lake Abiyata is composed of a mixture of surface water (80%) and groundwaters of the Bulbula alluvial plain (20%; Travi et al. 1997; Chernet 1998).

Under modern evaporative conditions, lake waters evolve from an alkaline to a highly alkaline type chemical facies due to the disequilibrium between HCO_3^- and concentrations of earth-alkali elements. This is in agreement with what is known about terminal lakes in ignimbrite areas (Travi et al. 1997). In such catchments, calcite saturation is quickly reached, and the resulting calcite—or magnesium calcite—precipitation leads to a very low chemical activity of calcium. Lake Abiyata modern waters are sodi-carbonate-dominated. The waters are characterized by a temperature of 22.5 °C, an electric conductivity of 25.5 mS cm^{-1}, and a pH of 10.1 (at sampling time and coring site; Figure 1). Therefore, the hyper-alkalinization process, marked and enhanced by evaporation, affirms the small content in dissolved calcium of Lake Abiyata surface waters (Table 1; $IS_{\text{Lake Abiyata surface water}} = 0.215$; PC-Wateq-4 Programme: Ball and Nordstrom 1991; AQUA Programme: Valles and de Cockborne 1992). Calcite precipitation cannot take place in such a system without any other additional source of calcium which may arise from mixing with Ca-type waters.

MATERIALS

The 12.6-m piston-cored sequence studied was taken in 1995 from the southeastern part of Lake Abiyata (core ABII) as far as possible from detrital input from former rivers (Figure 1), and under a 7-m water depth. The core consists mainly of shell-free, homogeneous clayey and organic muds,

Table 1 Chemical contents of some surface water and groundwater—Lake Abiyata basin, Ethiopia. Sampling date November 1994. See Figure 1 for sample locations. The chemical analyses in bold type correspond to the ones used for the calculation of the respective mixing proportions (see Discussion).

Sample	pH	Na^+ (mg L^{-1})	K^+ (mg L^{-1})	Ca^{2+} (mg L^{-1})	Mg^{2+} (mg L^{-1})	HCO_3^- (mg L^{-1})	CO_3^{2-} (mg L^{-1})	Cl^- (mg L^{-1})	SO_4^{2-} (mg L^{-1})	F^- (mg L^{-1})
Lake Ziway	8.6	73	18	12.9	6.8	266	11	11.1	2.4	1.4
Lake Langano	9.2	361	34	4.6	6.5	649	72	151.0	11.2	14.0
Lake Abiyata	**10.1**	**6019**	**519**	**0.35**	**1.9**	**7712**	**3500**	**2654.0**	**242.0**	**243.0**
Meki River	8.5	35	5	19.8	40.3	204	6	6.6	12.5	0.6
Bulbula borehole	8.2	297	16	12.1	91.1	966	0	17.6	8.2	18.3
Neguele Arsi Borehole	7.5	29	4	14.5	2.9	198	0	2.6	1.5	0.8
Gonde spring (ignimbrite)	7.4	8	4	8.1	3.8	40	0	2.3	2.2	0.2
Soda Ash plant	9.1	1167	11	0.6	15.7	2008	30	481.0	189.9	51.5
Kertefa borehole	**8.3**	**365**	**24**	**6.3**	**0.2**	**603**	**8**	**154.1**	**99.5**	**0.7**

Table 2 Palynofacies observations and countings (%) of selected samples from core ABII

Depth (cm)	Planktonic AOM	Algae	Zooclasts	Slightly oxidized debris	Pyro-fusinite	Preserved brownish	Gelified debris	Mushroom mycelium tissues	Pyrite
46.0	77	2	2	15	0	0	4	0	0
228.5	84	1	0	11	0.5	0	1	0	2.5
339.5	63	0	1	31	1.5	0	2.5	1	0
450.4	50	1	0	42	5	1	1	0	0
744.5	56	2	0	31	9	1	1	0	0
1036.5	50.5	2	1	27.5	7	1	3	1	6

alternating with finely (260–270, 710–720 and 770–780 cm) or coarsely (320–380 cm) laminated sandy marls (Figure 2). The increase in the number of tephra layers downwards correlates to increasing numbers of coarse sandy layers.

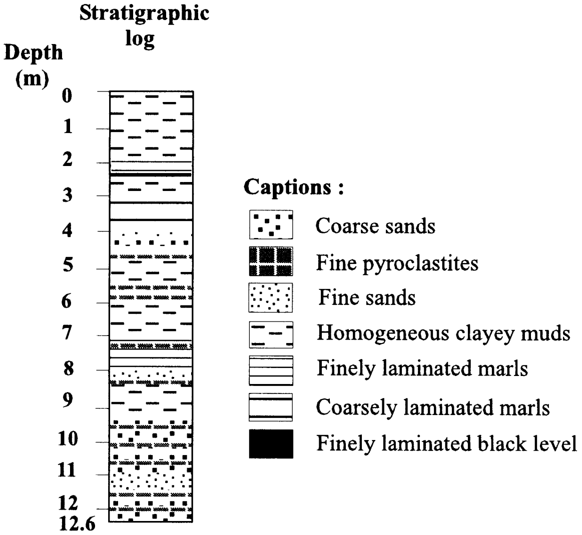

Figure 2 Core ABII, Lake Abiyata, Ethiopia (1995)

The modern sedimentation of Lake Abiyata is dominated by lacustrine organic matter due to an intense primary productivity. Its reduced catchment area and the lack of inflowing rivers preclude inputs of important detrital vegetation debris, and reduce the sedimentation of pedogenetic organic matter. These field observations have been confirmed by palynofacies analyses of organic compounds from 6 specific ^{14}C-dated levels[4] (Table 2). These levels contain mainly phytoplanktonic organic matter, with a maximum of 84% at 228.5 cm depth. On the other hand, the most common well preserved ligno-cellulosic (LC) debris, either translucent or brownish, is almost absent from the samples (<1%), showing the lack of input from emergent plants or local vegetation growing around the lake.

Gelified debris result from organic remains having been submitted to a very fast evolution toward colloid formation in water. These debris have the same origin as oxidized fragments, and reveal 1) a short residence time in soils; 2) anoxic conditions at the lake bottom; and 3) no (or very low) diagenetical effects before sedimentation. Pyrofusinite particles result from allochthonous influx and can be windblown from a far distance. Although the particles are increasing downwards and are roughly associated with the increasing numbers of ash layers, they are absent from the top of the core, likely indicating no present-day aeolian inputs to the lake.

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[4] For palynofacies observations, the total organic matter (TOM) is isolated from the mineral matrix by acidic attacks (HCl and HF acids), and mounted on slides as according the standard procedure. The OM was observed through transmitted light-microscopy for a characterization of the origin and preservation degree of the different organic components. In order to achieve countings of each organic fraction, samples were also submitted to a K-NaOH treatment to remove humic acids, and bromide-alcohol mixing for density separation.

RESULTS

Modern Samples

The selection of authigenic materials for ^{14}C dating has been based on microscopic observations of the deposits. Since no charcoal or macrophyte remains have been found, our selection has concentrated on total organic matter (TOM). Samples from sedimentary zones characterized by coarse deposits, either marls or sands, have been eliminated to the extent possible.

The ^{14}C activity of modern lake surface waters (^{14}C Act. =111.6 percent modern carbon [pMC]; $\delta^{13}C_{TDIC\ [‰\ vs.\ PDB]} = +3.5$; $\delta^{13}C_{CO2\ eq.\ [‰\ vs.\ PDB]} = -4.6$ with T = 22 °C) demonstrates the present-day equilibrium between the TDIC and the atmospheric reservoir (Levin et al. 1992, 1980). The slight difference between $\delta^{13}C_{TDIC}$ and $\delta^{13}C_{CO2\ eq.}$ is likely due to the respective ^{13}C re-equilibration rates between the pairs *primary organic matter/dissolved* ^{13}C and *dissolved* ^{13}C*/atmospheric* CO_2. In addition to palynofacies observations, which have highlighted the very short residence time of organic input from the lake catchment area, phytoplanktonic organic matter at sampling time (mid-day algal bloom) develops in equilibrium with surface lake waters (^{14}C $Act._{modern\ plankton\ [pMC]}=111.4$; $\delta^{13}C_{modern\ plankton\ [‰\ vs.\ PDB]}= -21.5$), and represents an authigenic material. We have assumed that the ^{14}C activity of present-day primary production has been constant through time. Although they represent maximum ages due to the presence of pedogenetic organic debris, these calculated ^{14}C dates from bulk samples should be very close to the true ages (very small amounts of pedogenetic remains) and thus can be considered valid.

The ^{14}C chronology of the AB-II core is based on 13 datings performed on TOM, and completed on low organic content levels with 9 datings on inorganic carbonates[5] (Table 3). Although both sets of data from both organic and carbonate components are in agreement with the stratigraphy, the 2 chronologies present a systematic and significant (although fluctuating) discrepancy. The time-lag is between 600 and 1500 yr, the ^{14}C ages defined on carbonates always being older than the ones on TOM (Tables 3 and 4; Figure 3).

We aim to examine this anomaly within the hydrological, hydrochemical, and sedimentological background of the Lake Abiyata basin. Such aging of the inorganic carbonates can only be explained through an input of ^{14}C-free/^{14}C-depleted carbon, either during precipitation or by diagenetical processes. In our case, and in contrast to what is proposed by Geyh et al. (1998) for most of the chronological interpretation of ^{14}C dates in lakes, this ^{14}C-free/^{14}C-depleted carbon input does not have to be considered constant through time. For a given level, the comparison between ^{14}C ages on TOM and ^{14}C ages on carbonates allows for the calculation of q, a coefficient that can be considered to reflect the proportion of ^{14}C-free/^{14}C-depleted carbon at that level (Table 3):

For TOM:

$$A = A_0 \cdot e^{-\lambda t} \qquad (1)$$

with A and A_0, the measured and initial ^{14}C activities of authigenic organic matter, respectively.

For inorganic carbonates:

$$A' = A'_0 \cdot e^{-\lambda t} \qquad (2)$$

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[5] The ^{14}C preparation for organic samples has consisted of a strong acid-alkali-acid (AAA) treatment, which was applied on both samples and reference blanks. For carbonated samples, the removal of the low organic content has been achieved through a weak H_2O_2-leaching.

Table 3 Measured and calibrated ^{14}C datings of sediments from core ABII

Depth (cm)	Type[a]	Sample nr (Orsay)	^{14}C activity (pMC)	Measured ^{14}C age (yr BP)	Calibrated ^{14}C ages (cal yr BP)[b]	Ref.[c]	δ^{13}C TOM (‰ vs. PDB)	δ^{13}C CaCO$_3$ (‰ vs. PDB)
7.0	TOM	H-1945	101 ± 0.8	100 ± 60	40 (270/0 [present day])	(1)		
7.0	**CaCO$_3$**	**H-1941**	**93.1 ± 0.7**	**580 ± 60**				**+2.21**
46.0	TOM	H-1372	89.8 ± 0.6	865 ± 60	740 (890/700)	(1)	−19.31	
127.1	**CaCO$_3$**	**H-1949**	**80.3 ± 0.6**	**1760 ± 60**				**+3.75**
228.5	TOM	H-1415	80.0 ± 0.9	1790 ± 70	1710 (1810/1610)	(1)		
293.0	**CaCO$_3$**	**H-1904**	**64.9 ± 0.6**	**3480 ± 70**				**+3.75**
339.5	TOM	H-1365	68.0 ± 0.7	3100 ± 60	3310 (3370/3220)	(2)	−17.33	
450.4	TOM	H-1418	64.6 ± 1.0	3510 ± 80	3775 (3880/3650)	(2)	−19.23	
551.8	**TOM**	**H-2010**	**61.3 ± 0.6**	**3900 ± 90**	**4345 (4425/4155)**	**(2)**	**−22.16**	
551.8	CaCO$_3$	H-2007	**51.1 ± 0.7**	**5400 ± 90**				
671.5	**TOM**	**H-2011**	**53.7 ± 0.6**	**4900 ± 100**	**5630 (5730/5500)**	**(1)**	**−23.75**	
671.5	**CaCO$_3$**	**H-2008**	**45.1 ± 1.5**	**6400 ± 100**				
748.5	CaCO$_3$	H-1944	48.6 ± 0.5	5800 ± 80				+2.50
794.8	TOM	H-1480	51.5 ± 0.4	5330 ± 60	6140 (6195/5995)	(1)	−24.12	
794.8	**CaCO$_3$**	**H-1906**	**47.3 ± 0.5**	**6010 ± 80**				**+2.80**
824.6	**CaCO$_3$**	**H-1902**	**43.6 ± 0.5**	**6660 ± 80**				**+2.80**
838.5	TOM	H-1481	46.2 ± 0.3	6200 ± 60	7100 (7175/7010)	(1)	−25.11	
885.1	TOM	H-1419	41.3 ± 1.0	7100 ± 80	7910 (7940/7805)	(1)	−23.54	
939.0	TOM	H-1374	37.4 ± 0.4	7900 ± 90	8625 (8950/8550)	(3, 4)	—	
941.5	**CaCO$_3$**	**H-2009**	**33.8 ± 0.9**	**8700 ± 90**				**+2.23**
984.5	TOM	H-1483	33.3 ± 0.3	8830 ± 80	9870 (9920/9660)	(5)	−25.81	
984.5	**CaCO$_3$**	**H-1903**	**29.4 ± 0.4**	**9830 ± 110**				**+2.00**
1024.0	**CaCO$_3$**	**H-1901**	**28.6 ± 0.4**	**10,050 ± 100**				**+2.00**
1036.5	TOM	H-1417	31.8 ± 1.2	9200 ± 100	10,205 (10,300/10,035)	(5)	—	
1115.5	TOM	H-1416	28.6 ± 1.3	10,050 ± 100	11,350 (11,885/11,010)	(5, 6)	−23.19	
1253.5	**CaCO$_3$**	**H-1893**	**21.7 ± 0.4**	**12,270 ± 130**				**+1.71**

[a] TOM: Total organic matter. CaCO$_3$: calcite (and low magnesium calcite). Error bars represent one sigma deviation. Accuracies on ^{13}C measurements are of ±0.05 and ±0.02‰ PDB for carbonates and organic matter, respectively.

[b] CALIB 3.0: Stuiver and Reimer (1993): 215–230.

[c] References: Calibration 1993, *Radiocarbon* 35(1), 1993. (1) Stuiver M, Pearson GW, p 1–23; (2) Pearson GW, Stuiver M, p 25–33; (3) Pearson GW, Becker B, Qua F, p 93–104; (4) Linick TW, Long A, Damon PE, Ferguson CW, p 943–53; (5) Kromer B, Becker B, p 125–35; (6) Bard E, Arnold M, Fairbanks RG, Hamelin B, p 191–9; (4) Pearson GW, Stuiver M, p 25–33.

with A' and A'_0, the measured and initial ^{14}C activities of of inorganic carbonates, respectively. The true ^{14}C age that would have to be found for inorganic carbonates is as follows:

$$A = A'_0 \cdot q \cdot e^{-\lambda t} = A_0 \cdot e^{-\lambda t} \tag{3}$$

with q, proportion of ^{14}C-free/^{14}C-depleted carbon at that level. Therefore,

$$1 = \frac{A_0 \cdot e^{-\lambda t}}{A'_0 \cdot q \cdot e^{-\lambda t}} = \frac{A_0}{A'_0 \cdot q} \tag{4}$$

$$q = \frac{A_0}{A'_0} \quad . \tag{5}$$

Consequently, q is constant for a given sample. Considering that A_o, is equal to 100 pMC (^{14}C activity BP), the initial ^{14}C activity of water in which the inorganic carbonates have precipitated is thus equal to A_o/q, i.e. 100/q (Table 4).

Mixing Processes

Based on the 6 pairs of samples analyzed (Table 4), the interpolated curve highlights that the ^{14}C-free/^{14}C-depleted carbon input fluctuates along the core (Figure 3). These fluctuations are likely related to the lake level variations in both amplitudes and origin (tectonic and/or climatic). Several hypotheses can be put forward (Figure 4):

1. *Carbonate precipitation in surface waters, without any post-sedimentary evolution.* As presented above and despite strong evaporative conditions (mean $\delta^{18}O_{\text{modern lake surface waters [‰ vs. SMOW]}}=8.5$; n=8), the crystallization of inorganic calcite could not have occurred in Lake Abiyata modern surface water due to its very low calcium content. However, during the rainy season, flooding events could have supplied the lake water with enough calcium to induce the significant precipitation of carbonates (Travi et al. 1997). This water input would have reinforced the equilibrium between the surface water TDIC and the atmospheric CO_2. This cannot account for the ^{14}C lag observed between fossil organic matter and carbonates.
   Superimposed on this eventual calcium input, the consumption of ^{13}C-depleted CO_2 by organic matter during photosynthesis (midday algal bloom occurring in the first 10 cm of the water column) would raise in the displacement of the calco-carbonic equilibria, and would also induce calcite precipitation. This process would have enhanced the precipitation of ^{13}C-enriched carbonates, the ^{13}C fractionation factor between the precipitated calcite and dissolved CO_2 being $-10.4‰$ at 25 °C. Although this seems to fit with the $\delta^{13}C$ values measured for inorganic carbonates (Table 3), the co-existing fractionation factor with respect to ^{14}C would have produced an aging of the initial ^{14}C activity of only 2 pMC (Saliège and Fontes 1983), lower than the 6.9 pMC observed for the core top (7-cm level).
2. *Early diagenetical processes at the water/sediment interface.* Although no chemical or isotopic data are available on the present-day interstitial water, CO_2 is generally released at the water/sediment interface during the very first steps of lacustrine organic matter oxidation. Following the calco-carbonic equilibria, and with no evidence of pH control by other chemical species, this increase in CO_2 concentration would immediately induce a decrease in pH, which would tend to inhibit early-diagenetical calcite precipitation. Finally, although complex dissolution/precipitation processes may occur at the surface of the newly crystallized calcite, the very low content of dissolved calcium and carbonated fraction cannot account for the observed ^{14}C discrepancies.
3. *Deep ^{14}C-free CO_2 rising along the main faults of the basin.* The ^{14}C activities of inorganic carbonates can be easily interpreted by considering the hypothesis of deep ^{14}C-free CO_2 rising along faults. On one hand, the modern chemical contents allow the calculation of the CO_2 partial pressure (pCO_2) of groundwater from the Kertefa borehole, i.e. $\sim10^{-2.88}$ (Table 1; PC-Wateq-4 Programme; Ball and Nordstrom 1991). This calculated value is higher than that of the atmosphere ($pCO_{2\ \text{atmosphere}} = 10^{-3.5}$), and does not demonstrate a deep CO_2 input in the aquifer. On the other hand, it seems very unlikely that deep CO_2 could have accounted for the post-sedimentary aging of fossil inorganic carbonates since the Lake Abiyata area constitutes the more stable part of the basin and does not present any active fault. However, the modern release of deep CO_2 observed in the deeper part of Lake Langeno (Figure 1), and greatly influencing its CITD all along the water column (EG, unpublished results), would not have modified that of Lake Abiyata even during highstands: 1) the geochemical influence of this deep CO_2 rapidly

decreases on both sides of the fault in Lake Langano; and 2) during highstands, the Lake surface/Lake deepness ratio increases, allowing for better equilibration of TDIC with atmospheric CO_2.

4. *Mixing with groundwater.* The participation of old groundwater remains the only possible source of 1) enough dissolved calcium to allow for calcite precipitation; and 2) enough ^{14}C-free carbon via the calco-carbonic system to account for the older carbonate fraction. In such a case, calcite precipitation likely occurs through the mixing of lake water and groundwater at the water/sediment interface since the 1) low groundwater flow; 2) re-equilibration of groundwater with lake water, through degassing process (although very low); and 3) difference in temperature between the 2 end-members of the mixing allow for this calcite precipitation.

The following calculation can demonstrate that the hypothesis of mixing between lake water and groundwater is valuable, although such a hypothesis would require an exhaustive study of the chemical and physical evolution of groundwater in the basin. Previous and ongoing studies (Travi et al. 1997; Chernet 1998; Chernet et al. 1999) indicate that groundwater sampled in the Kertefa borehole drilled in the central part of the Bulbula plain can be considered representative of the aquifer under the lake and is taken as the groundwater end-member.

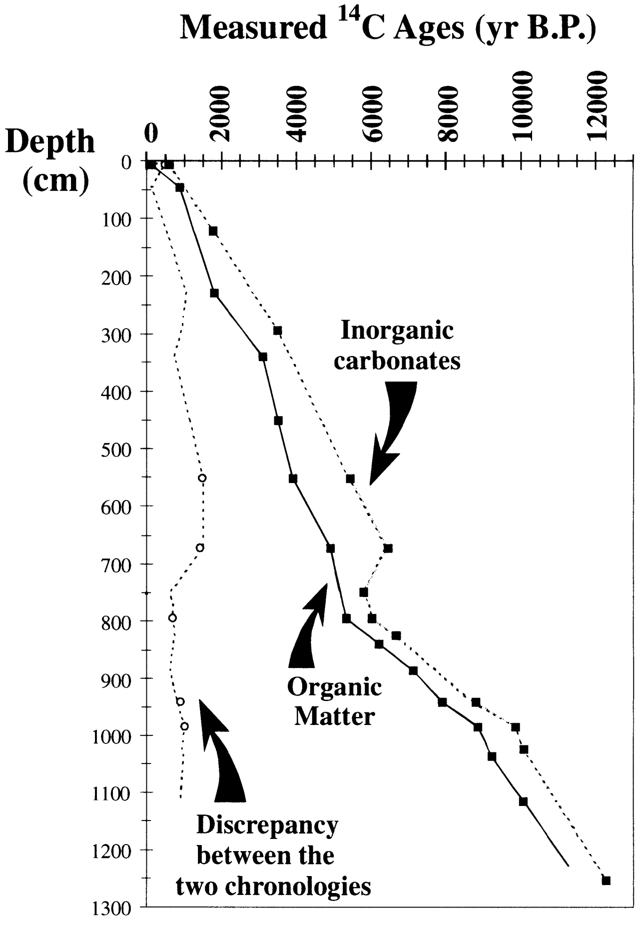

Figure 3 Radiocarbon chronology of core ABII–1995. Black and dashed curves correspond to datings on organic matter and inorganic carbonates, respectively. Empty circles represent the radiocarbon discrepancy for every pair of datings.

Table 4 Comparison of the discrepancy between ages on inorganic carbonates and organic matter at specific levels, and calculation of the dilution factor, q

Depth (cm)	^{14}C Age TOM (yr BP)	Measured activity of inorganic carbonates (pMC)	^{14}C Age CaCO$_3$ (yr BP)	Δ (yr)	Initial activity of inorganic carbonates (pMC)	q
7.0	100 ± 60	*93.1 ± 0.7*	*580 ± 60*	480	94.3	1.061
551.8	3900 ± 90	*51.1 ± 0.7*	5400 ± 90	1500	83.0	1.204
671.5	4900 ± 100	*45.1 ± 1.5*	6400 ± 100	1500	83.0	1.205
794.8	5330 ± 60	*47.3 ± 0.5*	6010 ± 80	680	91.8	1.089
939.0/941.5	7900 ± 90	33.8 ± 0.9	8700 ± 90	800	90.4	1.107
984.5	8830 ± 80	*29.4 ± 0.4*	9830 ± 110	1000	88.2	1.133

Considering that the equilibrium between the TDIC of the lake surface water and the atmospheric CO$_2$ remained constant through time, the respective parts of lake water and groundwater in the mixing can be calculated as follows:

$$N_{\text{mixed water}} = X \cdot N_{\text{POLE I}} + (1 - X) \cdot N_{\text{POLE II}} \tag{6}$$

with $N_{\text{mixed water}}$, $N_{\text{POLE I}}$, $N_{\text{POLE II}}$, the number of ^{14}C atoms in the mixed water, POLE I and POLE II, respectively; X, fraction of lake water in the mixing; 1–X, fraction of groundwater in the mixing.

On one hand, the measured ^{14}C activity of a sample expressed in pMC can be converted in true activity as follows:

$$A = \frac{^{14}\text{C Activity}_{\text{measured}} \cdot 13.56}{100} \quad \text{in dpm} \cdot \text{g}^{-1}. \tag{7}$$

On the other hand, the true activity corresponds to

$A = \lambda \cdot N$
with λ, radioactive decay constant, in mn^{-1}
and N, number of ^{14}C atoms.

From equations (6) and (7), we define the number of ^{14}C atoms of a specific sample

$$N = \frac{1}{\lambda} \cdot \frac{^{14}\text{C Activity}_{\text{measured}}}{100} \cdot 13.56 \quad \text{for 1 g of carbon.} \tag{8}$$

In order to describe the mixing between lake water and groundwater at the water sediment interface, we have to express the number of ^{14}C atoms versus 1 L of water, knowing the total dissolved inorganic carbon content of the solution (TDIC = Total Carbon = TC, in mol · L^{-1})

$$N = \frac{1}{\lambda} \cdot \frac{^{14}\text{C Activity}_{\text{measured}}}{100} \cdot 13.56 \cdot (\text{TC} \cdot 12) \quad \text{for 1 L of water} \tag{9}$$

with λ, in mn^{-1}
and 12, molar weight of carbon.

In that case, every end-member can be defined as follows:

$$N_{\text{POLE I (Lake Abiyata)}} = \frac{\left(\dfrac{A_{\text{POLE I}}}{100}\right)\cdot 13.56\cdot(TC_{\text{POLE I}}\cdot 12)}{\lambda} = \frac{(TC_{\text{POLE I}}\cdot 12)\cdot 13.56}{\lambda} \tag{10}$$

$$N_{\text{POLE II (groundwater)}} = \frac{\left(\dfrac{A_{\text{POLE II}}}{100}\right)\cdot 13.56\cdot(TC_{\text{POLE II}}\cdot 12)}{\lambda} \tag{11}$$

with
$A_{\text{POLE I}}$, measured ^{14}C activity of POLE I,
$A_{\text{POLE II}}$, measured ^{14}C activity of POLE II,
λ, in mn^{-1}.

A simple mass balance model between the 2 end-members of the mixing, i.e. lake water and groundwater, allow first for the calculation of the respective ^{14}C atoms number and total carbon content resulting mixed water. As feedback in the calculations, it then allows for the establishment of the ^{14}C activity evolution curve with respect to the respective contributions of the 2 end-members (Figure 5; Table 5).

$$^{14}C\ \text{Activity}_{\text{(inorganic carbonates)}} = \frac{100\cdot N_{\text{mixed water}}\cdot\lambda}{13.56\cdot(TC_{\text{mixed water}}\cdot 12)} \tag{12}$$

with $N_{\text{mixed water}}$, number of ^{14}C atoms per liter in the resulting water,
and $TC_{\text{mixed water}}$, Total Carbon Content (mol L^{-1}) in the resulting water.

The calculations have been realized entering the 2 extreme values of $[HCO_3^-]_{\text{Lake}}$ in the model, as deduced from the modern hydrological behavior of the basin, the modern geochemical evolution between Lakes Ziway and Abiyata having been considered to reflect the evolution of Lake Abiyata through time. These 2 values are 500 and 7000 mg L^{-1}, and represent respectively the present-day HCO_3^- concentration of Lake Ziway (*open* system end-member) and Lake Abiyata (*closed* system end-member). The buffered value of groundwater from the Kertefa borehole (groundwater end-member) has been chosen for the calculation.

Following the model, the transposition of these HCO_3^- concentrations in percentages of water volumes indicates mixing proportions compatible with the hydrological and hydrogeological *open* system, which seems to have prevailed during the Late and Middle Holocene (Table 5). Both water fluxes and total carbon concentration of the aquifer can be considered constant through time in the whole basin, and they define the basic level. Conversely, in Lake Abiyata, water inputs in the lake and the total carbon content of surface water evolve in opposite phases since a decrease in the HCO_3^- concentration corresponds to a high lake level. This would thus lead to an increase in the discrepancy between the 2 chronologies defined on carbonates and organic matter during lacustrine highstands.

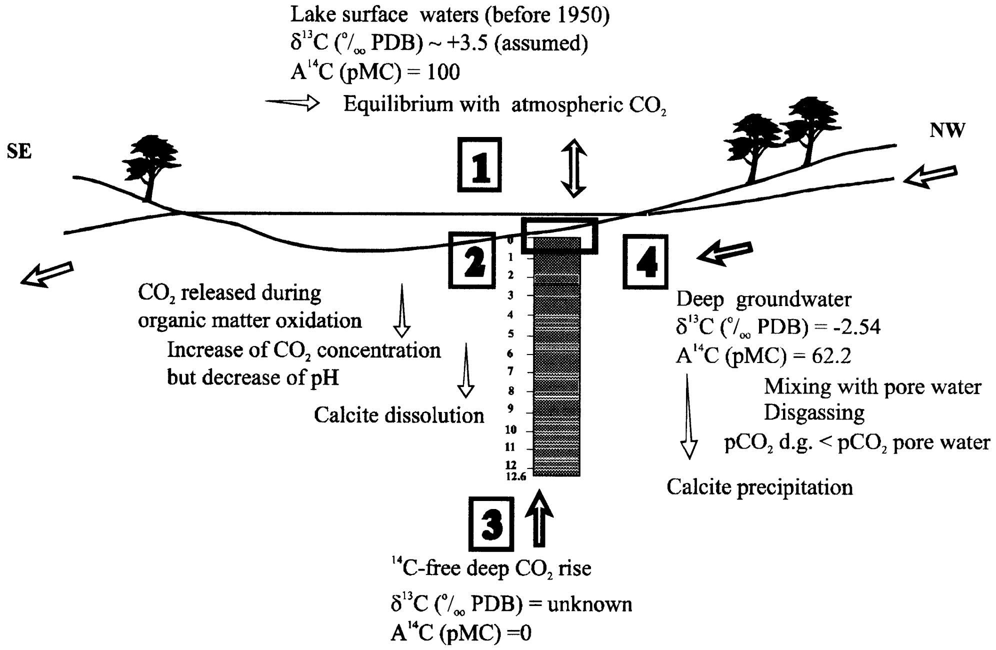

Figure 4 Processes of ^{14}C-free carbon incorporation in inorganic carbonates (Core ABII, 1995)

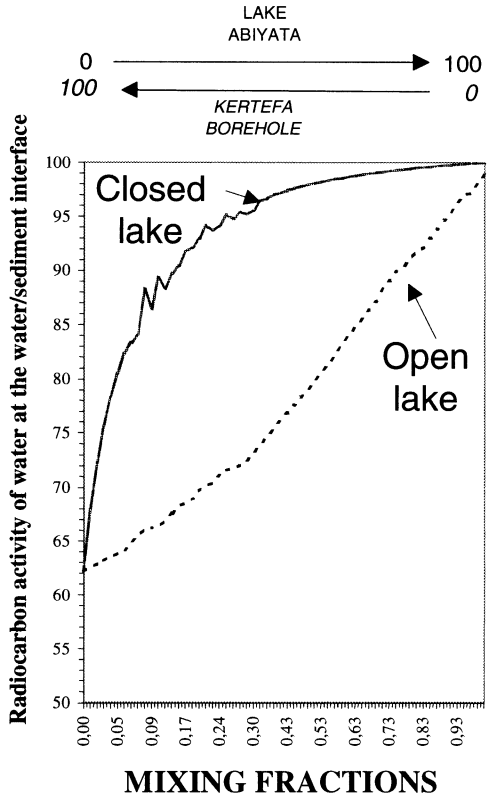

Figure 5 Mixing proportion between Lake Abiyata water and Kertefa groundwater at the water/sediment interface

Table 5 Mixing proportion between Lake Abiyata water and Kertefa groundwater at the water/sediment interface for every TOM/$CaCO_3$ ^{14}C-dated level. See Figure 1 and Table 1 for sample locations and chemical characteristics, respectively.

Depth (cm)	Measured ^{14}C Age on TOM (yr BP)	Δ (yr BP)	Mixing part (%)			
			CLOSED LAKE		OPEN LAKE	
			POLE I (Lake)	**POLE II (Groundwater)**	**POLE I (Lake)**	**POLE II (Groundwater)**
7.0	100	480	**17.7**	**82.3**	81.5	18.5
551.8	3900	1500	6.6	93.4	*59.0*	*41.0*
671.5	4900	1500	7.1	92.9	**60.1**	**39.9**
794.8	5330	680	16.7	83.3	**80.5**	**19.5**
941.5	7900	800	14.1	85.9	**77.0**	**23.0**
984.5	8830	1000	11.1	88.9	**71.8**	**28.2**

▭ *Open Lake*

⬭ *Nearing closure*

▨ *Closed Lake*

CONCLUSION

Since organic matter is considered authigenic, the understanding of the carbon isotopic anomalies related to [CO_2(aq.)] as well as the relationships between and the $CaCO_3$-CO_2-organic matter system is crucial when comparing the isotopic signatures and discriminating or confirming the authigeny of the materials dated.

The ^{14}C datings, as performed either on authigenic inorganic carbonates or on organic matter, show systematic discrepancies, ages obtained on carbonates always being older than those on organic matter. These differences are due to the geological and hydrogeological conditions and behavior of the study area. Although submitted to a very low hydraulic gradient, the presence of groundwater in the sedimentary basement of the lake brought us to consider the influence played on the crystallization and *geochemical* effects on the age of inorganic carbonates.

However, the observed discrepancy fits relatively well with the palaeohydrological phases already known on the basin (Street 1981; Gasse and Street 1978), and thus may help to validate paleoenvironmental reconstruction in the Lake Abiayta basin. The complete Holocene period is found within the first 12.6-m of core ABII. Despite a mean sedimentary rate of about 1.1 m/ka (Figure 3), which is in agreement with what is presently known about tropical lakes, the sedimentation rate is highly fluctuating: 1) 0.94 m ka^{-1} before 10 ka BP; 2) 0.43 m ka^{-1} between 10 and 4 ka BP; 3) 3.76 m ka^{-1} between 4 and 3 ka BP; and 4) 1.42 m ka^{-1} from 3 ka BP to present.

Since the ^{14}C activity of lakebottom water reaches 111.8 pMC, it likely confirms that 1) the lake waters are well mixed, and 2) the mixing with groundwater takes place at the water-sediment interface. The precise, accurate study of chemical and isotopic contents of interstitial waters might allow for the validation of such paleoenvironmental reconstructions, and the modeling of the eventual diagenetical evolution during burial.

The important groundwater input, characterized by a specific isotopic signature quite different from the one of surface waters, can make very hazardous the paleoenvironmental reconstructions currently used and based on inorganic carbonates. In the present study, the comparison of the 2 chronologies, through the calculation of the respective proportions of ^{14}C and total carbon brought by each end-member in the mixing, allows for the quantitative estimation of this groundwater input.

In future perspectives, the study of interstitial waters would help to both 1) determine the stable isotope contents of pore waters, since they must be in agreement with those of groundwaters, and as a result; 2) solve the eventual problem of early diagenetical re-crystallization during burial.

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^{14}C AGE OF GLACIATION IN ESTRECHO DE MAGALLANES–BAHÍA INÚTIL, CHILE

C J Heusser

100 Clinton Road, Tuxedo, NY 10987 USA

ABSTRACT. Basal ^{14}C dates from a core of the mire at Puerto del Hambre (53°36′21″S, 70°55′53″W), located within the area of glaciation in Estrecho de Magallanes–Bahía Inútil, Chile, are no older than 14,455 ± 115 yr BP. The ^{14}C dates are on samples from which screening isolated autochthonous plant remains. Previous ^{14}C dates of 15,800 ± 200, 16,590 ± 320, and 16,290 ± 140 yr BP are from bulk samples collected from similar basal increments at the site during 3 separate, independent coring operations. The previous suite of ^{14}C dates was suspected to be contaminated by older carbon in the light of chronological evidence, which indicates a ^{14}C age of approximately 14,850 yr BP for glaciation elsewhere in southern Chile. Contamination by "infinitely old" carbon reworked from nearby Tertiary beds and redeposited at Puerto del Hambre is evidently the cause for the older ^{14}C dates.

INTRODUCTION

Basal ^{14}C dates for the mire at Puerto del Hambre (53°36′21″S, 70°55′53″W), located about 50 km south of Punta Arenas (Figure 1), have been problematical with regard to the age of deglaciation in Estrecho de Magallanes–Bahía Inútil. The site, originally ^{14}C-dated to 15,800 ± 200 yr BP (Heusser 1984), later gave dates of 16,590 ± 320 yr BP (Porter et al. 1992) and 16,290 ± 140 yr BP (McCulloch and Bentley 1998). The original ^{14}C dating was found to be older than expected, which explains the subsequent effort to recore Puerto del Hambre to confirm its age. Other ^{14}C dates for deglaciation from within the glaciated area are between 14,260 and 13,280 yr BP (McCulloch and Bentley 1998), and deglacial ^{14}C dates relevant elsewhere in southern Chile are 14,640 yr BP at 54°52′S in Canal Beagle (Heusser 1998) and 14,355 yr BP at 46°25′S on Península de Taitao (Lumley and Switsur 1993). The glacial maximum in the Southern Lake District–Isla Chiloé (41°00′–42°30′S) is ^{14}C-dated to 14,870–14,810 yr BP (Denton et al. 1999).

Contamination by an allochthonous component serves to explain the antiquity of the deposit at Puerto del Hambre. The contaminant occurs as black, amorphous, noncrystalline, microscopic particulates without cellular differentiation, found only in basal core samples. It is believed to be carbonaceous material derived from Tertiary fossil beds that crop out in the vicinity of Estrecho de Magallanes and Bahía Inútil (Servicio Nacional de Geología y Minería 1982). The material, constituting a form of palynodebris (Boulter 1994), is part of rock flour produced by glacial abrasion, suspended in proglacial lake waters, and redeposited during the lacustrine phase of sedimentation.

An opportunity was provided in 1998 to recore the mire at Puerto del Hambre, as part of a high-resolution reconstruction of late-glacial, subantarctic paleoclimate and chronology (Heusser et al. 2000). AMS ^{14}C dates reported from the new core HE98–1C are on the remaining organic matter after removal of suspected Tertiary contaminant.

METHODS

Core HE98–1C was taken from the late-glacial portion of the deposit with a 5-cm-diameter square-rod piston sampler (Wright 1967). Increments were extruded and wrapped successively in plastic film and aluminum foil, boxed, and air-freighted directly to the Deep-Sea Sample Repository at Lamont-Doherty Earth Observatory, Palisades, New York, for archiving and storage. Cores were cut lengthwise into working and archival halves, photographed, and stored in plastic film in sealed containers under refrigeration prior to sampling. Total length of core (not including section breaks) is 262 cm at a depth below surface of between 534 and 816 cm.

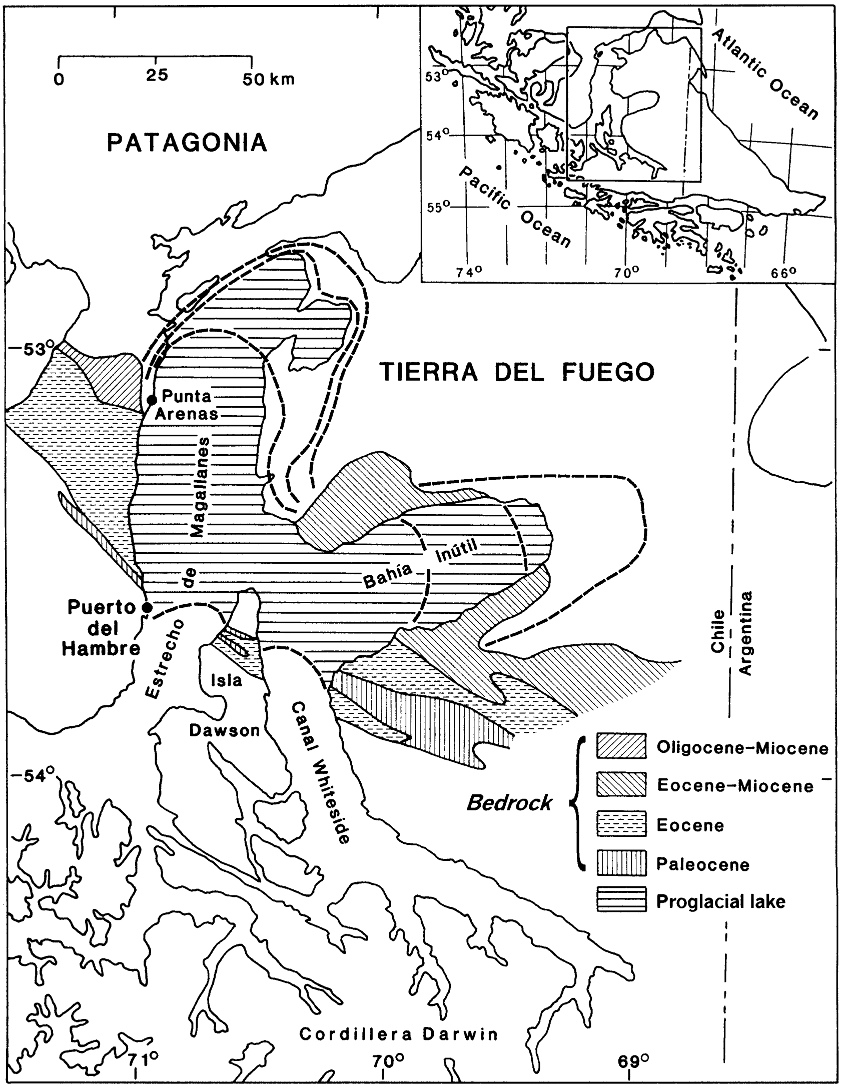

Figure 1 Location of the mire at Puerto del Hambre in southern Patagonia, subantarctic Chile. At the time of the last ice age, glaciers (– – –), whose source was largely in the Cordillera Darwin, terminated in and about Estrecho de Magallanes and Bahía Inútil (Clapperton et al. 1995). During the last glacial maximum, ice fronts extended to just beyond Punta Arenas in the Estrecho de Magallanes and at midpoint in Bahía Inútil. Following retreat of the ice, a proglacial lake occupied much of the area prior to the latest glacial advance, which appears to have culminated between 12,010 and 10,050 ^{14}C yr BP (McCulloch and Bentley 1998). Ages and locations of Tertiary bedrock are from Mapa Geológico de Chile (Servicio Nacional de Geología y Minería 1982).

Samples of the core taken at 2-cm intervals were processed for black microscopic particulates, pollen analysis, loss on ignition, and carbonate content (Berglund 1986; Faegri et al. 1989). Particulates were measured under the microscope (μm^2 gm^{-1} dry weight) from known concentrations of exotic spores added to samples during laboratory preparation.

Upon completion of processing and diagramming of results, samples from 20 horizons were selected for AMS ^{14}C dating. Deflocculation was effected by boiling in dilute KOH solution, after which suspensions were passed through 120-µm nylon screens and the black particulates measuring ≤90 µm removed in the process. Screened autochthonous material for dating was thoroughly washed with filtered water and oven-dried overnight at 105°C. Fibrous plant remains, leaf fragments, indeterminate plant detritus, and organic silt were AMS ^{14}C-dated at the NSF-Arizona AMS Facility. INTCAL98 (Stuiver et al. 1998) data were used for calibration (cal BP).

RESULTS AND DISCUSSION

Black particulate contaminant was found only in the basal 40 cm (300–340 cm) of core HE98–1C. The contaminant measured as much as 25.8 µm^2 gm^{-1} × 10^6 at depth (322 cm), but less than 1 µm^2 gm^{-1} × 10^6 at the top of the interval (Table 1). Of 7 ^{14}C AMS dates applicable to screened samples from this portion of the core, none is older than 14,455 ± 115 yr BP (332 cm). While the entire suite of dates lacks a consistent age-depth relationship, 2 other dates at 14,251 ± 91 (306 cm) and 14,204 ± 124 ^{14}C yr BP (310 cm) are similar within statistics to the date of 14,455 ± 115 ^{14}C yr BP. A much younger AMS ^{14}C date of 11,834 ± 186 yr BP is from the deepest level dated in the core (336 cm).

Figure 2 shows an age-depth plot of the 20 AMS ^{14}C dates for the screened samples from core HE98–1C. The average sedimentation rate of 16.2 ^{14}C yr cm^{-1} is based on selected ^{14}C dates of 10,089 ± 74 yr BP at 540 cm and 14,204 ± 124 yr BP at 786 cm. At the average rate, the age of the core is estimated at approximately 14,700 ^{14}C yr BP. Noticeably younger, with reference to dates closely allied to the average rate, are the variable ^{14}C dates in the lower third of the core. Variability appears to be attributable to a reservoir effect, which created a low ^{14}C/^{12}C ratio or an impoverished quantity of ^{14}C from atmospheric CO_2 versus total dissolved inorganic carbon (Olsson 1979, 1986; Geyh et al. 1998). Carbonate at Puerto del Hambre (Table 1), mostly <3.5% and not more than 8.4%,

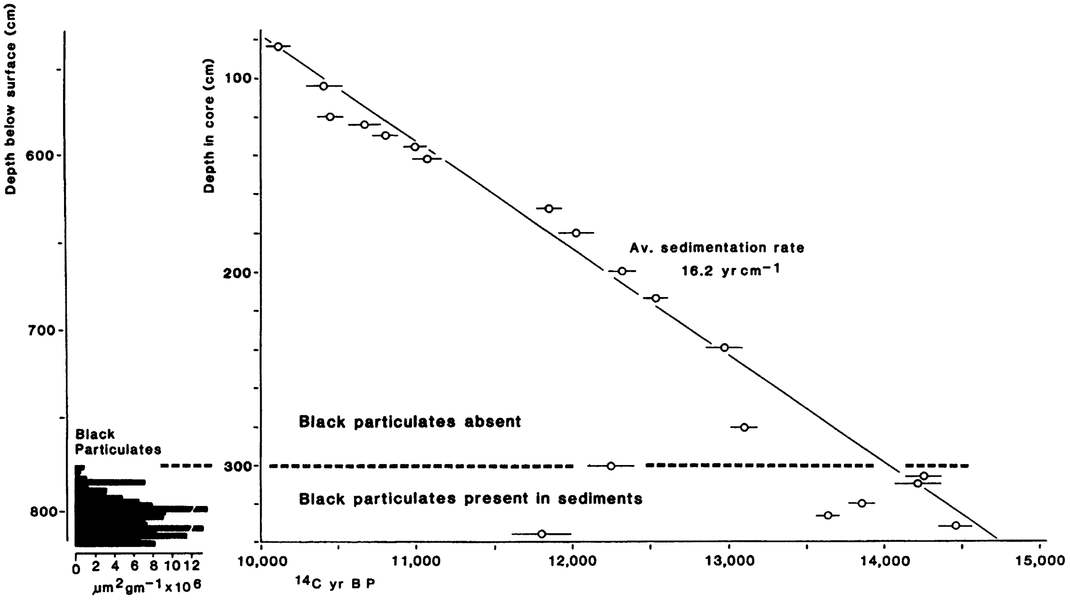

Figure 2 Age-depth plot of AMS ^{14}C dates of screened samples from core HE98-1C and distribution of black particulates in naturally occurring sediments at depth

may have contributed to the variable chronology at depth. Groundwater, a supplemental factor for the initial age-inverted ^{14}C dates, is possibly the cause of the much younger ^{14}C date of 11,834 ± 186 yr BP at 336 cm.

Among $\delta^{13}C$ values (Table 1), ranging from approximately −10.6‰ to −22.5‰, isotopic enrichment apparent in the set of samples is suggestive of low productivity, which is also implied by low loss-on-ignition measurements (2.4–7.3% at 314 cm and below; 11.4–17.0% at 300–312 cm). A C_4 pathway of carbon assimilation in the vegetation may also be involved where values are between −10‰ and −19‰ (Olsson 1986). Pessenda et al. (1998) report amounts of $\delta^{13}C$ between −15‰ and −21‰ for C_4-type, non-forested vegetation, whereas amounts for C_3-type forest communities range from −27‰ to −29.5‰. Pollen data from Puerto del Hambre (Heusser et al. 2000) imply the presence of dominant grass and heath communities in the basal part of core HE98-1C, consistent with δ^{13} enrichment from C_4-type nonarboreal vegetation.

The sequence of late-glacial pollen assemblages is consistent with previously published ^{14}C-dated pollen diagrams (Clapperton et al. 1995; Heusser 1995). Although depths differ in the respective diagrams, a consequence of coring locations on the mire, each records the presence of southern beech (*Nothofagus*) in the basal increment. The initial ^{14}C-dated assemblage containing southern beech, presumed to be a product of long-distance wind transport, is taken as the basis for both biostratigraphic and chronostratigraphic correlation.

Contamination by 50% old carbon, causing age determinations to be a half-life too old (Olsson 1986), can account for the excessive ages of unscreened bulk samples first ^{14}C-dated at Puerto del Hambre. The maximum age difference between the unscreened and screened samples amounts to >2300 ^{14}C yr. As Olsson and Eriksson (1965) indicate, removal by screening of the fine-grained matrix reduces the chance of error in assigning an age to the autochthonous component. Sources of old carbon in the case of Puerto del Hambre ^{14}C-dated samples are evidently coal beds and associated organic remains contained in regional Paleogene rock formations (Figure 1). The formations, underlying Estrecho de Magallanes-Bahía Inútil, were scoured by overriding glacial ice. Carbonaceous matter, which was in suspension when the proglacial lake formed as the ice wasted, became part of sediment deposited in the early lacustrine phase at Puerto del Hambre.

That reworking has taken place is made more convincing by the presence in the basal core sediment of palynomorphs similar to those described and figured from the local Loreto Formation of Oligocene-Miocene age by Fasola (1969). These include *Cyathidites*, *Phyllocladidites*, *Podocarpidites*, *Nothofagidites*, *Tricolpites*, and dinoflagellate cysts. The palynomorphs are readily distinguishable from similar late-glacial pollen by their differential staining, thicker and denser exines, and otherwise altered morphological features. Moreover, *Cyathidites* and *Podocarpidites* are unrelated in the existing flora, while taxa resembling *Phyllocladidites* are extinct.

CONCLUSION

^{14}C dates of 14,251 ± 91, 14,204 ± 124, and 14,455 ± 115 yr BP as maximum ages for screened samples from the basal portion of core HE98-1C at Puerto del Hambre are uniformly younger than the previous series given at 15,800 ± 200 yr BP (Heusser 1984), 16,590 ± 320 yr BP (Porter et al. 1992), and 16,290 ± 140 yr BP (McCulloch and Bentley 1998). Samples on which the ^{14}C dates were determined are from the same basal pollen zone and thus biostratigraphically correlative among cores collected.

Table 1 AMS ^{14}C dates, $\delta^{13}C$ values, and lithological data for basal 300–340 cm of core HE98-1C

Core depth (cm)	Material dated[a]	^{14}C Age (yr BP)	cal age (yr BP)[b]	$\delta^{13}C$ (PDB‰)	Laboratory number	Black particulates ($\mu m^2 gm^{-1} \times 10^6$)	Loss on ignition (%)[c]	Carbonate (%)[c]
300	Detritus silt	12,247 ± 126	14,270	−20.909	AA-30646	0.9	14.7	2.7
302						0.5	12.7	2.4
304						0.2	11.4	7.7
306	Detritus silt	14,251 ± 91	17,070	−12.001	AA-30647	1.3	17.0	2.3
308						7.3	13.8	8.4
310	Detritus silt	14,204 ± 124	17,040	−17.604	AA-30648	1.4	12.6	6.8
312						3.5	11.7	2.7
314						3.2	7.3	7.2
316						4.8	5.9	6.3
318						6.5	4.6	3.5
320	Organic silt	13,865 ± 85	16,640	−15.712	AA-30649	7.7	5.1	2.1
322						25.8	3.9	2.0
324						9.4	3.1	2.0
326	Leaf fragments	13,625 ± 80	16,360	−12.978	AA-30650	9.0	4.6	1.4
328						7.1	5.2	2.3
330						7.5	3.3	3.5
332	Organic silt	14,455 ± 115	17,320	−10.582	AA-30651	15.8	3.9	1.9
334						8.3	2.4	1.3
336	Organic silt	11,834 ± 186	13,830	−22.482	AA-30652	11.5	3.3	1.1
338						6.5	2.9	1.7
340						8.1	2.7	1.4

[a] Sample ±1 cm interval centered at core depth.

[b] From INTCAL98 extended ^{14}C calibration set (Stuiver et al. 1998).

[c] Data provided by T V Lowell.

The older series of ^{14}C dates, now considered to be unreliable, is attributed to contamination of samples by allochthonous, "infinitely old" carbon derived from nearby Tertiary deposits. With the retreat of the glacier at Puerto del Hambre, contamination occurred during the initial lacustrine phase of sedimentation, when a proglacial lake formed along the receding ice front. Redeposited Tertiary palynomorphs associated with the old carbon are similar to types found in the regional rock formation of Oligocene-Miocene age.

Glaciation of Estrecho de Magallanes–Bahía Inútil is dated close to 14,455 ± 115 ^{14}C yr BP. The event is apparently coeval with ^{14}C-dated glaciation in the Chilean Lake District–Isla Chiloé at 14,870–14,810 yr BP (Denton et al. 1999). From similar ^{14}C dates at other sites, the inference is that much of southern Chile at latitudes beyond 42°S was glaciated at this time.

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THE SPATIAL AND TEMPORAL PATTERNS OF STICK-NEST RAT MIDDENS IN AUSTRALIA

Stuart Pearson[1] • Ewan Lawson[2] • Lesley Head[3] • Lynne McCarthy[3] • John Dodson[4]

ABSTRACT. The spatial and temporal distribution of 145 radiocarbon dates on 66 Australian stick-nest rat middens (*Muridae: Leporillus* spp.) range from modern to 10,900 ± 90 BP. As in American packrat middens, age frequency follows a logarithmic decay, both continentally and at major sites. This is probably a result of natural decay processes. Unlike American middens of similar age, relatively few range changes in plant distribution have been detected in Australia. The distribution of ^{14}C ages and the associated midden materials provide important paleoenvironmental information from the arid interior of Australia. The middens record subtle changes in vegetation and dramatic changes in the fauna unlike those interpreted from sites on the coastal rim or the southeastern periphery of the arid zone.

INTRODUCTION

Stick-nest rats are in eclipse; *Leporillus apicalis* is extinct, and *Leporillus conditor* is the subject of an intensive recovery program from its refuge on Franklin Island, South Australia (Pedler and Copley 1993). However, the stick-nest rat middens have survived on the mainland and are a feature of many caves and overhangs in arid Australia. To our knowledge, the first published analysis of fossil middens in Australia is that of Green et al. (1983). The materials in the middens, and the distributions of the middens themselves, provide useful biological information about the surrounding area. This information is readily dated using radiocarbon methods (Berry 1993; Pearson and Dodson 1993; McCarthy et al. 1996). Head et al. (1998) found that a weak relationship existed between midden appearance and age, although a stronger relationship existed between appearance and the resolution of the paleoecological record in the midden. The majority of middens in Australia are found in deeply weathered rocks that erode to form shallow caves, rock shelters and overhangs along a low cliff-line or breakaway (Copley 1988). Other animals, such as rock wallabies and brush-tailed possums, may also be responsible for ancient excretory deposits in rock shelters, although the distinctive stick-nests discussed in this paper can be attributed to stick-nest rats (Pearson 1997).

From the sizeable number of conventional and accelerator mass spectrometry (AMS) ^{14}C analyses, we review the temporal distribution of the Australian midden record. Similar reviews (Mead et al. 1978; Van Devender et al. 1985; Webb 1986; Webb and Betancourt 1990) on packrat middens in the United States have helped focus subsequent efforts in North America. The North American findings include: reconstruction of vegetation distributions over the last 40,000 yr (e.g. Betancourt et al. 1990; Van Devender et al. 1990), isotopic analysis (e.g. Long et al. 1990; Pendall et al. 1999), analysis of insect communities (e.g. Elias et al. 1995), vertebrate fauna reconstructions using preserved remains (e.g. Mead and Phillips 1981; Mead et al. 1994), protein analysis on desiccated urine (Rogers and Bendich 1985; Lowenstein et al. 1991), stomatal responses to prehistoric atmospheric change (Van de Water et al. 1994), and atmospheric radionuclide production rates (Plummer et al. 1997). Many of the analyses are possible without the cost of additional fieldwork or ^{14}C dating because the material is not destroyed in analysis and much of it is stored in archives. Here we review the patterns of stick-nest rat midden dates from Australia, highlight the spatial and temporal extent of the record, and discuss new applications to the material that has already been ^{14}C dated.

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[1] School of Geography, University of New South Wales, Sydney, Australia. Present address: Department of Geography and Environmental Science, University of Newcastle, Callaghan 2308, Australia
[2] Antares AMS Physics Division (Lucas Heights), ANSTO, PMB 1 Menai, 2234, Australia
[3] School of Geosciences, University of Wollongong, Wollongong, 2522, Australia
[4] Department of Geography University of Western Australia, Stirling Highways, Nedlands, 6907, Australia

METHODS

We list all ^{14}C analyses known to us from published and unpublished work of researchers pursuing stick-nest rat midden analysis through March 1998. Sample collectors and submitters include Sandra Berry, John Dodson, Lesley Head, Lynne McCarthy, Stuart Pearson, Jay Quade, and Robert Webb. Table 1 records all known ^{14}C dates; analyses have not been subject to any exclusion criteria. For various reasons, not all the sample data we wished to review could be obtained from submitters or laboratories. A number of dates have been revised (in all cases by the laboratory). Details of samples sent to Beta Analytic Inc. were not available due to confidentiality and the destruction of their data warehouse by Hurricane Andrew in 1992.

The majority of the analyses have been done through the Australian Nuclear Science and Technology Organisation (ANSTO) using AMS. There was good analog evidence from the packrat work that this would allow rapid advances in midden analysis by identifying contamination, taphonomic processes and direct dates on special material (Van Devender et al. 1985). There is some debate about the selection of ^{14}C samples in packrat middens (see Mead and Phillips 1981). In North America most analyses have dated specific material from plants that are not found in the local area and, for the last decade, packrat scats have been dated to save other macrofossil material for analysis (Webb and Betancourt 1990).

The early conventional ^{14}C analyses from stick-nest rat middens were on bulk unwashed material and may have included mixtures of non-contemporaneous materials (Green et al. 1983; Berry 1993). Pearson (1989) used pads of finely chewed organic material (either grasses or *Acacia* phyllodes) for his conventional analyses. These layers appeared to have been accumulated quickly because they lacked a dusty coating or induration by desiccated urine or scats. Compared to larger woody debris, this finer material was also less likely to survive long on cave floors or be reworked through deposits. Samples were between 7 g and 20 g dry weight. Pretreatment by Beta Analytic Inc. included: examination for contamination such as rootlets, acid soaking to remove carbonates, rinsing to neutrality, drying, and combustion in an enclosed line before benzene synthesis and counting.

Pearson (1997) used AMS on the scats to date the rodent activity more directly than the analysis of other materials that may have been recycled from elsewhere. The usual protocol was for 1 scat (about 1 g) to be separated for ^{14}C, and 5 other scats, closely associated in the matrix of the dated sample, were prepared for pollen analyses. This provided the closest context for interpreting the pollen record. Pretreatment of the (AMS) ^{14}C samples was done at the ANSTO ANTARES Facility. Other material was pretreated at the NWG Macintosh Centre for Quaternary Dating at the University of Sydney, the NSF–Arizona AMS Laboratory at the University of Arizona, and at Beta Analytic Inc.

The facilities at Beta Analytic Inc. (liquid scintillation counters) and ANSTO report machine background contamination levels that would only affect samples older than about 40,000 yr. The correction due to contamination from combustion and graphitization was that associated with 2.5 μg modern carbon equivalent. ^{14}C ages reported here are determined using the NIST Oxalic Acid I (HOx1) standard. Counting errors are quoted to $\pm 1\sigma$. The estimation of the δ^{13}C correction ratio for samples was left to the laboratory and measured δ^{13}C corrections are not yet available for many samples. The estimated normalized δ^{13}C correction ranged from $-12‰$ to $-27‰$ and reflected the expectation that the material dated was isotopically similar to wood. The measured corrections that are available ($n=22$) are within this range ($-24.7 \pm 2.1‰$). The ratio of C-3 to C-4 pathway plants in a sample may significantly affect the δ^{13}C correction. However, these differences in corrections could, at worst, result in a 60-yr change in the age result within the ranges of the age determinations.

We separated the dates on specific parts of stick-nest rat middens ("selected dates") from the population of ^{14}C analyses ("all dates") in order to show the different distribution of the ^{14}C samples and the middens. Webb and Betancourt (1990) analyzed the patterns of all ^{14}C analyses because selection of one date to represent a midden was intractable. However, with the analysis of stick-nest rat midden dates, we have been able to select the oldest and youngest dates available from a midden. This corrects the number of repeated dates on a few middens and provides information about initiation construction and abandonment of a specific midden. We will refer to these 3 groups of dates as "all dates", "selected maximal dates" and "selected minimal dates".

Organic material found in the midden is certain to predate, and possibly postdate, the construction of the midden. The history of ^{14}C in samples from middens must be carefully considered on an individual basis (Pearson 1999) and is not considered in this summary. Amberat is a black matrix of desiccated urine, including phytoliths (Bowdery 1998), pollen and other midden debris. Presumably it contains a range of organic material from many sources; contaminates may make samples appear older. Instances of younger contaminates entering the midden have been recognized (Pearson 1997; Pearson et al. 1999) and this demands careful consideration of each sample, and greater understanding of the taphonomy of the deposits (Pearson 1999). The only sampling bias we are aware of is that Stuart Pearson avoided middens with no induration because they are likely to be contaminated and tend to disintegrate in sampling (Pearson 1989)—this may have resulted in relatively modern middens being under-represented in his samples.

We subtracted 24 yr from the dates older than 100 BP as the Southern Hemispheric offset, assuming the offset is unimportant after AD 1850 (Stuiver et al. 1998). We used CALIB 4.0 (Stuiver and Reimer 1993, 1998) to calibrate each ^{14}C date with the 1998 atmospheric decadal dataset (Stuiver et al. 1998) and created the 1σ probabilities of each date. This was done in 2 batches to resolve the data-entry limitations of Calib 4.0. The probabilities were summed to show the maximum amount of data from the ^{14}C analyses. We did not use a moving average in the calibration curve because the material sampled spanned an unknown period. The probabilities of each ^{14}C estimate were summed into 10-yr categories then exported into Excel® for graphing.

RESULTS

The spatial distribution of stick-nest rat middens is described in Copley (1988). The maximum likely midden extent follows the subfossil distribution of stick-nest rats in Australia (Strahan 1995). Middens recently found in Queensland (G Medlin and C Dollery, personal communication 1998) extend the range of fossil middens. The distribution shows the effects of an extensive search effort across central Australia and a concurrent intense effort in the Flinders Ranges and western New South Wales.

Table 1 lists all the ^{14}C analyses on material from stick-nest rat middens. Spreadsheet versions are available from the corresponding author—empty cells indicate unavailable information. The temporal distribution of all calibrated ^{14}C ages is shown on Figure 1; Figure 2 shows the selected samples that show the minimal and maximal ages on middens, and Figure 3 shows the spatial distribution of study sites and ^{14}C results. Figure 4 shows the spatial distribution of all ^{14}C ages during 4 periods in Australia. The oldest Flinders Ranges dates, 10,000–8000 BP, are based on 2 middens, SAJQ85 (dated 8 times) and SAJQ79 (dated once). Nine Flinders Ranges middens dated older than 5000 BP. Figure 5 shows the summed probability distributions from the maximal and minimal ^{14}C dates for 4 sites. These sites were graphed because a number of dates were available. At Umbeara Station and Gap Range all the middens found were dated. Figure 5 shows the relation between both initial construction dates (maximal ^{14}C dates) and abandonment (minimal) dates in a variety of locations.

Table 1 Radiocarbon analyses on material in stick-nest rat middens

^{14}C age (yr BP ± 1 σ)	[a]	Lab code	Material used	δ^{13}C[b]	[c]
10,900 ± 90	A	OZA-318	Scats	−25.0	max
10,870 ± 80	C	ANU-8109	Plant & scat		
10,870 ± 80		BETA-67214	Plant	−25.0	
10,630 ± 100	C	ANU-8138	Plant	−24	
10,550 ± 460	A	OZA-317	Plant	−24.0	
9473 ± 319	A	OZA-319	Plant	−25.0	
9420 ± 80	C	ANU-8110	Plant & scat	*−20.0*	
9060 ± 70	C	ANU-8137	Amberat	−27.0	
8820 ± 150	C	ANU-7754	Plant	−24.0	
7720 ± 90	A	BETA-81092	Scat		max
7400 ± 90	A	BETA-8108	Scat		
7310 ± 90	A	BETA-81090	Scat		max
7260 ± 100	A	OZC-6152	Scat		only
7200 ± 220	A	OZA-316	Scat	−25.0	max
6930 ± 80	A	BETA-81091	Scat		min
6920 ± 50	A	BETA-87302	Leaf		
6880 ± 50	A	BETA-87301	Leaf		
6800 ± 70	A	OZB-292	Plant		min
6710 ± 80	A	OZB-291	Plant		
6650 ± 80	A	OZB-297	Plant		
6610 ± 90	A	OZB-296	Scat		
6580 ± 60	A	BETA-87300	Leaf		
6520 ± 50	A	BETA-87299	Leaf		
6520 ± 50	A	BETA-91808	Plant		
6472 ± 163	A	OZA-31?	Leaf	−27.0	
6470 ± 60	A	BETA-91809	Scats		max
6380 ± 70	C	ANU-8136	Plant	−12.0	
6380 ± 70	C	ANU-8138	Amberat		min
6360 ± 100	C	BETA-89611	Plant		
6360 ± 100	C	BETA-89613	Plant		
6290 ± 190	A	OZB-295	Plant		
5790 ± 80	A	OZC-047	Scat	−26.5	
5710 ± 90	C	BETA-89609	Plant		
5690 ± 70	A	OZB-294	Scat		
5540 ± 110	A	OZA-315	Plant	−27.0	min
5340 ± 70	C	ANU-8139	Plant	−25.0	only
5340 ± 90	C	BETA-91810	Plant		
5240 ± 90	C	BETA-91807	Plant		
5020 ± 70	A	OZC-049	Scat	−27.8	only
4350 ± 60	A	OZC-046	Scat	−24.2	max
3790 ± 90	A	OZB-300	Scat		
3690 ± 180	A	OZB-298	Plant		max
3670 ± 70	A	OZC-6172	Scat		max
3640 ± 70	A	OZB-293	Scat		

Table 1 Radiocarbon analyses on material in stick-nest rat middens *(Continued)*

^{14}C age (yr BP ± 1 σ)	a	Lab code	Material used	δ^{13}C^{b}	c
3570 ± 120	A	OZB-299	Plant		min
3500 ± 90	C	BETA-23022	Plant		max
3450 ± 70	A	OZA-189	Amberat	–23.8	max
3430 ± 70	A	BETA-89612	Plant		
3430 ± 50	A	OZB-168	Amberat	–25.0	only
3350 ± 80	A	OZA-1882	Scat	–25.0	
3330 ± 190	A	OZA-190	Plant	–24.4	
3310 ± 80	A	OZA-1881	Scat	–25.0	min
3040 ± 70	C	BETA-89606	Amberat		max
2960 ± 100	C	BETA-97075	Amberat		
2950 ± 200	A	OZA-309		*–25.0*	
2780 ± 100	C	A-3990	Plant		max
2680 ± 60	C	BETA-80610	Amberat		max
2430 ± 70	A	OZA-308	Plant	–23.6	
2430 ± 80	A	OZC-618	Scat		min
2400 ± 290	A	OZA-980	Scat	–25.0	
2380 ± 40	A	OZA-963	Plant	–25.0	max
2330 ± 120	A	OZA-187	Plant	–27.0	max
2280 ± 120	A	OZA-186	Scat	–25.0	
1950 ± 70	C	BETA-67212	Plant	–25.0	max
1920 ± 110	C	ANU-7753	Plant	*–24.0*	only
1840 ± 100	C	ANU-2385	Charcoal		
1820 ± 40	A	OZA-978	Wood	–25.0	max
1720 ± 60	A	OZA-969	Scat	–25.0	only
1700 ± 90	C	BETA-23023	Plant		min
1660 ± 100	C	A-3989	Plant		min
1610 ± 50	A	OZA-975	Scat	–25.0	max
1600 ± 50	A	OZA-310		–25.0	max
1540 ± 110	A	OZA-181	Plant	–27.0	
1530 ± 50	A	OZA-182	Scat	–25.0	
1480 ± 140	C	BETA-30954	Plant	–25.0	only
1470 ± 70	C	BETA-76575	Plant	–25.0	only
1430 ± 50	A	OZA976	Scat	–25.0	max
1380 ± 60	A	BETA-91811	Scats		min
1310 ± 60	A	OZA964	Bark	–25.0	only
1300 ± 80	C	ANU8140	Plant	–27	max
1300 ± 40	A	OZA312	Wood		
1280 ± 70	A	OZC620	Scat		
1150 ± 100	C	BETA-30956	Plant	–25.0	max
1150 ± 50	A	OZA-962	Scat	–25.0	
1140 ± 40	A	OZA-967	Scat	–25.0	only
1130 ± 60	A	OZA-183	Scat	–21.3	max
1120 ± 50	A	OZA-974	Scat	–25.0	min
1000 ± 70		BETA-62204	Plant	–25.0	only

Table 1 Radiocarbon analyses on material in stick-nest rat middens *(Continued)*

^{14}C age (yr BP ± 1 σ)	a	Lab code	Material used	$\delta^{13}C^{b}$	c
1000 ± 70	A	OZA-313	Wood	−24.6	
961 ± 145	A	OZA-311	Plant	−24.6	min
940 ± 60	A	OZA-184	Plant	−27.0	min
900 ± 90	C	BETA-30957	Scat	−25.0	max
900 ± 45	A	OZA-167	Scat	−25.0	min
880 ± 75	A	OZC-616	Plant		only
880 ± 150	A	OZC-6211	Scat		
800 ± 80	C	BETA-68230	Plant	−25.0	max
780 ± 80	C	BETA-89607	Wood		only
770 ± 70	A	OZC-041	Scat	−27.2	max
710 ± 80	C	BETA-30955	Plant	−25.0	min
700 ± 95	A	OZA-977	Scat	−25.0	min
630 ± 100		BETA-62205	Plant	−25.0	min
620 ± 55	A	OZC-044	Plant	−27.5	max
610 ± 40	A	OZA-973	Scat	−25.0	
590 ± 55	C	BETA-76571	Plant	−25.0	max
590 ± 45	A	OZC-040	Scat		min
590 ± 50	A	OZC-623	Plant		
560 ± 60	A	OZC-048	Scat	−19.4	min
530 ± 80	C	BETA-86307	Wood		
530 ± 70	A	OZA-185	Scat	−25.8	min
510 ± 70	C	BETA-59079	Plant		
510 ± 50	A	OZA-966	Scat	−25.0	only
510 ± 70	A	OZA-979	Scat	−25.0	min
470 ± 65	A	OZC-045	Scat	−24.1	min
470 ± 130	A	OZC-619	Scat		only
460 ± 90	C	BETA-59080	Plant		
450 ± 60	A	OZC-043	Plant	−26.3	max
430 ± 35	A	OZB-164	Scat	−25.0	only
420 ± 50	A	OZC-042	Plant	−27.4	min
410 ± 165	A	OZA-192	Plant	−27.0	max
410 ± 40	A	OZA-314	Scat	−25.0	max
410 ± 65	A	OZC-625	Scat		
385 ± 140	A	OZC-614	Plant		
380 ± 60	C	BETA-89608	Amberat		only
360 ± 50	A	OZA965	Plant	−25.0	
360 ± 75	A	OZB165	Scat	−25.0	only
340 ± 40	A	OZA968	Scat	−25.0	min
340 ± 55	A	OZB166	Scat	−25.0	only
310 ± 70	C	BETA-59078	Plant		max
310 ± 40	A	OZA-961	Scat	−25.0	only
300 ± 90	C	BETA-30958	Plant	−25.0	min
300 ± 50	A	OZA970	Scat	−25.0	only
280 ± 40	A	BETA-86305	Wood		only

Table 1 Radiocarbon analyses on material in stick-nest rat middens *(Continued)*

^{14}C age (yr BP ± 1 σ)	[a]	Lab code	Material used	δ^{13}C[b]	[c]
270 ± 40	A	BETA-86306	Scats		min
250 ± 60	C	BETA-76573	Plant	–25.0	
230 ± 105	A	OZC-6221	Scat		
220 ± 70	C	BETA-59077	Wood		min
200 ± 55	A	OZA-972	Scat	–25.0	min
170 ± 60	C	BETA-76577	Plant	–25.0	
80 ± 50	A	OZA-968	Plant	–25.0	
80 ± 50	C	BETA-76576	Plant	–25.0	min
20 ± 80	C	BETA-68231	Plant	–25.0	min
104.34 pMC ± 0.7%	A	OZC-624	Seeds		
101.1 pMC ± 0.7%	C	BETA-76574	Plant	–25.0	min
101.55 pMC ± 0.4%	A	OZA-191	Skin	–21.8	
See text	A		Scat	–25.0	

[a] AMS or conventional method

[b] Corrections that are estimated rather than measured are shown in italics.

[c] Interpreted as **min**imal, **only** or **max**imal age (see text)

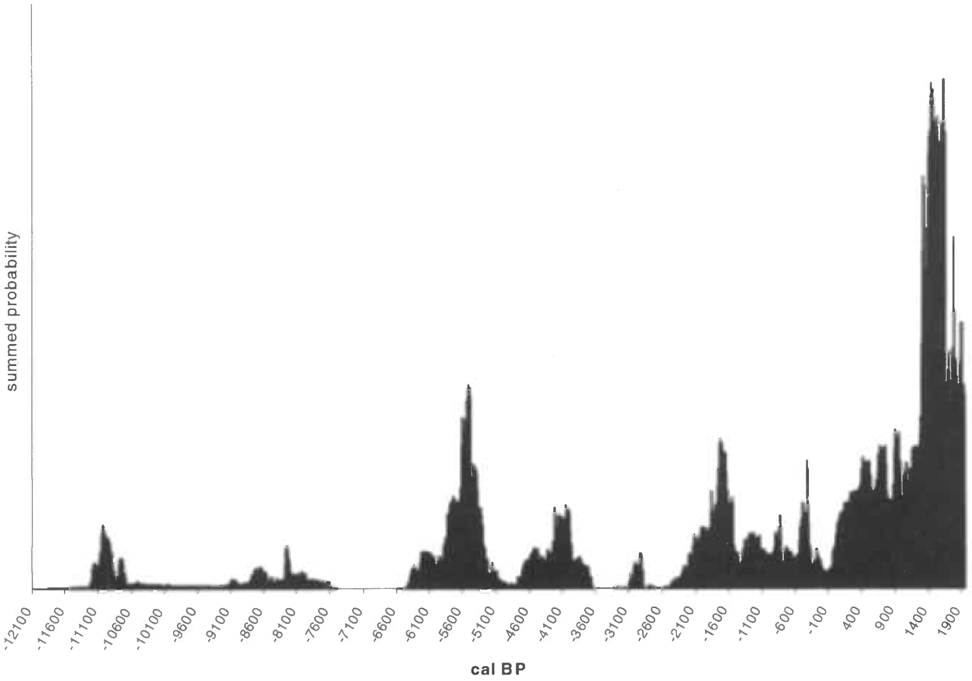


Figure 1 Distribution of calibrated dates on stick-nest rat middens

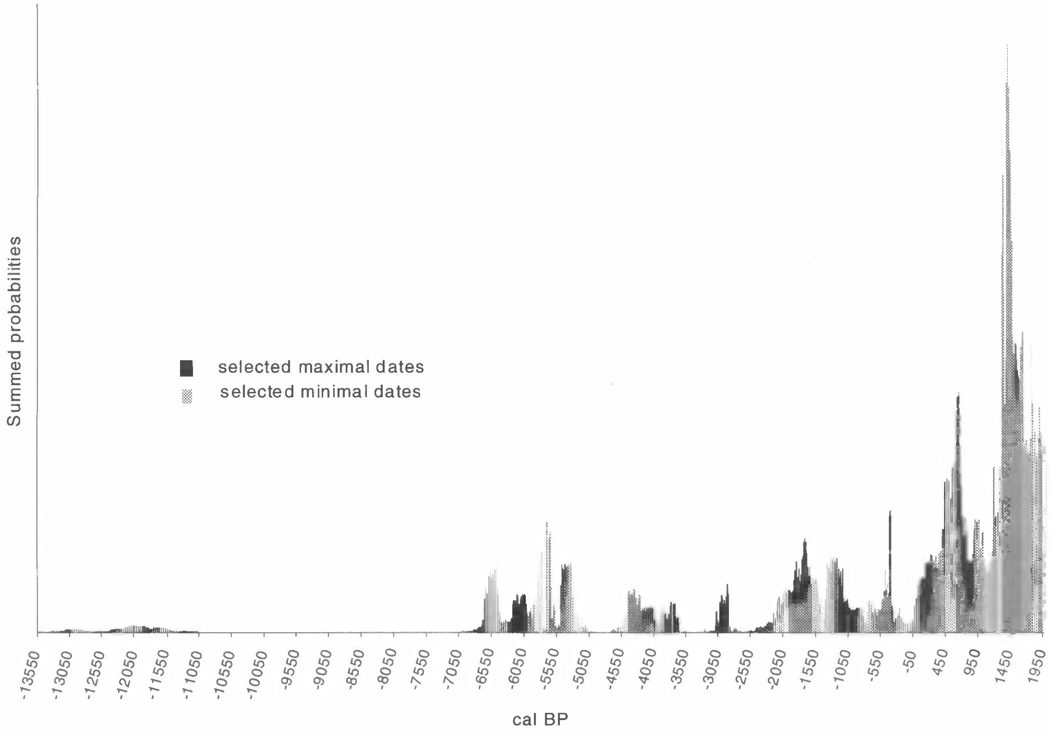
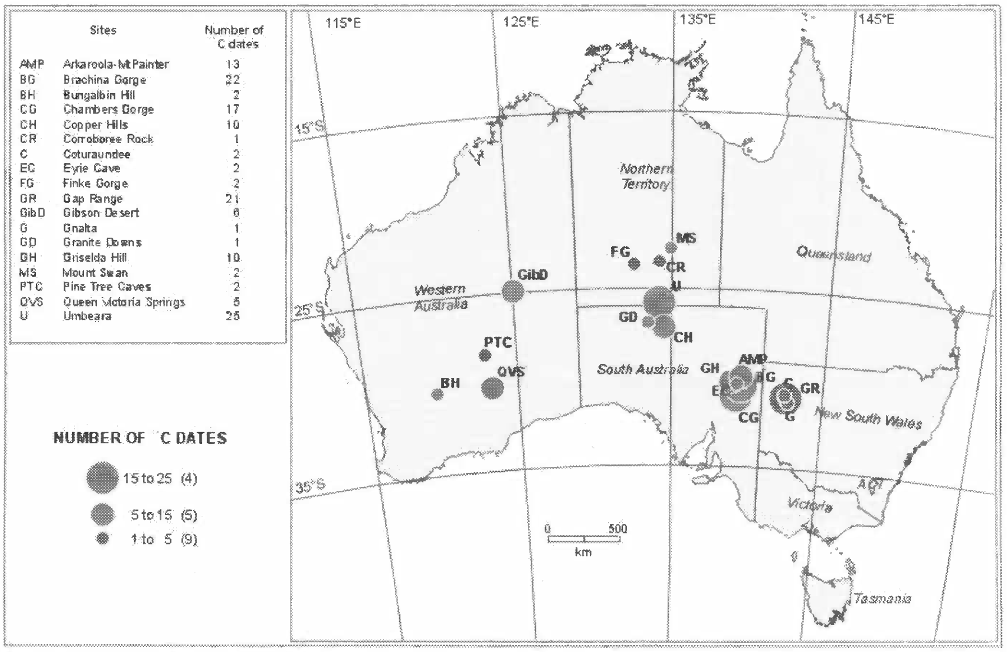


Figure 2 Distribution of maximal dates and minimal dates on middens



	Sites	Number of C dates
AMP	Arkaroola-Mt Painter	13
BG	Brachina Gorge	22
BH	Bungalbin Hill	2
CG	Chambers Gorge	17
CH	Copper Hills	10
CR	Corroboree Rock	1
C	Coturaundee	2
EC	Eyrie Cave	2
FG	Finke Gorge	2
GR	Gap Range	21
GibD	Gibson Desert	6
G	Gnalta	1
GD	Granite Downs	1
GH	Griselda Hill	10
MS	Mount Swan	2
PTC	Pine Tree Caves	2
QVS	Queen Victoria Springs	5
U	Umbeara	25

Figure 3 Map showing the distribution of stick-nest rat midden study sites and radiocarbon dates

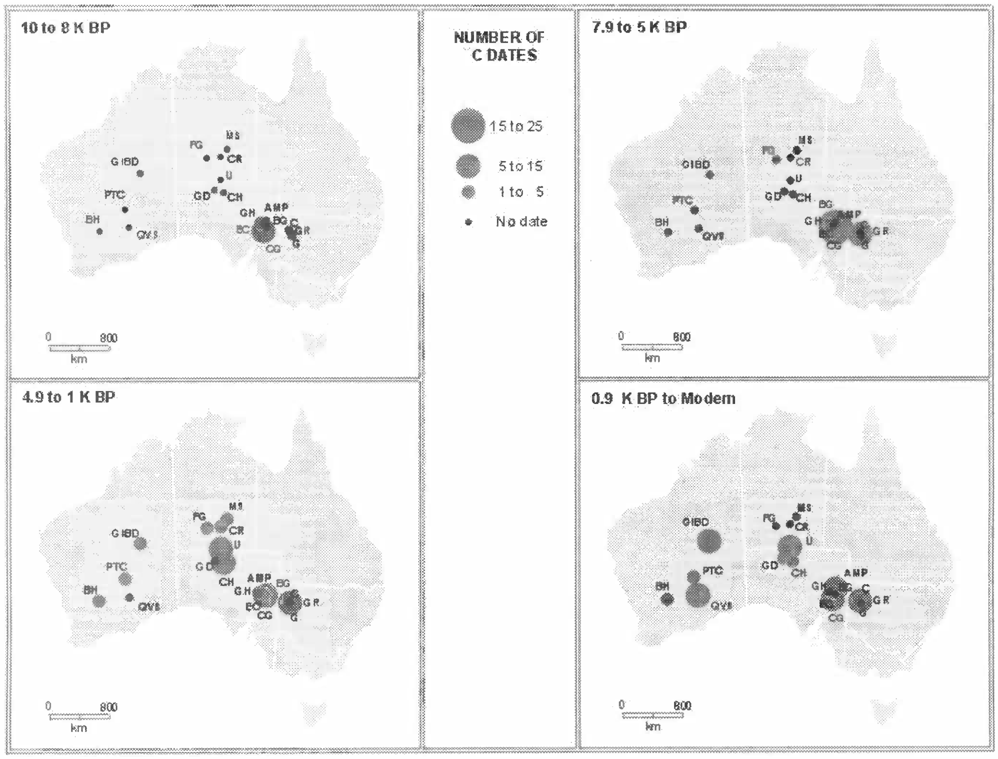


Figure 4 Maps showing the changing distribution of radiocarbon dates on middens

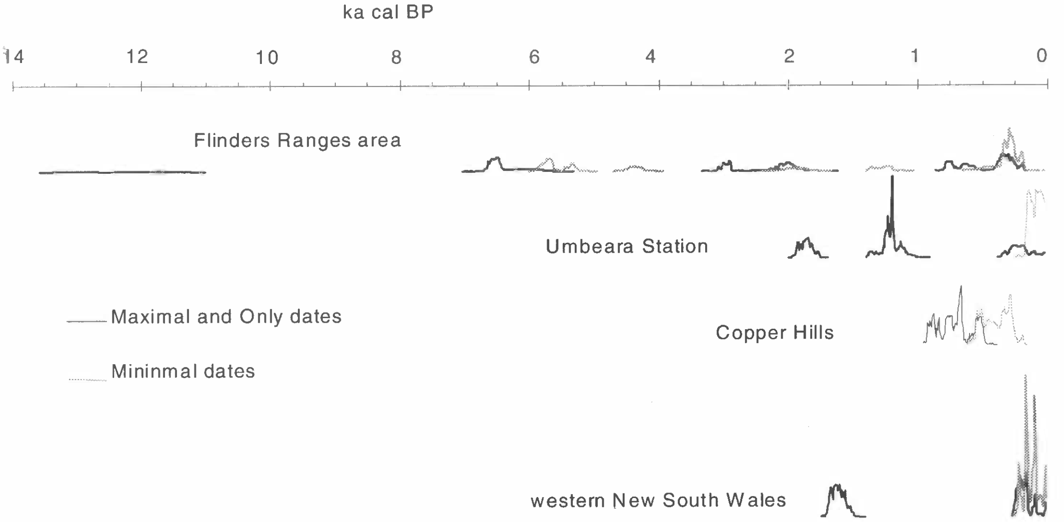


Figure 5 Maximal and minimal calibrated radiocarbon dates on middens from 4 sites: Copper Hills, Umbeara Station, Flinders Ranges area and western New South Wales

DISCUSSION

Analysis of the spatial and temporal distribution of stick-nest rat midden ages suggests that some important issues still need to be addressed by researchers. The advances in midden analysis are clearly evident in the changes in the material being dated, the detailed information now available about the preservation bias underlying the distribution of middens, the awareness of the ecological significance of the midden record, the detailed understanding of the age of individual middens, and regional difference between groups of middens at the few sites studied in detail. The direction of future research is suggested by these results and through comparisons with North America (Pearson and Betancourt 1999).

The extensive sampling of stick-nest rat middens is remarkable. However, only a fraction of the known distribution has been sampled (Pearson 1999). Accessibility by road has played an important role in midden collection and possibly in accelerating midden decay. Middens were destroyed by subfossil collectors and dingo hunters using access roads and tracks. As a result, the accessible middens are possibly the most damaged and also the most sampled. If so, there are likely to be older and less disturbed midden deposits in more isolated areas, but they would be very costly to sample.

^{14}C analyses of stick-nest rat middens (Table 2) have changed from bulk conventional dates of unsorted midden material (Green et al. 1983), to AMS dates using small samples (Pearson 1997; McCarthy et al. 1996). Stick-nest rat skin and pollen residues have also been dated. The American trend has been towards dating scats (21% by 1985; Webb and Betancourt 1990) and in Australia, dates are frequently (38%) on scats. Dating of a single taxon (e.g. most often *Juniperus* sp., an indicator of cooler climates when recovered from a fossil midden in low desert thorn scrub) is unique to America [Van Devender 1973; Mead et al. 1978]) and accounted for over 41% of all the midden dates in 1985 (Webb and Betancourt 1990). Special grants from the Australian Institute for Nuclear Science and Education (AINSE) yielded a much higher proportion of AMS dates from stick-nest rat middens (66%) than from packrat middens (4%), and Australian researchers have had more control on questions of contamination and resolution.

TABLE 2 Types of material dated from middens

Material	Australia (n=145)	American (n=1113)[a]
Scat	35%	0%
Scats	3%	21%
Plant fragments	>25%	38%
Single taxa	4%	41%

[a] Webb and Betancourt (1990)

Midden analysts use ^{14}C to constrain the age of interesting material by direct dating (e.g. the skin of the stick-nest rat), indirectly dating the material by assuming adjacent material is contemporaneous (e.g. analyzing selected macrofossils next to a pollen sample), and bracketing samples above (i.e. minimal date) and below (i.e. maximal date) the material of interest. All 3 methods have been used in stick-nest rat midden research, although packrat researchers have largely used direct dating. Most exploratory dates in Australia have used material at the base of the deposit to find the maximum length of the record (Table 1; Figure 2), so older samples are possibly over-represented. This is the most efficient way to find the oldest materials. Fewer dates have been used to constrain the minimal age of the deposits (Table 1). Many middens have been dated once to ascertain if further research and dating would be worthwhile.

Accumulation rates can be calculated using ^{14}C dates that constrain accumulations of measured depth. ^{14}C dates from a number of middens and sites in central Australia suggest accumulation rates are about 0.31 ± 0.7 mm per year, so a midden of 300 mm depth may reflect over 1000 yr of accumulation (Pearson 1997). Flinders Ranges middens have a depositional rate that may be more rapid over discrete periods of time. For example, at Brachina Gorge, a midden accumulated a few hundred years of material in a depth of 600 mm. Analysts in Australia appear to have routinely explored the stratigraphy of middens in more detail than their American counterparts (e.g. Pearson 1989; Head et al. 1998). However, it is clear that middens do not accumulate continually or vertically because ^{14}C results are anomalous and dusty weathering layers occur throughout the midden. The American packrat researchers have not used interpolated ages for samples in their analyses; instead each sample is selected to avoid layers separated by weathering rinds. The samples are then ^{14}C-dated and assumed to be contemporaneous. This difference reflects the ready access to ^{14}C analyses in the United States and perhaps the dominantly palynological background of the current researchers in Australia.

Systematic bias in sampling packrat middens for ^{14}C resulted in a marked bimodal distribution of American midden dates (Webb and Betancourt 1990). This is probably the result of an exponential decline in midden preservation through time, the abundance and easy identification of extra-local species around 8500 BP to 10,000 BP and older, and a lack of ecological change in the late Holocene. However, even late prehistoric dominance of arid land shrubs may change and be detected in late-Holocene middens. Packrat middens older than 8000 BP routinely contain macrofossils of plants that no longer occur at the site, or groups of species that are no longer sympatric. These data are used to preselect middens that will address the questions about vegetation composition change. Since these changes are ecologically the most interesting, the older middens have received more attention reflecting the efficiency of paleoecological sampling when indicator macrofossils are obvious. This results in a bimodal distribution of packrat midden dates with 2 underlying decay curves (Webb and Betancourt 1990). Hall (1997) has said that this selective process, along with the cliff-edge position of ancient middens, causes distortion in the vegetation reconstructed from middens. American researchers have also sought to identify contaminates through dating and redating some samples; this tends to further distort the distribution of ^{14}C dates.

In contrast, the samples in Australian stick-nest rat middens have not been preselected by indicator taxa. Instead, middens are dated "blind" because the midden contents appear very uniform regardless of their antiquity. The appearance and shape of middens had been assumed to be poor predictors of midden age. However, Head et al. (1998) suggest a categorization of midden structure to preselect middens for reliable paleoecological analysis in western New South Wales. The decay of middens through time is reflected by a monotonic curve of ^{14}C analyses (Figure 1). The pattern is the same when maximal and minimal midden ages are considered (Figure 2). Pearson's (1997) Umbeara sites and Head et al.'s (1998) Gap Range sites in western New South Wales do not suffer this selection bias but the distributions still follow a similar pattern of all the midden dates (Figure 5).

The existence of middens is predicated on the presence of a population of midden-building animals and the availability of suitable and sufficient biomass for midden construction. From the observations of midden construction elsewhere, middens will generally be built (and therefore more likely found) at times of increased productivity (Betancourt and Van Devender 1981). Factors controlling stick-nest rat midden activity include the existence of a viable metapopulation to occupy suitable sites, mortality below recruitment, and the satisfaction of nutritional and habitat requirements. Interpretation of the existence of middens in the landscape must take into consideration all these aspects. It is possible that stick-nest rat activity correlates with climatic variables of interest.

In the United States, there is a period between 4000 and 8000 BP during which there is a decline in the number of ^{14}C analyses available on middens. Webb and Betancourt (1990) suggested that a single cause was unlikely because the phenomenon is widespread across the Chiuahuan, Sonoran and Mojave Deserts, as well as the Great Basin and the Colorado Plateau. There is also a dearth of packrat middens from higher altitude sites (2500 m) at peak aridity (10,000 BP). Low productivity of the ecosystems probably makes the survival of packrats or accumulation of organic material unlikely. At one site, Betancourt and Van Devender (1981:656) suggested that, "packrat middens measure resource availability" and that a lack of middens in a particular age range may indicate a period of low productivity. The brief occurrence of rodent middens in the Atacama Desert of Chile reflects a small window of Holocene productivity (J Betancourt, personal communication 1999). Although gaps in the midden record in Australia may relate to periods of very low productivity, it is unlikely that this aspect of the record can be understood from current Australian research due to the extinction of stick-nest rats. The record may be partly solved through the continuing work at other sites.

We believe that there are sufficient ^{14}C samples, selected from a variety of sites including volcanic, metamorphic and sedimentary substrates, to show the pattern of midden decay in Australia (Figure 1). The spatial distribution of middens is discontinuous and there are sites with caves suitable for midden preservation that do not contain middens. The absence of middens could relate to extremely long periods of midden decay or a real absence of stick-nest rats at the sites. Factors that may speed the process of midden decay include periods of high humidity, bioturbation, and severe fires. Further work needs to be done to compare the distribution of subfossil stick-nest rat bone and the midden material to identify where the midden builders have been but the middens have not survived. The dearth of Glacial or early Holocene midden dates (Figure 2) may reflect a real lack of middens built during this period. Records from the continental margins suggest the arid cores suffered very low plant productivity and high ecological stress during the Last Glacial Maximum (Kershaw 1995). If productivity was extremely low, the small rodent population would build few middens and those that were built would be unlikely to survive long enough to contribute to our understanding of the arid zone's paleoecology. There are patterns in the maximal and minimal dates of middens suggesting that periods of accumulation and abandonment may be cyclic (Figures 2 and 5), although there is no synchronicity in either maximal or minimal dates between sites.

Most of the paleoenvironmental records and models suggest that Australian ecosystems have probably been stable for the last 4000 yr (Markgraf et al. 1992). So, the late Holocene age of most middens analyzed in Australia reduces the likelihood of major ecological change being encountered in the record. The one midden of late Glacial Maximum age does not appear to record a major environmental change in either the pollen or the macrofossil record (McCarthy et al. 1996). A comparison of northern and central Flinders Ranges middens by McCarthy et al. (1996) indicated more spatial variability in vegetation composition and structure between sites after 2000 BP, suggesting that local topography may buffer vegetation change. Apart from these middens, and other evidence from the arid zone (Smith et al. 1995), this suggests that at some sites the distribution of many arid plants and the composition of communities predate the mid-Holocene.

CONCLUSION

In Australia, extra-local taxa that would help in building a chronology have not been identified, and the age distribution of middens is less distorted by an "early-Holocene push" that characterizes the ^{14}C results on American packrat middens. We have not been able to identify with certainty any major shift in taxa, perhaps because the midden record is currently limited to the late Holocene–Modern period. This may be because the changes in the late Holocene are within the natural variability of the modern ecosystem or in the small to medium-scale spatial response of the vegetation signal.

Generally, appearance and architecture have not been found to be reliable indicators of stick-nest rat midden age, so middens have been dated without effective preselection. This has resulted in a unimodal distribution of ^{14}C ages in Australia rather than a bimodal distribution as in America. In the future, that pattern may change with the typologies developed by Head et al. (1998) to identify the stick-nest rat middens more likely to hold paleoclimatic records.

The availability of a paleoecological record determines which research questions can be addressed. Midden analysts may now expand from the search for biogeographic changes to study the inertia and plastic responses of taxa in the Australian arid zone. Further work using chemical and structural characteristics of macrofossils, or more emphasis on macrofossil identifications with adequate reference material, may be fruitful—particularly given the availability of well-dated midden materials. There is certainly still scope for the discovery of middens in areas likely to be sensitive to particular kinds of regional changes. The most dramatic finds would be at sites where changes in species composition were forced by a variable of interest (e.g. rainfall), and the changes were then recorded in midden materials. The search continues for these sites, or records from sites already sampled. The authors hope this paper contributes to the greater efficacy of stick-nest rat midden analysis in Australia.

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COMMENTS ON "AMERICA'S OLDEST BASKETRY"

Thomas J Connolly

State Museum of Anthropology, University of Oregon, Eugene, Oregon 97403 USA

William J Cannon

Bureau of Land Management, Lakeview District, PO Box 151, Lakeview, Oregon 97630 USA

ABSTRACT. A recent publication on directly dated basketry specimens from the western United States (Berger et al. 1998, *Radiocarbon* 40(2):615–20) contained some misleading information, and in a few cases discussed radiocarbon ages from unacknowledged sources. We provide the missing original citations along with some clarifications. We focus especially on the age of distinctive Fort Rock and Multiple Warp–style sandals, for which we provide additional previously unreported ^{14}C ages. Direct dates on fibers from Fort Rock sandals from 3 different sites range in age from 10,500 cal BP to about 9200 cal BP. Contextual evidence suggests that Multiple Warp sandals may date as early as 6600 cal BP, but the few directly dated specimens are less than 1000 yr old.

INTRODUCTION

In "America's Oldest Basketry," a short contribution by Berger et al. (*Radiocarbon* 40(2):615–20 [1998]), calibrated ranges are presented for previously reported radiocarbon ages on archaeological basketry specimens from western North America. The article contains a number of factual errors. Further, some dates, but not all, are cited as reports from ^{14}C labs by the senior author and others (Berger et al. 1965; Berger and Libby 1966), but some primary sources are not acknowledged. In no case are published reports by the sample submitter credited, although these would lead the reader to important contextual information and technical descriptions of the specimens.

The ^{14}C ages discussed by Berger et al. (1998) were all reported in works published between 1951 and 1982 (Table 1), and calibrated ranges for some of the dates have been previously reported (e.g. Connolly et al. 1995). We offer here a brief comment on the material mentioned from the central and southern Great Basin, then discuss the twined sandals from the south-central Oregon caves in greater detail.

DESCRIPTIVE BACKGROUND

In the caption to their Figure 1, Berger et al. identify an age of 2440 BP as the "earliest date for coiled technique". This may be the case for the Falcon Hill, Nevada, sites where this dated specimen was recovered (Hattori 1982), but it is not true for the general region (Figure 1). Coiled basketry was recovered from strata considerably older than 5000 yr at Hogup Cave, Danger Cave, and other sites in the eastern Great Basin (Adovasio 1970, 1986; Aikens 1970). While it is possible that this and other statements were intended to specifically reference the Falcon Hill basketry assemblage, with which the authors were familiar, this is not made clear in the text, and is not the case implied by the paper's title. In another case, the Berger et al. Figure 3 caption identifies plain twined, s-twist basketry, dated to 3900 BP, as the "last known occurrence" of this type. Again, this may be true for directly dated specimens from the Falcon Hill sites, but examples of this type are common in late prehistoric and ethnographic collections from the Great Basin (e.g., Adovasio 1986; Connolly et al. 1998; Fowler and Dawson 1986).

Berger et al.'s Figure 7 identifies the pictured artifacts as "Fort Rock, Oregon" sandals; the upper photo is a Fort Rock–style sandal, as defined by Cressman (1942, p 57–8). While the pictured specimen may indeed have come from Fort Rock Cave, specific source information, such as accession or specimen number, is not provided. Sandals of this type have been found in a number of northern

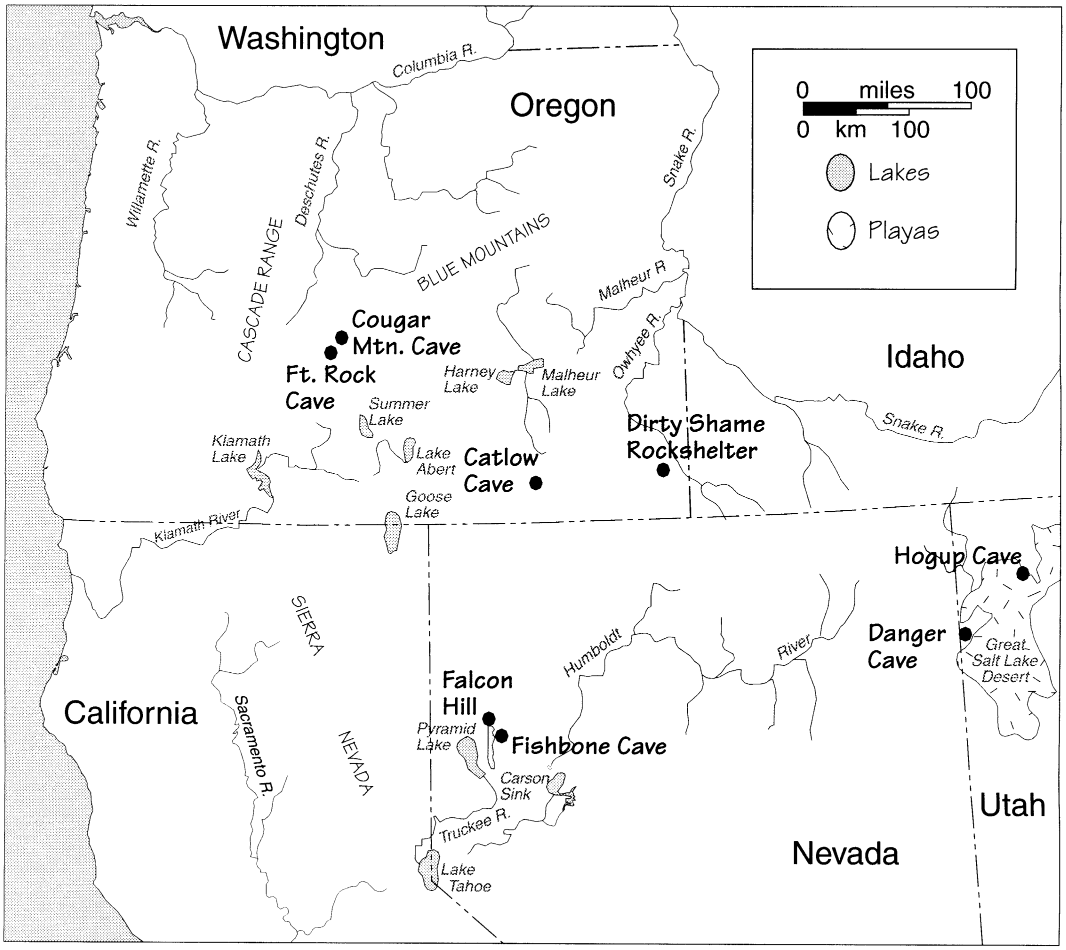

Figure 1 Locations of basketry-producing archaeological sites mentioned

Great Basin sites (Andrews et al. 1986; Connolly 1994; Cowles 1959; Cressman 1942). Berger et al. (1998, p 620) identify an unacknowledged date of 8410 ± 250 BP from a Fort Rock–style sandal from Cougar Mountain Cave; this was originally reported by Fergusson and Libby (1962) as 8510 ± 250 BP (Table 1).

The lower photo in Berger et al.'s Figure 7 shows sandals that are neither the Fort Rock type, nor from Fort Rock Cave; this pair of child-sized sandals was recovered from the upper levels of Catlow Cave by a crew under the supervision of Luther Cressman in 1937, and reported and illustrated in his 1942 publication. These sandals fit into the "Multiple Warp" type described by Cressman (1942, p 58). One sandal from this pair (Berger et al. 1998, Figure 7b; University of Oregon Museum of Natural History Accession 56, specimen 1-3112) has only recently been dated to 950 ± 45 BP (Table 1), effectively identical to the 959 ± 150 BP age reported by Cressman (1951, p 308) on a fire-hardened digging stick also collected from the upper levels of the site, and believed by him to be "from near the end of occupation" of the cave.

Table 1 Directly dated Fort Rock and Multiple Warp style sandals, northern Great Basin

Lab no.	^{14}C age (yr BP)	Age range (cal BP, 1 σ)	Material	Site	Reference(s), comments
Fort Rock–Style Sandals					
C-428a	9188 ± 480[a]	10,920–9650	Sagebrush bark	Fort Rock Cave	Arnold and Libby 1951
C-428b	8916 ± 540[a]	10,440–9380	Sagebrush bark	Fort Rock Cave	Cressman 1951; Bedwell and Cressman 1971
AA-30056[b]	8308 ± 43	9380–9240	Sagebrush bark	Catlow Cave	Not previously reported
UCLA-112	8510 ± 250	9840–9240	Tule	Cougar Mtn. Cave	Fergusson and Libby 1962; Connolly 1994
I-1917	8500 ± 140	9530–9380	Sagebrush bark	Fort Rock Cave	Bedwell and Cressman 1971
AA-9249	9215 ± 140	10,360–10,020	Sagebrush bark	Fort Rock Cave?[c]	Not previously reported
AA-9250	8715 ± 105	9870–9520	Sagebrush bark	Fort Rock Cave?[c]	Not previously reported
Multiple Warp Sandals					
WSU-4198	820 ± 60	780–670	Tule	S. Warner Cave	Fowler and Cannon 1992; Eiselt 1997
AA-30055	950 ± 45	930–790	Sagebrush bark[d]	Catlow Cave	Not previously reported

[a] The commonly cited 9053 ± 350 age for the "Fort Rock sandal" is actually an average of these 2 dates, run on "several pairs of woven rope sandals" (Arnold and Libby 1951, p 117). The weighted average of these 2 ages produces an age range of 10,390–9650 cal BP.

[b] Specimen 1-3583, Accession 56, State Museum of Anthropology, Eugene, Oregon.

[c] Reportedly recovered from Fort Rock Cave ca. 1928 by local residents, subsequently donated to the Lake County Museum, Lakeview, Oregon.

[d] Child-sized sandal, specimen 1-3112, accession 56, Oregon State Museum of Anthropology; pictured by Berger et al. (1998), Figure 7.

DISCUSSION

Fort Rock Sandals

Named for the site where several dozen examples were recovered from a cache buried beneath the approximately 7500-yr-old Mazama volcanic ash, Fort Rock–style sandals are described as having a flat close-twined sole, usually with 5 rope warps (although variation has been noted; Connolly 1994, p 73). Twining proceeded from the heel to the toe, where the warps are subdivided into finer warps and turned back to form an open-twined toe flap (refer to Berger et al. 1998, Figure 7a). A tie rope attached to one edge of the sole presumably wrapped around the ankle and fastened to the opposite edge (Cressman 1942, p 57).

Andrews et al. (1986) suggest that Fort Rock–style sandals may occur in Dirty Shame Rockshelter as late as about 6000 yr ago, based on stratigraphic associations. Table 1 summarizes all known ^{14}C ages from directly dated Fort Rock sandals. Most dated specimens are from Fort Rock Cave, but directly dated sandals of this type are also known from Cougar Mountain and Catlow Caves. Directly dated Fort Rock style sandals range in age from at least 10,500 cal BP to about 9200 cal BP.

Multiple Warp Sandals

Multiple Warp–style sandals are open or close twined from heel to toe, but have from 8 to more than a dozen warps "arranged in a series of parabolas around the heel" to form a pocket (Cressman 1942, p 58). Loose warps are bent back to form a toe cover, but are rarely twined. Loops built into the sole were pulled together across the top of the foot and secured with a tie (see also Berger et al. 1998, Figure 7b).

Adovasio (1986, p 197) has reported that a sandal similar to the northern Great Basin Multiple Warp type was recovered from Fishbone Cave in western Nevada in strata predating about 8200 cal BP. Andrews et al. (1986) report that Multiple Warp sandals were recovered from Zone VI in Dirty Shame Rockshelter in southeast Oregon, which predates about 6600 cal BP. While these associations appear to be valid, cave deposits can be quite disturbed, and Multiple Warp sandals from the northern Great Basin that have been directly dated—or are clearly associated with ^{14}C-dated features (Table 1; also see Eiselt 1997; Marchesini 1994)—are consistently much younger. Andrews et al. (1986, p 126) suggest that Multiple Warp sandals first appear in the northern Great Basin after 8000 cal BP, and were made up to the time of Euroamerican contact.

CONCLUSION

The distinctive Fort Rock and Multiple Warp sandal types appear to be of dramatically different ages. All directly dated Fort Rock style sandals predate 9000 cal BP, although it is possible sandals of this type were made until 7000 cal BP or later (Andrews et al. 1986). Multiple Warp sandals may have first appeared prior to 6600 cal BP, and sandals of this type were evidently made up to the historic period (Andrews et al. 1986). The small number that have been directly sampled or have unambiguous ^{14}C associations date to within the first half of the last millennium.

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NOTE FROM AUTHORS OF ORIGINAL ARTICLE

"We welcome the improvements presented by Connolly and Cannon filling in the archaeological/archival record. Some of their information had not been available to us directly. Other data in the literature proved to be inaccurate in the end." — *Rainer Berger, Millie Bendat, and Andrea Parker.*

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DATING ORGANIC TEMPER OF CERAMICS BY AMS: SAMPLE PREPARATION AND CARBON EVALUATION

Denise C Gomes

Museu de Arqueologia e Etnologia–USP, Avenida Prof. Almeida Prado, 1466, Cidade Universitária, São Paulo 05508-900, Brazil. Email: denisecavalcante@yahoo.com

Oscar Vega

Instituto de Pesquisas Energéticas e Nucleares–USP, Travessa R, 400, Cidade Universitária, São Paulo 05508-900, Brazil. Email: ovega@net.ipen.br

ABSTRACT. We describe a new methodology for separating organic temper from archaeological ceramics from Brazilian Amazonia. These experimental procedures were designed to directly date ceramic samples by accelerator mass spectrometry (AMS). An evaluation of the total carbon indicates the samples' potential for dating.

INTRODUCTION

Since the 1980s, accelerator mass spectrometry (AMS) has made it possible to date organics using smaller samples than those necessary for conventional radiocarbon dating. However, AMS encounters problems in dating archaeological ceramics because of the different origins of carbon present in pottery. These sources of carbon include the carbon in clays, as well as carbon contributed by temper, fuel, food remains, and geochemical contamination, any of which may affect ^{14}C dates (Gabasio et al. 1986; Evin et al. 1989; Johnson et al. 1988; Hedges et al. 1992).

Among these sources of carbon, clays are especially significant, for most of them are said to contain amounts of carbon of diverse "geological" ages (Gabasio et al. 1986; Johnson et al. 1988), which can influence the results of dating, sometimes increasing the apparent age of the potsherd. Depending on the firing temperature (600–900 °C) this carbon may or may not be oxidized (Johnson et al. 1986). In order to avoid invariable contamination by the detrital carbon from the clay and to ensure the real source of carbon to be dated, the organic temper, when available, should be extracted from the clay matrix.

Hedges et al. (1992), discussing the possibility of dating selected pottery fractions by the AMS method, have noted the importance of using organic temper removed from the sherds as a reliable dating material. Although they describe painstaking methods of microscopic excavation of sherds, the separation process presented here, for 2 different kinds of organic temper, can be considered low-cost and relatively fast.

METHODS

The Separation Process

In archaeological sites in lowland South America, especially in Amazonia, 2 kinds of organic temper are conspicuous in ceramics: spicules of freshwater sponges (*cauixi*) of different species rich in silica (Bergquist 1978), and bark ashes (*cariapé*) also containing silica (Linné 1932; Rye 1981; Shepard 1985). In this experiment, the sherds tempered with sponge spicules came from Santarém region, in the state of Pará. They belong to a museum collection, without contextual information. Only for the 4th sample, tempered with bark ashes, do we have the precise geographic location: site Marabitana II, Vaupés river (00°26′00″N, 68°50′05″W).

Almost 40 years ago, Evans and Meggers (1962, p 244) described a pioneering experiment in ^{14}C dating using these same organic tempers: "... the sherds were broken by mallet into fragments, and further reduced in size with mortar and pestle to facilitate filtration and rinsing during and after hydrochloric acid treatment for removal of inorganic carbon compounds. The residue was then burned and CO_2 collected and purified in the usual way."

In our experiment, different procedures were adopted for each of these elements, this time in order to prepare samples for AMS dating. Ceramic sherds weighing from 20 to 30 g, tempered with sponge spicules, were powdered with a mortar and pestle (Figure 1). The material was sieved with 2 sieves of different sizes, the first one with 100 mesh and the second with 200 mesh. The sample obtained was successively washed with distilled water, then dried on a 250 W infrared lamp.

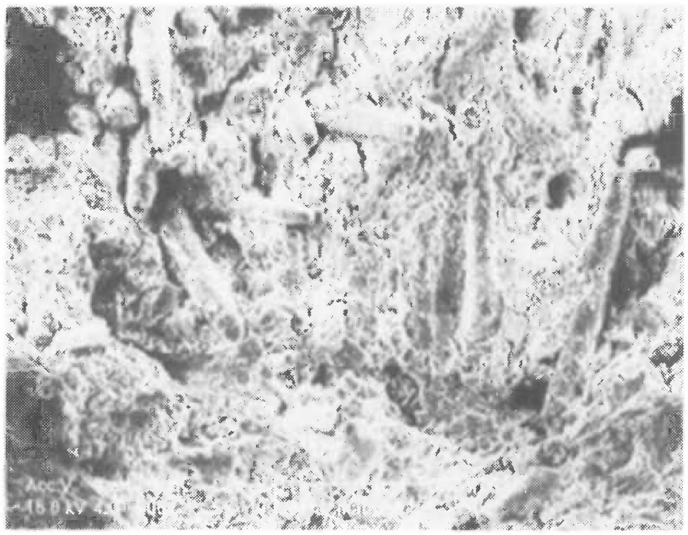

Figure 1 Sample 2 (clay matrix) showing the spicules of freshwater sponges.
Legend:

```
Acc.V      Spot Magn   Det  WD ________   100 µm
15.0 kV    4.0  200x    SE  9.7 clay matrix
```

Because of the biological characteristics and morphology of the sponge spicules, the sample was tentatively passed through a Frantz magnetic separator (model L1, 115 volts) at 1.5 A and a lateral angle of 10 degrees. The process was repeated 2 times and resulted in 2 samples: nonmagnetic and magnetic. A high concentration of spicules 300–400 µm long and 40 µm wide (Volkmer-Ribeiro and Costa 1992) was found in the nonmagnetic sample (Figure 2), while the magnetic one presented sparse quantities of spicules and a more generalized mineral composition (Figure 3). Owing to other elements still present in the nonmagnetic sample (quartz, humic substances, and bits of clay) pretreatment is required.

For the second organic temper, bark ashes, the process adopted was similar to the 1st experiment. The sherds were powdered with a mortar and a pestle, but in a very coarse granularity, in order to preserve the grains of temper as much as possible. As in the first case, the sample was sieved with 2 sieves, the first with 100 mesh and the second with 200 mesh. The content was washed several times with distilled water only.

The sample was filtered through 3 µm pore diameter qualitative analysis paper, then dried under a 250 W infrared lamp. The last step was separating the large grains of temper (0.5–2 mm) mixed in the powdered clay, using metallic tweezers. Since the light-gray-colored grains of bark ashes were preserved, their visual recognition was possible (Figure 4).

Figure 2 Sample 2 (nonmagnetic): concentration of spicules; some of them were broken during the separation process. Legend:

Acc.V	Spot	Magn	Det	WD	____________ 200 µm
5.0 kV	4.0	100x	SE	10.0	nonmagnetic

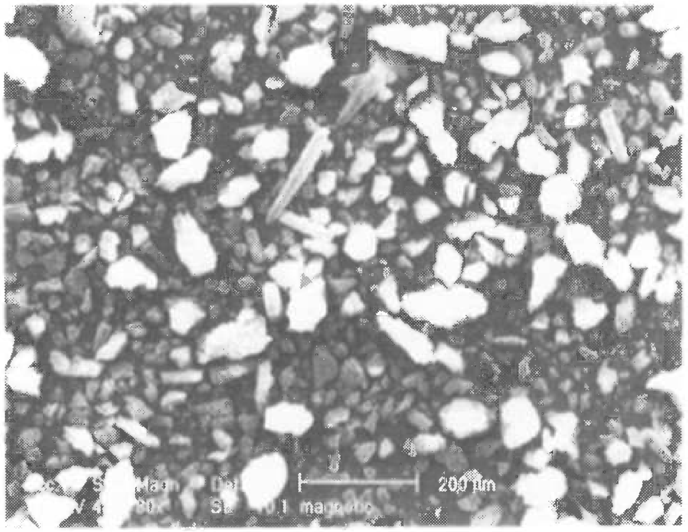

Figure 3 Sample 2 (magnetic): sparse quantities of spicules, bits of clay and quartz. Legend:

Acc.V	Spot	Magn	Det	WD	____________ 200 µm
5.0 kV	4.0	80x	SE	10.1	magnetic

Analytical Equipment and Methodology

The carbon analyses were done using an absorption infrared analyzer, model CS-400, made by Laboratory Equipment Company (LECO). The process was begun by weighing the sample out into a ceramic crucible using an electronic balance. After the addition of an accelerator material, the sam-

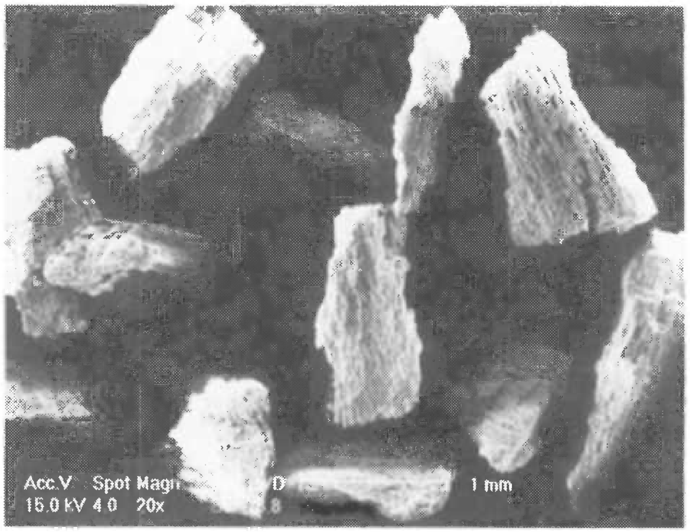

Figure 4 Sample 4: grains of bark ashes. Legend:

Acc. V	Spot	Magn	Det	WD______________	1 mm
15.0kV	4.0	20x	SE	8.8	bark ashes

ple crucible was placed in a high-frequency furnace, where sample combustion takes place in an oxidizing atmosphere. During combustion all elements of the sample oxidize. Carbon-bearing elements are reduced releasing the carbon, which immediately binds with the oxygen to form CO and CO_2.

Subsequently, sample gases are swept into the carrier steam. CO is converted to CO_2 in the catalytic heater assembly. Carbon is measured as CO_2 in the infrared cell as gases flow through it. The total carbon, as CO_2, is detected on a continuous and simultaneous basis. The cell consists of an infrared source, a chopper motor, a narrow bandpass filter, a condensing cone, an infrared energy detector, and the cell body.

Radiation energy is chopped at a rate of 87.5 Hz before it enters the cell body. The chopped energy enters the cell body through a window, travels through the cell body, then exits through a second window and a precise wavelength filter. The selective filter passes only the CO_2 absorption wavelength into a condensing cone that concentrates the energy at the detector. The solid-state detector is AC-coupled to a preamplifier. As the gas concentration increases the voltage to the preamplifier decreases.

RESULTS AND DISCUSSION

The ceramic samples tempered with sponge spicules were classified as magnetic or nonmagnetic, according to the process of separation described above. The mass used for each analysis was around 100 mg. Three to five parts of the original samples were analyzed to determine reproducibility.

The results (Table 1) indicate that the nonmagnetic samples, which contain the higher concentration of sponge spicules and therefore potentially of organic material to be dated, have the lower amount of carbon. Sample 3 was 30% reduced in carbon compared to its magnetic counterpart. This confirms a pattern already inferred by Evans and Meggers (1962), who reported 7 ^{14}C dates for lowland South America ceramic samples with organic temper (charcoal, *cariapé,* and *cauixi*), with carbon percentages ranging from 0.3% to 1.8%.

Table 1 Carbon analyses of archaeological ceramics from Amazonia

Sample nr	Sample description	Total carbon (%)[a]
1	Sponge spicules/nonmagnetic: *Metania reticulata* [b] *Drulia uruguayensis* *Trochospongilla paulula* *Trochospongilla pensylvania*	0.20
1a	Sponge spicules/magnetic	0.56
2	Sponge spicules/nonmagnetic *Metania reticulata* *Drulia uruguayensis* *Oncosclera navicella*	0.27
2a	Sponge spicules/magnetic	0.54
3	Sponge spicules/nonmagnetic *Metania reticulata* *Drulia uruguayensis* *Radiospongilla amazonensis* *Corvospongilla seckti* *Oncosclera petricola*	0.09
3a	Sponge spicules/magnetic	0.30
4	Bark ashes	0.25

[a] Units are in percentage (%) mass/mass.

[b] The sponge spicules' species were identified by Dr Cecília Volkmer-Ribeiro (MCN).

They discarded 2 dates, explaining the erroneous results as a consequence of the samples' small percentage of carbon, 0.3% and 0.6%, respectively. The authors also suggested the sponge spicules' deficiency in ^{14}C as a reason for these results. Although Junk and Furch (1985) registered a deficiency in carbonates (and probably in ^{14}C) in waters of the lower and middle Amazon and its tributaries of clear waters (Tapajós) and dark waters (Negro), Evans and Meggers' statement cannot be generalized. Moreover, we have to take in consideration the natural deficiency in organic matter of some species of freshwater sponges, whose preferred habitat is in the rocky bottoms of Amazonian rivers (Volkmer-Ribeiro and Tavares 1993; Tavares and Volkmer-Ribeiro 1997). These sponges are remarkable for their large spicules, rich in silica, and their reduced amount of spongine (organic matter) binding the spicules. Two examples are the species *Drulia uruguaiensis* and *Oncosclera navicella*. On the other hand, the same characteristics can be found in *Metania reticulata*, a species of floodwater habitats (Volkmer-Ribeiro, personal communication 1999).

However, the remaining 5 dates were all acceptable for the authors, including even 1 date obtained from a sample with 0.6% of carbon. Comparing our results to Evans and Meggers', only sample number 3 seems to be problematic. We observed the reduction of carbon in nonmagnetic relative to magnetic samples in samples 2 and 2a: the former contained only 50% as much carbon as the latter. Sample number 4, bark ashes, also indicates potential for dating, comparable to the nonmagnetic sponge spicule samples.

In their field and laboratory experiments, Evin et al. observed that in oxidizing conditions "the carbon from the temper disappears almost completely whatever the temper material. Only very little remained" (1989, p 279). Nevertheless, when present in sufficient amounts and extracted from the sherds, organic temper seems to be a reliable source for AMS dating (Hedges et al. 1992).

CONCLUSION

Further dating of these samples will test the hypotheses presented here. Pretreatment is still necessary to eliminate inorganic carbon, especially in the sponge spicule samples. The choice of ceramic samples tempered with sponge spicules belonging to *Metania reticulata, Drulia uruguaiensis,* and *Oncosclera navicella* will help verify Evans and Meggers' conclusion about the deficiency of ^{14}C in these species. Finally, we believe that the process of separation of the organic temper from ceramics can isolate fractions that correspond to culturally introduced carbon, contributing to the potential of AMS as a technique for directly dating archaeological artefacts.

ACKNOWLEDGMENTS

The first author is indebted to Dr Koji Kawashita, Geochronological Research Center of the Geoscience Institute–USP, Brazil, for supervising the separation process of organic ceramic temper. She also would like to express her gratitude to Vasco Antônio P. Loios, technician of the Geochronology Separation Laboratory, Geoscience Institute–USP, Brazil, for his support. Special thanks are due to Dr Cecília Volkmer-Ribeiro, from the Museu de Ciências Naturais do Rio Grande do Sul, Brazil, for the zoological analysis of the freshwater sponges contained in the ceramics. Finally, this manuscript benefits from the comments and encouragement of Eduardo Góes Neves, from MAE-USP, and the reviewers from *Radiocarbon.*

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BOOK REVIEW

Harry E Gove. *From Hiroshima to the Iceman: The Development and Applications of Accelerator Mass Spectrometry.* Philadelphia, Institute of Physics Publishing, 1999: 226 p. ISBN 0-7503-0558-4. List price $27 US (paperback) and $99 US (hardback).

Reviewed by: Walter Kutschera, Vienna Environmental Research Accelerator, Institut für Radiumforschung und Kernphysik, University of Vienna, Vienna, Austria

Harry E Gove, Professor Emeritus of Physics at the University of Rochester, is one of the pioneers of accelerator mass spectrometry (AMS). He was personally involved in many "firsts" in this field, which was pioneered in 1977. Ever since, Gove has followed the field closely, and from the early beginnings he was one of the most outspoken advocates of AMS: For example, he clearly was responsible for the excitement about the ^{14}C dating of the Shroud of Turin, which has triggered countless discussions about the problems of dating this religious relic. A separate account of this issue is given in a previous book by the same author: *Relic, Icon or Hoax? Carbon Dating the Turin Shroud* (IOP Publishing 1996. Reviewed by R E Taylor in *RADIOCARBON* Vol 39, Nr 1, 1997).

Gove calls his book "a semi autobiographical account of the historical development of accelerator mass spectrometry". It is a fascinating book because in every sentence one feels the passionate involvement of the author. In this sense it is a very personal account of the history of AMS, so one should not expect a strictly objective and rational view of the method and its applications.

In a way, the book reminded the reviewer of *The Double Helix* by James Watson (1968), "a personal account of the DNA discovery and the roles of the people involved in it, which aroused some controversy" (quote from *Encyclopaedia Britannica*). It was said that those who were involved in the race to decipher the structure of DNA did not like the book, unlike those who were not directly involved.

Gove describes AMS in a similar fashion. His book is full of his personal experiences, including details about what people said, and did, or did not do. He does not try to be objective. There is nothing wrong with such an approach, but the prospective reader should be aware that this is not a textbook on AMS. What one learns is something else, perhaps equally—if not more—important than a cool description of AMS from a purely scientific book: it is a beautiful but necessarily biased view of how the development of AMS happened with real people. Along the way, one learns about the basics of the technological development of AMS, and about some of the more interesting—if not to say recondite—applications. This includes ^{14}C dating in connection with the Shroud of Turin, the Iceman "Ötzi", the Dead Sea Scrolls, the initial peopling of the Americas, and the arrival of the Vikings in Newfoundland. The applications of heavier radionuclides such as ^{36}Cl are discussed in connection with the atomic bombing of Hiroshima and Nagasaki (re-establishing the neutron fluence). Both ^{36}Cl and ^{129}I are also applied to nuclear-waste monitoring in the hydrosphere. A brief mention of ice-core measurements in connection with the ^{36}Cl "bomb-peak" is also included. The final chapter of the book presents an interesting outlook on the future of AMS.

AMS is one of the most successful spin-offs of nuclear accelerator technology. It became an analytic tool of truly global dimensions. Gove's book is capable of instilling into the reader the fascination of analyzing our world atom by atom, aiming for a deeper understanding of the underlying physical and chemical processes. Together with the more analytical monograph of AMS, *Accelerator Mass Spectrometry: Ultrasensitive Analysis for Global Science* by C Tuniz, J R Bird, D Fink, and G F Herzog

(CRC Press, Boca Raton, 1998), there are now two books on AMS on the market, which complement one another well. (For an extensive review of the latter see *RADIOCARBON* Vol 41, Nr 1, 1999.)

Anyone interested in learning about AMS is well advised to read Gove's book and the other mentioned above, and, in addition, the excellent but little-known review article "AMS in the Earth Sciences: Techniques and Applications" by R C Finkel and M Suter in *Advances in Analytical Geochemistry*, Volume 1 (JAI Press Inc. 1993, p 1–114). This review article and the book by Tuniz et al. provide the necessary ingredients to fully appreciate Gove's more colloquial book. However, the latter is certainly the more entertaining one. It is simply fun to read. Overall, I believe, it conveys well the passion of scientific involvement.

RADIOCARBON UPDATES

Internet Resources

Received from Caitlin Buck of Cardiff University:

> The School of History and Archaeology, Cardiff University, Wales, United Kingdom, would like to invite readers of *Radiocarbon* to make use of BCal, the first online Bayesian radiocarbon calibration service, at
>
> http://bcal.cf.ac.uk/
>
> The Bayesian framework is acknowledged as offering considerable benefits over traditional calibration methods as it allows the integration of prior relative chronological information along with radiocarbon evidence to arrive at coherent and integrated archaeological interpretations.
>
> BCal can be accessed from any computer on the Internet with a frame-enabled Web browser (e.g. Netscape 2 or above) and is not dependent on the power of the user's machine, since all calculations are undertaken on the dedicated BCal server.
>
> We believe that this is the first example of a site offering online Markov chain Monte Carlo Bayesian data analysis, and we encourage the international research community to take advantage of the resources we are able to offer.
>
> BCal is available for use free of charge by members of the international academic research community and for a small fee by individuals, institutions, and companies who wish to use it for commercial purposes. Please do visit the site and register as a user. We will be glad to share our resources with you and to hear of the results of your research.

Grant Award

Received from NERC:

> NERC Grant Award GR3/JIF/01: An Accelerator Mass Spectrometer Facility for ^{14}C and Cosmogenic Isotope Analysis Applications: £3,912,303.00.
>
> In July 1998 the outcome of discussions between the Governors of the Wellcome Trust and the United Kingdom's Chancellor of the Exchequer was to create a joint fund of £1.4 billion to provide a programme towards addressing the infrastructure problems of the universities. This Joint Infrastructure Fund (JIF) has targeted the biological, physical, engineering and social sciences, and is administered through the relevant Research Councils. It provides for buildings, major equipment and other elements of infrastructure of the universities.
>
> Under the direction of Professor Tony Fallick (Director of Scottish Universities Environmental Research Centre), a successful bid was made to the JIF by the Scottish radiocarbon dating community (Drs Doug Harkness, Gordon Cook, Marian Scott and Charlotte Bryant), together with the wider cosmogenic isotope community (Prof. Mike Summerfield, Dr Dick Kroon and Dr Sarah Metcalfe from the University of Edinburgh and Prof. Paul Bishop and Dr Colin Braithwaite from the University of Glasgow) to establish an AMS facility for applied research in ^{14}C and other cosmogenic isotopes.

Birth Announcement

Irka and Wojtek Hajdas are pleased to announce the arrival of their baby boy, Jon Stanislaw, born on 13 November 1999.

RADIOCARBON, Vol 41, Nr 3, 1999, p 325–351 © 1999 by the Arizona Board of Regents on behalf of the University of Arizona

RADIOCARBON LABORATORIES

This is *RADIOCARBON*'s annual list of active radiocarbon laboratories and personnel known to us. Conventional beta-counting facilities are listed in Part I, and accelerator mass spectrometry (AMS) facilities in Part II. Laboratory code designations, used to identify published dates, are given to the left of the listing. (See p 349 ff. for a complete list of past and present lab codes.)

Please notify us of any changes in staff, addresses, or other contact information.

I. CONVENTIONAL ^{14}C COUNTING FACILITIES

ARGENTINA

AC Dr. Héctor Osvaldo Panarello
Pabellón INGEIS
Ciudad Universitaria
1428 Buenos Aires, Argentina
Tel: +54 11 4783 3021/23; Fax: +54 11 4783 3024
Email: hector@ingeis.uba.ar

LP Prof. Aníbal Juan Figini
Laboratorio de Tritio y Radiocarbono-LATYR
Facultad de Ciencias Naturales y Museo-UNLP
Paseo del Bosque S/N°
1900 La Plata, Argentina
Tel/Fax: +54 21 270648

AUSTRALIA

ANU Dr. Rainer Grün
Quaternary Dating Research Centre
Australian National University
Research School of Pacific Studies
Canberra ACT 0200 Australia
Tel: +61 6 249 3122; Fax: +61 6 249 0315
Email: rainer.grun@anu.edu.au

SUA Dr. Mike Barbetti
The NWG Macintosh Centre for Quaternary Dating
Madsen Building F09
The University of Sydney
NSW 2006 Australia
Tel: +61 2 9351 3993; Fax: +61 2 9351 4499
Email: m.barbetti@emu.usyd.edu.au

AUSTRIA

VRI Dr. Edwin Pak
Institut für Radiumforschung und Kernphysik
Universität Wien
Boltzmanngasse 3
A-1090 Vienna, Austria
Tel: +43 1 4277 51764; Fax: +43 1 4277 51752
Email: pak@ap.univie.ac.at

Franz Schönhofer
Federal Institute for Food Control and Research
Kinderspitalgasse 15
A-1090 Vienna, Austria
Tel: +43 1 40491 520; Fax: +43 1 40491 540

IAEA Dr. Manfred Gröning
International Atomic Energy Agency (IAEA)
Isotope Hydrology Laboratory
Wagramerstrasse 5
P.O. Box 100
A-1400 Vienna, Austria
Tel: +43 1 2600 21740/21766; Fax: +43 1 20607
Email: M.Groening@iaea.org

Dr. Roland Tesch
Austrian Research and Testing Centre Arsenal
Environment Division
Faradaygasse 3
A-1030 Vienna, Austria
Tel: +43 1 79747 516
Fax: +43 1 79747 587
Email: tesch.r@arsenal.ac.at

BELGIUM

ANTW Prof. R. Vanhoorne
Department of General Botany
State University Centre Antwerp
Groenenborgerlaan 171
B-2020 Antwerp, Belgium

IRPA M. Van Strydonck
Royal Institute for Cultural Heritage
Jubelpark 1
B-1000 Brussels, Belgium
Tel: +32 2 739 67 11 (institute), +32 2 739 67 02 (lab)
Fax: +32 2 732 01 05
Email: mark.vanstrydonck@kikirpa.be

Prof. L. Moens, K. Vandeputte
University of Ghent - Dept. of Analytical Chemistry
Proeftuinstraat 86
B-9000 Gent, Belgium
Tel: +32 9 264 65 25
Fax: +32 9 264 66 99
Email: vdputte@inwchem.rug.ac.be

LAR Prof. Dr. Jean Govaerts
Lab. d'Application des Radioéléments
Chimie B6, Sart Tilman
Liège, Belgium

BELARUS

IGSB Dr. N. D. Michailov
Institute of Geological Sciences of the
National Academy of Sciences of Belarus
Kuprevich Street 7
Minsk 220141 Belarus
Tel: +375 0172 63 81 13; Fax: +375 0172 63 63 98
Email: mihailov@ns.igs.ac.by

BRAZIL

FZ Prof. M. F. Santiago
Departamento de Física - UFC
Campus do Pici - Cx. Postal 6030
60455-760 Fortaleza-CE, Brazil
Tel: +55 85 288 9913; Fax: +55 85 287 4138
Email: marlucia@fisica.ufc.br

CENA Dr. Luiz Carlos Ruiz Pessenda
Radiocarbon Laboratory
Centro de Energia Nuclear na Agricultura
Universidade de São Paulo
Avenida Centenario 303
Caixa Postal 96 – CEP 13400-970
Piracicaba, São Paulo, Brazil
Tel: +55 19 429 4656; Fax: +55 19 429 4610
Email: lcrpesse@pira.cena.usp.br

BULGARIA

Prof. Yanko Yanev
Department of Chemistry
Radiochemical Laboratory
University of Sofia
1, A Ivanov Av.
1126 Sofia, Bulgaria

CANADA

GSC Dr. Roger N. McNeely
Radiocarbon Dating Laboratory
Geological Survey of Canada
601 Booth Street
Ottawa, Ontario K1A 0E8 Canada
Tel: +1 613 995 4241; Fax: +1 613 992 6653
Email: mcneely@gsc.nrcan.gc.ca

BGS Howard Melville
Department of Earth Sciences
Brock University
St. Catharines, Ontario L2S 3A1 Canada
Tel: +1 905 688 5550 ext. 3522; Fax: +1 905 682 9020
Email: hmelvill@spartan.ac.BrockU.ca

WAT Robert J. Drimmie
Department of Earth Sciences
Environmental Isotope Laboratory
University of Waterloo
Waterloo, Ontario N2L 3G1 Canada
Tel: +1 519 888 4567 ext. 2580; Fax: +1 519 746 0183
Email: rdrimmie@sciborg.uwaterloo.ca

UQ Serge Occhietti and Pierre Pichet
Radiocarbon Laboratory
GEOTOP
University of Québec at Montréal
P.O. Box 8888, Succursale Centre Ville
Montréal, Québec H3C 3P8 Canada
Tel: +1 514 987 4080; Fax: +1 514 987 3635
Email: occhietti.serge@uqam.ca

CHINA

CG Drs. Yijian Chen and G. Peng
Radiocarbon Laboratory
Institute of Geology
State Seismological Bureau
P.O. Box 634
Beijing 100029 China
Tlx: 6347

Prof. Qiu Shihua
Radiocarbon Laboratory
Insitute of Archaeology, CASS
27 Wangfujing Dajie
Beijing, China 100710
Tel: +86 010 65135532
Fax: +86 010 65135532

Prof. Shouli Yang
China-Japan Friendship Hospital
Beijing, China 100013

Dr. Wu Xiaohong
Archaeometry & Archaeological Dating Laboratory
Department of Archaeology
Peking University
Beijing, China 100871
Email: wuxh@pku.edu.cn

Prof. Li Xingguo
Institute of Vertebrate Paleontology and Paleoanthropology
Academia Sinica
Beijing, China

Dr. Guan San Yuan
Beijing Nuclear Instrument Factory
P.O. Box 8800
Beijing, China

Prof. Chen Yijian
Radiocarbon Laboratory
Institue of Geology
State Seismological Bureau
Beijing, China

Prof. Li Chongling
Changchun Institute of Geography
Academia Sinica
Changchun, China

HL Yunzhang Yue
Second Institute of Oceanography
State Oceanic Adminstration
P.O. Box 1207
Hangzhou, Zheijiang 310012 China
Tel: +86 571 8076924 ext. 328; Fax: +86 571 8071539
Tlx: 35035 NBOHZ CN; Cable: 3152

Dai Kaimei
Department of Physics
Nanjing University
Nanjing 210093 China
Tel: +86 25 3596746
Fax: +86 25 307965; Tlx: 34151 PRCNU CN
Email: postphys@nju.edu.cn

Wang Jian
Department of Geography
Nanjing Normal University
Nanjing 210024 China
Tel: +86 25 3303666 ext 3202; Fax: +86 25 3307448

Prof. Gao Zhonghe or Sr. Engineer Chen Xiaoming
Seismological Bureau of Jiangsu Province
3 Weigang
Nanjing 210014 China
Tel: +86 25 4432919 ext. 3028; Fax: +86 25 4432585; Tlx: 77777, Nanjing

Ms. Ruan Chengwen, Director
Mr. Liu Jingyuan
Seismological Bureau of Xinjiang Uygur Autonomous Region
42 South Beijing Road
Urumqi, Xinjiang, 830011 China
Tel: +86 991 3838126; Fax: +86 991 3835623
Email: xjdzj@mail.wl.xj.cn

Dr. Shen Chengde
Institute of Geochemistry
Chinese Academy of Sciences
Wushan, Guangzhou 510640 China
Tel: +86 20 85519755 ext. 2179; Fax: +86 20 85514130
Email: cdshen@public.guangzhou.gd.cn

XLLQ Zhou Weijian
Institute of Earth Environment
XiYing Lu 22-2
Xi'an 710054, Shaanxi, China
Tel: +86 29 5512264 (work); 86 29 5256429 (home); Fax: +86 29 5522566
Email: weijian@loess.llqg.ac.cn; weijian@public.xa.sn.cn

Prof. Huang Qi
Radiocarbon Laboratory, Insitute of Salt Lakes
Academia Sinica
6 Xiying Road
710043 Xi'an, Shanxi Province, China
Tel: +86 29 5520397 (H)

Asst. Prof. Liang Qinsheng
Radiocarbon Laboratory, Institute of Salt Lakes
Academic Sinica
810008 Xining, Qinghai Province, China
Tel: +86 971 6301414; Fax: +86 971 6306002
Email: qingsheng.liang@isl.ac.cn

CROATIA

Z Drs. Bogomil Obelić and Nada Horvatinčić
Ruđer Bošković Institute
P.O.B. 1016, Bijenička 54
10001 Zagreb, Croatia
Tel: +385 1 4680 219; Fax: +385 1 4680 239
Email: Bogomil.Obelic@irb.hr and Nada.Horvatincic@irb.hr
WWW: http://www.irb.hr/zef/c14-lab/

CZECH REPUBLIC

CU Jan Šilar
Department of Hydrogeology
Charles University
Albertov 6
CZ-12843 Prague 2 Czech Republic
Tel: +42 2 21952139 or +42 2 21951111
Fax: +42 2 21952180
Email: silar@prfdec.natur.cuni.cz

DENMARK

K Dr. Kaare Lund Rasmussen
^{14}C Dating Laboratory
National Museum
Ny Vestergade 11
DK-1471 Copenhagen K, Denmark
Tel: +45 33 47 3176; Fax: +45 33 47 3310
Email: kaare.lund.rasmussen@natmus.dk

ESTONIA

Tln Dr. Enn Kaup
Radiocarbon Laboratory
Institute of Geology at Tallinn Technical University
Estonia pst 7
10143 Tallinn, Estonia
Tel: +372 645 4679; Fax: +372 631 2074
Email: kaup@gi.ee; rajamae@isogeo.gi.ee

Dr. Jaan-Mati Punning
Institute of Ecology
Kevade 2
Tallinn 10137, Estonia
Tel: +372 2 451 634; Fax: +372 2 453 748
Email: mati@eco.edu.ee

Ta Prof. Volli Kalm and Dr. Arvi Liiva
Radiocarbon Laboratory
Institute of Geology
University of Tartu
Vanemuise St. 46
51014 Tartu, Estonia
Tel/Fax: +372 7 375 836
Email: geol@ut.ee

FINLAND

Su Tuovi Kankainen
Geological Survey of Finland
P.O. Box 96
FIN-02151 Espoo, Finland
Tel: +358 205 50 11; Fax: +358 205 50 12
Email: tuovi.kankainen@gsf.fi

Hel Högne Jungner
Dating Laboratory
P.O. Box 11, Snellmaninkatu 3
FIN-00014 Helsinki University, Finland
Tel: +358 9 191 23436; Fax: +358 9 191 23466
Email: hogne.jungner@helsinki.fi

FRANCE

Gif Dr. Michel Fontugne
Centre des Faibles Radioactivités
Laboratoire mixte CNRS-CEA
F-91198 Gif sur Yvette, Cedex, France
Tel: +33 1 69 82 35 25; Fax: +33 1 69 82 35 68
Email: Michel.Fontugne@cfr.cnrs-gif.fr
and
Laboratoire Souterrain de Modane
Laboratoire mixte IN2I 3-CNRS/DSM-CEA
90, Rue Polset
F-73500 Modane, France

Ly Mr. Jacques Evin
CDRC - Centre de Datation par le RadioCarbone
Université Claude Bernard Lyon I, Batiment 217
43, Boulevard du 11 Novembre 1918
F-69622 Villeurbanne Cedex, France
Tel: +33 472 44 82 57; Fax: +33 472 43 13 17
Email: jacques.evin@cismsun.univ-lyon1.fr

GEORGIA

TB Dr. S. Pagava
Radiocarbon Laboratory
I.Javakhishvili Tbilisi State University
I.Chavchavadze av., 3
Tbilisi 380008 Georgia
Tel: +995 32 222105
Email: spagava@access.sanet.ge

GERMANY

Bln Dr. Jochen Görsdorf
Deutsches Archäologisches Institut
Eurasien-Abteilung
Postfach 330014
D-14191 Berlin, Germany
Tel: +49 30 203 77 275: Fax: +49 30 203 77 275
Email: goerc14@zedat.fu-berlin.de

Fra Prof. Dr. Dr. Reiner Protsch von Zieten
Radiocarbon Laboratory
J. W. Goethe-Universität
Siesmayerstrasse 70
D-60323 Frankfurt am Main, Germany
Tel: +49 69 798 24764 / 24767; Fax: +49 69 798 24728

Fr Prof. Dr. Detlef Hebert
Institut für Angewandte Physik
Technische Universität Bergakademie Freiberg
D-09596 Freiberg/Sa., Germany
Tel: +49 3731 39 2371 / 2594; Fax: +49 3731 39 4004
Email: hebert@tu-freiberg.de

HAM Dr. Peter Becker-Heidmann
Institut für Bodenkunde
Universität Hamburg
Allende-Platz 2
D-20146 Hamburg, Germany
Tel: +49 40 42838 2003; Fax: +49 40 42838 2024
Email: PBeckerH@Uni-Hamburg.de
WWW: http://www.geowiss.uni-hamburg.de/i-boden/tt14c.htm

Hv Prof. Dr. M. A. Geyh
Niedersächsisches Landesamt für Bodenforschung
Postfach 510153
D-30655 Hannover-Stillweg 2, Germany
Tel: +49 511 643 2537; Fax: +49 511 643 2304
Email: Mebus.Geyh@BGR.de

Hd Dr. Bernd Kromer
Heidelberger Akademie der Wissenschaften
c/o Institut für Umweltphysik
Universität Heidelberg
Im Neuenheimer Feld 229
D-69120 Heidelberg, Germany
Tel: +49 6221 5 46 357; Fax: +49 6221 5 46 405
Email: Bernd.Kromer@iup.uni-heidelberg.de

KI Dr. Helmut Erlenkeuser and Prof. Dr. Pieter M. Grootes
Leibniz-Labor
Christian-Albrechts-Universität
Max-Eyth-Str. 11
D-24118 Kiel, Germany
Tel: +49 431 880 3894 (P.M.G.); +49 431 880 3896 (H.E.)
Fax: +49 431 880 3356
Email: pgrootes@leibniz.uni-kiel.de; herlenkeuser@leibniz.uni-kiel.de
WWW: http://www.uni-kiel.de:8080/leibniz/indexe.htm

KN Dr. Bernhard Weninger
Labor für ^{14}C-Datierung
Institut für Ur-und Frühgeschichte
Universität zu Köln
Weyertal 125
D-50923 Köln, Germany
Tel: +49 221 470 2880 / 2881; Fax: +49 221 470 4892

LZ Dr. Achim Hiller
UFZ-Umweltforschungszentrum Leipzig-Halle GmbH
Sektion Hydrogeologie
Arbeitsgruppe Paläoklimatologie
Theodor-Lieser-Strasse 4
D-06120 Halle, Germany
Tel: +49 345 5585 226; Fax: +49 345 5585 559
Email: hiller@hdg.ufz.de

GREECE

DEM Dr. Yannis Maniatis
Laboratory of Archaeometry
Institute of Materials Science
National Centre for Scientific Research "Demokritos"
153 10 Aghia Paraskevi Attikis
Greece
Tel: +30 1 6503389 or +30 1 6524821; Fax: +30 1 6519430
Email: maniatis@ims.demokritos.gr
WWW: http://www.ims.demokritos.gr/archae

LIH Dr. Nicolaos Zouridakis
Laboratory of Isotope Hydrology
Institute of Physical Chemistry
National Centre for Scientific Research "Demokritos"
153 10 Aghia Paraskevi Attikis
POB 60228
Greece
Tel: +30 1 6503969; Fax: +30 1 6511766
Email: nizouri@cyclades.nrcps.ariadne-t.gr

HUNGARY

Deb Dr. Zsusa Szanto
Institute of Nuclear Research of the Hungarian Academy of Sciences
H-4026 Bem tér 18/c,
P.O. Box 51
H-4001 Debrecen, Hungary
Tel: +36 52 417266; Fax: +36 52 416181
Email: aszanto@moon.atomki.hu

ICELAND

Dr. Páll Theodórsson
Science Institute
University of Iceland
Dunhaga 3
IS-107 Reykjavík, Iceland
Tel: +354 525 4800; Fax: +354 552 8911
Email: pth@raunvis.hi.is

INDIA

PRLCH R. Bhushan, S. Krishnaswami and B. L. K. Somayajulu
Physical Research Laboratory
Chemistry Department
Oceanography and Climatic Studies Area
Navrangpura
Ahmedabad 380 009 India
Tel: +91 79 6462129; Fax: +91 79 6560502
Email: bhushan@prl.ernet.in; swami@prl.ernet.in; soma@prl.ernet.in

PRL Mr. M. G. Yadava
Radiocarbon Dating Research Unit
Oceanography and Climate Studies Area
Earth Sciences and Solar System Division
Physical Research Laboratory
Navrangpura
Ahmedabad 380 009 India
Tel: +91 79 462129; Fax: +91 79 6560502
Telegram: "Research"
Email: myadava@prl.ernet.in

JUBR Prof. S. D. Chatterjee, Dr. R. C. Sastri and Dr. Haradhan De
Biren Roy Research Laboratory for Archaeological Dating
Department of Physics
Jadavpur University
Calcutta 700 032 India
Tel: +91 33 473 4044; Fax: +91 33 473 4266; Tlx: 21-4160 (VC JU IN)

BS Dr. G. Rajagopalan
Radiocarbon Laboratory
Birbal Sahni Institute Palaeobotany
PO Box 106, 53 University Road
Lucknow 226 007 India
Tel: +91 522 32 4291; Fax: +91 522 37 4528, +91 522 38 1948
Email: bsip@bsip.sirnetd.ernet.in

INDONESIA

Mr. Wandowo
Section Hydrology
National Atomic Energy Agency
Pasar Jumat, P.O. Box 2
Kebayoran Lama
Djakarta, Indonesia

Dr. Wisjachudin Faisal
Staff of Research and Development Center for Advance Technology
National Nuclear Energy Agency
Jl. Babarsari PO Box 1008
Yogyakarta 55101 Indonesia
Tel: +62 274 515435
Fax: +62 274 561824
Email: p3tm@indo.net.id *or* wisya@batan.go.id

IRAN

TUNC Dr. A. Mahdavi
Tehran University Nuclear Centre
P.O. Box 2989
Tehran, Iran

IRELAND

UCD
Drs. Peter I. Mitchell and Edward McGee
UCD Radiocarbon Laboratory
Department of Experimental Physics
University College Dublin
Belfield, Dublin 4, Ireland
Tel: +353 1 706 2220 / 2225 / 2222
Fax: +353 1 283 7275
Email: Peter.Mitchell@ucd.ie; Edward.Mcgee@ucd.ie
WWW: http://www.ucd.ie/~radphys

ISRAEL

RT
Mr. Israel Carmi, Dr. Elisabetta Boaretto
Department of Environmental Sciences and Energy Research
Weizmann Institute of Science
76100 Rehovot, Israel
Tel: +972 8 342544; Fax: +972 8 344124
Email: cicarmii@wis.weizmann.ac.il; elisa@wis.weizmann.ac.il

ITALY

ENEA
Dr. Agostino Salomoni
ENEA Radiocarbon Laboratory
Via dei Colli, 16
I-40136 Bologna, Italy
Tel: +39 51 6098168; Fax: +39 51 6098187

R
Dr. Salvatore Improta
Dipartimento di Fisica
Università "La Sapienza"
Piazzale Aldo Moro, 2
I-00185 Rome, Italy
Tel: +39 6 49914208
Fax: +39 6 4957697
Email: Salvatore.Improta@roma1.infn.it
and
Dr. Giorgio Belluomini
Radiocarbon Laboratory
Istituto per le Tecnologie Applicate ai Beni Culturali
Consiglio Nazionale delle Ricerche
Area della Ricerca di Roma
CP 10 – Via Salaria Km 29,300
I-00016 Monterotondo St., Rome, Italy
Tel: +39 06 90672469; Fax: +39 06 90672373
Email: belluomi@mlib.cnr..it

Rome
Prof. Gilberto Calderoni
Department of Earth Sciences
University of Rome "La Sapienza"
Piazzale Aldo Moro, 5
I-00185 Rome, Italy
Tel: +39 6 499 14580; Fax: +39 6 499 14578
Email: calderoni@axrma.uniroma1.it

UD
Dr. Piero Anichini, Dr. Valerio Barbina, per.ind. Ennio Virgili
Azienda Speciale Servizi Laboratorio e CRAD
Via Nazionale, 33
I-33040 Pradamano UD, Italy
Tel: +39 432671061; Fax: +39 432671176
Email: laboratorio@azservizi.cciaa-ud.xnet.it

JAPAN

KEEA
Dr. Yoshimasa Takashima
Kyushu Environmental Evaluation Association
1-10-1, Matsukadai, Higashiku
Fukuoka 813-0004, Japan
Tel: +81 92 662 0410; Fax: +81 92 662 0990
Email: kawamura@keea.or.jp

KSU
Prof. Osamu Yamada
Faculty of Science
Kyoto Sangyo University
Kita-ku, Kyoto 603 Japan

OR
Setsuko Shibata
Research Center of Radioisotopes
Research Institute for Advanced Science and Technology
University of Osaka Prefecture
1-2, Gakuen-cho, Sakai, Japan
Tel: +81 722 36 2221; Fax: +81 722 54 9938
Email: shibata@riast.osakafu-u-ac.jp

GaK
Dr. Kunihiko Kigoshi
Radiocarbon Laboratory
Gakushuin University
Mejiro Toshima-ku, 1-5-1, Faculty of Science
Tokyo 171, Japan
Tel: +81 3 3986 0221 ext. 6482; Fax: +81 3 5992 1029
Email: kunihiko.kigoshi@gakushuin.ac.jp

PAL
Dr. Shigemoto Tokunaga
Radiocarbon Laboratory
Palynosurvery Co.
Nissan Edobashi Bld.
1-10-5 Honcho, Nihonbashi
Chuoku, Tokyo, Japan
Tel (office): +81 3 3241 4566; (lab) +81 274 42 8129
Fax: +81 3 3241 4597
Email: palynoa@blue.ocn.ne.jp

TK
Dr. Kunio Yoshida
C-14 Dating Laboratory
The University Museum
The University of Tokyo
7-3-1 Hongo, Bunkyo-ku
Tokyo 113 Japan
Tel: +81 3 3812 2111
Fax: +81 3 3814 4291
Email: gara@um.u-tokyo.ac.jp

NU
Dr. Kunio Omoto
Radiocarbon Dating Laboratory
Department of Geography
College of Humanities and Science
Nihon University
25-40, 3 Chome, Sakurajosui
Setagaya-ku, Tokyo 156 Japan
Tel: +81 35317 9273 *or* +81 33303 1691
Fax: +81 35317 9429 *or* +81 33303 9899
Email: omoto@chs.nihon-u.ac.jp

JGS
Dr. Hajime Kayanne
Department of Geography
University of Tokyo
Mongo 113-0033, Tokyo, Japan
Tel: +81 3-5841-4573
Fax: +81 3-3814-6358
kayanne@geogr.s.u-tokyo.ac.jp

KOREA

Dr. Jung Sun Ahn
Advanced Atomic Energy Research Institute
150, Duk-Jin Dong, Seo-Ku
Daejeon, Chung Nam, Korea

KCP
Dr. Hyung Tae Kang and Kyung Yim Nah
Archaeological Studies Division
National Cultural Property Research Institute
1-57 Sejongno Chongno-gu
Seoul, Korea 110 050
Tel: +82 2 735 5281 ext. 323; Fax: +82 2 735 6889
Email: vvyckht1@chollian.net

LATVIA

Riga
Dr. V. S. Veksler and Dr. A. A. Kristin
Institute of Science - Application Research
Riga 50 Merkelya 11
Riga 226 050, Latvia
Tel: +371 7 212 501 *or* +371 7 213 636

LITHUANIA

Dr. T. Bitvinskas
Institute of Botany
Lithuanian Academy of Sciences
Laieves Str. 53
Kaunas 233 000 Lithuania

Dr. Jonas Mažeika
Radioisotope Laboratory
Institute of Geology
Sevcenkos 13
Vilnius 2600 Lithuania
Tel: +370 2 236103
Fax: +370 2 236710
Email: jonmaz@geologin.lt

Dr. R. Krenyavichus
Institute of Physics and Mathematics
Lithuanian Academy of Sciences
K. Pozelos Str. 54
Vilnius 232 000 Lithuania
Tel: +370 2 641836

MONACO

IAEA-MEL
Prof. Pavel Povinec (*see also* Slovakia)
International Atomic Energy Agency
Marine Environmental Laboratory
4 Quai Antoine 1er
MC-98012 Monaco
Tel: +377 979 77216; Fax: +377 979 77273
Email: p.povinec@iaea.org

THE NETHERLANDS

GrN
Dr. J. van der Plicht
Centre for Isotope Research
University of Groningen
Nijenborgh 4
NL-9747 AG Groningen, The Netherlands
Tel: +31 50 3634760; Fax: +31 50 3634738
Email: plicht@phys.rug.nl

NEW ZEALAND

Wk
Dr. A. G. Hogg and Dr. T. F. G. Higham
Radiocarbon Laboratory
University of Waikato
Private Bag
Hamilton, New Zealand
Tel: +64 7 838 4278; Fax: +64 7 838 4192
Email: ahogg@waikato.ac.nz, *or* thigham@waikato.ac.nz
WWW: http://c14.sci.waikato.ac.nz

NZ
Dr. Rodger Sparks
Rafter Radiocarbon Laboratory
Institute of Geological and Nuclear Sciences, Ltd.
P.O. Box 31-312
Lower Hutt, New Zealand
Tel: +64 4 570 4671; Fax: +64 4 570 4657
Email: R.Sparks@gns.cri.nz

NORWAY

T
Steinar Gulliksen
Radiological Dating Laboratory
Norwegian University of Science and Technology
N-7491 Trondheim, Norway
Tel: +47 73 593310; Fax: +47 73 593383
Email: Steinar.Gulliksen@vm.ntnu.no

POLAND

Gd
Prof. Anna Pazdur and Dr. Tomasz Goslar
Radiocarbon Laboratory
Silesian University of Technology
Institute of Physics
Krzywoustego 2
PL-44-100 Gliwice, Poland
Tel: +48 32 2372254; Fax: +48 32 2372488
Email: pazdur@zeus.polsl.gliwice.pl

KR
Tadeusz Kuc
Krakow Radiocarbon Laboratory
Environmental Physics Department
University of Mining and Metallurgy
PL-30-059 Krakow, Poland
Tel: +48 12 6172979 *or* 6333740; Fax: +48 12 6340010
Tlx: 0322203 agh pl
Email: kuc@novell.ftj.agh.edu.pl

LOD
Paweł Trzeciak and Ireneusz Borowiec
Radiochemical Laboratory
Archaeological and Ethnographical Museum in Lódz
Pl. Wolnšci 14
PL-91-415 Lódz, Poland
Tel: +48 42 6328440 *or* +48 42 6334307
Fax: +48 42 6329714
Email: jotmol@krysia.uni.lodz.pl

PORTUGAL

Sac
A. M. Monge Soares
Laboratório de Isótopos Ambientais
Instituto Tecnológico e Nuclear
Estrada Nacional 10
P-2686 Sacavém Codex, Portugal
Tel: +351 1 9550021; Fax: +351 1 9441455
Email: amsoares@itn1.itn.pt

REPUBLIC OF CHINA

NTU Dr. Tsung-Kwei Liu
Department of Geology
National Taiwan University
245 Choushan Road
Taipei, Taiwan, Republic of China
Tel/Fax: +886 2 3657380
Email: liutk@ccms.ntu.edu.tw

RUSSIA

KRIL Dr. E. Starikov
Radiocarbon Dating Laboratory
Krasnoyarsk Institute of Forest and Wood
Russian Academy of Sciences
Prospect Mira 53
Krasnoyarsk 660036 Russia

MAG Dr. Anatoly V. Lozhkin
Quaternary Geology and Geochronology Laboratory
Northeast Interdisciplinary Scientific Research Institute
Russian Academy of Sciences, Far East Branch
16 Portovaya St
Magadan 685000 Russia
Email: lozhkin@neisri.magadan.su

GIN Dr. L. D. Sulerzhitsky
Geological Institute
Russian Academy of Sciences
Pyzhevsky 7
Moscow 109017 Russia
Tel: +7 095 230 8136
Email: suler@geo.tv-sign.ru, suler@ginran.msk.su

IEMAE Prof. L. Dinesman
Institute of Ecology and Evolution
Russian Academy of Sciences
Leninsky Prospect 33
Moscow 117071 Russia
Fax: +7 095 954 5534
Email: sevin@sovamsu.sovusa.com

IGAN Dr. O. A. Chichagova
Institute of Geography
Russian Academy of Sciences
Staromonetnyi 29
Moscow 109017 Russia
Tel: +7 095 230 8366; Fax: +7 095 959 0033
Email: ochichag@mtu-net.ru

IORAN Dr. V. Kuptsov, Chief of Isotope Group
P.P. Shirshov Institute of Oceanology
Russian Academy of Sciences
Nakhimovsky Prospekt, 23
Moscow 117851 Russia
Fax: +7 095 1245983

IWP Dr. Yu. A. Karpytchev
Isotope Laboratory
Institute of Water Problems
Russian Academy of Sciences
13/3 Sadovo-Tschernogryazskaya
Moscow 103064 Russia
Tel: +7 095 208 5471

MSU
Prof. P. A. Kaplin and Dr. O. B. Parunin
Laboratory of Recent Sediments and Pleistocene Paleogeography
Moscow State University
Vorobyovy Gory
Moscow 119899 Russia
Email: g1706@mail.ru

Dr. M. Alekseev
Russian Section, INQUA
Pyshevsky 7
Moscow 109017 Russia
Tel: +7 095 230 8026 / 8188

Dr. V. Polyakov
All-Union Hydrogeological Institute
Moscow District
Zelyonyi
Noginskyi Rayon 142452 Russia

SOAN
Dr. L. Orlova
United Institute of Geology, Geophysics and Minerology (UIGGM SB RAS)
Universitetsky pr. 3
630090 Novosibirsk 90 Russia
Tel: +7 3832 352 654; +7 3832 357 363; Fax: +7 3832 352 692
Email: vitaly@uiggm.nsc.ru; Tlx: 133 123 KORA SU

IVAN
Drs. O. A. Braitseva, S. N. Litasova
I. V. Melekestev and V. N. Ponomareva
Institute of Volcanology
Bul Piipa 9
Petropavlovsk-Kamchatsky 683006 Russia
Tel: +7 5 91 94

LE
Dr. Ganna Zaitseva
Institute of the History of Material Culture
Russian Academy of Sciences
Dvortsovaya Naberezhnaya, 18
191186 St. Petersburg, Russia
Tel: +7 812 311 8156; Fax: +7 812 311 6271
Email: ganna@mail.wplus.net

LU
Dr. Prof. Kh. A. Arslanov
Geographical Research Institute
St. Petersburg State University
Sredniy Prospect 41
St. Petersburg 193004 Russia
Tel/Fax: +7 812 218 7904
Email: kozyrev@mail.nevalink.ru

Prof. G. E. Kocharov
A. F. Ioffe Physico-Technical Institute
Russian Academy of Sciences
Polytechnicheskaya 26
St. Petersburg 194021 Russia
Tel: +7 812 247 9167; Fax: +7 812 247 7928
Email: Grant.Kocharov@pop.ioffe.rssi.ru

Prof. Dr. Barbara Yakheemovich or Dr. E. K. Latypova
Institute of Geology
Russian Academy of Science
October Revolution 10
Ufa 450025 Russia
Tel: +7 3472 220712; Fax: +7 3472 223569

Dr. A. Korotky
Institute of Geography
Russian Academy of Sciences
Radio 7
Vladivostok 690032 Russia

Dr. Yaroslav V. Kuzmin
Pacific Institute of Geography
Far Eastern Branch of the Russian Academy of Sciences
Radio St. 7, Vladivostok 690041 Russia
Fax: +7 423 2312159
Email: ykuzmin@tigdvo.marine.su

PI Dr. V. Kostyukevich
Permafrost Institute, Siberian Branch
Russian Academy of Sciences
Sergelyakh
Yakutsk 677010 Russia

SLOVAKIA

Ba Prof. Pavel Povinec (*see also* Monaco)
Department of Nuclear Physics
Comenius University
Mlynská dolina F1
842 15 Bratislava, Slovakia
Fax: +42 7 725882

SOUTH AFRICA

Pta S. Woodborne
Quaternary Research Dating Unit (QUADRU)
c/o Enviromentek, CSIR
P.O. Box 395
0001 Pretoria, South Africa
Tel: +27 12 841 3380; Fax: +27 12 349 1170
Email: swoodbor@CSIR.co.za

SPAIN

UBAR Prof. Gemma Rauret and Dr. Joan S. Mestres
Laboratori de Datació per Radiocarboni
Departament de Química Analítica
Universitat de Barcelona
Facultat de Química, 3a. Planta
C/. Martí i Franquès, 1-11/Avda. Diagonal, 647
E-08028 Barcelona, Spain
Tel: +34 3 402 1281; Fax: +34 3 402 1233
Email: jmestres@d3.ub.es

UGRA Prof. M. Purificación Sánchez and Dr. Elena Villafranca
Laboratorio de Datación por C-14
Centro de Instrumentación Científica
Campus Fuentenueva, Ed. Mecenas
Universidad de Granada
E-18071 Granada, Spain
Tel: +34 58 244229; Fax: +34 58 243391
Email: mpsansan@goliat.urg.es *and* jlazuen@goliat.urg.es

CSIC Dr. Fernán Alonso and Antonio Rubinos
Geochronology Laboratory
Instituto de Química-Física Rocasolano - CSIC
Serrano, 119
28006 Madrid, Spain
Phone: +34 91 561 9400
Fax: +34 91 564 2431
Email: f.alonso@iqfr.csic.es or rubinos@iqfr.csic.es

SWEDEN

Lu Göran Skog
Radiocarbon Dating Laboratory
University of Lund
Tornavägen 13
SE-223 63 Lund,
Tel: +46 46 222 7885; Fax: +46 46 222 4830
Email: Goran.Skog@c14lab.lu.se

U Dr. Ingrid U. Olsson
Department of Physics
Uppsala University
Box 530
SE-751 21 Uppsala, Sweden
Tel: +46 18 4713571; Fax: +46 18 4713524
Email: ingrid.olsson@fysik.uu.se

SWITZERLAND

B Thomas Stocker
Climate and Environmental Physics
Physics Institute
Sidlerstrasse 5
CH-3012 Bern, Switzerland
Tel: +41 31 631 44 64; Fax: +41 31 631 44 05
Email: stocker@climate.unibe.ch
WWW: http://www.climate.unibe.ch

THAILAND

Director
Chemistry Department
Office of Atomic Energy for Peace
Vibhavadi Rangsit Road
Anabang, Bangkok, Thailand

TURKEY

METU Dr. Mustafa Özbakan
Radiocarbon Dating Laboratory
Middle East Technical University
Department of Physics
06531 Ankara, Turkey
Tel: +90 312 210 32 76; Fax: +90 312 210 12 81
Email: ozbakan@metu.edu.tr

UKRAINE

Ki Dr. Nikolai N. Kovalyukh and Dr. Vadim V. Skripkin
National Academy of Sciences and Ministry of Extraordinary Situation of Ukraine
State Scientific Centre of Environmental Radiogeochemistry
Kyiv Radiocarbon Laboratory
Palladin 34
Kyiv-142
252680 Ukraine
Tel/Fax: +38 0 44 444 0060
Fax: +38 0 44 444 1465
Email: kyiv14c@radgeo.freenet.kiev.ua

URCRM Dr. Michael Buzinny
Ukrainian Research Center for Radiation Medicine
Academy of Medical Sciences of Ukraine
04050, Melnikova str. 53
Kiev, Ukraine
Tel: +380 44 2445874; Fax: +380 44 2137202
Fax: +1 413 473-3008
Email: buzinny@bigfoot.com; buzinny@hotmail.com
WWW: http://bigfoot.com/~buzinny

UNITED KINGDOM

Birm R. E. G. Williams
Department of Geological Sciences
P.O. Box 363
University of Birmingham
Birmingham B15 2TT England

Q Prof. Roy Switsur
Cambridge Radiocarbon Dating Research Laboratory
Environmental Sciences Research Centre
East Road
Cambridge CB1 1PT England
Tel: +44 1223 363271 x2594
Email: vrs1@cam.ac.uk

BM Janet Ambers
Department of Scientific Research
The British Museum
London WC1B 3DG England
Tel: +44 171 323 8332; Fax: +44 171 323 8276
Email: J.Ambers@british-museum.ac.uk

RCD R. L. Otlet / A. J. Walker
RCD – Radiocarbon Dating
The Old Stables
East Lockinge, Wantage
Oxon OX12 8QY England
Tel/Fax: +44 1235 833667

UB Dr. Gerry McCormac
Radiocarbon Dating Laboratory
School of Archaeology and Palaeoecology
The Queen's University of Belfast
Belfast BT7 1NN Northern Ireland
Tel: +44 2890 335141; Fax: +44 2890 315779
Email: f.mccormac@qub.ac.uk
WWW: http://www.qub.ac.uk/arcpal

GU Dr. G. T. Cook
SURRC Radiocarbon Dating Laboratory
Scottish Universities Research & Reactor Centre
Scottish Enterprise Technology Park
East Kilbride G75 0QF Scotland
Tel: +44 13552 23332 or +44 13552 70136; Fax: +44 13552 29898
Email: g.cook@surrc.gla.ac.uk

SRR Professor A. E. Fallick
NERC Radiocarbon Laboratory
Scottish Enterprise Technology Park
Rankine Avenue
East Kilbride, Glasgow G75 0QF Scotland
Tel: +44 1355 260037; Fax: +44 1355 229829
Email: radiocarbon@nercrcl.gla.ac.uk
WWW: http://www.gla.ac.uk/nercrcl

Dr. E. M. Scott
Department of Statistics
University Gardens
University of Glasgow
Glasgow, G12 8QW, Scotland
Tel: +44 141 330 5125; Fax: +44 141 330 4814
Email: marian@stats.gla.ac.uk

SWAN Quentin Dresser
Department of Geography
University of Wales, Swansea
Singleton Park, Swansea
West Glamorgan SA2 8PP Wales
Tel: +44 1792 295148; Fax: +44 1792 295955
Email: P.Q.Dresser@swansea.ac.uk

UNITED STATES

A Dr. Austin Long
Laboratory of Isotope Geochemistry
Geosciences Department
The University of Arizona
Tucson, Arizona 85721 USA
Tel: +1 520 621 8888; Fax: +1 520 621 2672
Email: along@geo.arizona.edu

UCI Dr. Ellen Druffel and Ms. Sheila Griffin
Radiocarbon Laboratory
Department of Earth System Science
University of California, Irvine
PSRF 207
Irvine, California 92697-3100 USA
Tel (Druffel/office): +1 949 824 2116
Fax: +1 949 824 3256; Email edruffel@uci.edu

UCLA Prof. Rainer Berger
Institute of Geophysics and Planetary Physics
University of California
Los Angeles, California 90024 USA
Tel: +1 310 825 4169; Fax: +1 310 206 3051

UCR Prof. R. E. Taylor
Radiocarbon Laboratory
Department of Anthropology
Institute of Geophysics and Planetary Physics
University of California, Riverside
Riverside, California 92512 USA
Tel: +1 909 787 5521; Fax: +1 909 787 5409
Email: retaylor@citrus.ucr.edu

Beta Dr. M. A. Tamers and Mr. D. G. Hood
Beta Analytic Inc.
4985 SW 74 Court
Miami, Florida 33155 USA
Tel: +1 305 667 5167; Fax: +1 305 663 0964
Email: beta@radiocarbon.com

UGA John E. Noakes
Center for Applied Isotope Studies
The University of Georgia
120 Riverbend Road
Athens, Georgia 30602-4702 USA
Tel: +1 706 542 1395; Fax: +1 706 542 6106

ISGS Chao-li Liu and Hong Wang
Isotope Geochemistry Section
Illinois State Geological Survey
615 E. Peabody Drive
Urbana, Illinois 61820 USA
Tel: +1 217 333 9083; Fax: +1 217 244 7004
Email: jliu@geoserv.isgs.uiuc.edu

NIST Lloyd A. Currie and George A. Klouda
Atmospheric Chemistry Group
National Institute of Standards and Technology

Currie:
100 Bureau Dr., Stop 8370
Gaithersburg, Maryland 20899-8370 USA
Tel: +1 301 975 3919; Fax: +1 301 216-1134
Email: Lloyd.Currie@nist.gov

Klouda:
100 Bureau Dr., Stop 8372
Gaithersburg, MD 20899-8372 USA
Tel: +1 301 975 3931; Fax: +1 301 216-1134
Email: George.Klouda@nist.gov

GX Alexander Cherkinsky
Geochron Laboratories, a division of
Krueger Enterprises, Inc.
711 Concord Avenue
Cambridge, Massachusetts 02138 USA
Tel: +1 617 876 3691; Fax: +1 617 661 0148
Email: acherkinsky@geochronlabs.com

I James Buckley
Teledyne Brown Engineering Environmental Services
50 Van Buren Avenue
Westwood, New Jersey 07675 USA
Tel: +1 201 664 7070; Fax: +1 201 664 5586
Tlx: 134474

QL Prof. Emeritus Minze Stuiver
Quaternary Isotope Laboratory
Box 351360
Quaternary Research Center
University of Washington
Seattle, Washington 98195-1360 USA
Tel: +1 206 685 1735; Fax: +1 206 543 3836
Email: minze@u.washington.edu

WIS Dr. David M. McJunkin
Center for Climatic Research
University of Wisconsin-Madison
1225 W. Dayton Street
Madison, Wisconsin 53706 USA
Tel: +1 608 262 7328; Fax: +1 608 262 5964
Email: mcj@facstaff.wisc.edu

URUGUAY

URU Drs. Ma. Cristina Ures and Roberto Bracco
Laboratorio de ^{14}C
Facultad de Química
Universidad de la República
Gral. Flores 2124
Montevideo, Uruguay
Tel: +598 2 924 8571; Fax: +598 2 924 1906
Email: radquim@bilbo.edu.uy

II. ^{14}C ACCELERATOR FACILITIES (AMS)

AUSTRALIA

ANSTO Dr. Ewan Lawson
ANTARES AMS Centre
Physics Division
Australian Nuclear Science and Technology Organisation (ANSTO)
New Illawarra Road
Lucas Heights, NSW 2234 Australia
Tel: +61 2 9717 3025; Fax: +61 2 9717 3257
Email: eml@ansto.gov.au

ANUA Dr. L. Keith Fifield
Department of Nuclear Physics, RSPhysSE
Australian National University
Canberra, ACT 0200 Australia
Tel: +61 2 6249 2095; Fax: +61 2 6249 0748
Email: keith.fifield@anu.edu.au

AUSTRIA

VERA Prof. Walter Kutschera
VERA-Laboratorium
Institut für Radiumforschung und Kernphysik
Universität Wien
Währingerstrasse 17
A-1090 Vienna, Austria
Tel: +43 1 4277 51700; Fax: +43 1 4277 9517
Email: Walter.Kutschera@univie.ac.at

CANADA

TO Dr. Roelf P. Beukens
IsoTrace Laboratory
University of Toronto
60 St. George Street
Toronto, Ontario, Canada M5S 1A7
Tel: +1 416 978 4628; Fax: +1 416 978 4711
Email: isotrace.lab@utoronto.ca
WWW: http://www.physics.utoronto.ca/~isotrace

CHINA

PKU Prof. Kun Li and Prof. Zhiyu Guo
Department of Technical Physics
Institute for Heavy Ion Physics
Peking University
Beijing 100871 China
Tel: +86 10 2501881 or +86 10 62501875
Fax: +86 10 2501873 or +86 10 62501875
Email: puihip@sun.ihep.ac.cn

DENMARK

AAR Dr. Jan Heinemeier and Dr. Niels Rud
AMS ^{14}C Dating Laboratory
Institute of Physics and Astronomy
University of Aarhus
DK-8000 Aarhus C, Denmark
Tel: +45 8942 3718; Fax: +45 8612 0740
Email: jh@dfi.aau.dk
WWW: http://www.c14.dk

FRANCE

Gif A GDR Tandetron
Domaine du CNRS
Avenue de la Terrasse, Bat. 30
F-91198 Gif sur Yvette Cedex, France
Tel: +33 1 69 82 39 15; Fax: +33 1 69 82 36 70

Director: Jean-Claude Duplessy
Centre des Faibles Radioactivités
Laboratoire mixte CNRS-CEA
F-91198 Gif sur Yvette Cedex, France
Tel: +33 1 69 82 35 26; Fax: +33 1 69 82 35 68
Email: Jean-Claude.Duplessy@cfr.cnrs-gif.fr

AMS ^{14}C
Maurice Arnold
Centre des Faibles Radioactivités (CEA-CNRS)
Tandetron
Avenue de la Terrasse
BP 1
F-91198 Gif sur Yvette Cedex, France
Tel: +33 1 69 82 35 63; Fax: +33 1 69 82 36 70
Email: Maurice.Arnold@cfr.cnrs-gif.fr

AMS ^{10}Be, ^{126}Al, ^{129}I
Françoise Yiou and Grant Raisbeck
CSNSM Bat. 108
F-91405 Campus Orsay Cedex, France
Tel: +33 1 69 15 52 64; Fax: +33 1 69 15 52 68
Email: yiou@csnsm.in2p3.fr; raisbeck@csnsm.in2p3.fr

GERMANY

Dr. Wolfgang Kretschmer
Physikalisches Institut
Universität Erlangen-Nürnberg
Erwin-Rommel-Str. 1
D-91054 Erlangen, Germany
Tel: +49 9131 857075; Fax: +49 9131 15249
Email: kretschmer@physik.uni-erlangen.de

Gert Jaap van Klinken
^{14}C AMS Laboratory
Max-Planck-Institut für Biogeochemie
PO Box 10 01 64
D-07701 Jena, Germany
Tel: +49 3641 643708/3806; Fax: +49 3641 643710
Email: vklinken@bgc-jena.mpg.de
WWW: www.bgc-jena.mpg.de

KIA Prof. Dr. Pieter M. Grootes, Dr. Marie-Josee Nadeau
Leibniz-Labor
Christian Albrechts Universität
Max-Eyth-Str. 11
D-24118 Kiel, Germany
Tel: +49 431 880 3894 (P.M.G.); +49 431 880 7373 (M.-J.N., M.S.)
Fax: +49 431 880 3356
Email: pgrootes@leibniz.uni-kiel.de (P.M.G.)
WWW: http://www.uni-kiel.de:8080/leibniz/indexe.htm

KOREA

SNU Prof. Jong Chan Kim
The Inter-University Center for National Science Research Facility
Seoul National University
Seoul 151-742 Korea
Tel: +82 2 880 5774; Fax: +82 2 884 3002
Email: jckim@phya.snu.ac.kr
WWW: http://npl3.snu.ac.kr

JAPAN

NUTA Dr. Toshio Nakamura
Tandetron AMS Laboratory
Dating and Materials Research Center
Nagoya University
Chikusa, Nagoya 464-8602 Japan
Tel: +81 52 789 2578; Fax: +81 52 789 3095
Email: g44466a@nucc.cc.nagoya-u.ac.jp

THE NETHERLANDS

GrA Dr. J. van der Plicht
Centre for Isotope Research
University of Groningen
Nijenborgh 4
NL-9747 AG Groningen, The Netherlands
Tel: +31 50 3634760; Fax: +31 50 3634738
Email: plicht@phys.rug.nl

UtC Dr. K. van der Borg
R. J. Van de Graaff Laboratorium
Universiteit Utrecht
Princetonplein 5
P.O. Box 80.000
3508 TA Utrecht, The Netherlands
Tel: +31 30 53 2238 or +31 30 53 1492
Fax: +31 30 2532532; Email: k.vanderborg@fys.ruu.nl

NEW ZEALAND

NZA Dr. Rodger Sparks
Rafter Radiocarbon Laboratory
Institute of Geological and Nuclear Sciences, Ltd.
P.O. Box 31-312
Lower Hutt, New Zealand
Tel: +64 4 570 4671; Fax: +64 4 570 4657; Email: R.Sparks@gns.cri.nz

SWEDEN

LuA Göran Skog
Radiocarbon Dating Laboratory
University of Lund
Tornavägen 13
SE-223 63 Lund, Sweden
Tel: +46 46 222 7885; Fax: +46 46 222 4830
Email: Goran.Skog@C14lab.lu.se

Ua Dr. Göran Possnert
Tandem Laboratory
University of Uppsala
Box 533
SE-751 21 Uppsala, Sweden
Tel: +46 18 4713059; Fax: +46 18 555736
Email: possnert@material.uu.se

SWITZERLAND

ETH Georges Bonani
ETH/AMS Facility
Institut für Teilchenphysik
Eidgenössische Technische Hochschule Hönggerberg
CH-8093 Zürich, Switzerland
Tel: +41 1 633 2041; Fax: +41 1 633 1067
Email: bonani@particle.phys.ethz.ch

UNITED KINGDOM

OxA R. E. M. Hedges / C. Bronk Ramsey
Oxford Radiocarbon Accelerator Unit
Research Laboratory for Archaeology and the History of Art
Oxford University
6 Keble Road
Oxford OX1 3QJ England
Tel: +44 1865 273939; Fax: +44 1865 273932
Email: orau@rlaha.ox.ac.uk
WWW: http://www.rlaha.ox.ac.uk

UNITED STATES

AA Dr. D. J. Donahue, Dr. P. E. Damon and Dr. A. J. T. Jull
NSF-Arizona AMS Facility
PO Box 210081
The University of Arizona
Tucson, Arizona 85721-0081 USA
Tel: +1 520 621 6810; Fax: +1 520 621 9619
Email: ams@physics.arizona.edu
WWW: http://www.physics.arizona.edu/ams

CAMS Dr. John Knezovich
Center for Accelerator Mass Spectrometry
Lawrence Livermore National Laboratory
P.O. Box 808, L-397
Livermore, California 94550 USA
Tel: +1 510 422 4520; Fax: +1 510 423 7884
Email: knezovich1@llnl.gov

NSRL Dr. Scott Lehman
INSTAAR Laboratory for AMS Radiocarbon Preparation and Research (NSRL)
CU-Boulder
1560 30th St.
Campus Box 450
Boulder, Colorado 80309-0450
Telephone: +1 303 492-0362 Fax: +1 303 492-6388
Email: Scott.Lehman@colorado.edu
WWW: http://www.Colorado.EDU/INSTAAR/RadiocarbonDatingLab

PL Dr. David Elmore
Purdue Rare Isotope Measurement Laboratory
Purdue University
1396 Physics Building
West Lafayette, Indiana 47907-1396 USA
Tel: +1 765 494 6516; Fax: +1 765 494 0706
Email: elmore@primelab.physics.purdue.edu
WWW: http://primelab.physics.purdue.edu

WHAMS John M. Hayes
National Ocean Sciences AMS Facility
Woods Hole Oceanographic Institution
McLean Laboratory, Mail Stop #8
Woods Hole, Massachusetts 02543-1539 USA
Tel: +1 508 289 2649; Fax: +1 508 457 2183
Email: shandwork@whoi.edu
WWW: http://nosams.whoi.edu

LABORATORIES – CODE DESIGNATIONS*

A	Arizona	USA
AA	NSF-Ariz. AMS Facility	USA
AAR	University of Aarhus	Denmark
AC	Ingeis	Argentina
AECV	Alberta Environmental Center of Vegreville	Canada
AERIK*	Atomic Energy Res. Inst.	Korea
ALG	Algiers	Algeria
ANL*	Argonne Nat. Lab., Ill.	USA
ANTW	Antwerp	Belgium
ANU	Australian National University	Australia
ANUA	ANU Accelerator	Australia
AU*	University of Alaska	USA
B	Bern	Switzerland
Ba	Bratislava	Slovakia
BC*	Brooklyn College	USA
Beta	Beta Analytic	USA
Birm	Birmingham	USA
Bln	Berlin	Germany
BM	British Museum	England
BONN*	Universität Bonn	Germany
BS	Birbal Sahni Institute	India
BSG	Brock University	Canada
C*	Chicago	USA
CAMS	Center for Accelerator Mass Spectrometry	USA
CAR*	Univ. College, Cardiff	Wales
CENA	Centro Energia Nuclear na Agricultura	Brazil
CG	Institute of Geology	China
CH	Chemistry Laboratory	India
CRCA	Cairo	Egypt
CSIC	Geochronology Lab, IQFR-CSIC, Madrid	Spain
CSM*	Cosmochemistry Lab. USSR Academy of Sciences	USSR
CT*	Caltech, Calif. Inst. Tech.	USA
CU	Charles University	Czech Republic
D*	Dublin, Trinity College	Ireland
Dak*	Univ. de Dakar	République du Sénégal
DAL*	Dalhousie University	Canada
DE*	USGS, Denver	USA
Deb	Debrecen	Hungary
DEM	NCSR Demokritos	Greece
DIC*	Dicar Corp and Dicarb Radioisotope Company	USA
DRI	Desert Research Institute	USA
ENEA	ENEA, Bologna	Italy
ETH	ETH/AMS Facility	Switzerland
F	Florence	Italy
Fr	Freiberg	Germany
Fra	Frankfurt	Germany
FSU*	Florida State University	USA
FZ	Fortaleza	Brazil
G*	Göteborg	Sweden
GAK	Gakushuin University	Japan
Gd	Gliwice	Poland
GD*	Gdansk	Poland
Gif	Gif sur Yvette	France
Gif A	Gif sur Yvette and Orsay	France
GIN	Geological Institute	Russia
GL*	Geochronological Lab.	England
Gro*	Groningen	The Netherlands
GrN	Groningen	The Netherlands
GrA	Groningen Accelerator	The Netherlands
GSC	Geological Survey	Canada
GU	Scottish Universities Research & Reactor Centre (formerly Glasgow University)	Scotland
GX	Geochron Laboratories	USA
H*	Heidelberg	West Germany
HAM	Hamburg	Germany
HAR*	Harwell	England
Hd	Heidelberg	Germany
Hel	Helsinki	Finland
HIG*	Hawaii Inst. of Geophys.	USA
HL	Second Institute of Oceanography	China
HNS	Hasleton-Nuclear, Palo Alto, California	USA
Hv	Hannover	Germany
I	Teledyne Isotopes	USA
IAEA	International Atomic Energy Agency	Austria
IAEA-MEL	Marine Environmental Laboratory	Monaco
ICEN	Instituto Tecnológico e Nuclear	Portugal
IEMAE	Institute of Evolutionary Morphology and Animal Ecology	Russia
IGAN	Institute of Geography	Russia
IGS*	Inst. of Geological Sci.,	Sweden
II*	Isotopes, Inc., Palo Alto	USA
IOAN	Institute of Oceanography	Russia
IORAN	Institute of Oceanology	Russia
IRPA	Royal Institute of Cultural Heritage	Belgium
ISGS	Illinois State Geological Survey	USA
IVAN	Institute of Volcanology	Ukraine
IVIC*	Caracas	Venezuela
IWP	Institute of Water Problems	Russia

*Indicates laboratories that are closed, no longer measuring ^{14}C, or operating under a different code designation.

JGS	Geological Survey of Japan	Japan
JUBR	Biren Roy Research Laboratory	India
K	National Museum	Denmark
KAERI*	Korean Atomic Energy Research Institute	Korea
KCP	National Cultural Property Research Institute	Korea
KEEA	Kyushu Environmental Evaluation Association	Japan
KI	Kiel	Germany
KIA	Kiel AMS	Germany
Ki (KIEV)	Institute of Radio-Geochemistry of the Environment	Ukraine
KN	Köln	Germany
KR	Krakow	Poland
KRIL	Krasnoyarsk Institute	Russia
KSU	Kyoto Sangyo University	Japan
L*	Lamont-Doherty	USA
LAR	Liège State University	Belgium
LE	St. Petersburg	Russia
LIH	NCSR Demokritos	Greece
LJ*	Scripps (UCSD) La Jolla	USA
LOD	Lodz	Poland
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LZ	Umweltforschungszentrum Leipzig-Halle	Germany
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Ma*	University of Winnepeg	Canada
MAG	Quaternary Geology and Geochronology Laboratory	Russia
MC*	Centre Scientifique de Monaco	Monaco
METU	Middle East Technical University	Turkey
ML*	Miami	USA
Mo*	Verdanski Inst. of Geochemistry, Moscow	USSR
MOC*	Archaeological Institute, Czechoslovak Acad. of Sci.	Czechoslovakia
MP*	Magnolia Petroleum	USA
MRRI*	Marine Resources Research Institute	USA
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NPL*	National Physical Laboratory, Middlesex	England
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PRL	Physical Research Laboratory	India
Pta	Pretoria	South Africa
Q	Cambridge	England
QL	Quaternary Isotope Laboratory	USA
QC*	Queens College	USA
QU*	Centre de Recherches Minérales, Québec	Canada
R	Rome	Italy
RCD	Radiocarbon Dating	England
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It is our great pleasure to invite you to participate in the 17th International Radiocarbon Conference, scheduled for June 18–23, 2000, in Israel.

The Conference will be held at a beautiful location, in the rural setting of Kibbutz Ma'ale Hachamisha in the Judean Hills, just west of Jerusalem. The Kibbutz offers an attractive self-contained arrangement of excellent accommodation and conference facilities, which will enable a high degree of interaction between conference participants. The City of Jerusalem with its unique history and tourist attractions is a short drive away and is easily reached by bus or taxi.

The first ^{14}C Conference at the dawn of a new millennium will undoubtedly include exciting new scientific developments. Keeping the tradition of past Radiocarbon conferences, the scientific program will include a wide variety of topics. Sessions will be devoted to:

- Archaeology – with a special session on ^{14}C data of historical periods in the Near East
- Calibration of the ^{14}C time scale
- Sample treatment and measurement techniques
- Geophysics and Geochemistry of ^{14}C
- Cosmogenic radionuclides
- Environment past and present
- Global change
- Glaciology
- Hydrology
- Oceanography
- Geology
- Soils

Participants are welcome to indicate their preference for either oral or poster presentation of their papers. However, the final decision regarding form of presentation will be made by the Organizing Committee. Detailed information will be included in the Second Announcement.

The social program of the Conference will include an afternoon walking tour in the Old City of Jerusalem and a one-day tour in the unique Dead Sea area (lowest point on the earth's surface). A floating swim in the Dead Sea – which is seven times saltier than the ocean – is indeed a unique fun experience.

Sincerely, The Organizing Committee:

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Elisabetta Boaretto, Secretary, The Weizmann Institute of Science
Hendrik J. Bruins, Ben Gurion University of the Negev
Michael Paul, Hebrew University of Jerusalem
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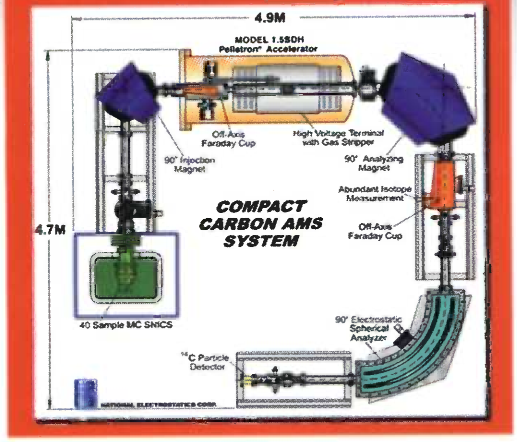

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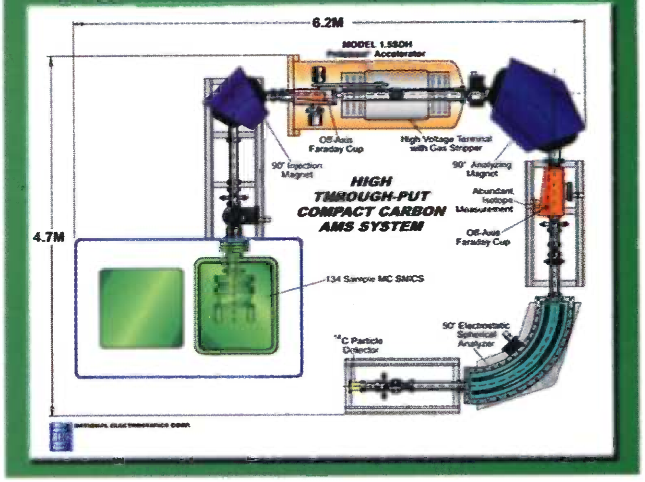

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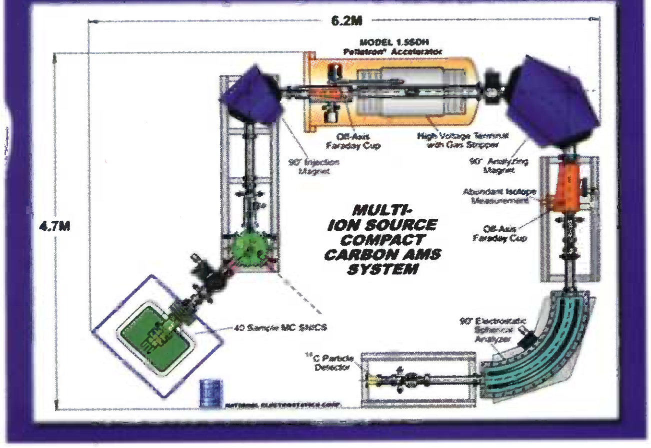

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