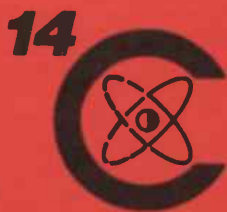


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# Radiocarbon

An International Journal of Cosmogenic Isotope Research

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**$^{14}\text{C}$  AND SOIL DYNAMICS: Special Section**

**Guest Editors:**

D. D. Harkness

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# Announcement

*RADIOCARBON* has reduced its price for individual subscribers down to 1988 levels! Effective 12 January 1997, the subscription price for new subscribers is \$55/year (plus \$10 surface postage for non-U.S. addresses). All other individual subscriptions beginning with Volume 40 will also cost \$55. (Institutional subscription rates are not affected.)

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## TO ALL MY FRIENDS

After a little more than a year of fighting two bouts with malignant tumors—first in the lung, then in the brain—I think it is high time that you read a few words directly from me.

Cancer is a disease of both physical and emotional losses. Sometimes I don't know which are worse. Both are devastating and most are permanent, changing only in degree and stage in my disease, treatment and mental attitude. The trick is to deal with the changes and losses as they occur, and to deal with the concomitant shock, fear, anger, frustration, anxiety, panic, depression, loneliness, despair and thousands of other burning emotions. The idea is to translate the turbulence into something meaningful, hopeful and almost acceptable. This is a chore of constant hard work and help from professionals, friends and family. And just when you think you have one issue resolved, something changes, and you have to battle it again. The excellent doctors who care for me cannot promise miracles, but they can give me endless encouragement, which they do. All we can hope for is daily progress and a good prognosis for the near future. So far, I have both.

Cancer is also a disease of gains. I have learned a great deal about myself, as an individual and in relation to others. I learned a great deal about cancer—the dreadful statistics and the anecdotal miracles. When I visited the *RADIOCARBON* office the other day, I met a professor whom I hadn't seen for some time. He was extremely happy to see me and said, "By this time you must be the wisest person in the world." Sometimes I feel wise, but other times I feel just as confused as when this all started.

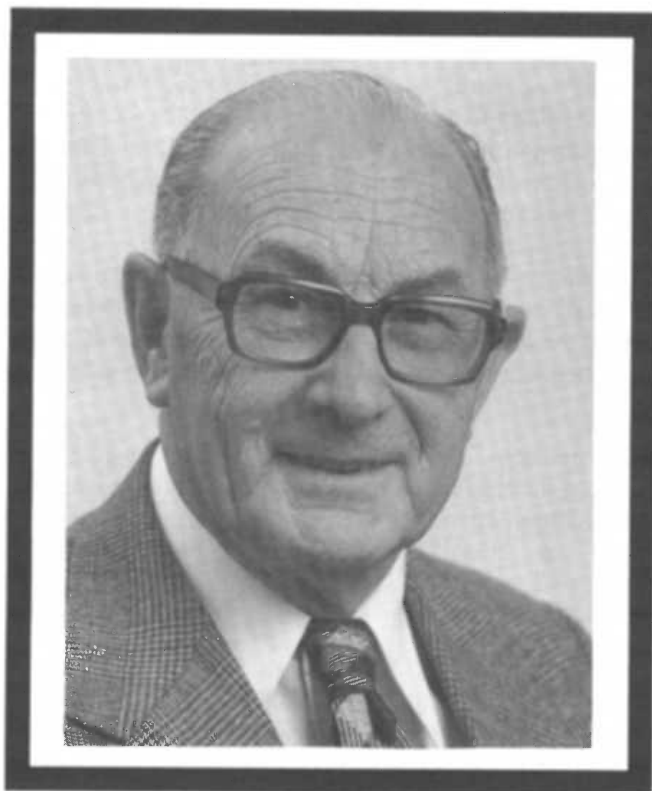
I learned how wonderful my family could be, especially my daughters, who crossed the country time after time, as if they rode a shuttle bus instead of an airplane; my friends from the east coast and beyond, who dropped their busy careers, and came to care for me for a week or more following surgery and during treatment; my many friends in Tucson for their endless support and devotion; the *RADIOCARBON* staff who took charge of all the backlogged work and completed one project after another, and set up the "Delicious Deliveries" fund (which has not yet run out!); the Geosciences Department, in particular, and the University of Arizona, in general, for showing so much kindness, helpfulness and generosity with all the "red tape" of claims and forms, *etc.*; and my *RADIOCARBON* friends for sending tons of letters, cards, notes, e-mail messages, gifts and contributions. I mention here another personal event relevant to my department and university. Recently, I read an article on the front page of our local newspaper. The photograph of a woman with a bald head caught my attention. The article described how this woman was fired from her job because she did not have the energy to keep up with her usual performance due to chemotherapy treatments for breast cancer. I cannot describe my feelings and my gratitude for my good fortune!

A new world opened up to me when I became involved with therapy at the Arizona Cancer Center and the University Medical Center. How lucky can a person possibly be to have ongoing in Tucson, state-of-the-art cancer research and to benefit from new and aggressive programs? But to build bonds with courageous patients, brilliant and compassionate doctors who became friends, skilled and caring technicians and nurses is an experience I would not want to have missed.

It will take me a while to re-enter the *RADIOCARBON* world, and I cannot promise that I will, fully. David Sewell is doing a splendid job of being Acting Managing Editor, which he could not manage without the full support and expertise of Kim Tanner Elliott. My presence will occasionally pervade the hallways of *RADIOCARBON*, overseeing and advising on some of the activities and projects, wherever possible. I look forward to our renewals (of friendship, of course)!

*Renee Kra*





**ATHOL RAFTER, 1913-1996**

One of the pioneers of radiocarbon, Thomas Athol Rafter died peacefully on 26 September 1996, at the age of 83.

Athol Rafter was born in Wellington, New Zealand, on 5 March 1913. He grew up in Wellington, attended school there (St. Patrick's College), and graduated from Victoria University of Wellington, then a College of the University of New Zealand, in 1938. Athol first attracted attention while at secondary school, not so much for his academic achievements as for his abilities as a middle distance athlete. In his early career, he worked as a schoolteacher, and in 1940 he was appointed to the staff of the Dominion Laboratory of the New Zealand Department of Scientific and Industrial Research (DSIR). At the end of World War II, Athol was one of a small group of scientists selected to investigate the possibilities opened up by the new field of nuclear science. As a chemist, Athol learned the techniques necessary for handling and using radioactive isotopes. Gordon Fergusson designed and constructed instruments for monitoring background radioactivity, and was joined by Graham McCallum. Others, particularly Bill McCabe, assisted Athol in such projects as using labeled  $^{32}\text{P}$  for studying the action of superphosphate as a soil fertilizer. This isotope was also investigated as a therapeutic tool for cancer treatment.

According to Athol's own account, it was the head of the DSIR, F. R. Callaghan, who asked him, in 1950, if he would look into a new method of measuring the ages of volcanic ash showers using radioactive carbon, "to stop the geologists arguing". Accordingly, Athol assembled a small team

comprising himself, Alex Wilson and Bill McCabe to work on the chemistry side, while Fergusson, McCallum and Fred Knox developed a suitable counting system. These initial attempts basically copied Libby's original method of using a solid carbon counter, and Athol corresponded with Libby on the best method of depositing a thin carbon layer on the inner copper surface. However, the New Zealand team soon encountered problems with this approach. One of the problems was that the chemical preparation laboratory was in Wellington, but the counting laboratory was in Lower Hutt, about 18 km away, and it was necessary to carry the counter with its delicate carbon layer by car along a road that bore little resemblance to the highway of today. All too often, the carbon had been shaken off the counter surface by the time it reached the Lower Hutt laboratory. It was at the end of such a trip that Bill McCabe recalls Athol remarking that there had to be a better way to do this! Other problems involved the difficulties of keeping the carbon free of radioactive contaminants introduced during the carbon reduction stage, and so attention shifted to developing a counter using CO<sub>2</sub> as the counter gas.

At this time, Hessel de Vries and G. W. Barendson were also working on a CO<sub>2</sub> counter, but their system was successful only for small volumes of gas. Athol and his team were in contact with de Vries, and they realized that the purity of the CO<sub>2</sub> was of paramount importance. By concentrating their efforts as much on the quality of the counting gas as on the configuration of the counter itself, they finally succeeded in producing a 7.7-liter counter system that could measure ages to a precision of better than  $\pm 50$  yr, a major achievement for the time.

With the radiocarbon laboratory now fully established at Lower Hutt, Athol turned his attention to monitoring natural <sup>14</sup>C levels in the environment. Regular sampling programs to measure <sup>14</sup>C in the atmosphere and the southern ocean were begun. This work began sufficiently early (in fact just in time) to establish a baseline of atmospheric <sup>14</sup>C before nuclear weapons testing changed the picture entirely. Athol reported the first measurements of environmental radiocarbon produced by nuclear weapons testing in 1957, but this was not just a lucky observation. He had signaled his intentions in his previous 1955 paper on <sup>14</sup>C variations in nature, the last sentence of which reads, "Samples for <sup>14</sup>C activity of the atmosphere are being constantly taken to study any trends with seasons or possible enrichment of the atmosphere in <sup>14</sup>C from atomic explosions that may not as yet have reached this part of the Earth's atmosphere."

This account of Athol Rafter's career has concentrated entirely on his work with <sup>14</sup>C, and does not cover his achievements in isotope geochemistry or his later connections with teaching. But I think it is important to finish with a view of him as man and a leader. Athol was in many respects a plain man, with a plain man's propensity to go straight to what is important, unhampered by a veneer of sophistication or undue intellectualizing. For him there was no conflict between being an eminent scientist and having a devoutly religious view of life. His postwar work culminated in his being appointed foundation Director of the Institute of Nuclear Sciences, a position he held until his retirement in 1978. In this role, his leadership was clear and firm, without in any way conforming to the stereotype of the autocratic director. He always maintained an easy relationship with his staff, and seemed to see them as part of his extended family. In the early days of the Institute, a tradition grew of Athol entertaining the entire staff at his home on the last day before the Christmas shutdown. One year, in a return gesture, the staff presented him with a glass candleholder shaped in the form of a hydrogen atom, complete with nucleus and orbiting electron. The following year it seemed appropriate to repeat the gesture, this time with a representation of a helium atom. Once the pattern was established, year by year, the candle progressed inexorably through the periodic table, to the growing despair of the Institute glassblower. Spontaneous fission set in at  $Z = 5$ , and the custom was reluctantly abandoned.



The leadership Athol gave spread beyond Lower Hutt. Alex Wilson (now at The University of Arizona) went on to become professor of chemistry at Waikato University, where he set up the  $^{14}\text{C}$  laboratory now ably managed by Alan Hogg. Henry Polach left the Institute of Nuclear Sciences to establish the radiocarbon laboratory at the Australian National University. Athol retired before the full impact of accelerator mass spectrometry was felt in the  $^{14}\text{C}$  world. But even in retirement, he took a lively interest in this new way of doing things, although, to be honest, accelerators were always a bit of a mystery to him. No matter. All of us who knew him and worked with him will recall the universal greeting he had when he passed by in the corridors, whether to junior technician or senior scientist: "How's it going?"

### Rodger Sparks

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# Radiocarbon

1996

## <sup>14</sup>C AND SOIL DYNAMICS

### INTRODUCTION: CHALLENGES IN THE SOIL

The decision to proceed with compilation of a special Soils Issue was taken in June 1992 during a planning workshop hosted in Tucson by the editors of *RADIOCARBON*. The undertaking was intended as a prelude to a wider and more far-reaching initiative to establish the International Radiocarbon Soils Database (IRSDB). In concept, this electronic archive, to be serviced by a worldwide cooperative of soil scientists and managed *via* the established expertise at the University of Arizona Department of Geosciences, would provide the international scientific community with a comprehensive, freely accessible and routinely updated source of unambiguously defined carbon isotope data. Justification for the database seemed well founded in the growing public concern and political awareness over the possible environmental impacts from steadily increasing global temperatures due to the so-called "greenhouse effect". Geoscientists had recognized a moral and/or professional responsibility to respond; in the first instance with the input necessary to understand and quantify the natural system, as the basis for both predictive climate modeling and immediate management of the natural carbon cycle. For this exercise it was already apparent, however, that a major constraint in objective modeling lay in defining the extent to which the pool of carbon stored in soils can influence concentrations of the carbonaceous gases (CO<sub>2</sub> and CH<sub>4</sub>) in the Earth's atmosphere. While it could be accepted that soils would function as either a carbon source or a carbon sink in relation to the atmosphere and in response to prevalent climate, insufficient information was available concerning the precise inventory, global distribution or mean residence times for the several forms of carbon retained in soils. Previous enquiries among the several <sup>14</sup>C laboratories known to have had a direct involvement in the measurement of <sup>14</sup>C in soils were encouraging in this respect. A wealth of essential data does exist but a significant proportion is latent due to either its diverse publication throughout the scientific literature and including foreign (non-English) language text or its retention unpublished in laboratory archives. It was particularly encouraging that positive responses as to the potential availability of essential data were received from a number of scientific colleagues in Eastern Europe, the states of the former Soviet Union and China. Given the established role of the journal *RADIOCARBON* as the focus for international consensus and collaboration in applied <sup>14</sup>C measurement, it was considered opportune that work towards the benefits of a soils database should proceed using existing facilities for the collection and collation of appropriate carbon isotope values and related data.

Advantage was taken of the collective expertise of the Workshop participants in drawing up the detailed blueprint for a database that would attract input and facilitate access across the broad community of "environmental sciences". The agenda for that exercise and its agreed outcome are reported by Peter Becker-Heidmann in his paper entitled "Requirements for an International Radiocarbon Soils Database" (see pages 177 to 180 of this issue).

In the event the IRSDB proposal failed to attract the necessary funding and, for various reasons, this volume has suffered an unduly long gestation. However, these factors do not reflect any diminution of the original objectives. Scientific publications over the past decade bear witness to a steady increase in the volume and diversity of academic study aimed at our better understanding of the glo-

bal distribution, composition, geochemistry and residence times of the carbon retained in organic and mineral rich soils. Likewise, the concern over man-made changes to the global environment has not abated; in its most recent report (IPCC 1996) the Intergovernmental Panel on Climate Change dispels any previous uncertainty by stating that, "... the balance of evidence suggests that there is a discernible human influence on the global climate." However, even faced with the various threats to economic stability and quality of life, there remains continuing debate over such fundamental issues as the possibility of a substantial "missing sink" of carbon associated with the plant/soil regime of the northern hemisphere temperate zone. The scientific challenge to researchers working within or in collaboration with  $^{14}\text{C}$  laboratories is clear. It remains twofold, viz. 1) to ensure that existing information is made readily available for multidisciplinary access, and 2) to exploit and publicize advances in the technology of natural  $^{14}\text{C}$  measurement that are particularly relevant to the study of soil carbon.

The specific role of the naturally occurring isotopes of carbon as quantitative tracers was highlighted further in the proceedings of a Soils Workshop session convened during the 15th International Radiocarbon Conference (Glasgow 1994). From a lively discussion forum (summarized in Becker-Heidmann and Harkness (1995)) it was evident that such diverse interests as paleoenvironment reconstruction and modern land management priorities, when allied with new technological opportunities, in particular  $^{14}\text{C}$ -AMS, were stimulating exciting new approaches in soil-based research. Nevertheless, and by way of general warning, it was agreed that even with the experience of over 30 years, several of the fundamental questions posed in those early days remained unanswered or at best only partly resolved. The immediate call was for the promotion of *RADIOCARBON* as the most suitable and internationally recognized platform for the reporting and discussion of advances and project priorities in the applied carbon isotope geochemistry of soils.

In the absence of the intended IRSDB, the old specter of ready access to data seemed set to persist and compound. However, a recent revision of policy by the editors of *RADIOCARBON* offers a welcome and best possible alternative, viz. the launch in electronic format of the new on-line journal *Radiocarbon Date Lists*, backed by the decision to reserve the established *RADIOCARBON* format for the publication of research articles. As highlighted by the Editor (Long 1995) the new arrangement is now available for the positive benefit of all  $^{14}\text{C}$  laboratories and their users. However, the onus for success, perhaps the greatest challenge, is on practicing  $^{14}\text{C}$  laboratories to publicize and take full advantage of these facilities in making their work generally known and readily available to the international science community. We hope that the research papers contained in this dedicated issue will be both a stimulus for and a herald of improved dialogue among geochemists and other research disciplines who find an intellectual challenge in the soil. At least one major listing of  $^{14}\text{C}$  measurements and related pedagogical data ("NERC Soil Organic Matter Studies; 1977 till 1997") is being prepared currently for presentation in the *Radiocarbon Date Lists* format.

The community will meet soon (June 1997) in Groningen to convene the 16th International Radiocarbon Conference. The provisional program has a slot reserved for the consideration of  $^{14}\text{C}$  in soils—let us hope that progress in the reporting of results, old and new, can feature as positive feedback in the general discussion.

*Doug Harkness*

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- Long, A. 1995 From the Editor. *Radiocarbon* 37(3): iii.

## REQUIREMENTS FOR AN INTERNATIONAL RADIOCARBON SOILS DATABASE

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### INTRODUCTION

Up to now, Global Carbon Cycle Models (GCCM) have only represented the soil and biosphere in a very simplified way. For example, the High Resolution Biosphere Model (HRBM) (Esser 1987; Esser and Lautenschlager 1994) distinguishes five subreservoirs and determines the fluxes between them for selected area of a global grid. The models have not yet been sufficiently tested against global observations. Such testing is difficult because the observed variables are in turn dependent on the behavior of other geological carbon pools, *e.g.*, the atmosphere and ocean.

Data on the following variables are readily available in the scientific literature:

- Atmospheric CO<sub>2</sub> concentration since 1860 (or for the past 160,000 yr from the Vostok ice core)
- Atmospheric δ<sup>13</sup>C since 1860
- Atmospheric <sup>14</sup>C since 1860 (or for the past 8000 yr from the dendrochronological record)

The data and/or information that is necessary as input for effective GCCM testing include:

- How much carbon is/can be stored in soil
- How much carbon is/may be released as CO<sub>2</sub>, CH<sub>4</sub> and CO
- Which factors influence the net storage and/or release of carbon, especially climatic factors (feedback effects)
- What are the rates of net storage or release of carbon as a function of soil type and in response to the prevailing environmental conditions.

These data are available neither in direct form nor for every point or area on the globe. They may be derived from secondary data by calculation, estimation or a submodel. A minimum set of these secondary data is, for example, the one used in the HRBM:

- Soil type
- Composition of vegetation
- Type of land use
- Mean temperature
- Annual precipitation

There are several sources of these secondary data:

- FAO soil classification and soil map (Driessen and Dudal 1989)
- USDA Soil Taxonomy (Soil Survey Staff 1994) and related maps
- Agroclimate maps
- Estimations of Bohn (1979), Buringh (1984) and others on global soil carbon amounts and distributions
- Estimations on a soil system depending on carbon depth profiles (Cherkinsky and Chichagova 1990)
- Maps of net primary plant production (Lieth 1978, *etc.*)
- Soil carbon models (Parton *et al.* 1995, *etc.*)
- Soil radiocarbon dates

Because  $^{14}\text{C}$  dates of soil organic matter (SOM) represent a fingerprint of the history of its dynamics, they can be useful in reconstructing its major parameters, especially in determining whether a soil sample has been a source or a sink of carbon. To serve the purpose of GCCMs, the data should cover the major soil classes and regions of the Earth and be available in digital and unambiguous structured form.

#### **THE STRUCTURE OF THE IRSDB**

Existing  $^{14}\text{C}$  dates of soils have been produced for different reasons, mostly to answer pedogenetic questions and without regard to the evaluation of carbon cycling. Therefore, additional information on the environmental context of these data is necessary to ascertain whether and how they may be useful for the purpose of GCCMs.

In Tucson on June 5–7, 1992 a NASA-sponsored workshop was held to discuss and plan the structure and installation of an International Radiocarbon Soils Database (IRSDB). As a result, the following hierarchically ordered list of database fields was proposed by the participants. The field names were designed to be compatible with the proposed International Radiocarbon Data Base (IRDB) (Kra 1986; Walker and Kra 1988). Data that are essential for assessing carbon turnover and, therefore, global change calculations, are marked by an asterisk (\*); those that might overload the database are marked with a question mark (?). All other fields were recognized as generally useful and should be added to the database if available.

#### **PROPOSED LIST OF THE IRSDB DATABASE ENTRY FIELDS:**

##### **General**

- \* Labcode and no.
- \* Single sample or sample set (profile or catena, set no.)
- \* Submitter (name and address)
- \* Collector (name and address)
- \* Date of sampling
- \* Date of measurement
- \* Sampling location (longitude, latitude)
- \* Pretreatment for dating
- \* Type of soil fraction(s) or other material used for dating

##### **Results**

- \*  $^{14}\text{C}$  result ( $\text{pM} \pm \text{error}$  (as defined by Stuiver and Polach 1977); raw data)
- \*  $\delta^{13}\text{C}$
- \* Possible contamination
- \* Submitter's comment
- \* Lab's comment

##### **Context**

- \* Literature References
- Original field of study
- ? Quality control figure

##### **Environmental Data**

- Country
- Site description

Parent material, time of deposition  
 Topography (landform?)  
 Ecosystem type and climatic conditions (mean temperature and annual rainfall)  
 Vegetation (actual, history and potential)  
 Land use

#### Profile-Related Data

- \* Soil order and type (both FAO and local classification)
- Profile description (data related to carbon turnover and migration)
  - Humus form
  - Rooting pattern
  - Stratigraphy
  - Physical barriers to water penetration
- Reference to Soil Conservation Service file

#### Sample-Related Data

- \* Sampling depth interval, depth and horizon
  - Method of sampling
- \* Bulk density
- ? Soil color (wet, moist, dry)
- \* Particle size distribution (<2 mm, >2 mm) (at least texture)
  - Clay type
- \* pH (and method)
  - Eh
- ? CEC
- \* C content (organic and inorganic)
  - C<sub>org</sub>/N ratio
- ? Lignin content
- ? Phosphorus (and method)
- ? Microbial biomass
- ? Data on other isotopes (heavy, stable)

In addition, a standard exchange format for the <sup>14</sup>C and additional data of the IRSDB, which serve as input to GCCMs, should be defined (*cf.* Wilcock *et al.* 1986; Walker *et al.* 1990).

#### ACKNOWLEDGMENTS

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## APPENDIX

### Ecosystem Type and Climatic Conditions

Boreal forest	Tropical moist forest
Tundra and alpine meadow	Tropical seasonal forest
Temperate evergreen forest	Tropical woodland
Temperate deciduous forest	Tropical grassland
Temperate woodland	Swamps and marshes
Temperate grassland	Desert

### Soil Order and Type

#### A. Major Soil Groupings of the FAO System (Driessen and Dudal 1989):

Histosols	Fluvisols	Plinthosols	Solonchaks	Luvisols
	Gleysols	Ferralsols	Solonetz	Podzoluvisols
Anthrosols	Leptosols	Nitisols	Gypsisols	Planosols
	Regosols	Acrisols	Calcisols	Podzols
Andosols		Alisols		
Arenosols	Cambisols	Lixisols	Kastanozems	
Vertisols			Chernozems	
			Phaeozems	
			Greyzems	

#### B. Orders of Soil Taxonomy (Soil Survey Staff 1994):

Alfisols	Entisols	Mollisols	Ultisols
Andisols	Histosols	Oxisols	Vertisols
Aridisols	Inceptisols	Spodosols	

## USING BULK SOIL RADIOCARBON MEASUREMENTS TO ESTIMATE SOIL ORGANIC MATTER TURNOVER TIMES: IMPLICATIONS FOR ATMOSPHERIC CO<sub>2</sub> LEVELS

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**ABSTRACT.** Although soil contains about three times the amount of carbon present in the preindustrial atmosphere, determining how perturbations (e.g., changing land use, CO<sub>2</sub> fertilization, changing climate and anthropogenic nitrogen deposition) alter soil carbon storage and influence atmospheric CO<sub>2</sub> levels has proved elusive. Not knowing the soil carbon turnover times causes part of this uncertainty. I outline a strategy for using radiocarbon measurements to estimate soil organic matter turnover times and inventories in native soil. The resulting estimates of carbon exchange produce reasonable agreement with measurements of CO<sub>2</sub> fluxes from soil. Furthermore, derivatives of the model are used to explore soil carbon dynamics of cultivated and recovering soil. Because the models can reproduce observed soil <sup>14</sup>C measurements in native, cultivated, and recovering ecosystems (i.e., the underlying assumptions appear reasonable), the native model was modified to estimate the potential rate of additional carbon storage because of CO<sub>2</sub> fertilization. This process may account for 45–65% of the “missing CO<sub>2</sub> sink.”

### INTRODUCTION

Many researchers have estimated the global inventory of soil carbon using a variety of techniques: Schlesinger (1977) used vegetation types to estimate an inventory of 1456 Gt C; Post *et al.* (1982) estimated soil humus to hold 1395 Gt C using climatic life zones; and Eswaran, den Berg and Reich (1993) used soil orders to estimate an inventory of 1576 Gt C. Although there are differences among the techniques, the results generally agree. How this large pool of carbon influences atmospheric CO<sub>2</sub> is uncertain, because the discrete turnover times of the multitude of soil compounds are not well known. These turnover times may range from days to millennia. The purpose of this paper is to introduce a strategy for estimating the turnover time of soil organic matter (SOM) using bulk soil radiocarbon measurements.

The technique builds on the approaches of others: the Century and Rothamsted models, mass balance approaches and soil humus fractionation approaches. The Century and Rothamsted models use measurements of soil carbon decomposition as the foundation for sophisticated ecosystem models. The model structures are similar, having SOM consisting of fast, active and passive fractions. The Century model (Parton *et al.* 1987, 1989, 1993; Schimel *et al.* 1994) has an active carbon turnover time ranging from 20 to 50 yr, and, it assigns an 800- to 1200-yr turnover time for passive carbon. The Rothamsted model (Jenkinson 1990) uses a 20-yr turnover time for active carbon and a near infinite turnover time for passive carbon.

O'Brien and Stout (1978) use a sophisticated model to interpret their New Zealand soil <sup>14</sup>C measurements. Their model is constrained by the depth distribution of total carbon and <sup>14</sup>C and includes carbon input, decomposition rates and soil diffusiveness. They assign a 50-yr turnover time for active carbon and a near-infinite time for passive carbon.

Researchers have tried to separate active and passive components using physical and chemical fractionation techniques (Paul *et al.* 1964; Campbell *et al.* 1967; Martel and Paul, 1974b; Goh *et al.* 1976; Goh, Stout and Rafter 1977; Goh, Stout and Brown 1984; Scharpenseel, Ronzani and Pietig 1968; Scharpenseel, Tamers and Pietig 1968; Trumbore, Vogel and Southon 1989; Trumbore, Bonani and Wölfli 1990). Trumbore (1993) summarizes the results of various fractionation techniques. One way to test the effectiveness of these fractionation schemes is to see if the amount of



bomb  $^{14}\text{C}$  increases in the soil as predicted by estimates of the residence time for SOM. To date, the available fractionation schemes cannot do this (Trumbore 1993).

#### ESTIMATING SOIL CARBON TURNOVER TIMES USING BULK RADIOCARBON MEASUREMENTS

In my research, I use a time-step one-box model and bulk soil  $^{14}\text{C}$  measurements to estimate turnover times and inventories of active and passive carbon. The model has atmospheric  $^{14}\text{C}$  values and  $\text{CO}_2$  concentrations for every year from AD 1800 until the present. The user selects the carbon inventory and the turnover time. The turnover time equals the carbon inventory divided by the exchange flux. The exchange flux equals the amount of carbon that is added to the box (from photosynthesis) or lost from the box (respiration). Losses through erosion and dissolution are thought to be small (Schlesinger 1986) and are not considered. The model can be run in either a steady-state mode (where the flux in equals the flux out) or in a non-steady-state mode (in which carbon is either accumulating or decreasing). I use this model and soil  $^{14}\text{C}$  data to show that soil carbon has more than one component and to estimate the turnover time of the passive fraction, the proportions of active and passive carbon in surface soil, and the active soil carbon residence time.

Many researchers have concluded that soil consists of a complex mix of organic molecules whose turnover times range from a few years to thousands of years. This pool cannot be characterized by a single turnover time (O'Brien 1984; Balesdent, Wagner and Mariotti 1988; Parton *et al.* 1987, 1989, 1993; Jenkinson and Raynor 1977). Figure 1 shows how a single residence time of 650 yr fails to characterize soil humus. Six prebomb values of surface soil had an average  $^{14}\text{C}$  content of 92% modern (pMC) (Table 1). This 92 pMC value translates into a 650-yr turnover time, which shows very little increase in bomb  $^{14}\text{C}$  with time. The soil  $^{14}\text{C}$  values increase in the 1960s and then level off

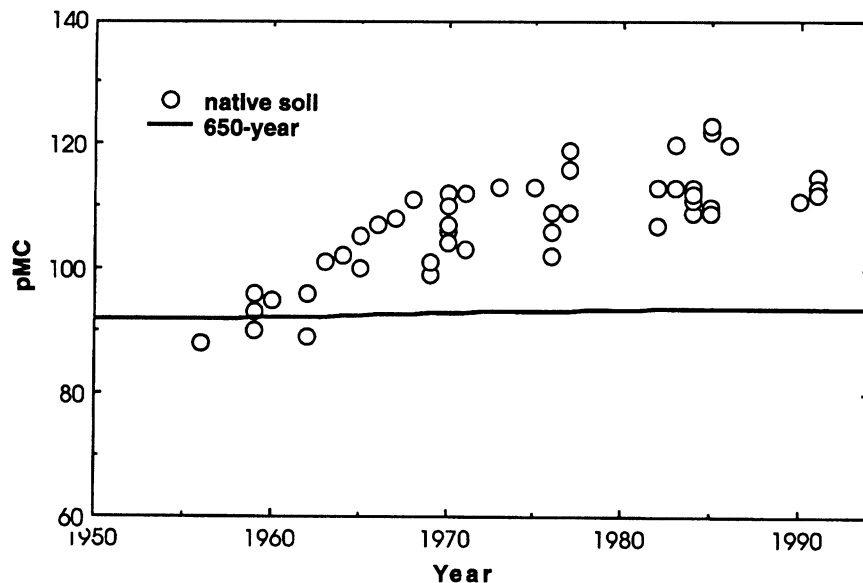


Fig. 1. Soil  $^{14}\text{C}$  values vs. time. Measured soil  $^{14}\text{C}$  values for non-cultivated soils are plotted against time from 1950 to 1991. The values tend to increase during the 1960s and then level off. Model results for a theoretical carbon pool having an 650-yr residence time are shown by the line. If soil carbon consisted of components having this single residence time, one would expect the observations to fall around this line. Data for these figures from Tables 1 and 2 and Harrison (1994).

(Fig. 1). This increase suggests that soil organic material contains an active component with a turnover time significantly <650 yr. This active component must be diluted with a passive component having a turnover time of >650 yr.

TABLE 1. Prebomb Soil <sup>14</sup>C Values for Uncultivated Soil

pMC	Depth (cm)	Year	Reference	Soil type	Location
96	1–8	1959	O'Brien (1986)	Mollisol	New Zealand
96	0–23	1959	Trumbore (1993)	Temp. forest	USA
82	0–12	1927	Trumbore <i>et al.</i> (1990)	Spodosol	FSU
96	A-hor.	1962	Campbell <i>et al.</i> 1967	Chernzemec	Canada
90	A-hor.	1962	Campbell <i>et al.</i> 1967	Mollisol	Canada
94	0–2	1959	Vogel 1970	Forest	Germany

The passive soil turnover time can be estimated from soil <sup>14</sup>C measurements made at depths where little or no active soil carbon is present. <sup>14</sup>C concentrations decrease with depth, reflecting a decrease in the proportion of active to passive carbon. Both carbon content and <sup>14</sup>C soil values tend to decrease with increasing depth (Harrison, Broecker and Bonani 1993a). At some depth, the soil <sup>14</sup>C measurements approach an inflection value below which they tend to decrease very slowly. These values and depths vary for different locations (see Table 2). The average value for the sites listed in Table 2 was 55 pMC, which corresponds to a 4700-yr turnover time for passive soil carbon. However, some tropical soils have active soil carbon several meters below the surface (Nepstad *et al.* 1994; Fisher *et al.* 1994)

TABLE 2. Deep Soil <sup>14</sup>C Values for Cultivated and Uncultivated Soil

pMC	Depth (cm)	Reference	Soil type	Location
49	65–105	Becker-Heidmann <i>et al.</i> (1988)	Mollisol	China
43	74–94	O'Brien and Stout (1978)	Mollisol	New Zealand
60	60–140	Scharpenseel and Becker-Heidmann (1989)	Vertisol	Israel
60	60–140	Becker-Heidmann (1989)	Udic	India
62	85–110	Tsutsuki <i>et al.</i> (1988)	Mollisol	Germany

For the prebomb condition, one can estimate the proportions of active and passive components in surface soil using the 55% modern passive soil <sup>14</sup>C value and the 92% modern values measured for the bulk soil (Table 1). If the active component turns over quickly (<100 yr), enough so that we can assume that its <sup>14</sup>C value is almost 100 pMC (<sup>14</sup>C has a half-life of 5700 yr), a mixture of 17% passive and 83% active leads to the observed average <sup>14</sup>C value of 92 pMC (Fig. 2).

The postbomb increase in soil <sup>14</sup>C concentrations can be used to estimate the active soil carbon turnover time (Harrison 1993a). A 25-yr turnover time produces the best fit to the available data. Most of the points are for temperate ecosystems, so warmer tropical ecosystems may have faster turnover times, whereas cooler boreal turnover times may be slower. The proportions of active and passive soil may differ for tropical and boreal climates.

This approach can be validated by looking at a specific site where deep, prebomb, and a time series of surface soil data can be used to compare model predictions with soil <sup>14</sup>C measurements. O'Brien

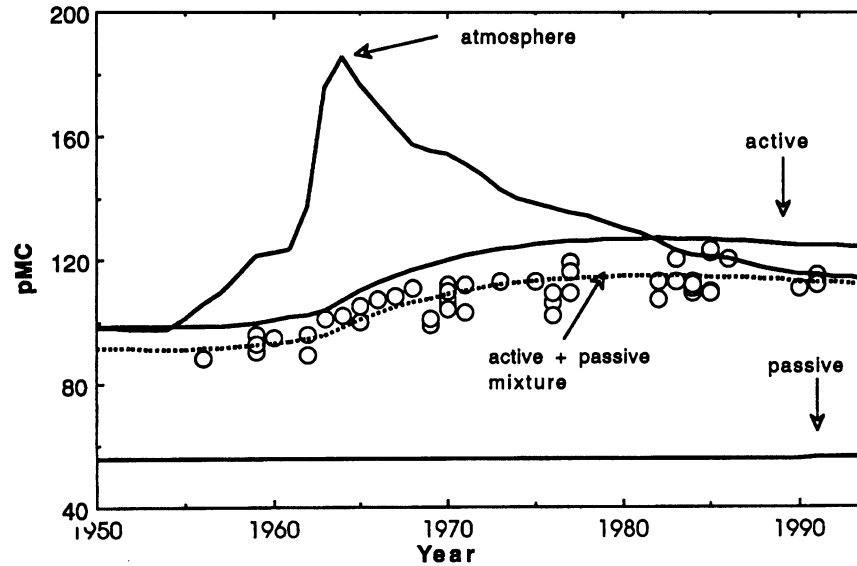


Fig. 2. Soil  $^{14}\text{C}$  model predictions and soil  $^{14}\text{C}$  observations vs. time. A mixture of 83% active and 17% passive soil carbon produces the best visual fit to the native soil  $^{14}\text{C}$  measurements ( $\circ$ ). The concentration of atmospheric  $^{14}\text{C}$  almost doubled because of nuclear bomb testing.

and Stout (1978) reported measurements for a New Zealand grassland soil that included a deep soil value and a time series of surface soil values that extended from prebomb times into the mid-1970s. The model that best fits the data has a 12% passive component plus a 88% active portion that turns over every 25 yr. Figure 3 shows the agreement between the model and the data. Although this model reproduces the prebomb soil  $^{14}\text{C}$  values and the postbomb increase in  $^{14}\text{C}$  values in native soil, the model's robustness can be evaluated by seeing if derivatives can explain  $^{14}\text{C}$  measurements in cultivated and recovering soil.

#### SOIL CARBON DYNAMICS IN CULTIVATED ECOSYSTEMS

Another way to test the model's assumption is to see if it can be modified to reflect carbon dynamics in cultivated ecosystems. Soil loses *ca.* 25% of its carbon when cultivated (Schlesinger 1986; Post and Mann 1990; Davidson and Ackerman 1993). This loss stems from reduced inputs of organic matter and increased rates of organic matter mineralization. Cultivated soil generally has lower  $^{14}\text{C}$  values than native soil that has been sampled at the same time (Fig. 4; Martel and Paul 1974a). Hsieh (1992, 1993) has developed a two-component model that reproduces temporal changes in  $^{14}\text{C}$  values in cultivated soil. Using a similar approach, Harrison, Broecker and Bonani (1993b) assumed that carbon lost from soil due to cultivation would be lost from the active carbon pool. However, the oxidation and loss of a fraction of the active-soil carbon could only explain about half the observed  $^{14}\text{C}$  depletion. Mixing subsurface soil with the shallow surface soil through cultivation (*i.e.*, the plow mixes up the soil) can account for the remaining depletion in  $^{14}\text{C}$  values. The model, which included mixing and oxidation, produced good agreement with the available data for changes in soil  $^{14}\text{C}$  upon cultivation (Fig. 4).

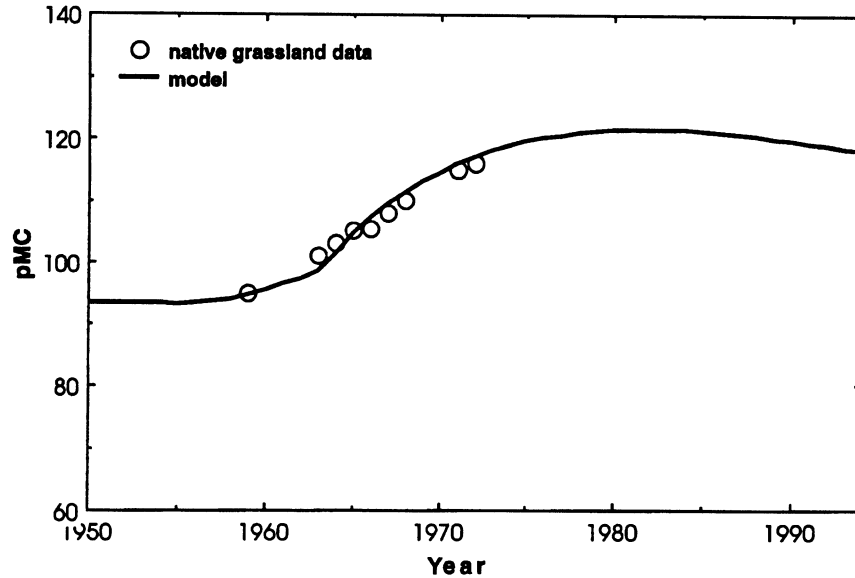


Fig. 3. New Zealand test case. O'Brien and Stout (1978) published  $^{14}\text{C}$  data for a New Zealand grassland site comprising a time series of surface soil, including one early-bomb, and one deep soil  $^{14}\text{C}$  value. I used this information to attempt to validate the model for a specific site. This model that best fit the data consisted of 88% active and 12% passive carbon. The active carbon had a 25-yr turnover time.

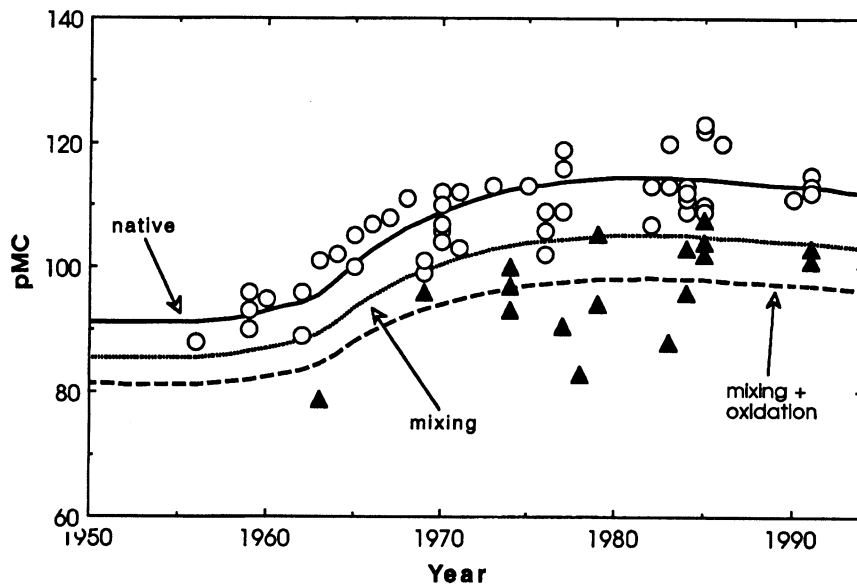


Fig. 4. Native vs. cultivated soil  $^{14}\text{C}$  values. Cultivated soil ( $\blacktriangle$ ) has lower  $^{14}\text{C}$  values than native soil ( $\circ$ ). This difference is caused by mixing and oxidation. The farmer's plow mixes deeper  $^{14}\text{C}$  depleted soil with  $^{14}\text{C}$  rich surface soil, diluting the amount of active soil carbon in the surface. Increased SOM oxidation further reduced the inventory of active soil carbon.

## SOIL CARBON TURNOVER IN RECOVERING ECOSYSTEMS

To test the model further, the baseline model was modified to explore the carbon dynamics of a recovering soil that was increasing its carbon stores. One example includes a recovering temperate forest located in the Calhoun National Forest, described by Richter *et al.* (1994; 1995), Harrison (1994), and Harrison *et al.* (1995). This site contains a Loblolly pine (*Pinus taeda*) forest that was planted in 1959 on land that had been cultivated for the previous 150 yr. The soil has been accumulating carbon since the pines were planted. From 1962 to 1968, the surface carbon concentration increased from 5.9 to 8.0 Mg ha<sup>-1</sup>. The native soil carbon model was modified to take into account this carbon accumulation by increasing the flux of carbon into the active-soil carbon pool. The turnover time that best reproduced the observed <sup>14</sup>C measurements was 12 yr, which is about twice as fast as carbon turnover in native ecosystems. Figure 5 shows the agreement between the model and the data for this accumulating ecosystem.

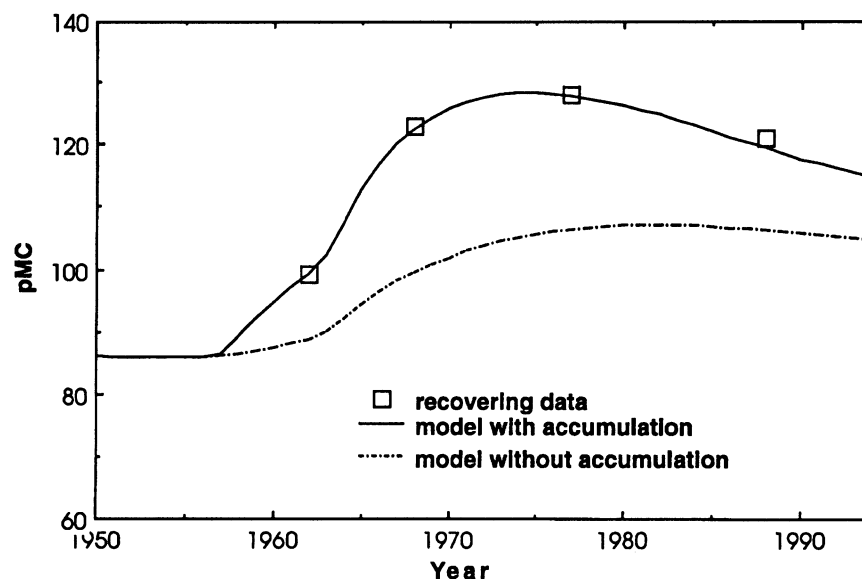


Fig. 5. <sup>14</sup>C measurements and model results for South Carolina. The model results that best fit the surface soil <sup>14</sup>C measurements were those from an accumulation model that took into account the increase in carbon inventory. The carbon increased from 5.9 Mg ha<sup>-1</sup> to 8.0 Mg ha<sup>-1</sup>. The active reservoir turnover time is about twice as fast as the global average for native soils.

## DETERMINING THE GLOBAL INVENTORY OF ACTIVE SOIL ORGANIC MATTER

Table 3 lists the current best estimates of the global inventory of non-wetland SOM. These values can be translated to give a global inventory of active soil carbon. The active-to-passive proportions found in surface soil cannot be applied to all carbon present in non-wetland soil because the proportion of active to passive carbon decreases with increasing depth. The integrated inventories suggest that the global pool is *ca.* 50% passive and 50% active (Harrison 1993a). Although extrapolating these distributions involves uncertainty (*i.e.*, the proportions are likely to differ for other climates and types of vegetation), the global inventory of active carbon in soils ranges between 500 and 625 Gt C.

TABLE 3. Soil Carbon Sequestration Due to  $\text{CO}_2$  Fertilization\*

Category	Reference		
	Schlesinger (1977, 1991)	Post <i>et al.</i> (1982)	Eswaran <i>et al.</i> (1993)
Total soil carbon (Gt C)	1500	1400	1600
Non-wetland soil carbon (Gt C)	1000	1200	1250
Active soil carbon (Gt C)	500	600	625
Exchange flux ( $\text{Gt C yr}^{-1}$ )	20	24	25
Potential C sequestration due to $\text{CO}_2$ fertilization ( $\text{Gt C yr}^{-1}$ )	0.5	0.6	0.7
% "missing $\text{CO}_2$ sink"	45	55	65

\*This table uses estimates of the inventory of active soil carbon and a simple  $\text{CO}_2$  fertilization model to estimate the potential carbon sequestration in soil due to  $\text{CO}_2$  fertilization. Only non-wetland soil was included in the calculation. The inventory of active soil was estimated assuming that a soil profile contains *ca.* 50% active carbon; a  $\text{CO}_2$  fertilization factor of 0.35 was used in the  $\text{CO}_2$  fertilization model to calculate the amount of carbon sequestered during an average year in the 1980s (Harrison 1993a). Dixon *et al.*'s (1994) missing sink estimate for the 1980s of  $1.1 \text{ Gt C yr}^{-1}$  was used.

A way to test the model is to determine if fluxes measured from the soil agree with those values predicted by the model. A 500 Gt C pool turning over every 25 yr emits 20 Gt C from the soil annually, *i.e.*, a flux of  $150 \text{ g C m}^{-2} \text{ yr}^{-1}$ . The 600 and 625 Gt C values result in  $24 \text{ Gt C yr}^{-1}$  ( $180 \text{ g C m}^{-2} \text{ yr}^{-1}$ ) and  $25 \text{ Gt C yr}^{-1}$  ( $190 \text{ g C m}^{-2} \text{ yr}^{-1}$ ), respectively. These estimates are significantly lower than the observed flux from a temperate forest soil, which ranged from 400 to  $500 \text{ g C m}^{-2} \text{ yr}^{-1}$  (Raich and Schlesinger 1992). It must be realized that the measured values record sources of  $\text{CO}_2$  besides microbial respiration of SOM, such as root respiration and the oxidation of litter and fine roots. While it is impossible to resolve the contribution from these additional  $\text{CO}_2$  sources, it is unlikely that they account for >50% of the total flux. Furthermore, soils such as desert soil that have low organic carbon contents make it difficult to compare global and regional values. The ecological complexities make it virtually impossible to get better agreement between the  $\text{CO}_2$  fluxes predicted by the model and those measured for a temperate forest ecosystem. However, even if one considers the measured flux to represent an upper limit, the predicted values fall well below it.

#### CARBON DIOXIDE FERTILIZATION

Having estimated the turnover time and inventory of fast-cycling carbon, it is possible to assess the amount of carbon that might be stored in soil as a consequence of  $\text{CO}_2$  fertilization.  $\text{CO}_2$  fertilization occurs when plants increase their growth when exposed to elevated  $\text{CO}_2$  levels (Strain and Cure 1985; Bazzaz and Fajer 1992). A convenient way of expressing the effect is *via* a  $\text{CO}_2$  fertilization factor (*i.e.*, the percentage increase in growth that results from a doubling of  $\text{CO}_2$  concentration).

Although many indoor  $\text{CO}_2$  fertilization experiments have shown increased growth at elevated  $\text{CO}_2$  levels (Strain and Cure 1985), the extrapolation of these results to natural vegetation is highly controversial (Bazzaz and Fajer 1992). Further, the application of these  $\text{CO}_2$  fertilization factors to the carbon input to soil involves speculation. Zak *et al.* (1993) have shown that the soil carbon under plants grown in doubled  $\text{CO}_2$  concentrations in open-top chambers was greater than their nonelevated paired counterparts, although their results were not statistically significant. Norby *et al.* (1992) found evidence of increased fine root density for trees grown in elevated  $\text{CO}_2$  concentrations,

lending credibility to the belief that if plant growth is stimulated, the inventory of soil carbon will be as well.

For this study, a CO<sub>2</sub> fertilization model has been developed to estimate the additional amount of carbon stored in soil because of CO<sub>2</sub> fertilization. The flux of carbon into soil organic matter can be increased by adding the term

$$\beta \delta p\text{CO}_2 \text{ EF}, \quad (1)$$

where  $\beta$  = CO<sub>2</sub> fertilization factor (0.35 after Harrison *et al.* 1993a)  
 $\delta p\text{CO}_2$  = fractional change in CO<sub>2</sub>  
 EF = exchange flux.

As the flux of carbon into the box increases in the box, the decay flux (*i.e.*, the decay constant times the amount of carbon in the box) also increases. If the level of atmospheric CO<sub>2</sub> stops increasing, the soil will attain a higher steady-state carbon content with a turnover time of 25 yr.

Table 3 lists the model results. The amount of carbon sequestered in soil because of CO<sub>2</sub> fertilization ranges from 0.5 to 0.7 Gt C yr<sup>-1</sup> for the 1980s. Dixon *et al.* (1994) estimate that the "missing sink" is 1.1 Gt C yr<sup>-1</sup> for this time period. Thus, CO<sub>2</sub> fertilization might well provide the mechanism to explain much of the "missing CO<sub>2</sub> sink" as carbon held in soil.

## CONCLUSION

I have presented a strategy for estimating the global inventory and turnover time for carbon stored in non-wetland soil. The available <sup>14</sup>C data suggest that active soil carbon has a 25-yr turnover time and a 500–625 Gt C inventory. Therefore, active soil carbon is well placed to respond significantly to perturbations such as CO<sub>2</sub> fertilization, changing climate and anthropogenic nitrogen deposition. The effects of CO<sub>2</sub> fertilization on the flux of detrital organic matter input to soil can potentially store 0.5–0.7 Gt C yr<sup>-1</sup>, thus explaining a major portion of the "missing CO<sub>2</sub> sink."

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## THE USE OF CARBON ISOTOPES ( $^{13}\text{C}$ , $^{14}\text{C}$ ) IN SOIL TO EVALUATE VEGETATION CHANGES DURING THE HOLOCENE IN CENTRAL BRAZIL

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**ABSTRACT.** This paper presents carbon isotope data measured in three soil profiles from the Salitre area, Central Brazil. The study forms part of a research project on tropical and subtropical soils in Brazil, in which the main objective is to use carbon isotopes to provide information about vegetation changes that have occurred in relation to climate changes during the Holocene.  $^{14}\text{C}$  data from charcoal samples and soil organic matter (SOM) indicate that the organic matter in the soils studied is of Holocene age at least. Furthermore, the presence of a significant amount of charcoal in the soils suggests that forest fire was a significant occurrence during the Holocene and probably had an important role in determining the dynamics of forest vegetation in the study area. Correspondingly,  $^{13}\text{C}$  data indicate that  $\text{C}_3$  plants provided the dominant vegetation of the study area, even during the dry periods when savanna vegetation is supposed to have replaced the forest communities. This study contributes to our better understanding of the relation between climatic changes and vegetation in the subtropical region of Brazil.

### INTRODUCTION

Reconstruction of past vegetation changes and their relation to climate in tropical and subtropical forest is essential for understanding the response of these ecosystems to future climatic change. Different approaches involving geomorphological (Ab'Saber 1977, 1982; Servant *et al.* 1981; Bigarella and de Andrade Lima 1982), biological, botanical (Haffer 1969; Prance 1973; Gentry 1982) and palynological studies (Absy *et al.* 1991; Ledru 1993) have been used to infer past climatic changes in the Amazonia and Central region of Brazil.

The naturally occurring isotopes  $^{13}\text{C}$  and  $^{18}\text{O}$  are used widely as tracers of paleoenvironmental processes, mainly from lacustrine sediments, such as carbonate and organic sediments (Cerling 1984; Hollander, McKenzie and Haven 1992; Aravena *et al.* 1992) and recently this approach has been extended to soils (Schwartz *et al.* 1986; Becker-Heidmann and Scharpenseel 1989, 1992).

The stable carbon isotope composition ( $^{13}\text{C}/^{12}\text{C}$ , or  $\delta^{13}\text{C}$ ) of soil organic matter (SOM) records information regarding the occurrence of  $\text{C}_3$  and/or  $\text{C}_4$  plant species in past plant communities, and their relative contribution to net primary productivity by the plant community (Troughton, Stout and Rafter 1974; Stout, Rafter and Troughton 1975). Such information has been used to document vegetation change (Hendy, Rafter and MacIntosh 1972; Dzurec *et al.* 1985), to infer climate change (Hendy, Rafter and MacIntosh 1972; Krishnamurthy, De Niro and Pant 1982) and to estimate rates of SOM turnover (Cerri *et al.* 1985).

These applications are based on isotope effects occurring during photosynthesis that imprint plants with a carbon isotopic composition that is characteristic of the photosynthetic pathway.

$\delta^{13}\text{C}_{\text{PDB}}$  values of  $\text{C}_3$  plant species range from *ca.*  $-32$  to  $-20\text{‰}$ , with a mean of  $-27\text{‰}$ , whereas  $\delta^{13}\text{C}_{\text{PDB}}$  values of  $\text{C}_4$  species range from  $-17$  to  $-9\text{‰}$ , with a mean of  $-13\text{‰}$ . Thus,  $\text{C}_3$  and  $\text{C}_4$  plant species have distinct  $\delta^{13}\text{C}$  values and differ from each other by *ca.*  $14\text{‰}$  (Boutton 1991).

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In this paper we report  $^{14}\text{C}$  and  $\delta^{13}\text{C}$  data measured using SOM and charcoal from three soil profiles collected under a natural forested slope in the Salitre area, central Brazil. The study site is one of several at which applied carbon isotope research is being used to evaluate vegetation and climatic changes during the late Pleistocene and Holocene in tropical and subtropical soils in Brazil.

## METHODS

The Salitre site is located ca. 300 km W of Belo Horizonte, capital of Minas Gerais State, in the Salitre de Minas ( $19^{\circ}\text{S}$ ,  $46^{\circ}46'\text{W}$ ), region of Central Brazil (Fig. 1). The present climate is characterized by a 4-month dry period and mean winter temperatures above  $15^{\circ}\text{C}$  (Ledru 1993). The native vegetation is Cerrado (wooded savanna) and includes *Byrsonima coccolobifolia*, *Solanum lycocarpum*, *Kielmeyera coriacea*, *Dalbergia mischlobium*, *Stryphnodedron adstrindens* and *Erythroxylum* spp. (G. Ceccantini, personal communication 1994). Extensive forest areas have been cleared for agricultural use in this region. The site lies within a paleolagoon basin that has been the focus of a paleoclimatic investigation in Central Brazil (Ledru 1993). The soil type according to the Brazilian soil classification is a Latossolo Vermelho Amarelo, and in the American and FAO classification an Oxisol and Ferralsol.



Fig. 1. Map showing the location of the study area in Central Brazil

Soil samples were collected from three excavations *ca.* 250 m apart on a forested (mesophytic semideciduous) slope (Fig. 2). We sampled up to 10 kg of soil at 10 cm intervals to a maximum depth of 2 m. The samples were dried to constant weight at 60°C and root and plant remains were discarded by hand-picking. Any remaining plant debris was removed by flotation in HCl 0.01M and the residual soil was then dried to constant weight, sieved to <200  $\mu\text{m}$  to minimize the effects of sample heterogeneity and homogenized for  $^{13}\text{C}$  and  $^{14}\text{C}$  analyses. The humin fraction was extracted from 2.5-kg aliquots using standard methods (Dabin 1971; Goh 1978; Anderson and Paul 1984), *viz.*, 1) acid digestion in 0.5 M hydrochloric acid at 70°C to 80°C for 4 h followed by washing with distilled water to a pH of 3–4; 2) reaction of the acid-insoluble residue with at least 30 liters (10 liters per extraction) of 0.1M  $\text{Na}_4\text{P}_2\text{O}_7$  for *ca.* 36 h (12 h per extraction) followed by washing to a pH of 3–4; 3) hydrolysis of the solid residue with 4 liters of 3M HCl at 100°C for 12 h followed by washing to a pH of 3–4; 4) drying of the solid residue at 40°C for 48 h followed by sieving to recover the <200- $\mu\text{m}$ -size fraction.

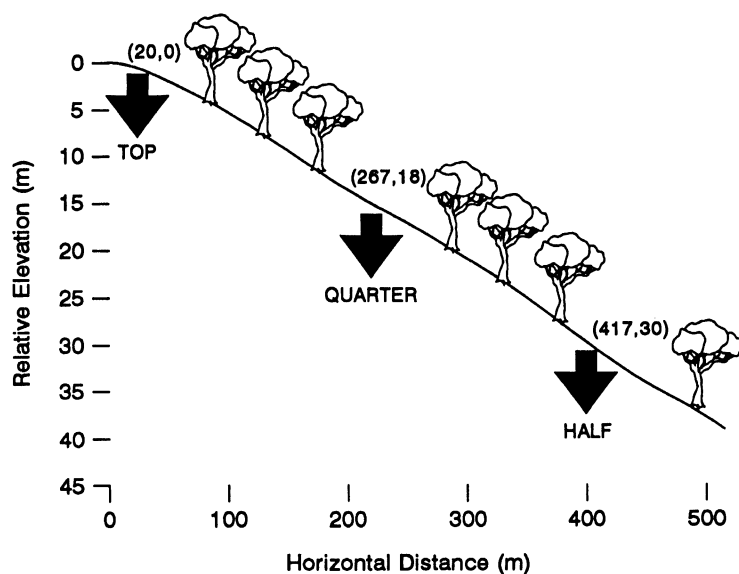


Fig. 2. Sampling points located under the forested slope

Charcoal samples were collected by hand-picking from 10-kg soil samples, oven dried at 90°C, weighed and treated using the conventional acid-alkali-acid treatment. Samples from between 2.20 m and 3.45 m depth were used only for  $\delta^{13}\text{C}_{\text{PDB}}$  measurements. These were collected from small amounts of soil by hand-picking and using a 5-mm sieve.

$^{14}\text{C}$  analyses on charcoal and humin samples were carried out at the Radiocarbon Laboratory, Centro de Energia Nuclear na Agricultura (CENA), by liquid scintillation counting of synthesized benzene (Pessenda and Camargo 1991). Benzene samples were counted for at least 48 h in a Packard 1550 low-level liquid scintillation counter.  $^{14}\text{C}$  ages are expressed in years BP and reflect normalization to  $\delta^{13}\text{C}_{\text{PDB}}$  of  $-25\text{‰}$  (Stuiver and Polach 1977) and an analytical precision of  $\pm 1.0$  pMC.  $^{13}\text{C}$  analyses were determined by isotope ratio mass spectrometry using  $\text{CO}_2$  generated by sample combustion at 900°C in an oxygen atmosphere. These analyses were undertaken at the Stable Isotopes laboratory at CENA and results were expressed in the delta per mil notation, with an analytical precision better than 0.2‰.

The carbon content in soil samples was determined using 1 to 5 gr of the  $<200 \mu\text{m}$  soil fraction by combustion in a carbon autoanalyzer or by the wet digestion method. These analyses were undertaken by the Soil Chemistry Laboratory at CENA and the results are expressed as weight percent of the dry sample.

## RESULTS AND DISCUSSION

### Soil Properties

Physical and chemical properties in the Salitre soil are presented in Figures 3 and 4. These soils are characterized by a high percentage of clay (78% to 83%), with sand and silt being the minor fractions (Fig. 3). Soil organic carbon decreased from *ca.* 4.5% at the surface (except at the half slope location of *ca.* 6%) to 1.5% at 2.0 m (Fig. 4). The relatively good preservation of organic matter in these tropical soils could be a consequence of their high clay content, giving rise to the formation of refractory organo-clay complexes.

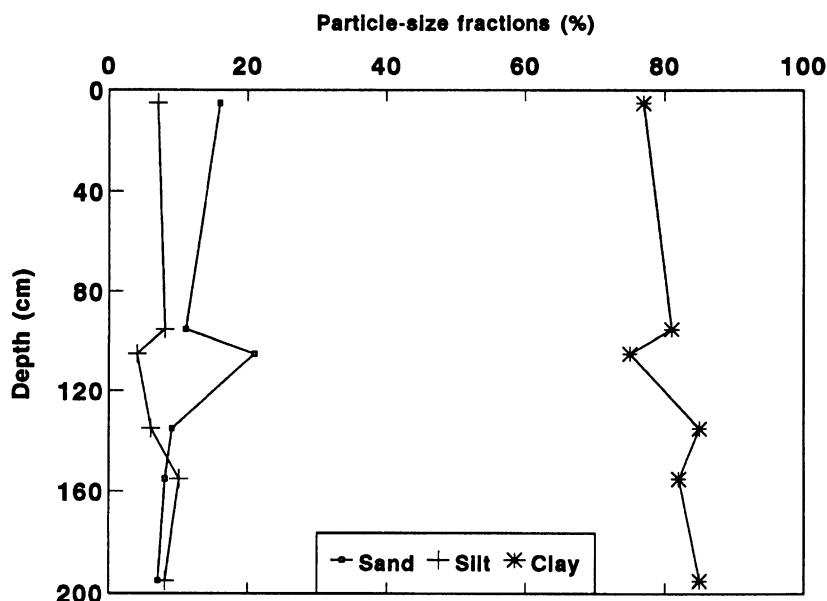


Fig. 3. Particle size fractions of soil from Salitre (half of slope)

Charcoal is present throughout the entire soil profile at each of the three sampling locations (Fig. 5). Peaks are observed at certain soil depths, but without a clear correlation among the three locations. The presence of charcoal in these soils is a clear indication that this area has been affected by frequent forest fires, probably throughout most of the recorded history. The extremely high content of charcoal in some soil horizons indicates that fires were much more prevalent during some periods, perhaps indicating much drier conditions. Charcoal found in forest soils has also been reported in the Upper Rio Negro region of Colombia and Venezuela, indicating the occurrence of frequent and widespread fires in the Amazon Basin, possibly associated with extremely dry periods and/or human disturbance (Saldarriaga and West 1986). Such events range from 6000 BP to the present and several coincide with dry phases recorded in the tropics during the Holocene (Absy 1982; Absy *et al.* 1991; Van der Hammen 1982).

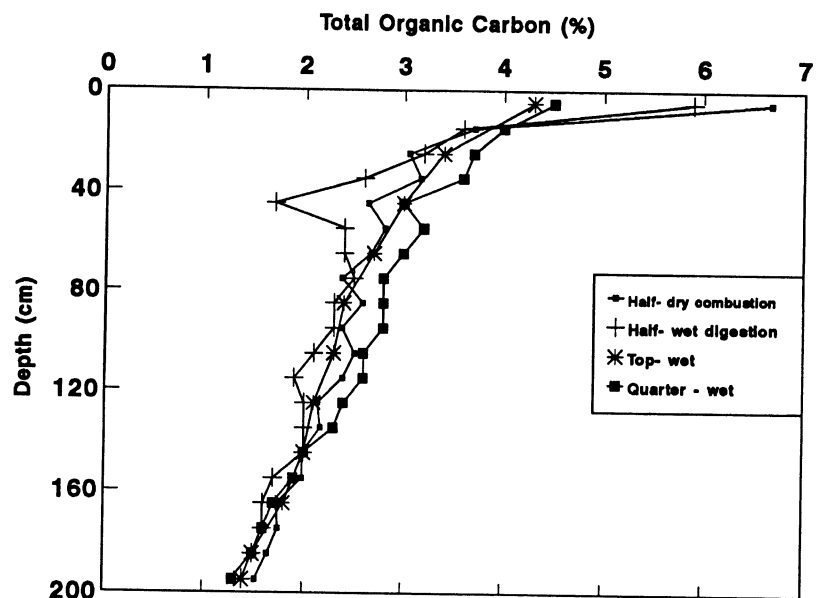


Fig. 4. Total organic carbon of soil samples

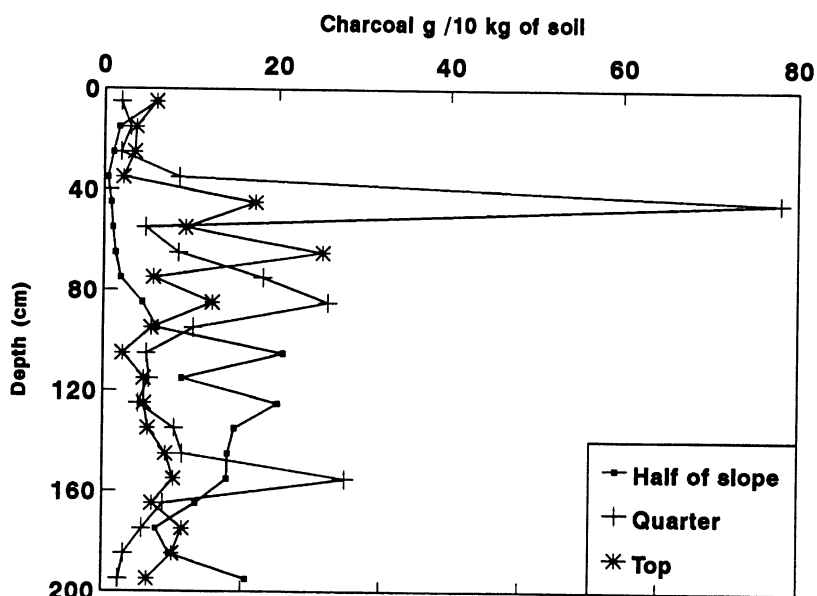


Fig. 5. Charcoal distribution in relation to soil depth

The presence of charcoal throughout the entire sampled depth of the Salitre area soil profiles indicates that translocation of organic matter has been a significant process during the development of these soils. Erosion is sometimes attributed to the formation of soil horizons (Oliveira, Menk and Rota 1985). However, the  $^{14}\text{C}$  dates on charcoal described in the following section eliminate this

possibility for the Salitre soils. The most likely mechanism is translocation of organic matter due to biological activity (Boulet *et al.* 1995). This mechanism has been postulated to explain the presence of charcoal in soils from Amazonia (Soubies 1980) and in the dark horizon of soils from the Botucatu region, Brazil (Miklos 1992).

### Carbon Isotope Data

#### <sup>14</sup>C Results

Charcoal <sup>14</sup>C ages range from 160 yr BP near the soil surface to 8790 yr BP at 2.0 m depth (Fig. 6). From the age/depth gradient, charcoal at the deepest locations should date to at least 12 ka BP. No clear differences are observed in the <sup>14</sup>C depth profiles at the three sampling locations, except that below 150 cm, the charcoal sample from the “quarter” location is older than charcoal at similar depth in the other locations. These data also show that no significant age differences are observed over certain depth intervals. This effect is most obvious at the top location between 150 and 180 cm, suggesting mixing of charcoal at this depth interval. <sup>14</sup>C dates of charcoal and humin do not show significant age differences to a depth of 155 cm (Fig. 7). It can be assumed therefore that the humin is a useful fraction for dating SOM. At 200 cm the humin is *ca.* 2000 yr younger than the charcoal, indicating a significant translocation of younger carbon into the deepest part of soil profile. <sup>14</sup>C dates of charcoal indicate that these materials represent vegetation that grew in the study area for at least the last 12 ka BP.

#### <sup>13</sup>C Results

$\delta^{13}\text{C}_{\text{PDB}}$  values obtained for SOM from the “half”, “quarter” and “top” slope locations range, respectively, from  $-24.7\text{‰}$  to  $-21\text{‰}$ ,  $-26.7\text{‰}$  to  $-21.7\text{‰}$  and  $-26\text{‰}$  to  $-21\text{‰}$  (Fig. 8). This general

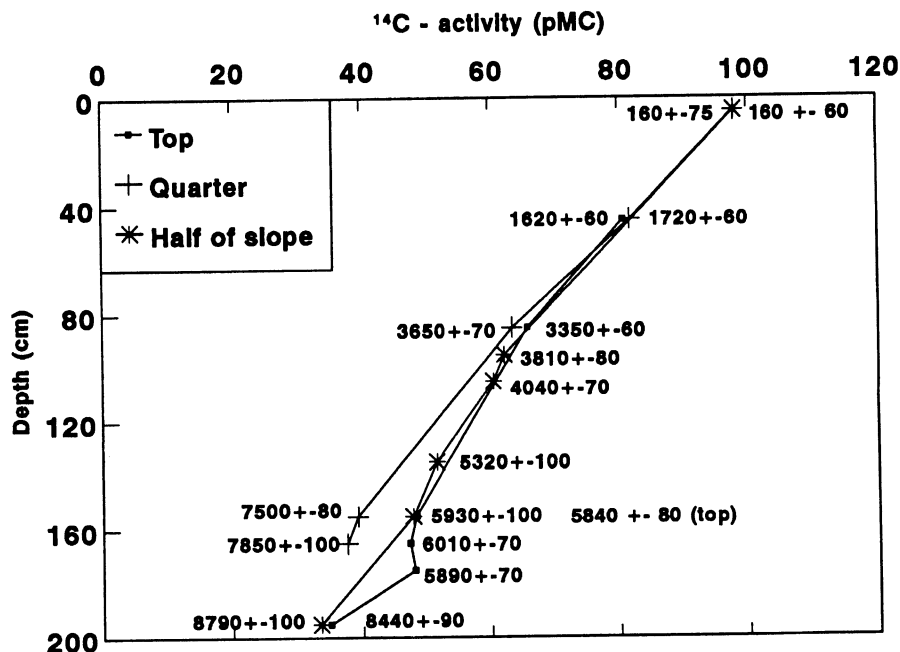
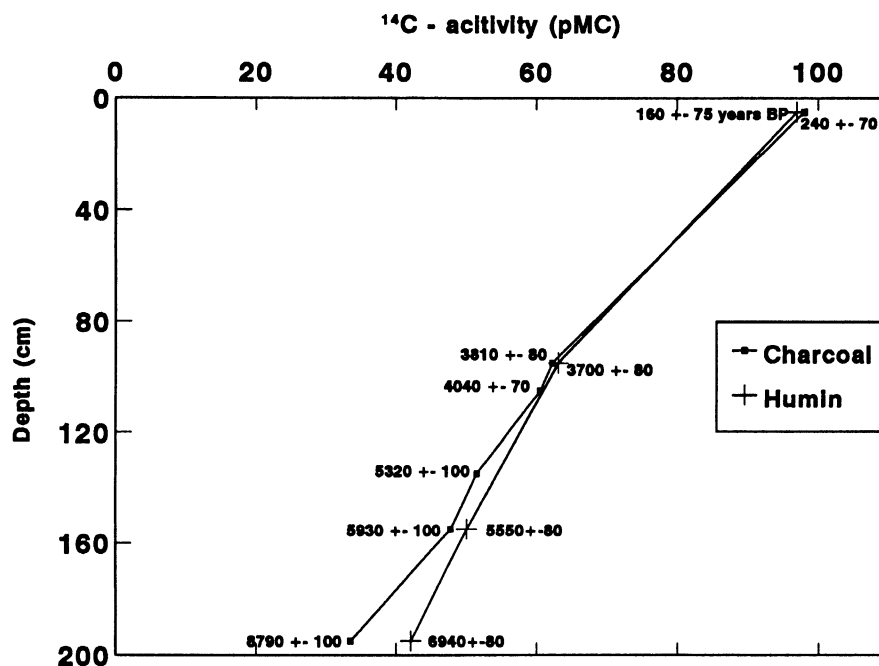
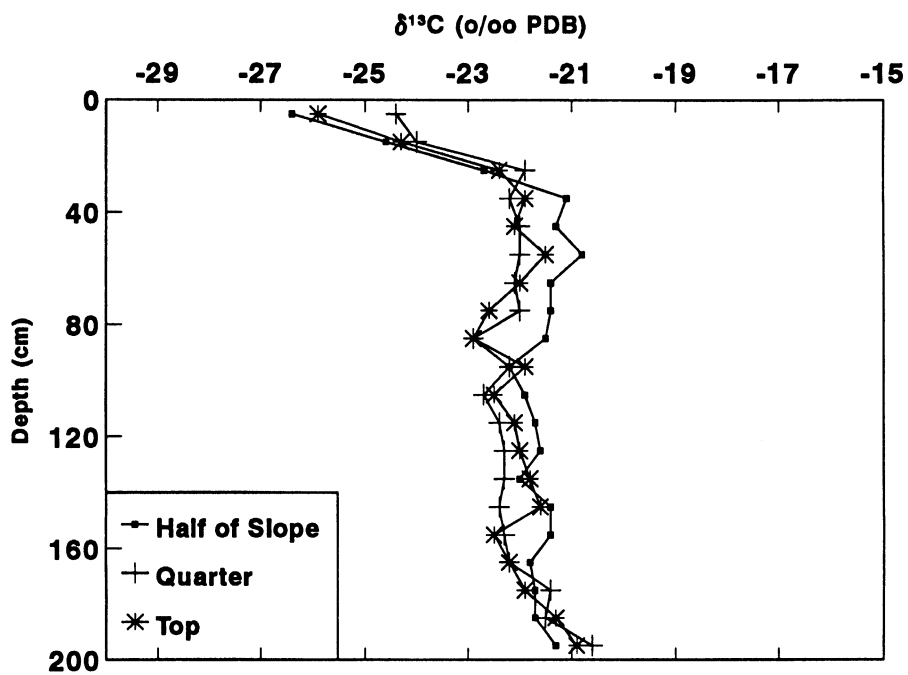


Fig. 6. <sup>14</sup>C dates (yr BP) of charcoal samples in relation to soil depth

Fig. 7.  $^{14}\text{C}$  dating of charcoal and humin samples (half of slope)Fig. 8. Natural variation of  $\delta^{13}\text{C}$  in relation to soil depth



trend toward heavier  $^{13}\text{C}$  enrichment with depth could be due to isotope effects occurring during decomposition of SOM (Nadelhoffer and Fry 1988; Becker-Heidmann and Scharpenseel 1992). The pattern is certainly typical for SOM generated by  $\text{C}_3$ -type vegetation (Cerri *et al.* 1985; Boutton 1991; Pessenda *et al.*)

The  $\delta^{13}\text{C}$  values of the charcoal show a wide variability within the three soil depth profiles, ranging between  $-28$  to  $-25$  ‰ (Fig. 9). Their  $^{13}\text{C}$  values clearly indicated that  $\text{C}_3$  plants have dominated in the area, probably for at least the last 12,000 yr. This pattern concurs with the interpretation of the independent  $^{13}\text{C}$  data measured for SOM.

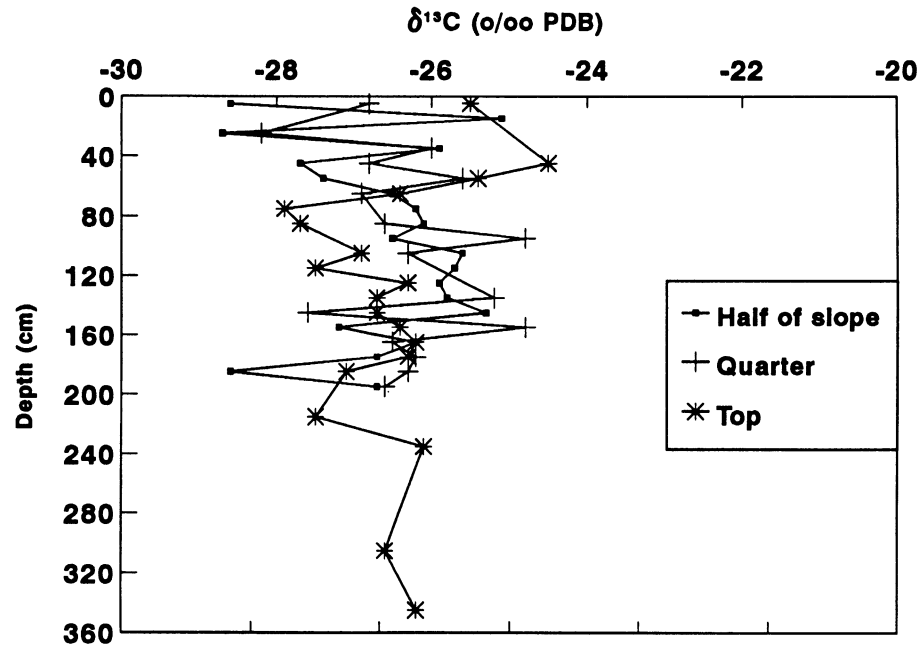


Fig. 9. Natural variation of  $\delta^{13}\text{C}$  of charcoal samples in relation to soil depth

#### Implication for Reconstruction of Paleovegetation

Reconstruction of paleovegetation and its relation to past climatic changes is one of the key research areas for our better understanding the response of tropical and subtropical forest to any future climate change in the Amazonia and Central regions of Brazil (Absy 1982; Absy *et al.* 1991; Ledru 1993; Pessenda *et al.*, in press). In the Salitre area, Central Brazil, Ledru (1993) used pollen analyses to record vegetational changes during the last 32,000 yr BP. She postulated two major episodes of forest retreat that were probably associated with very dry climatic conditions. These occurred between 11,000 and 10,000, and 6000 and 4500 yr BP. Dry periods have also been reported in the Central Amazon Basin and other areas of South America during the Holocene (Absy 1982; Van der Hammen 1982). The most significant of these phases appear to fall between 7500 and 6000; 4200 and 3500; 2700 and 2000; 1500 and 1200; and 700 and 400 yr BP (Bigarella 1971; Fairbridge 1976; Absy *et al.* 1991).

In the case of Salitre area, it is possible to expect that during the drier periods the vegetation was mainly composed of  $\text{C}_4$  grass species, since these are more resistant to low soil moisture (Tieszen *et*

al. 1979; Korner, Farquhar and Roksandic 1988). However, our  $^{13}\text{C}$  data from charcoal and SOM samples suggest that  $\text{C}_4$  grasses were not the predominant vegetation at any time in the SOM record, including the drier periods postulated by Ledru (1993). It seems that  $\text{C}_4$  grass has not been the dominant vegetation during the drier periods in the tropical Amazon region (Martinelli *et al.*, in press). However,  $^{13}\text{C}$  data from subtropical soils (Pessenda *et al.* 1995) suggest that  $\text{C}_4$  plants provided a significant part of the vegetation during the early and middle part of the Holocene in the subtropical region of Brazil. This in turn indicates that in the case of tropical regions the "dry phase" savanna vegetation was dominated by  $\text{C}_3$  grasses and/or woody vegetation. On the basis of pollen analyses, Absy (1991) postulated this hypothesis for vegetation changes in the Carajas, Amazon region during a dry phase between 7500 and 6000 BP. An alternative explanation is that the replacement of the forest by savanna vegetation was restricted to small areas. The presence of abundant charcoal in the soils in the Salitre area even during the dry periods postulated by Ledru (1993) suggests that forest and woody vegetation were present in this region at least throughout the entire Holocene. The presence of  $\text{C}_4$  plants inferred from the  $^{13}\text{C}$  data in subtropical regions (Pessenda *et al.* 1995) could indicate that during past episodes of climatic change this region was much drier than the tropical region.

## CONCLUSION

$^{14}\text{C}$  concentrations in charcoal and SOM collected from three soil profiles in the Salitre area, Central Brazil, indicate that the organic matter in these soils represents the Holocene at least. The presence of significant amounts of charcoal in the soil profiles suggests that forest fires were a significant process throughout the Holocene and probably had an important role in determining the dynamics of the forest vegetation in the study area.  $^{13}\text{C}$  data from both charcoal and SOM indicate that  $\text{C}_3$  plants provided the dominant vegetation, even during the dry periods postulated by Ledru (1993). This study provides additional information to our understanding of the relationship between climatic changes and vegetation in the tropical region of Brazil.

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## NATURAL RADIOCARBON MEASUREMENTS IN BRAZILIAN SOILS DEVELOPED ON BASIC ROCKS

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**ABSTRACT.** This paper presents <sup>14</sup>C, <sup>13</sup>C and chemical data of soil organic matter (SOM) in three soil profiles under native forests from Brazil: Londrina (southern), Piracicaba (southeastern) and Altamira (northern). The main objective is to use carbon isotopes in tropical and subtropical soils of Brazil to provide information about vegetation changes that occurred in relation to climate changes during the Holocene. <sup>14</sup>C data from SOM indicate that the organic matter in the soils studied is of at least Holocene age. <sup>13</sup>C data indicate that C<sub>4</sub> plants probably provided the dominant vegetation in Londrina and Piracicaba during the early and mid-Holocene and that C<sub>3</sub> plants provided the dominant vegetation in the Altamira region during the Holocene.

### INTRODUCTION

Radiocarbon dating has been used in soil work since 1950. The main emphasis has been on chronological problems of soil genesis (Martel and Paul 1974), carbon dynamics and identifying parameters for the evaluation of biologically resistant forms of organic matter (O'Brien 1984). Because SOM decomposition and mineralization are relatively slow processes, only a few methods can provide useful data, *e.g.*, long-term experiments or, in some cases, natural <sup>13</sup>C measurements (Balesdent 1987).

We report here a list of <sup>14</sup>C, <sup>13</sup>C and chemical data of organic matter from soil profiles in three regions of Brazil. The aim of the project, developed in the Radiocarbon Laboratory of the CENA, was to associate <sup>14</sup>C dates with the <sup>13</sup>C signature of SOM to study the evolution of local vegetation. <sup>14</sup>C data allowed us to estimate SOM chronology and <sup>13</sup>C indicated the vegetation types C<sub>3</sub> and C<sub>4</sub> of the local paleoenvironment.

### STUDY SITES

L. C. R. Pessenda, E. P. E. Valencia, P. B. Camargo and E. C. C. Telles collected 48 samples from soil profiles under natural forests. In July 1991, samples were collected from Londrina (51°10'W 23°18'S), state of Paraná, southern Brazil and Piracicaba (22°43'S 47°38'W), São Paulo, southeastern Brazil. The site of Altamira (52°58'W, 3°30'S), Pará, northern Brazil, was sampled in February 1992. The natural forest at Londrina and Piracicaba is a Mesophytic semideciduous type and Altamira is part of the Amazon forest (Fig. 1).

The soils of Londrina and Altamira are clayey, with kaolinite predominating, and are classed as "Terra Roxa Estruturada", according to the Brazilian soil classification, "Alfisol" in Soil Taxonomy (USDA) and "Nitosol" in the FAO soil classification system. The clayey and kaolinitic soil at Piracicaba is called "Latossolo Vermelho Escuro" (Dark Red Latosol), according to Brazilian soil classification, "Oxisol" in Soil Taxonomy; "Ferralsol" in FAO classification.

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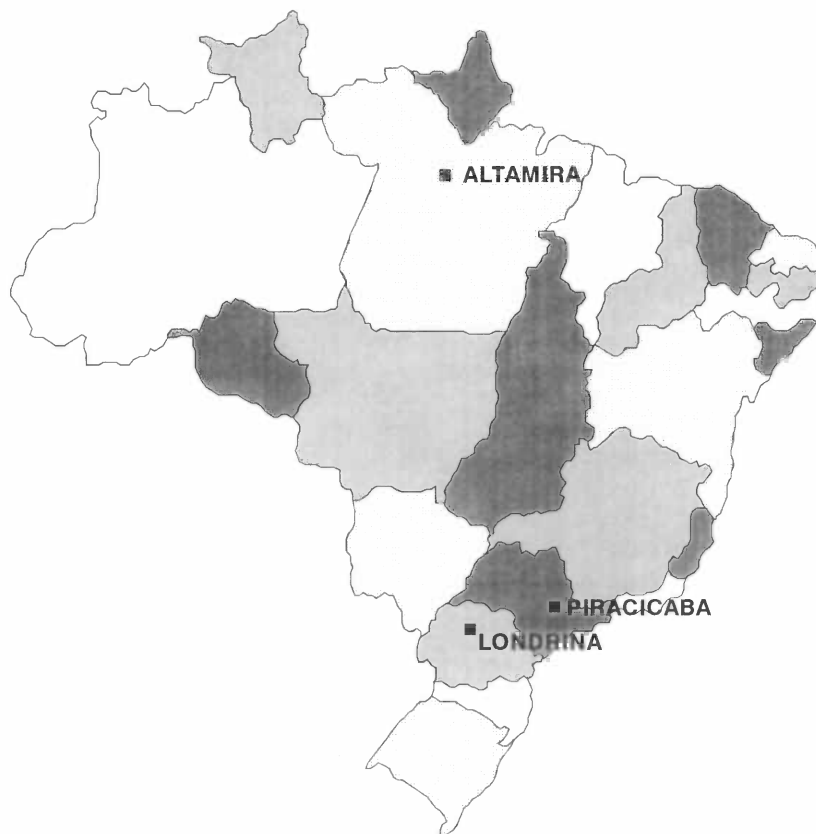


Fig. 1. Map of Brazil showing study sites

## METHODS

At all sites, soils were sampled by collecting up to 5 kg in 10-cm increments from the surface to 180-cm depth. Samples for total SOM analyses (1 kg) were dried at 60°C to constant weight and root fragments were discarded by handpicking. Any remaining plant debris was removed by flotation in HCl 0.01 M and redried to constant weight. In order to minimize the effects of sample heterogeneity, all samples were ground, sieved (<200  $\mu\text{m}$ ) and homogenized for  $^{13}\text{C}$  and  $^{14}\text{C}$  measurements. The humin fraction was extracted from the 200  $\mu\text{m}$  fraction (2.5 kg) according to conventional methods (Dabin 1971; Goh 1978; Anderson and Paul 1984): 1) acid digestion in hydrochloric acid 0.5 M at 70°C–80°C for 4 h and washing with distilled water until pH reaches 3–4; 2) reaction of solid residue with at least 30 liters (10 liters per extraction) of sodium pyrophosphate-sodium hydroxide 0.10 M for *ca.* 36 h (12 h per extraction) and washing with distilled water until the pH reaches 3–4; 3) hydrolysis of solid residue with 4 liters of 3 M HCl at 100°C for 12 h, washing until the pH reaches 3–4; and 4) the solid residue was dried at 40°C for 48 h and sieved (< 200  $\mu\text{m}$ ).

$^{14}\text{C}$  dating,  $\delta^{13}\text{C}$  and chemical analyses were performed between August 1991 and November 1992.  $^{14}\text{C}$  analyses were carried out on total SOM and the humin fraction using benzene and liquid scintillation counting (Pessenda and Camargo 1991). Benzene samples were counted for at least 48 h in a low-level Packard 1550 liquid scintillation spectrometer.  $^{14}\text{C}$  ages are expressed in conventional

years BP and percent modern carbon (pMC) relative to 95% of the activity of the NIST oxalic acid standard (HOxI) and normalized to a δ<sup>13</sup>C of -25‰ PDB (Stuiver and Polach 1977). The analytical precision is ± 1.0 pMC.

The stable carbon isotopic ratios (<sup>13</sup>C/<sup>12</sup>C) of SOM were determined by isotope ratio mass spectrometry using CO<sub>2</sub> from samples combusted at 900°C in an atmosphere of pure oxygen. Results are expressed as δ<sup>13</sup>C with respect to PDB standard in the conventional (‰) notation; the analytical precision is ± 0.2‰. Carbon contents of soil samples were determined, using 1–5 g of the <200 μm-sized fraction, using a C,H autoanalyzer. All samples were analyzed 2 or 3 times, with a coefficient of variation <4%; values are expressed as weight percent of dry sample. Nitrogen contents were determined using 0.7 g of <200 μm fraction using the Kjeldahl method; values are expressed as weight percent of dry sample. Soil density was calculated by collecting a mass of soil in a small cylinder of known volume. After drying to constant mass, the density was calculated. The soil pH was determined in water.

**RESULTS AND DISCUSSION**

Tables 1 through 3 show <sup>14</sup>C, δ<sup>13</sup>C, total organic carbon, total nitrogen, soil bulk density and soil pH values obtained for total SOM from Londrina, Piracicaba and Altamira, respectively. All the soil profiles show decreasing <sup>14</sup>C content with depth, except for the 140–150 cm sample interval of Altamira profile, which seems to record an age reversal. All surface samples have <sup>14</sup>C >100 pMC, showing the influence of thermonuclear <sup>14</sup>C in the recent organic matter. δ<sup>13</sup>C values between the surface and the 40–50 cm interval are indicative of C<sub>3</sub> plants, and this reflects the current local vegetation (forest) in all three regions. These values remained almost constant for the Altamira profile from the soil surface to 180 cm depth; however, the Londrina and Piracicaba soils show a significant change from -21.6‰ to -15‰ in <sup>13</sup>C values.

TABLE 1. Londrina Soil Profile

Lab code (CENA-)	Sample horizon (cm)	<sup>14</sup> C (pMC)	<sup>14</sup> C (yr BP)	δ <sup>13</sup> C (‰)	Total C (wt %)	Total N (wt %)	C/N	Soil bulk density (g/cc)	Soil pH
194	0 to 10	110 ± 1.2	--	-25.8	1.85	0.21	8.81	1.23*	5.4*
--	10 to 20	--	--	-25.1	1.43	0.16	8.94	--	--
--	20 to 30	--	--	-24.3	1.59	0.19	8.42	1.23*	5.3*
--	30 to 40	--	--	-24.2	1.29	0.15	8.60	--	--
193	40 to 50	90.3 ± 0.7	820 ± 60	-23.8	1.02	0.13	7.85	--	--
--	50 to 60	--	--	-23.2	1.00	0.11	9.09	--	--
--	60 to 70	--	--	-22.2	0.89	0.10	8.90	1.25*	5.5*
192	70 to 80	78.8 ± 1.1	1920 ± 60	-21.6	0.86	0.09	9.56	--	--
--	80 to 90	--	--	-21.0	0.85	0.09	9.44	--	--
196	90 to 100	74.3 ± 0.8	2390 ± 60	-21.3	0.99	0.10	10.00	--	--
--	100 to 110	--	--	-19.4	0.82	0.08	10.25	--	--
--	110 to 120	--	--	-18.5	0.71	0.07	10.00	--	--
195	120 to 130	50.8 ± 0.7	5450 ± 90	-16.8	0.71	0.06	11.83	--	--
--	130 to 140	--	--	-16.7	0.73	0.06	12.17	--	--
--	140 to 150	--	--	-15.5	0.78	0.06	13.00	1.43*	5.3*
--	150 to 160	--	--	-15.1	0.88	0.05	17.80	--	--
--	160 to 170	--	--	-14.9	0.87	0.05	17.40	--	--
218	170 to 180	31.3 ± 0.5	9340 ± 120	-15.0	0.93	0.05	18.60	--	--

\*Values obtained from Ref. 8



TABLE 2. Piracicaba Soil Profile

Lab code (CENA-)	Sample horizon (cm)	<sup>14</sup> C (pMC)	<sup>14</sup> C (yr BP)	δ <sup>13</sup> C (‰)	Total C (wt %)	Total N (wt %)	C/N	Soil bulk density (g/cc)	Soil pH
184	0 to 10	107.3 ± 0.8	--	-25.7	2.67	0.30	8.90	1.22*	5.8*
--	10 to 20	--	--	-25.7	1.59	0.16	9.94	1.35*	5.0*
--	20 to 30	--	--	-25.6	1.12	0.11	10.18	1.21*	5.0*
--	30 to 40	--	--	-23.8	0.96	0.09	10.67	1.16*	5.0*
191	40 to 50	85.8 ± 0.9	1230 ± 90	-22.7	0.79	0.07	11.29	1.29*	4.7*
--	50 to 60	--	--	-21.6	0.63	0.06	10.50	--	5.0*
--	60 to 70	--	--	-19.7	0.63	0.05	12.60	--	5.2*
223	70 to 80	71.8 ± 0.7	2680 ± 70	-19.0	0.64	0.05	12.80	--	5.2*
--	80 to 90	--	--	-18.7	0.63	0.05	12.60	--	--
222	90 to 100	68.8 ± 0.7	3030 ± 70	-17.2	0.56	0.04	14.00	--	--
--	100 to 110	--	--	-16.8	0.56	0.04	14.00	--	--
221	110 to 120	66.8 ± 0.7	3260 ± 70	-17.5	0.54	0.04	13.50	--	--
--	120 to 130	--	--	-16.8	0.47	0.04	11.75	--	--
--	130 to 140	--	--	-16.7	0.43	0.04	10.75	--	--
220	140 to 150	63.8 ± 0.6	3640 ± 70	-16.7	0.43	0.04	10.75	--	5.3*

\*Values obtained from Ref. 9

TABLE 3. Altamira Soil Profile

Lab code (CENA-)	Sample horizon (cm)	<sup>14</sup> C (pMC)	<sup>14</sup> C (yr BP)	δ <sup>13</sup> C (‰)	Total C (wt %)	Total N (wt %)	C/N	Soil bulk density (g/cc)	Soil pH
239	0 to 10	103.9 ± 0.8	--	-26.7	1.40	0.18	7.78	1.39*	5.5*
--	10 to 20	--	--	-25.6	0.78	0.12	6.50	--	--
--	20 to 30	--	--	-26.8	0.81	0.10	8.10	1.43*	5.1*
--	30 to 40	--	--	-25.7	0.68	0.09	7.55	--	--
237	40 to 50	84.1 ± 0.7	1440 ± 70	-25.7	0.57	0.08	7.12	--	--
--	50 to 60	--	--	-25.8	0.53	0.07	7.57	--	--
--	60 to 70	--	--	-25.6	0.41	0.07	5.86	--	--
236	70 to 80	70.8 ± 0.7	2790 ± 80	-25.4	0.39	0.06	6.50	--	--
--	80 to 90	--	--	-26.8	0.26	0.06	4.33	--	--
233	90 to 100	63.5 ± 0.7	3640 ± 90	-25.6	0.28	0.05	5.60	--	--
--	100 to 110	--	--	-25.7	0.23	0.05	4.60	--	--
232	110 to 120	55.0 ± 0.6	4800 ± 80	-25.0	0.25	0.04	6.25	--	--
--	120 to 130	--	--	-25.3	0.22	0.04	5.50	--	--
--	130 to 140	--	--	-25.5	0.25	0.04	6.25	--	--
231	140 to 150	57.9 ± 0.6	4390 ± 90	-26.5	0.31	0.04	7.75	1.41*	5.9*

\*Values obtained from Ref. 8

Taking into account the increase of <sup>14</sup>C age with depth, the increase of δ<sup>13</sup>C can be explained in two ways: 1) organic matter decomposition leads to the accumulation and transport of <sup>13</sup>C-enriched materials with depth in the profile; 2) during the pedological evolution of the soil profiles, the dominant vegetation changed from C<sub>4</sub>- to C<sub>3</sub>-dominant photosynthetic pathway. Similar <sup>13</sup>C records have also been documented in other studies in Brazil (Cerri 1986; Rocha 1990; Desjardins *et al.* 1991). Research is underway to test the postulated hypotheses, *e.g.*, phenol lignin analysis of selected soil samples to characterize past vegetation change in south and southeast regions of Brazil. Paleoenvironmental changes during the last 30 ka yr BP have been documented in Central Brazil and the Amazon basin (Absy *et al.* 1991; Ledru 1993). Based on pollen analyses, these studies indicate periods of forest regression changing to savanna-type vegetation. Such evidence supports vegetation

changes as an explanation for the  $^{13}\text{C}$  trend observed in the Piracicaba and Londrina soils. Work in progress in soils from Central Brazil will contribute to the understanding of the carbon isotopic record in Brazilian soils.

Table 4 shows  $^{14}\text{C}$  dates,  $\delta^{13}\text{C}$  and total carbon of the humin fraction from the same sites. In general, these data show less  $^{14}\text{C}$  content for the humin fraction compared to bulk SOM. This difference is most pronounced in the Altamira soil. Similar patterns have been reported in other studies (Martel and Paul 1974; Campbell *et al.* 1967; Nowaczyk and Pazdur 1990). This reflects that SOM is composed of fractions of different age. Humic and fulvic acids that are removed during humin extraction are more mobile and can be sources of younger carbon transported downward from the shallow part of the soil. In the case of  $^{13}\text{C}$ , we observed no significant differences between SOM and humin samples. The total carbon concentration of the humin fraction is significantly (up to five times) less than that of SOM. The results and ideas presented in the paper will be discussed in detail elsewhere, as a component of more comprehensive publications (Martinelli *et al.* 1996; Pessenda *et al.* in press).

TABLE 4. Humin Fraction of Soils of Londrina, Piracicaba and Altamira

Lab code (CENA-)	Site	Sample horizon (cm)	$^{14}\text{C}$ (pMC) humin	$^{14}\text{C}$ (pMC) total SOM	$\delta^{13}\text{C}$ (‰) humin	Total C (wt %) humin
241	Londrina	40 to 50	72.9 ± 0.7	90.3 ± 0.7	-22.9	0.22
230	Londrina	90 to 100	68.1 ± 0.6	74.3 ± 0.8	-21.3	0.24
240	Londrina	170 to 180	26.1 ± 0.4	31.3 ± 0.5	-14.5	0.25
243	Piracicaba	70 to 80	65.1 ± 0.9	71.8 ± 0.7	-19.3	0.13
242	Piracicaba	90 to 100	59.1 ± 0.8	68.8 ± 0.7	*	*
246	Piracicaba	110 to 120	57.8 ± 0.9	66.8 ± 0.7	-17.4	0.11
249	Altamira	50 to 60	56.6 ± 0.6	--	-26.4	0.12
250	Altamira	100 to 110	36.5 ± 0.5	--	-25.8	0.13
248	Altamira	130 to 140	29.5 ± 0.5	--	-26.2	0.10

\*Insufficient material

#### ACKNOWLEDGMENTS

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## THE EFFECT OF TILLAGE ON SOIL ORGANIC MATTER USING $^{14}\text{C}$ : A CASE STUDY

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**ABSTRACT.** We compared four adjacent soil plots in an effort to determine the effect of land use on soil carbon storage. The plots were located at the High Plains Agricultural Research Laboratory near Sidney, Nebraska. We measured  $^{14}\text{C}$ , total carbon, total nitrogen and  $^{137}\text{Ce}$  to determine the size and turnover times of rapid and stable soil organic matter (SOM) pools, and their relation to land-use practices. Results were consistent with the model produced by Harrison, Broecker and Bonani (1993a) in that the  $^{14}\text{C}$  surface soil data fell on the time trend plots of world  $^{14}\text{C}$  surface soil data, indicating that the natural sod and non-tilled plots had a rapidly turning over SOM pool, comprising *ca.* 75% of surface soil carbon, and the tilled plots had a rapidly turning over SOM pool, comprising only 50% of surface soil carbon.

### INTRODUCTION

The role of the terrestrial biosphere remains a major uncertainty in our understanding of the modern carbon cycle. Soil in particular represents a large and potentially variable carbon reservoir. Because soils are heterogeneous, extrapolating global carbon fluxes from limited, existing data can be only tenuous. Thus, it is crucial that we broaden our base of soil carbon data. To this end, we have researched carbon cycling in soils that have been subject to different land-use treatments. Atmospheric  $\text{CO}_2$  has been rising as a result of human activities, including fossil-fuel combustion, cement manufacture and land conversion (*i.e.*, deforestation). Further, concentrations of  $\text{CO}_2$  in the atmosphere are expected to double in the next century. Because  $\text{CO}_2$  is a greenhouse gas that could perturb the global climate, it is important to understand its sources, sinks and controls in the environment.

The loss of soil organic matter (SOM) as a result of the conversion of native land into agricultural land is a well-known phenomenon (Davidson and Ackerman 1993; Harrison 1993; Post and Mann 1990; Schlesinger 1991; Tate 1987). Harrison, Broecker and Bonani (1993a) developed a box model of SOM using surface soil "bomb"  $^{14}\text{C}$  data. This model allowed an estimation of soil carbon loss due to changes in land use. Based on the fact that soil pools with short residence times (<100 yr) will record a more pronounced response to atmospheric "bomb"  $^{14}\text{C}$  than those with long residence times (~1000 yr), it is possible to partition soils into fast- and slow-cycling soil pools with respect to a time trend of bulk surface soil  $^{14}\text{C}$  values (Harrison, Broecker and Bonani 1993a). Soils are a complex system comprised of various pools of organic material with a wide range of turnover times. Here we use "bomb"  $^{14}\text{C}$  as a tracer of soil organic carbon. Atmospheric  $^{14}\text{C}$  (as  $^{14}\text{CO}_2$ ) is incorporated into plant materials during photosynthesis. Death and subsequent decomposition of plant materials incorporates  $^{14}\text{C}$  into the SOM pool. Modeling SOM by partitioning it into two pools, a fast and a slow turnover pool, is a simplification, but it is adequate to yield an estimation of changes in carbon storage.

The purpose of this study is to evaluate the effect of land-use practices on carbon storage in cultivated soils subjected to different land-use practices. Related questions are whether agricultural soils have the potential to store excess  $\text{CO}_2$ , which soils are better candidates for increased carbon storage, and what cultivation/engineering practices would facilitate carbon storage. These questions must be resolved if we hope to understand and possibly mitigate the effects of rising atmospheric  $\text{CO}_2$ .

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## METHODS

### Site Description

Samples were collected from the High Plains Agricultural Laboratory near Sidney, Nebraska. The portion of the site used in this study is comprised of four adjacent plots, each 8.5 × 45.5 m. The soil has been characterized as a Duroc loam (fine, silty, mixed, mesic Pachic Haplustoll). The parent material was of mixed loess and alluvium; the site has a slope of <1%. All four plots were covered by native sod until 1970 when they were moldboard-plowed to permit the commencement of a long-term wheat/fallow rotation experiment. One plot was left in its natural sod state and the other three were planted with winter wheat (*Triticum aestivum*). One plot was subjected to plowing (plowing depth ~20 cm, no plant residue on surface), another to sub-till plowing in which most of the plant residue was left on the surface (tilled with V-shaped blades to a depth of 90–150 cm), and the third was a non-plowed plot on which weeds were controlled chemically. Wheat was harvested in odd years and the plots were left fallow in even years (Fenster and Peterson 1979). Samples were collected from each of the four plots (subsequently referred to as plowed, subtilled, sod and non-plowed) using a 5-cm diameter soil auger (hand) to represent depth intervals 0–5 cm, 5–10 cm, 10–20 cm, 20–30 cm, 30–40 cm, 40–50 cm, 50–75 cm and 75–100 cm. Soil was immediately transferred to plastic bags, excess air was expelled and the bags were sealed. Samples were processed following the procedure described by Trumbore (1988). Visible roots, organic debris and pebbles were removed at the time of sample collection. Samples were oven-dried at 60°C within one week of collection. Dry samples were passed through a 2-mm sieve to remove and discard remaining pebbles and bits of plant debris. Samples were further crushed and sieved through a .0092-mm mesh. We used distilled water to float out light organic debris and then added dilute HCl to remove carbonates and, finally, rinsed the samples with distilled water until neutral, at which point we re-dried and crushed the samples.

We measured total carbon and total nitrogen for each depth interval using a Carlo Erba carbon/nitrogen analyzer. Following the acid extraction of carbonates with HCl, we measured the samples' CaCO<sub>3</sub> contents on a Coulometrics coulometer. Because residual carbonate was present, we performed a second carbonate extraction and re-analyzed the samples by coulometry to ensure that all carbonate had been removed to avoid distortion of the organic <sup>14</sup>C signal. The second acid extraction resulted in complete removal of carbonates.

We prepared the samples for accelerator mass spectrometry (AMS) by sealing them in evacuated quartz tubes with CuO, Ag and quartz wool and combusting at 950°C for 2 h. We sent the CO<sub>2</sub> to the ETH lab in Zürich for target preparation and <sup>14</sup>C analysis. We transferred the remaining portion of each sample to aluminum cans and counted them for <sup>137</sup>Ce activities on either an intrinsic germanium detector or a lithium-drifted detector.

## RESULTS AND INTERPRETATION

Data reported in the study are presented in Figures 1 through 6. <sup>137</sup>Cs, total organic carbon (TOC) and “bomb” <sup>14</sup>C trends demonstrate the homogenization of the soil profile due to both tillage processes. <sup>137</sup>Cs measurements enable the determination of the extent of erosion and physical mixing processes in the soil column because this nuclide tends to be strongly adsorbed onto clay and organic particles (Ritchie and McHenry 1990). Adsorption occurs rapidly after deposition and is, for practical purposes, irreversible. In undisturbed soils, <sup>137</sup>Cs shows an exponential decrease in depth. It is useful as a tracer in that its distribution is virtually only a function of mechanical transport. Thus, it clearly reflects disturbance due to plowing and other physical disturbances. As a measure of erosion and deposition, it is useful to calculate the total deposition of <sup>137</sup>Cs onto the landscape. In this study,

we have integrated the activity of cesium over the entire sampling depth, divided by the cross-sectional area of the sample and compared the result to the known decay-corrected deposition value of  $87 \text{ m Ci km}^{-2}$  (as of 1993). The total deposition value was determined from measurements of strontium fallout over New York City multiplied by 1.5 (Olsen *et al.* 1978). We assume that the  $87 \text{ m Ci km}^{-2}$  value is a reasonable value for input to Nebraska soils in that our sample site is in the same latitudinal band as New York City. However, because our study site has a mean annual precipitation of 47 cm compared to a mean annual precipitation of 112 cm for New York City (Bair 1992), the  $^{137}\text{Cs}$  input value was validated against Nebraska soil  $^{137}\text{Cs}$  measurements reported by Graustein and Turekian (1986). Decay-correcting Graustein and Turekian's data to 1994 yielded a  $^{137}\text{Cs}$  inventory of  $87 \text{ m Ci km}^{-2}$ , implying a deposition rate comparable to that of New York City.

Cesium inventories for 3 of the 4 plots were fairly consistent with one another and concur with atmospheric input. However, an excess in cesium was observed for the sod plot, implying a local input that deviated from the known atmospheric input for this region, or an influx of  $^{137}\text{Cs}$  tagged soil particles to the sod plot (Fig. 1). The excess cesium appears to be mainly confined to the 0–5-cm depth interval, correlating well with the high percentage of organic carbon in this stratum (Figs. 2–3). Because cesium is associated with organic materials, mechanical transport could serve to reduce both SOM and  $^{137}\text{Cs}$  in the soil profile, *i.e.*, tillage practices are known to promote erosion, providing one explanation of the lower values for the non-tilled plots. Further, some of the soil lost during plowing may have accumulated on the adjacent, grass-covered, natural sod site, its grass cover allowing the entrainment of windblown soil particles. The  $^{137}\text{Cs}$  excess observed in the sod plot might also result from the fact that this plot was not plowed in 1970, before which most of the  $^{137}\text{Cs}$  had been deposited. The other three plots were plowed, which may have caused some loss of surface soil. The question remains, however, why the sod site has a higher  $^{137}\text{Cs}$  inventory than can be explained by the known deposition for this area.

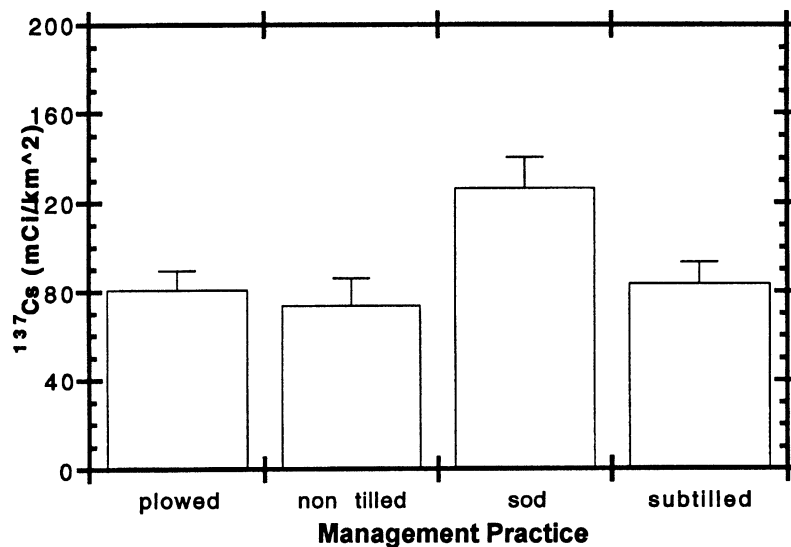


Fig. 1.  $^{137}\text{Cs}$  inventories. Three of the four plots show inventories in agreement with the known deposition (corrected to 1993). The sod plot shows excess  $^{137}\text{Cs}$ , possibly due to the input of  $^{137}\text{Cs}$  tagged organic material. Total inventories with  $1 \sigma$  errors are as follows (mCi km<sup>-2</sup>): plowed  $82.67 \pm 12.20$ , non-tilled  $73.60 \pm 12.73$ , subtile  $87.54 \pm 17.96$ , sod  $123.34 \pm 13.85$ .

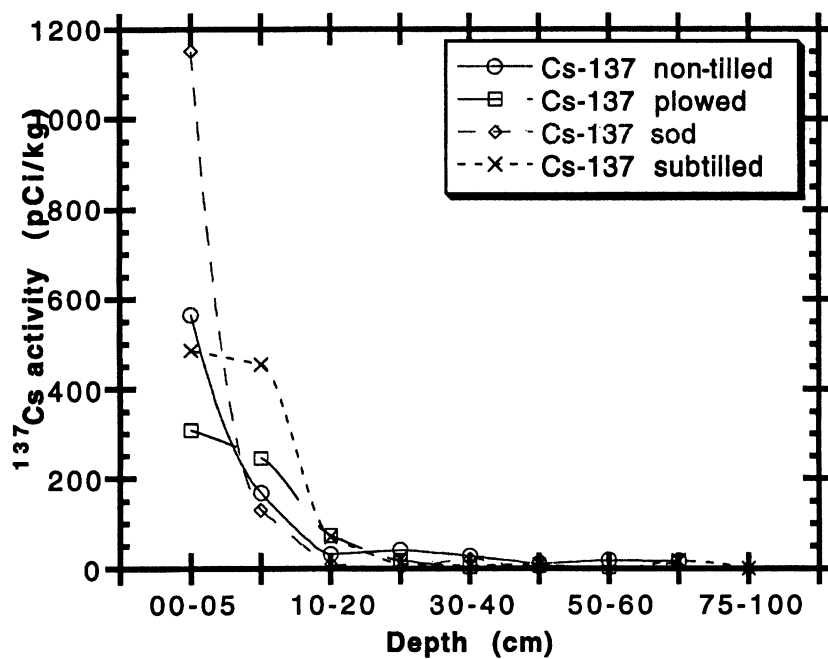


Fig. 2. <sup>137</sup>Cs profiles showing exponential decrease with depth

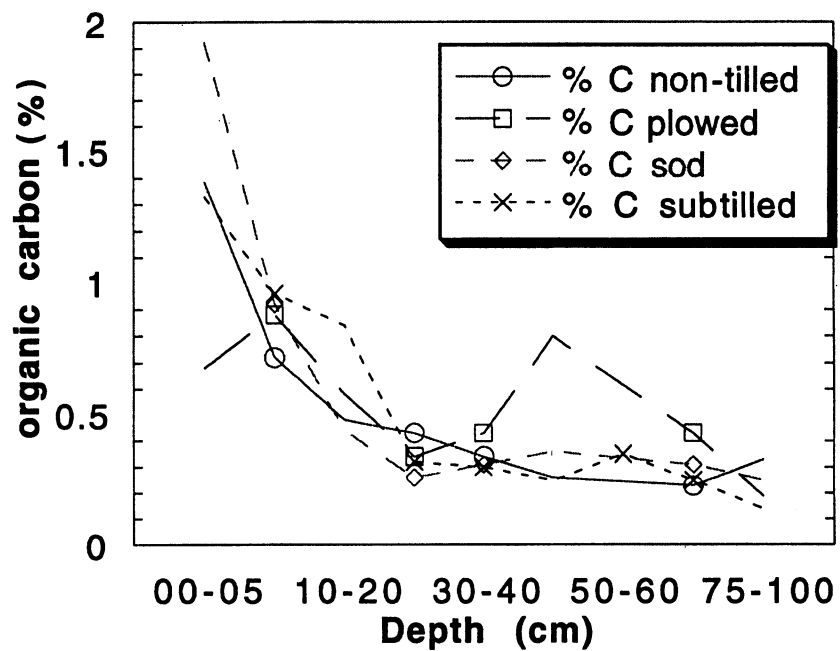


Fig. 3. Organic carbon profiles showing exponential decrease with depth

The  $^{137}\text{Cs}$  distribution in the soil profile clearly demonstrates the effect of mechanical mixing. The higher surface values of the non-tilled plots decreased rapidly with depth. The plowed plots show a more homogeneous cesium distribution through the upper 10 cm and a subsequent rapid decline similar to that observed in the non-plowed plots (Fig. 2). Carbon and nitrogen concentration data showed similar trends with depth, decreasing down the soil column (Figs. 3–4). As expected, carbon contents decrease more rapidly in the non-tilled plots.

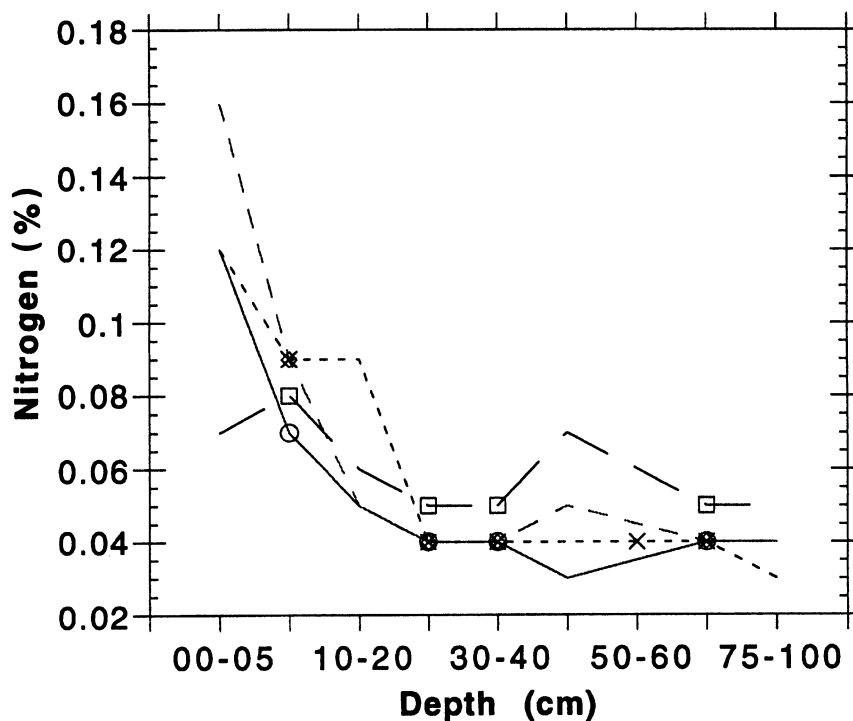


Fig. 4. Percent nitrogen plotted vs. depth. Percent nitrogen shows rapid decrease with depth.

$^{14}\text{C}$  surface values, expressed as percent modern carbon (pMC), were higher for the non-tilled plots. Furthermore, the non-tilled plots had a steeper gradient in the upper 10 cm than the plowed plots (surface  $^{14}\text{C}$  value ranges for non-tilled and tilled plots, respectively, are 102.5–108.2 and 101.8–103.4). The non-tilled plot, however, had a surface R value ( $R = \frac{^{14}\text{C}/^{12}\text{C}_{\text{sample}}}{(^{14}\text{C}/^{12}\text{C})_{1850 \text{ wood}}}$ , as in Harrison, Broecker and Bonani 1993b) closer to that of the tilled plots than the natural sod (Fig. 5). The removal of vegetation from the non-tilled plot during harvest is a probable cause of the difference between the sod and non-tilled plots'  $^{14}\text{C}$  values. All three treatments reduce the input of fresh organic material into the soil, whereas the litter produced on the sod plot is allowed to remain and enter the soil pool. Our deep soil  $^{14}\text{C}$  value is within the range of deep soil global  $^{14}\text{C}$  measurements (as cited in Harrison (1996)) and can be considered indicative of the presence of only a non-labile organic matter component. The intermediate  $^{14}\text{C}$  value ( $R = .87$ ) for the depth interval 30–40 cm of the sod plot indicates a decreased fraction of fast-turnover SOM at this depth. Taking this value to be a mixture between the fast cycling component and the slow-cycling component, we determine a fast-cycling component R value of 1.20 based on our deep soils measured R value, bulk surface  $^{14}\text{C}$  soil value and the assumption that 75% of surface soil is fast-cycling. If the 30–40 cm depth interval R



value of 0.87 is taken to represent a mixture between the fast-cycling (R value taken as 1.20) component and the slow-cycling component (R value that of deep soil, 0.71) we can calculate that this depth interval is comprised of roughly 33% fast cycling SOM and 67% slow cycling SOM, demonstrating a decreasing proportion of fast-cycling SOM with increasing depth in the soil column.

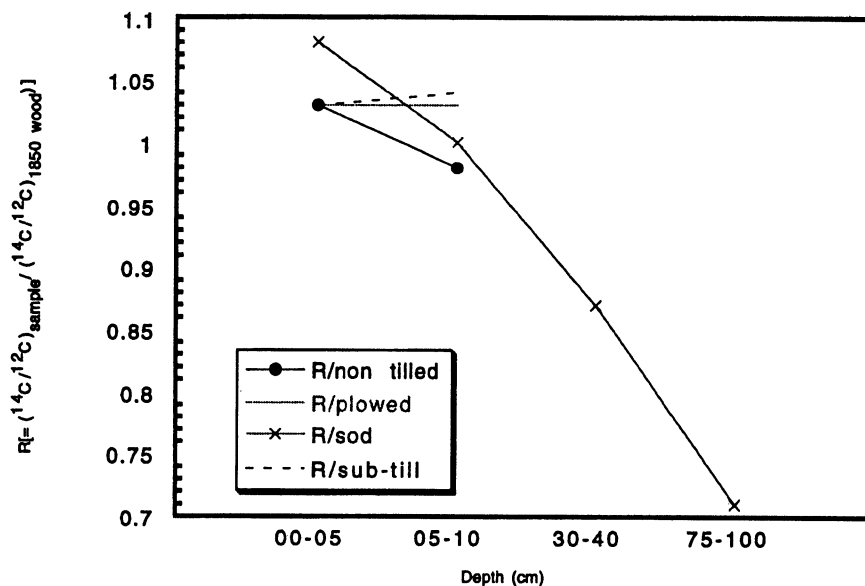


Fig. 5.  $^{14}\text{C}$  vs. depth. The natural (sod) plot shows a steeper  $^{14}\text{C}$  gradient. In addition, the surface  $^{14}\text{C}$  data point for the natural plot is also significantly higher than the others, implying a larger component of fast-cycling carbon.

## DISCUSSION

Based on a time trend of surface-soil  $^{14}\text{C}$  measurements, Harrison, Broecker and Bonani (1993a) showed that surface SOM can be modeled in terms of two pools, a labile pool having a short residence time of ~25 yr, and a slow-turnover pool having a residence time ~3700 yr. Natural (non-cultivated) surface soils were composed on average of ~75% fast turnover SOM and 25% slow turnover SOM. Cultivated surface soils, however, were interpreted to be only 50% fast turnover SOM and 50% slow turnover SOM, indicating the possibility that cultivated surface soils had lost a fraction of their initial fast turnover pool. Although here we use bulk surface  $^{14}\text{C}$  measurements to quantify the size and turnover times of carbon in SOM, the exponential decrease of “bomb”  $^{14}\text{C}$  in the biosphere has limited its usefulness as a geochemical tracer for future studies. The 1970s would have been the ideal decade in which to collect soil samples and measure their  $^{14}\text{C}$  activities, since this was the period when the maximum divergence in the various soil pools’  $^{14}\text{C}$  values could be observed. Since then, the difference in  $^{14}\text{C}$  values enhanced by the “bomb”  $^{14}\text{C}$  spike among individual SOM pools has been decreasing, making interpretation of the data progressively more tenuous (Fig. 6). The  $^{14}\text{C}$  data described herein appear to follow the “bomb”  $^{14}\text{C}$  surface trends for cultivated (in this case, plowed) and non-cultivated soils indicating a significant decrease in the proportion of fast cycling to slow cycling SOM (*i.e.*, a 3:1 mixture to a 1:1 mixture of fast:slow components). Indeed, the sod site surface  $^{14}\text{C}$  value of  $R=1.08$  indicates that the natural site (sod) does have the largest proportion of fast cycling surface SOM of the three plots. All of our surface  $^{14}\text{C}$  values follow the expected trends. However, the

weighted averages of the  $^{14}\text{C}$  values for the cultivated and uncultivated sites are very close together, arguably as a result of the atmospheric  $^{14}\text{C}$  inventory decrease as described above (Fig. 7).

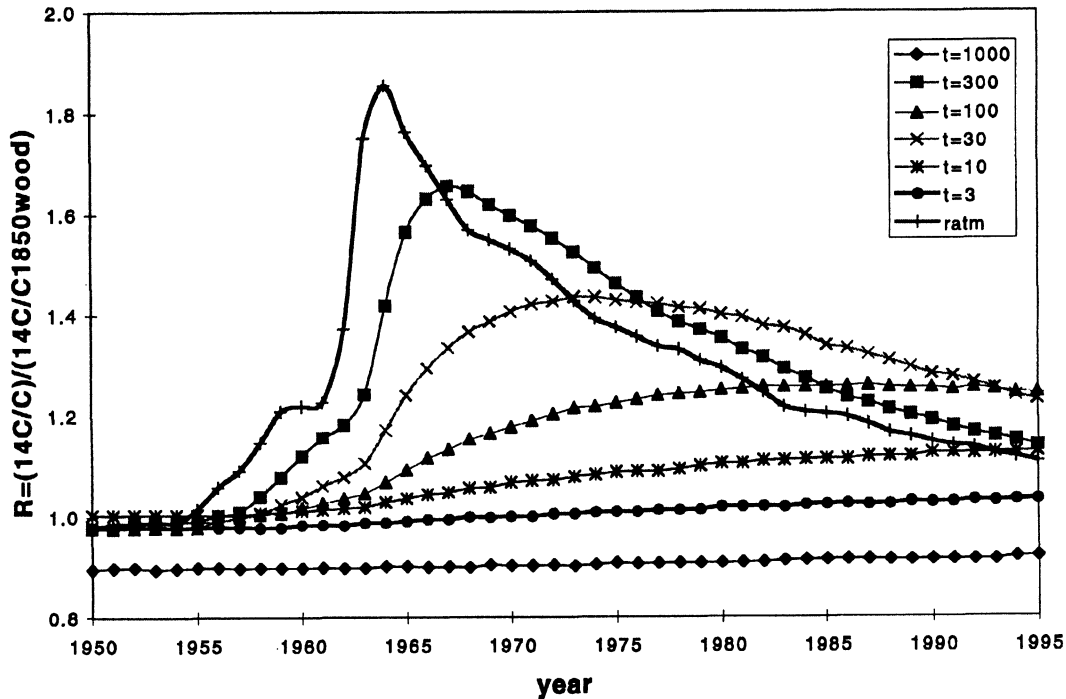


Fig. 6. Plot of  $^{14}\text{C}$  over time from 1950–1995 (projected). Atmospheric R shows greatest increase due to direct input. Reservoirs of various turnover times respond to atmospheric trends at different rates, shorter turnover reservoirs having more pronounced, faster responses. R values for different reservoirs had the maximum divergence during the 1970s and since then they appear to be converging, limiting the potential of bomb radiocarbon as a tracer of SOM. (Figure from Harrison, Broecker and Bonani 1993b).

Cultivation reduces SOM by a variety of mechanisms. First and most obvious is that cultivation removes plant debris from the soil, thereby restricting the natural cycling of material in the system. Tilling or plowing incorporates some or all plant residue respectively into the soil column, as well as providing air that enhances the rate of microbial decomposition. Tilling also serves to break up soil-aggregate complexes, which have an important role in stabilizing SOM. When soil aggregates are broken up, SOM becomes much more susceptible to degradation and subsequent loss (Martel and Paul 1974; Schimel *et al.* 1994).

Non-tillage farming systems have been shown to enhance crop production while maintaining SOM levels (Fenster and Peterson 1979; Young 1982). Since enhanced terrestrial carbon storage is an option for mitigating rising  $\text{CO}_2$  levels, it would be appropriate to further explore how cultivation practices can be used to facilitate such storage. Because it is the fast turnover pool that responds to atmospheric inputs on relatively short time scales, it is important that methods are developed that not only reduce its loss but possibly increase its proportion of the soil profile by taking advantage of  $\text{CO}_2$ -related growth enhancement in combination with fertilization.

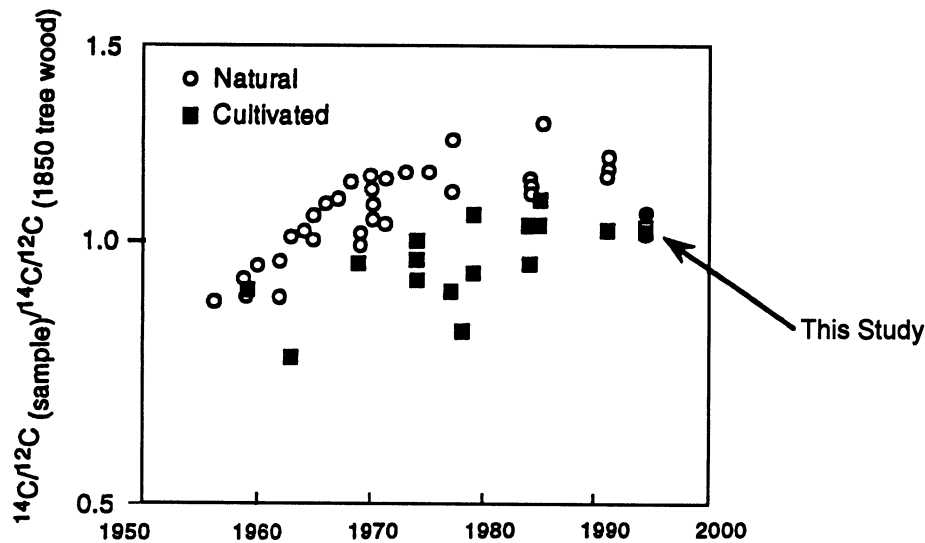


Fig. 7. Summary plot from Harrison *et al.* (1993a) showing the evolution of  $^{14}\text{C}/^{12}\text{C}$  ratios in topsoil from natural and agricultural plots (worldwide) from 1950 to present. The results of the Nebraska soils are shown for comparison.

## CONCLUSION

The TOC measurements show that the carbon content in all plots decreases rapidly with depth. Plowing and tilling processes serve to distribute organic material deeper in the soil column, as well as promote decay of detrital material contributing to the partial loss of fast-cycling SOM. These results are as expected, because plowing and tilling serve to physically homogenize the material in the soil column, mixing surface litter with deeper soil, as well as to promote removal of SOM.  $^{14}\text{C}$  surface data indicate that the cultivated plots lost a significant fraction of their fast-cycling surface organic matter. Results indicate that a non-tillage cultivation is advantageous in maintaining levels of fast cycling carbon in the soil profile.

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## COMPARISON OF FRACTIONATION METHODS FOR SOIL ORGANIC MATTER <sup>14</sup>C ANALYSIS

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**ABSTRACT.** <sup>14</sup>C measurements provide a useful test for determining the degree to which chemical and physical fractionation of soil organic matter (SOM) are successful in separating labile and refractory organic matter components. Results from AMS measurements of fractionated SOM made as part of several projects are summarized here, together with suggestions for standardization of fractionation procedures. Although no single fractionation method will unequivocally separate SOM into components cycling on annual, decadal and millennial time scales, a combination of physical (density separation or sieving) and chemical separation methods (combined acid and base hydrolysis) provides useful constraints for models of soil carbon dynamics in several soil types.

### INTRODUCTION

Models describing the dynamics of accumulation and turnover of organic carbon generally recognize components of soil organic matter (SOM) that turn over on annual (active), decadal (slow) and centennial to millennial (passive) time scales (Jenkinson and Raynor 1977; O'Brien and Stout 1978; Parton *et al.* 1987; Jenkinson, Adams and Wild 1991; Potter *et al.* 1993; Townsend, Vitousek and Trumbore 1995; Schimel *et al.* 1994). Although these concepts have proven useful in explaining the magnitude and timing of changes in SOM following a perturbation such as land clearing for cultivation (Parton *et al.* 1987; Cambardella and Elliott 1993; Davidson and Ackerman 1993; Schimel *et al.* 1994), no recognized method now exists for determining how to apportion SOM into compartments that turn over on different time scales. To be useful for dividing SOM into components for ecosystem modeling, a procedure must determine both the pool sizes and turnover rates.

Two methods are presently in use to partition SOM into active, slow and passive pools. The first models the observed increase of <sup>14</sup>C during the 30 yr since the end of atmospheric weapons testing, using additional constraints derived from knowledge of carbon inputs to the system (O'Brien and Stout 1978; Harkness, Harrison and Bacon 1986; Balesdent 1987; Scharpenseel *et al.* 1989; Trumbore, Vogel and Southon 1989; Vitorello *et al.* 1989; Jenkinson, Adams and Wild 1991; Harrison, Broecker and Bonani 1993; Trumbore 1993; Townsend, Vitousek and Trumbore 1995; Trumbore, Chadwick and Amundson 1996). The second relies on physical and/or chemical fractionation methods to separate SOM into pools that turn over on different time scales (Paul *et al.* 1964; Campbell *et al.* 1967; Scharpenseel, Ronzani and Pietig 1968; Martel and Paul 1974; Goh *et al.* 1976; Goh, Stout and Rafter 1977; Anderson and Paul 1984; Goh, Stout and O'Brien 1984; Scharpenseel *et al.* 1989; Trumbore, Vogel and Southon 1989; Trumbore, Bonani and Wölfli 1990; Scharpenseel and Becker-Heidmann 1992).

Radiocarbon measurements of pre-bomb SOM fractions are interpreted as indicative of their turnover rates, though this can cause serious overestimation of the turnover time in soils collected before 1963 (Balesdent 1987; Trumbore 1993; Trumbore and Druffel 1995). A test of the efficiency of any fractionation method at isolating a relatively homogeneous (in terms of turnover) organic matter pool is a comparison of the observed <sup>14</sup>C increase since atmospheric weapons testing (Goh, Stout and O'Brien 1984; Balesdent 1987; Trumbore, Vogel and Southon 1989; Trumbore 1993). These tests have shown that, although no fractionation method is completely successful, separation of organic matter by density, followed by hydrolysis in 6N HCl, does leave a residual collection of compounds, which, on average, turn over more slowly than the hydrolyzed portions of SOM (Campbell *et al.* 1967; Scharpenseel, Ronzani and Pietig 1968; Martel and Paul 1974; Goh *et al.* 1976;

Goh, Stout and Rafter 1977; Anderson and Paul 1984; Goh *et al.* 1984; Trumbore, Vogel and Southon 1989; Trumbore, Bonani and Wölfli 1990; Scharpenseel and Becker-Heidmann 1992; Trumbore 1993; Trumbore, Chadwick and Amundson 1996).

Here we compare measurements of  $^{14}\text{C}$  made of SOM fractionated using different physical and chemical separation methods. We analyzed both archived (pre-bomb) and contemporary soil samples. The implications of these data for soil carbon cycling are discussed in other publications (see Table 1 for references).

TABLE 1. Summary of Characteristics of Soils Analyzed in the Fractionation Study

Sample	Location	Year collected	Clay content	Soil order/horizon	Vegetation/climate	Parent material	Depth interval (cm)	Ref.*
BS-7	Paragominas, Brazil	1992	>85%	Oxisol/A	Tropical forest	Kaolinitic sediments	30–35	1
BS-9	Pará, Brazil	1992	>85%	Oxisol/B	Tropical forest	Kaolinitic sediments	100–105	1
H-13	Lapahoehoe, Hawaii	1992	ND	Andisol/A	Tropical pasture	Volcanic ash (15–25 ka)	0–10	2
H-15	Lapahoehoe, Hawaii	1992	ND	Andisol/B	Tropical pasture	Volcanic ash (15–25 ka)	40	2
NS-11	Shaver Lake, California	1992	7–8%	Ultisol/A	Dry temperate forest	Granodiorite	5–10	3
NS-13	Shaver Lake, California	1992	7–8%	Ultisol/B	Dry temperate forest	Granodiorite	20–40	3
OS-9	Shaver Lake, California	1959	7–8%	Ultisol/A	Dry temperate forest	Granodiorite	0–15	3
OS-10	Shaver Lake, California	1959	7–8%	Ultisol/B	Dry temperate forest	Granodiorite	15–58	3
A1-52	Michigan	1992	<2%	Ansposodosol/B	Moist temperate forest	Beach sand, 3 ka	52	4
B1-56	Michigan	1992	<2%	Ansposodosol/B	Moist temperate forest	Beach sand, 10 ka	56	4

\* References:

1. Trumbore *et al.* (1995)
2. Townsend, Vitousek and Trumbore (1995)
3. Trumbore, Chadwick and Amundson (1996)
4. Barrett and Schaetzl (1992)

## METHODS

### Summary of Fractionation Methods

All samples are sieved to remove components >2 mm. Chemical and density separation procedures have been described before (Trumbore, Vogel and Southon 1989; Trumbore 1993), and are summarized in Figure 1. Three fractionation techniques are used: separation by density, chemical extraction using acids and bases, and separation by size.

### Density Separation

The density separation extracts organic matter of low density (<1.6–2.0 g cm<sup>-3</sup>) by flotation in a heavy liquid. The low-density material consists of plant matter and charcoal (Sollins, Spycher and

Topik 1983; Spycher, Sollins and Rose 1983). Dense fractions contain mineral-associated organic matter, and some microbial cell debris (Sollins, Spycher and Topik 1983; Spycher, Sollins and Rose 1983). In general, the low-density material has higher  $^{14}\text{C}$  values and turns over more rapidly than much of the SOM. Charcoal may be isolated by dissolving other low-density material (except pollen) by treatment with strong acids and bases (Hammond *et al.* 1991; Gillespie *et al.* 1992a; Gillespie *et al.* 1992b).

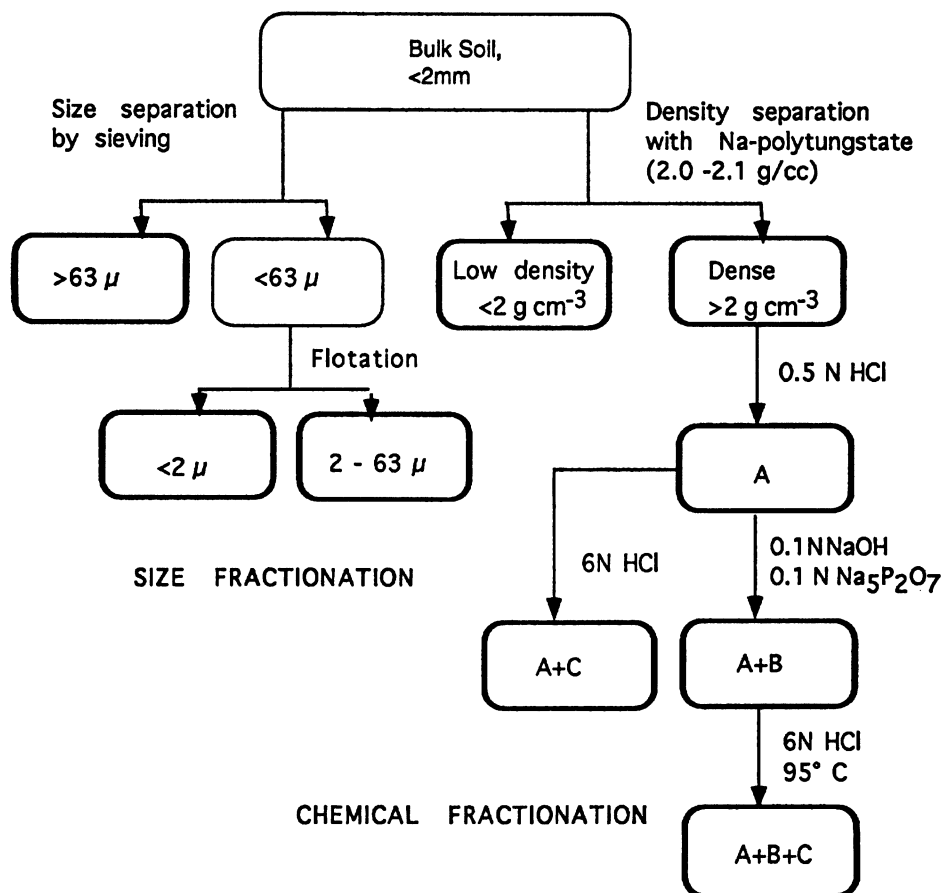


Fig. 1. Fractionation scheme used for samples in this study. See text for details of procedures and explanation of abbreviations.

### Chemical Fractionation

Operationally defined fractionation procedures for SOM often rely on extraction using acids and bases. More labile components are hypothesized to be soluble, leaving behind more refractory (and therefore more  $^{14}\text{C}$ -depleted) constituents. These chemical extraction procedures will also affect soil mineral content and can cause changes in SOM chemistry and structure. Extraction with Na-pyrophosphate ( $\text{Na}_4\text{P}_2\text{O}_7 \cdot 10\text{H}_2\text{O}$ ) and 6N HCl will dissolve sesquioxide minerals and remove organic matter complexed with iron and aluminum, and heating SOM in strong acid can cause condensation reactions. The data presented here compare the  $^{14}\text{C}$  content of residues after hydrolysis in acid (HCl)



of varying strength, and in base (NaOH-Na-pyrophosphate). Because the extraction of fresh plant material by these techniques leaves a residue (Ertel and Hedges 1984), we chose to perform acid and base hydrolysis on SOM that has been separated previously by density, and to extract only the denser portion of the soil. For organic-rich Andisol samples, we extracted the bulk soil (as nearly all had densities  $>2.1 \text{ g cm}^{-3}$ ). Soils developed on basic volcanic rock and containing minerals like allophane and imogolite should employ lower density liquids than we used for improved separation of mineral and organic constituents (Sollins, Spycher and Topik 1983; Spycher, Sollins and Rose 1983).

### Size Separation

Recent research has tied the dynamics of decadal-cycling SOM to different size fractions (Cambardella and Elliott 1993, 1994), which has led to speculation about the role of soil aggregates in limiting the decomposition of reactive SOM constituents by physically protecting organic matter from soil microbial activity (Oades 1993). In addition, the limited measurements of  $^{14}\text{C}$  in size-fractionated organic matter available from the literature (Anderson and Paul 1984) suggest a relation between the size of soil-associated organic material and stability of associated organic matter. Recently, Mayer (1994) has shown that organic matter content is related to surface area in many soils and sediments. If organic matter is protected by stabilization on clay surfaces, we might expect it to have lower  $^{14}\text{C}$  values than organic matter, which is more available for microbial consumption.

For the size separation, we used the  $<2\text{-mm}$  sieved samples, without prior density separation. We measured the  $^{14}\text{C}$  content of three size fractions:  $<2 \text{ mm}$  but  $>63 \mu$ ,  $<63 \mu$  but  $>2 \mu$ , and  $<2 \mu$ . The largest fraction was separated by dry sieving; the fractions  $>$  and  $<2 \mu$  were separated by settling from a suspension (Folk 1961). This procedure breaks apart soil aggregates.

### Carbon and $^{14}\text{C}$ Measurements

Percent carbon data were determined by  $\text{CO}_2$  evolution on *in-vacuo* combustion at  $900^\circ\text{C}$  with cupric oxide wire (Buchanan and Corcoran 1959).  $^{14}\text{C}$  analyses were made by accelerator mass spectrometry (AMS) at the Center for AMS, Lawrence Livermore Laboratory, Livermore, California (Southon *et al.* 1992). Graphite targets for AMS measurement were prepared from the purified  $\text{CO}_2$  using the sealed-tube, zinc reduction method modified from Vogel (1992).  $^{14}\text{C}$  data are reported as  $\Delta^{14}\text{C}$ , the per mil difference in  $^{14}\text{C}/^{12}\text{C}$  ratio between the sample and an absolute standard (oxalic acid decay-corrected to 1950) (Stuiver and Polach 1977; Donahue, Linick and Jull 1990).  $\delta^{13}\text{C}$  values used to correct for mass-dependent fractionation effects were assumed to be  $-25\text{‰}$  for all samples, as all sites had predominantly  $\text{C}_3$  vegetation.

### Nomenclature

We adopt a shorthand for describing the fractionation treatment experienced by a sample. The descriptors, denoted in Figure 1, are defined as follows:

BS = bulk soil. Due the expense of AMS measurements, we often rely on measurements of the constituent fractions to derive a bulk soil  $^{14}\text{C}$  number by mass balance.

LD = low-density fraction ( $< 2.0$  to  $2.1 \text{ g cm}^{-3}$ ).

HD = dense fraction ( $>2.0$  to  $2.1 \text{ g cm}^{-3}$ ).

All chemical fractionation procedures are performed on the HD fraction, and the data associated with them refers to the residue after a given chemical separation procedure. A refers to acid hydrolysis at room temperature in  $0.5 \text{ N HCl}$ ; B refers to extraction with  $0.1 \text{ N NaOH}$ – $0.1 \text{ N}$  sodium pyrophosphate; C refers to hydrolysis in  $6\text{N HCl}$  at  $95^\circ\text{C}$ . Often, we show data derived from the residue

of a combination of chemical fractionation procedures: for example, A+B+C refers to the residue after sequential hydrolysis in 0.5 N acid, 0.1 N NaOH and 6 N HCl. When performing these procedures, the residue after each extraction step is rinsed thoroughly in distilled H<sub>2</sub>O before moving on to the next procedure.

### Samples

We present here summaries of fractionation for several different kinds of soils. Table 1 summarizes data related to soil classification and site-specific characteristics, such as climate and parent material. References discussing the carbon cycling in these soils, or containing more information about the soils, as well as more detailed discussion of <sup>14</sup>C data with regard to soil carbon dynamics at each site, are also listed.

### RESULTS

Tables 2 and 3 show the carbon and <sup>14</sup>C content of the fractionated SOM. To better compare the effects of fractionation methods on the different soils, the results are also plotted in Figures 2 and 3.

#### Density Separation

Low-density components consist primarily of fine root hairs, charcoal (found in most LD fractions, but not quantified separately) and surface detritus mixed into the soil (in Oxisols BS-7, BS-9). Low-density material makes up a large portion of the total soil carbon in A horizons of more coarse-textured soils and the Andisol (which also had the highest total carbon content, Table 2). The <sup>14</sup>C content of LD material is close to atmospheric <sup>14</sup>CO<sub>2</sub> values at the time of collection in both pre- and post-bomb soils, indicating rapid turnover of this fraction.

In general, <sup>14</sup>C values of the dense fraction (mineral-associated) carbon decreased with depth in the soil, as has been observed (Scharpenseel *et al.* 1989). A notable exception is in the Spodosol Bs horizon (where sesquioxide accumulation is occurring), which shows increased <sup>14</sup>C values compared to the overlying eluviated horizon. Bomb <sup>14</sup>C is present in HD fraction for soils sampled in the 1990s, indicating the presence of at least some fast-cycling carbon associated with soil mineral phases.

#### Acid-Base Hydrolysis

In the heavier-textured soils (excluding the Shaver soil from the Sierra Nevada, California), most of the carbon present is associated with mineral surfaces (HD fraction). Hydrolysis of the HD fraction in relatively mild acids (0.5 N HCl) removes >30% of the mineral-associated carbon for all soils studied (see Fig. 2A). The removal is greatest in Spodic B horizons (the Michigan soils samples, B1-56 and A1-52), where >70% of the organic carbon is solubilized in 0.5 N HCl. The carbon remaining is depleted in <sup>14</sup>C compared to the starting dense fraction material to varying degrees (Fig. 3A). Least affected are the Andisol (Hawaii) samples; most affected are the spodic B horizons. Subsequent hydrolysis in base and strong acid removes more carbon and <sup>14</sup>C (Figs. 2B and 3A-C). The residue after A+B+C treatments is, in all but two cases, the most depleted in <sup>14</sup>C of any treatment. Figure 3 summarizes the difference in <sup>14</sup>C content of residual carbon after various treatments, compared to the starting dense material (1:1 line on figures). The A+B treatment (Fig. 3B) removes almost as much carbon and <sup>14</sup>C as the A+B+C treatment, while treatments using only acids (A+C; Fig. 3C) do not remove as much C and <sup>14</sup>C as treatments including a base hydrolysis step. The Andisols and Oxisols are affected by chemical fractionation less than coarser-textured soils. The difference between unfractionated dense material and the A+B or A+B+C residues is smaller in A horizons of soils than in B horizons. This is more noticeable in previously published profiles (Trumbore, Bonani and Wöflfi 1990).

TABLE 2. Results of Chemical Fractionation Procedures\*

Sample	C density $\text{gC m}^{-2} \text{cm}^{-1}$ (depth)		LD <2.0 g/cc		D (>2 g/cc)		A only		A + B		A + B + C		A + C	
	%C <sub>LD</sub>	$\Delta^{14}\text{C}$	%C <sub>g</sub>	$\Delta^{14}\text{C}$	%C <sub>d</sub>	$\Delta^{14}\text{C}$	%C <sub>d</sub>	$\Delta^{14}\text{C}$	%C <sub>d</sub>	$\Delta^{14}\text{C}$	%C <sub>d</sub>	$\Delta^{14}\text{C}$	%C <sub>d</sub>	$\Delta^{14}\text{C}$
BS-7	<5%	+170	1.0	-102.4	61.7	-195.0	32.3	-316.4	14.1	-302.5	37.4	-254.8		
BS-9	<1%	ND	0.4	-552.0	72.0	-615.0	49.9	-723.0	15.4	-852.6	18.6	-662.7		
H-13	ND	ND	11.0	4.0	46.5	-16.3	20.1	36.7	25.6	44.7	22.0	21.4		
H-15	ND	ND	4.2	-660.8	62.1	-705.9	12.6	-723.5	12.0	-758.9	13.8	-676.2		
NS-11	462	+109.3	1.5	92.6	12.2	4.9	8.2	-56.7	5.2	-86.5	7.0	-164.2		
NS-13	98	+169.5	0.4	32.7	40.9	-55.2	18.1	-153.7	11.4	-167.7	16.8	-115.1		
OS-9	383	-35.8	2.1	-36.5	13.0	-93.9	5.9	-167.2	4.0	-173.6	4.2	-91.5		
OS-10	92	-30.9	0.4	-123.0	39.4	-145.3	20.3	-249.7	13.6	-345.6	2.4	-193.4		
A1-52	ND	ND	0.7	-148.7	25.6	-298.6	7.3	-287.0	2.6	-324.7	8.8	-256.9		
B1-56	ND	ND	1.0	-123.7	12.4	-231.2	4.7	-316.8	3.8	-328.4	6.6	-309.3		

\*Sample identification given in Table 1. Carbon density (CD;  $\text{gC m}^{-2} \text{cm}^{-1}$ ) is calculated from bulk density and %C. ND = no data. The first fractionation procedure was separation by density into <2.0 g/cc (LD) and >2.0 g/cc (HD) fraction. %C<sub>LD</sub> denotes the percent of total carbon in the LD fraction. The percent of total carbon in HD fraction (not shown) is 100-%C<sub>LD</sub>. Hawaii (H-13 and H-15) samples were not separated by density. For these soils, the bulk data are listed in the LD column, since most of the soil had density <2.0 g/cc. The starting material for chemical fractionation procedures was the HD fraction, for which gravimetric percent carbon (%C<sub>p</sub>) data are shown.

Chemical procedures are as follows:

A = residue after extraction with 0.5 N HCl at room temperature for 12 h.

B = residue after extraction at room temperature for 24 h with 0.1 N NaOH-0.1 N Na<sub>4</sub>P<sub>2</sub>O<sub>7</sub> · 10H<sub>2</sub>O.

C = residue after extraction with 6 N HCl at 95°C for 12 h.

Combinations of chemical procedures are shown, e.g., results under the heading A+B are for the residual organic matter following treatment with first A, then B. %Cd is the percent of the carbon in the untreated HD fraction that remains after chemical treatment. 100% minus the %Cd gives the percent of total HD carbon extracted. <sup>14</sup>C data are reported as  $\Delta^{14}\text{C}$  (‰), assuming  $\delta^{13}\text{C}$  of -25‰ for all samples; vegetation was exclusively C<sub>3</sub> at all sites. The 1- $\sigma$  uncertainty of the <sup>14</sup>C measurements (based on repeated measurements of secondary standards) is  $\pm 10$  to 15‰.

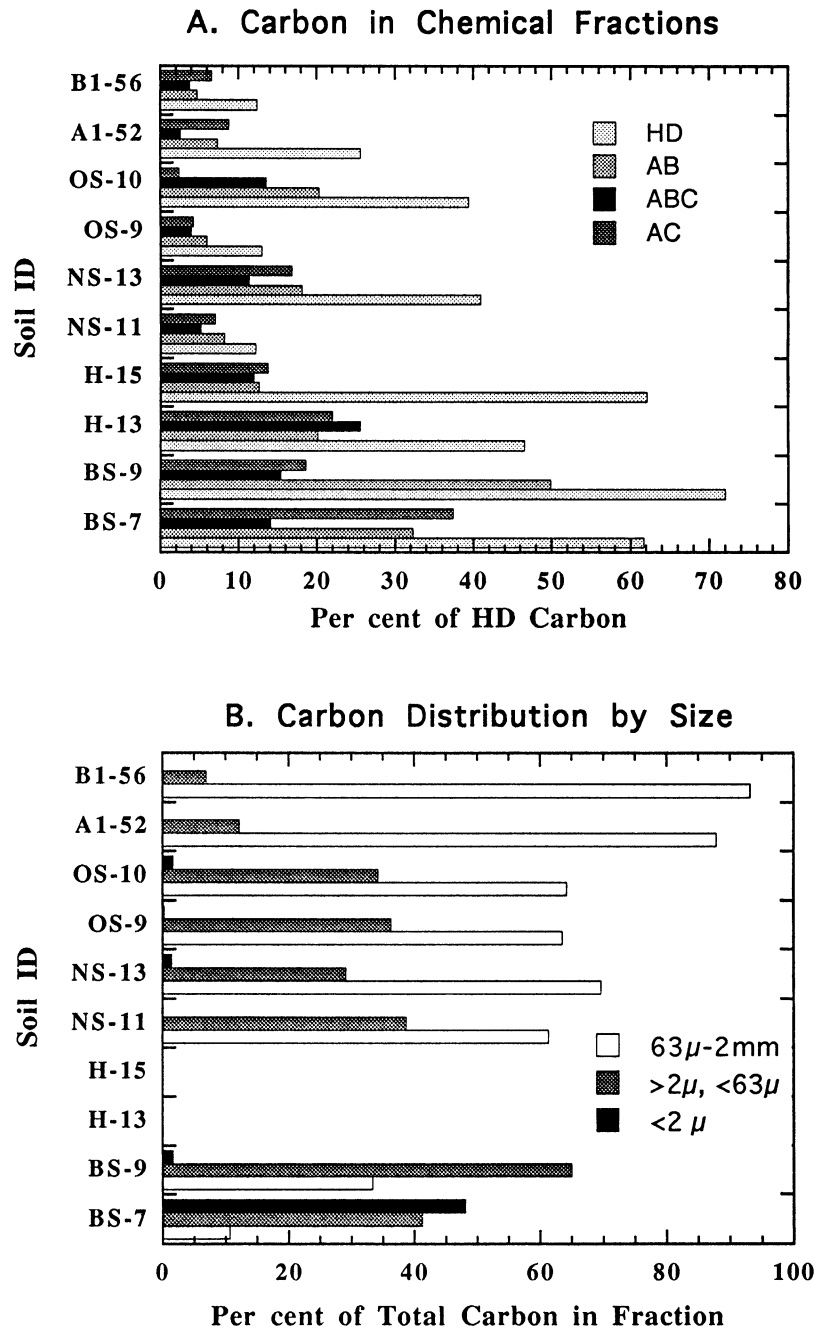


Fig. 2. Percent of total carbon in the residues left after acid and base hydrolysis treatments of the dense fraction (A) and after size fractionation of the bulk soil (B).

TABLE 3. Results of Size Fractionation Procedures\*

Sample	2 mm > S > 63 $\mu$			63 $\mu$ > S > 2 $\mu$			> 2 $\mu$		
	%C	%Ct	$\Delta^{14}\text{C}$	%C	%Ct	$\Delta^{14}\text{C}$	%C	%Ct	$\Delta^{14}\text{C}$
BS-7	3.9	10.6	7.6	1.5	41.3	-92.5	1.0	48.1	-107.4
BS-9	0.4	33.3	-465.8	0.4	65.0	-499.7	0.5	1.6	-368.6
NS-11	11.2	61.3	82.9	3.5	38.7	67.5	--	--	--
NS-13	0.8	69.5	11.0	1.7	29.1	41.7	4.5	1.4	73.8
OS-9	2.2	63.5	-54.4	4.7	36.3	-41.5	7.8	0.2	-55.1
OS-10	0.9	64.2	-108.1	1.8	34.2	-51.6	2.9	1.6	-271.3
A1-52	0.6	87.8	-128.8	12.6	12.2	-103.0	Very little clay		
B1-56	0.6	93.1	-113.3	15.2	6.9	-91.6	Very little clay		

\*%C is gravimetric carbon content, %Ct is the percent of total soil carbon in the size fraction and  $^{14}\text{C}$  data are reported as  $\Delta^{14}\text{C}$  (‰), assuming  $\delta^{13}\text{C}$  values of -25‰ for all samples.

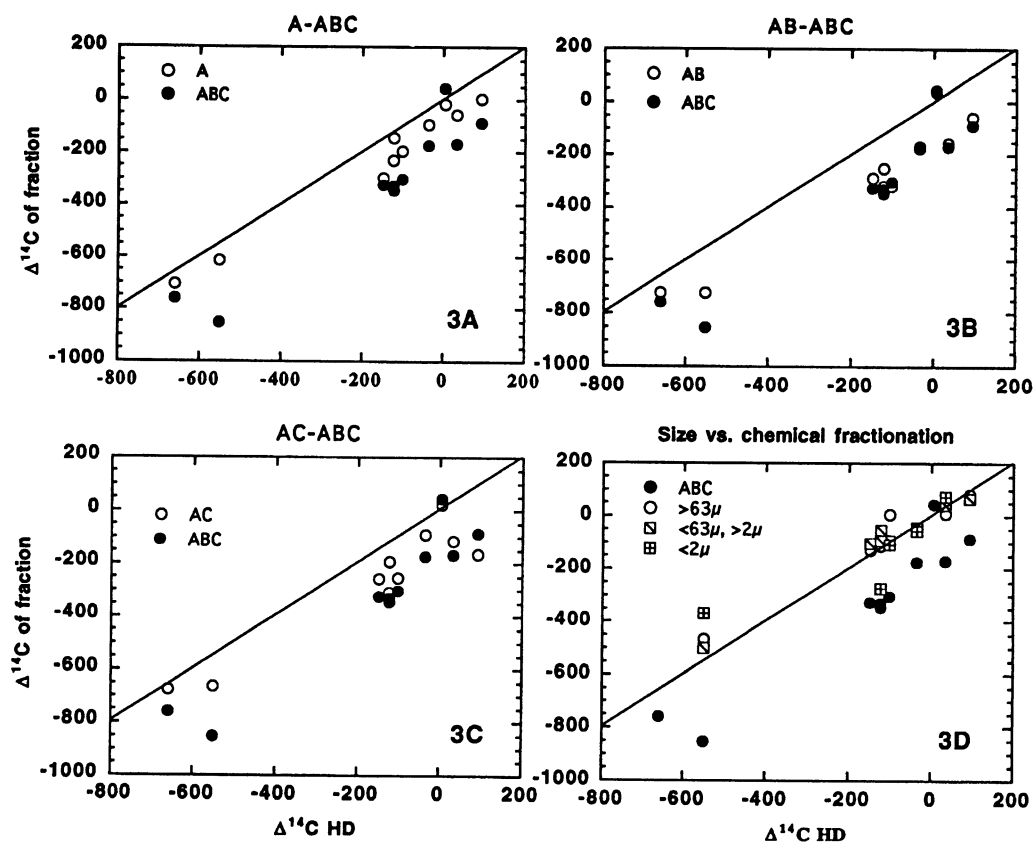


Fig. 3.  $\Delta^{14}\text{C}$  of the dense fraction (HD) compared to that measured in the residue after various chemical treatments. The 1:1 line (which signifies no difference in  $^{14}\text{C}$  between the residue and the untreated original material) and the  $\Delta^{14}\text{C}$  values for A+B+C treatments (normally the most  $^{14}\text{C}$ -depleted residues) are shown on each figure.

### Size Fractionation

The results of size fractionation on splits of the same soils in which density and chemical procedures were used are reported in Table 3. Most of the carbon in coarser-textured soils was associated with the  $>63 \mu$  fraction (Fig. 2B), whereas in more clay-rich, heavy-textured soils (Oxisols), most of the organic carbon is associated with the fine silt and clay fractions ( $<63 \mu$ ). Size separations were not performed on the Andisol (Hawaii) soils; these are dominated by fine-grained material. Size-fractionation of soils did not affect  $^{14}\text{C}$  values as much as chemical fractionation (Fig. 3D). We did not see any consistent tendency for clay-sized particles to have  $\Delta^{14}\text{C}$  values much greater or less than other size fractions, although clay fractions both enriched and depleted in  $^{14}\text{C}$  were observed.

### SUGGESTIONS FOR A STANDARD FRACTIONATION SCHEME

The usefulness of different fractionation methods depends in part on the type of soil, in particular, on soil texture (clay content). Separation by density will concentrate organic matter that turns over rapidly (decades and shorter time scales) in the low-density fraction. In coarse-textured soils with granitic parent material, such as the Shaver soil (Sierra Nevada), low-density organic matter can make up the majority of the total soil carbon in the A horizon. The remaining HD material shows significant  $^{14}\text{C}$  increases in A horizons due to incorporation of bomb  $^{14}\text{C}$ . Chemical treatment, especially hydrolysis in acid and base, removes relatively  $^{14}\text{C}$ -enriched components of the dense, mineral-associated organic matter. Chemical treatment is most effective in coarse-textured soils, in part because more of the total carbon is removed in the hydrolysis procedure. Chemical extraction with acids and bases did not change the  $^{14}\text{C}$  content of Andisol organic matter, even though up to 90–95% of the original carbon was removed.

Large differences in  $^{14}\text{C}$  content were not observed between different size fractions. Most of the carbon in coarse-textured soils is in the larger size fractions ( $>63 \mu$ ), and probably is mostly low-density vascular plant material, although  $^{14}\text{C}$  values indicate that this organic matter is diluted by other (mineral associated) constituents.

We suggest the most informative fractionation procedure to be used in separating SOM into faster and slower cycling components combines density and chemical separation procedures. Low-density organic material (except for charcoal) turns over on timescales of decades and less (Trumbore 1993; Trumbore, Chadwick and Amundson 1996). Extraction of dense material with acid and base will leave a residue that can provide a minimum estimate of the size and  $^{14}\text{C}$  content of a passive pool in  $^{14}\text{C}$  modeling. This fractionation will be most useful in soils of medium-to-coarse texture, with granitic parent material.

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## ESTIMATION OF SLOW- AND FAST-CYCLING SOIL ORGANIC CARBON POOLS FROM 6N HCl HYDROLYSIS

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**ABSTRACT.** Acid hydrolysis is used to fractionate the soil organic carbon pool into relatively slow- and fast-cycling compartments on soils from Arizona, the Great Plains states and Michigan collected for carbon isotope tracer studies related to soil carbon sequestration, for studies of shifts in C<sub>3</sub>/C<sub>4</sub> vegetation, and for “pre-bomb” soil-carbon inventories. Prior to hydrolysis, soil samples are first treated with cold 0.5–1N HCl to remove soil carbonates if necessary. Samples are then dispersed in a concentrated NaCl solution ( $\rho \approx 1.2 \text{ g cm}^{-3}$ ) and floated plant fragments are skimmed off the surface. After rinsing and drying, all remaining recognizable plant fragments are picked from the soil under 20 $\times$  magnification. Plant-free soils, and hot, 6N HCl acid-hydrolysis residue and hydrolyzate fractions are analyzed for carbon content,  $\delta^{13}\text{C}$  and  $^{14}\text{C}$  age, and the carbon distribution is verified within 1–2% by stable-carbon isotope mass balance. On average, the recalcitrant residue fraction is 1800 yr older and 2.6‰ more  $^{13}\text{C}$ -depleted than total soil organic carbon. A test of hydrolysis with fresh plant fragments produced as much as 71–76% in the acid-hydrolysis residue pool. Thus, if plant fragments are not largely removed prior to hydrolysis, the residue fraction may date much younger than it actually is.

### INTRODUCTION

An understanding of soil dynamics and carbon cycling in soils requires estimating the types and sizes of soil carbon pools as well as the mean residence times in each. For modeling purposes, the number of soil organic carbon pools is often simplified. For example, Parton *et al.* (1987) incorporated five organic carbon pools in their model including an “active” fraction of microbial products and rapidly cycling soil organic matter (SOM) (1–5 yr turnover time), a slowly cycling SOM pool (25 yr), a passive pool (200–1500 yr), a plant residue structural pool (1–5 yr) and a plant residue metabolic pool (0.1–1 yr). Schimel *et al.* (1993) modeled a rapid-cycling microbial matter pool with residence time of <1 to >4 yr, a “passive” carbon pool with residence time of 600 to >5000 yr, and detritus and “slow” organic carbon pools whose residence times are intermediate between the fast- and slow-cycling pools. Schimel *et al.* (1993) found the organic carbon residence time of each compartment to be correlated to the temperature, and the distribution of carbon among the compartments to be influenced by texture.

Researchers have attempted to define such organic carbon pools by means of physical and chemical methods. Anderson and Paul (1984) determined  $^{14}\text{C}$  activity on coarse and fine, silt and clay fractions, with the fine silt and coarse clay fractions containing the oldest carbon and the fine clay fraction somewhat younger, suggesting the fine clay is a site of short- to medium-term protection of labile humic compounds. Cambardella and Elliott (1992) used dispersion and collection on a 53  $\mu\text{m}$  sieve to isolate a “particulate organic matter” (POM) fraction in grassland soils that slowly cycled over a time frame of several decades. This cycling was much faster than the mineral-associated organic matter fraction, and Cambardella and Elliott (1992) suggested this fraction constitutes most SOM lost initially from cultivation of grassland soils.

Campbell *et al.* (1967) found distinctly different ages among chemical fractions with non-hydrolyzable, total humic acid and humin fractions being older than bulk unfractionated soil organic carbon, and fulvic acid generally being younger than the bulk value. Nissenbaum and Schallinger (1974) also isolated “traditional” organic fractions within the NaOH soluble portion of soil organic carbon in a variety of soil types by means of acetone and ethanol extraction. These included humic acid, ful-

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vic acid and their components, and they found humic acid more  $^{13}\text{C}$ -depleted than the fulvic acid (ca. 0.6–1.2‰). Martel and Paul (1974) opted for the products of acid hydrolysis rather than the traditional fractions because earlier incubation experimental work with labeled  $^{14}\text{C}$  showed only small differences of label concentration in the traditional fractions, but a much greater range of labeled  $^{14}\text{C}$  activities in the hydrolyzed fractions. They found that HCl hydrolysis effectively dissolves the young constituent carbon compounds, leaving older resistant molecules in the residue, and was thus a useful technique to explore soil organic carbon stability in nature. Scharpenseel and Schiffmann (1977) and Stout *et al.* (1981) also promoted acid hydrolysis as a standard procedure for soil fractionation. Trumbore (1993) combined density separation with acid hydrolysis, and found the latter to be fairly effective in isolating old carbon from certain soils (temperate), but with some “bomb” carbon making its way into the residue fraction since 1959. However, the acid hydrolysis procedure was totally ineffective in isolating old carbon from tropical soils. Trumbore (1993) suggested that acid hydrolysis may solubilize some refractory carbon, or permit exchange of young for old carbon on functional groups of the refractory organic matter. Scharpenseel (1977) had previously determined that acid hydrolysis residues may contain cellulose, but that increasingly greater ages were obtained with each of 8 successive 6N HCl hydrolyses.

Since 1991, we have been routinely using acid hydrolysis in our  $^{14}\text{C}$  and  $\delta^{13}\text{C}$  analysis to differentiate labile and recalcitrant soil organic carbon fractions. We present here a summary of the characteristics of these fractions as applied to soils from Arizona, Michigan and the Great Plains states. We also consider the question of hydrolysis of fresh plant matter as it relates to partitioning between the hydrolyzate and residue fractions of acid hydrolysis.

## METHODS

Surface soil samples were collected from all sites at depths of 0–10, 0–15 or 0–30 cm. For several sites, samples were variously taken at greater depths in the profile (from 10–20 to up to 90–120 cm). The Arizona (Entisol) soil samples from Maricopa County, Arizona, were collected in conjunction with the “Free-Air Carbon Dioxide Enrichment” (FACE) experiment with cotton in 1991 from control plots that received ambient air  $\text{CO}_2$ , and from enriched plots that received 550 ppm  $\text{CO}_2$  (Leavitt *et al.* 1994). Great Plains (dominantly Mollisol) soils were collected in Kansas, Nebraska, Colorado, North Dakota and Texas, and included both cultivated and uncultivated soils as well as some pre-“bomb” samples. For the Great Plains soils, exact locations, ages, variations of  $^{14}\text{C}$  and  $\delta^{13}\text{C}$  with depth and implications of these results to the vegetational history of the area and to modern processes will be reported in detail elsewhere (Paul *et al.* ms.; Follett *et al.* ms.). The Michigan (Alfisol) soils were collected at the Kellogg Long-Term Ecological Research (LTER) site near Kalamazoo. The grain size and soil taxonomic classification are detailed in Paul *et al.* (ms.) with soils ranging in clay content from ca. 11 to 34%, whereas organic carbon contents are in the range of ca. 0.4 to 3% (Follett *et al.* ms.).

Soil samples were first sieved at 1 mm to remove large plant fragments and pebbles, and a subsample of 50–100 g was acidified with 0.5–1N HCl to remove the carbonate fraction found in most of the samples. The samples were then immersed in a concentrated NaCl solution ( $\rho \approx 1.2 \text{ g cm}^{-3}$ ) and stirred. Following settling of the heavier solids, floating plant fragments were skimmed from the surface. This was repeated until the samples no longer had a component that floated, after which the soils were rinsed free of salt, dried and pulverized with a mortar and pestle.

Between 1 and 5 g of these processed soil samples were then further picked free of recognizable plant and root fragments under a binocular microscope at 20 $\times$ . Organic carbon content and  $\delta^{13}\text{C}$

were determined after combustion of 0.10–0.25 g subsamples of these soil samples in quartz tubes at 900°C in the presence of copper oxide, silver foil and copper turnings (Boutton 1991). Carbon yield was determined manometrically from the CO<sub>2</sub> combustion product, and CO<sub>2</sub> was analyzed primarily with a VG Micromass 602C isotope ratio mass spectrometer to determine δ<sup>13</sup>C (some analyses were performed with a Finnigan MAT Delta-S mass spectrometer). Isotope ratio mass spectrometer precision is *ca.* ± 0.05‰ for repeated analysis of the same CO<sub>2</sub> gas. Seven separate combustions and analyses of subsamples of one FACE soil sample produced a mean δ<sup>13</sup>C of –23.40‰ with respect to PDB (*s*=0.25‰), and a manometrically determined, mean organic carbon content of 0.60% (*s*=0.03%). Seven more recent runs with another soil standard gave a δ<sup>13</sup>C precision (± 1σ) of 0.33‰ and a carbon content precision (± 1σ) of ±0.06%. Acid hydrolysis was performed on most of the soils that were free of carbonates and plant fragments. One to 3 g of soil were placed into a 500-ml round-bottom flask with *ca.* 50 ml of 6N HCl. With a water-cooled condenser installed above the flask, the mixture was heated to boiling with an electric heating mantle for *ca.* 18 h. The soluble (hydrolyzate or “supernatant”) fraction was recovered following filtering and heating to evaporate the liquid. The resistant (“residue”) fraction was obtained by rinsing the material collected on the filter with deionized water and then drying. For samples for which δ<sup>13</sup>C was measured on both the acid-hydrolysis residue and hydrolyzate fractions, an isotope mass balance was calculated with the carbon yields to verify the procedure. On average, the calculated mass-balance total carbon δ<sup>13</sup>C was within 1–2% of the measured δ<sup>13</sup>C (mean difference between mass-balance and measured δ<sup>13</sup>C was –0.31‰, *s*=0.91‰, *n*=50). The results reported herein are all processed and analyzed in the above manner at the Laboratory of Tree-Ring Research/The University of Arizona, but there is a small bias (1–2‰) relative to measurements of the same standards at USDA-ARS/Ft. Collins and the Department of Crop and Soil Sciences/Michigan State University. We are in the process of resolving the differences by systematic examination of the procedures.

Processed soils and hydrolysis soil fractions were also combusted to CO<sub>2</sub> by the above method for tandem accelerator mass spectrometer (TAMS) <sup>14</sup>C analysis at the NSF-Arizona Accelerator Facility. The CO<sub>2</sub> samples were reduced to CO on hot zinc, and then to graphite on hot iron (Slota *et al.* 1987). Accelerator measurements of <sup>14</sup>C activity of the graphite were calculated as percent modern carbon (pMC) with a δ<sup>13</sup>C correction for fractionation.

The hydrolysis test of plant fragments employed two samples of FACE cotton field stubble collected 26 April 1992, one from an enriched CO<sub>2</sub> plot and the other from its paired control plot. The samples were ground to 20 mesh and then hydrolyzed with boiling 6N HCl for 18 h. Hydrolyzate and residue were separated by filtering, rinsing the residue and heating the hydrolyzate to dryness. Carbon distribution was calculated from the recovered dry masses and manometrically determined carbon contents.

## RESULTS AND DISCUSSION

We analyzed 65 soil samples: 2 from Michigan, 12 from Arizona and 51 from the Great Plains. Within this total, samples from different depths of the same profile are considered as separate samples. Of this set of samples, all but four had a <sup>14</sup>C measurement on at least one soil fraction. The 37 surface soil samples represent depths ranging from 0–10 cm to 0–30 cm, depending on the site and requirements of other companion experiments for soil sampling. Figure 1A shows the distribution of <sup>14</sup>C ages among the total carbon and acid-hydrolysis residue fraction of the surface soils. The <sup>14</sup>C activity of the total organic carbon (after floating and picking plant fragments) was “modern” (“post-bomb”) for eight of the samples, the highest being 118.73 ± 0.66 pMC (possible tracer contamination). Three others, including one subsurface soil, had activities >500 pMC, and were clearly

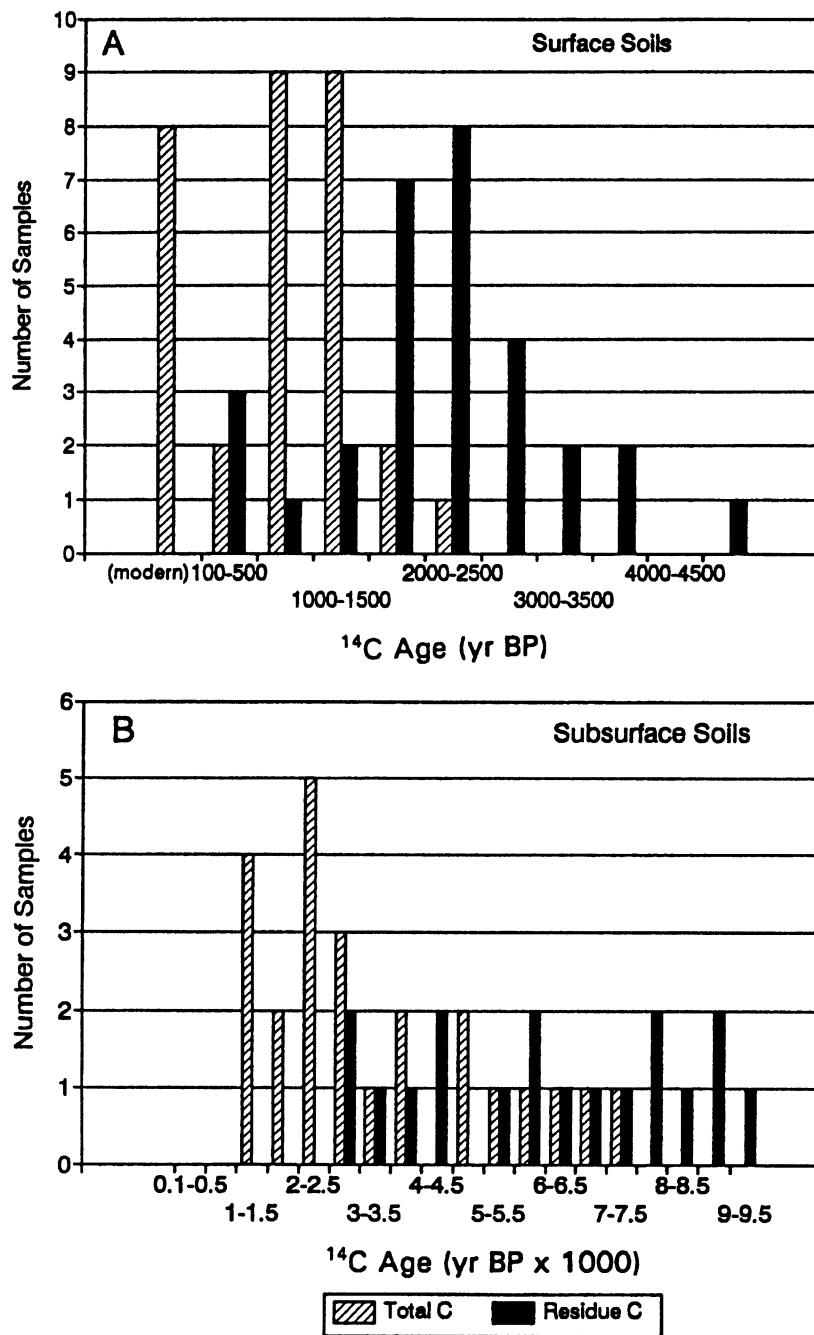


Fig. 1. Distribution of <sup>14</sup>C ages among soil organic carbon fractions of surface (A) and subsurface (B) soils. ▨ = total carbon; ■ = acid-hydrolysis residue.

contaminated with tracer  $^{14}\text{C}$ . This contamination, however, was restricted to 4 soils, and was not found in 40 or more subsequent samples. We thus believe the tracer contamination did not derive from The University of Arizona laboratories. The lowest  $^{14}\text{C}$  activity of surface-soil total organic carbon was  $77.22 \pm 0.49$  pMC ( $2075 \pm 50$  BP). In all cases, the  $^{14}\text{C}$  activity of the acid-hydrolysis residue fraction was older than the total carbon, with activity ranging from  $97.90 \pm 0.75$  pMC ( $170 \pm 60$  BP) to  $54.33 \pm 0.40$  pMC ( $4900 \pm 60$ ). The  $^{14}\text{C}$  activities of the acid-hydrolysis hydrolyzate fraction were measured on only six surface soils, and they ranged from  $107.7$  to  $93.3$  pMC (modern to  $340 \pm 60$  BP).

For the subsurface soils (Fig. 1B),  $^{14}\text{C}$  activity of the total organic carbon ranged from  $85.99 \pm 0.57$  pMC ( $1215 \pm 55$  BP) to  $39.48 \pm 0.33$  ( $7465 \pm 65$ ). The  $^{14}\text{C}$  activity of the acid-hydrolysis residue fraction of these soils ranged from  $70.15 \pm 0.54$  pMC ( $2850 \pm 60$  BP) to  $30.75 \pm 0.49$  ( $9475 \pm 125$ ). Table 1 summarizes the average differences in  $^{14}\text{C}$  between pairs of total organic carbon and the acid-residue fraction carbon. For all soils, the average activity difference is  $16.0$  pMC and the average age difference is  $1804$  yr. The average differences in  $^{14}\text{C}$  activity for surface and subsurface soils calculated separately are  $16.5$  and  $15.4$  pMC, respectively. However, the average age difference between these fractions for surface and subsurface soils is  $1485$  and  $2282$  yr, respectively. The greater age difference for the subsurface relative to surface soils, despite their similarity of activity difference, is simply a function of their lower overall  $^{14}\text{C}$  activity combined with the exponential nature of the  $^{14}\text{C}$  dating equation.

TABLE 1. Summary of Paired Differences in  $\delta^{13}\text{C}$  and  $^{14}\text{C}$  Activity of Soil Organic Carbon Fractions\*

Fraction	Surface soils	Subsurface soils	All soils
$\delta^{13}\text{C}_{\text{PDB}}\text{‰}$ (total-residue)	$1.77 \pm 0.91$ (30)	$4.09 \pm 2.05$ (17)	$2.61 \pm 1.80$ (47)
$\delta^{13}\text{C}_{\text{PDB}}\text{‰}$ (total-hydrolyzate)	$2.14 \pm 1.16$ (29)	$-1.53 \pm 0.93$ (15)	$-1.93 \pm 1.12$ (44)
$\delta^{13}\text{C}_{\text{PDB}}\text{‰}$ (residue-hydrolyzate)	$-3.89 \pm 1.80$ (29)	$-5.76 \pm 1.78$ (15)	$-4.53 \pm 1.99$ (44)
$^{14}\text{C}$ activity pMC (total-residue)	$16.5 \pm 9.3$ (27)	$15.4 \pm 7.7$ (18)	$16.0 \pm 8.6$ (45)
$^{14}\text{C}$ age yr BP (total-residue)	$-1485 \pm 877$ (27)	$-2282 \pm 1191$ (18)	$-1804 \pm 1077$ (45)

\*Values represent the mean  $\pm 1$  standard deviation (number of cases in parenthesis).

For  $\delta^{13}\text{C}$ , with only three exceptions in all the soils, the acid-hydrolysis residue was the most  $^{13}\text{C}$ -depleted, the hydrolyzate was the most  $^{13}\text{C}$ -enriched. On average, the  $\delta^{13}\text{C}$  of all total organic carbon, acid-hydrolysis residue and hydrolyzate fractions was  $-19.9$ ,  $-22.5$  and  $-18.2\text{‰}$ , respectively. However, the within-fraction variability is quite high (e.g.,  $\delta^{13}\text{C}$  of total carbon varied from  $-15.2$  to  $-27.0\text{‰}$ ) because of the variety of soil types and developmental histories; standard deviations are  $2.5$  ( $n=64$ ),  $2.2$  ( $n=47$ ) and  $2.8\text{‰}$  ( $n=44$ ), respectively. When the surface and subsurface soils are considered separately (Fig. 2), the three fractions are  $-20.7 \pm 2.1$  ( $n=37$ ),  $-22.5 \pm 2.0$  ( $n=30$ ) and  $-18.7 \pm 2.9\text{‰}$  ( $n=29$ ), and  $-18.7 \pm 2.7$  ( $n=27$ ),  $-22.5 \pm 2.6$  ( $n=17$ ) and  $-17.3 \pm 2.2\text{‰}$  ( $n=15$ ), respectively. The similarity of acid-hydrolysis residue and hydrolyzate  $\delta^{13}\text{C}$  values, but the difference (ca.  $2\text{‰}$ ) in total organic carbon  $\delta^{13}\text{C}$  between these depths, is a consequence of the large difference in average distribution of carbon at these two depths. For the surface soils,  $55.5 \pm 8.6\%$  of the carbon is in the residue and  $45.5 \pm 8.6\%$  ( $n=30$ ) is in the hydrolyzate, whereas for the subsurface soils, there is an average of  $44.3 \pm 9.5\%$  in the residue and  $55.7 \pm 9.5\%$  ( $n=18$ ) in the hydrolyzate. Given the greater age of the subsurface soils, these relations may reflect a shift in isotopic values during the soil-forming process, by which soils may become  $1\text{--}3\text{‰}$  more  $^{13}\text{C}$ -enriched by microbial decomposition (Dzurec *et al.* 1985; Bertram 1985; Becker-Heidmann and Scharpenseel 1986). However, because the fraction of carbon from acid-hydrolysis residue is on average less than that from the

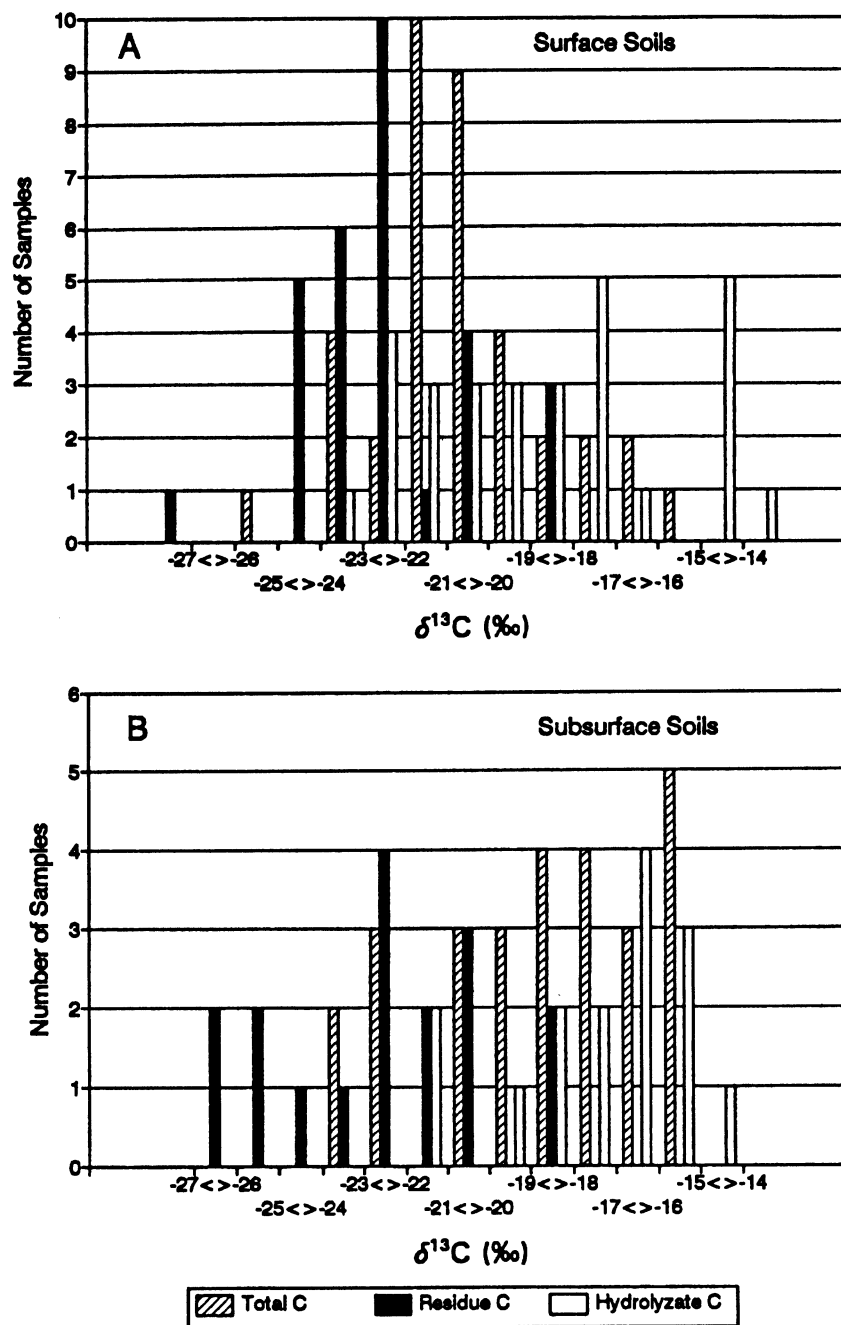


Fig. 2. Distribution of  $\delta^{13}\text{C}$  among soil organic carbon fractions of surface (A) and subsurface (B) soils. Symbols as in Fig. 1; □ = acid-hydrolysis hydrolyzate.

hydrolyzate for the subsurface soils, there is an implication that either an easily degradable form of carbon is being leached down into the subsurface, or a substantial amount of old carbon is converted into a form which may be recalcitrant but not with respect to 6N HCl. If the latter were true,  $^{14}\text{C}$  activity of the subsurface hydrolyzate fraction should be older than the hydrolyzate of the surface soils. Although we have no direct  $^{14}\text{C}$  measurements of the hydrolyzate fraction of the subsurface soils, we infer from isotopic balance of the total carbon and residue  $^{14}\text{C}$  that the hydrolyzate of the subsurface soils is indeed older than the surface hydrolyzate fraction by *ca.* 3500 yr, on average.

A more representative parameter than the average  $\delta^{13}\text{C}$  of fractions is the average isotopic difference between these fractions summarized in Table 1. The greatest  $\delta^{13}\text{C}$  difference is indeed between the acid-hydrolysis residue and the hydrolyzate ( $4.5 \pm 2.0\text{‰}$ ) for all soils, but the difference in subsurface soils is *ca.* 2‰ greater than in surface soils. Eight of the surface soils identified as virgin, uncultivated, had average  $\delta^{13}\text{C}$  differences between fractions that were within 0.4‰ of each of these overall average values. Thus, the consequences of cultivation do not seem to significantly alter this parameter.

Comparison of  $^{14}\text{C}$  ages and  $\delta^{13}\text{C}$  with soil organic carbon content yielded no significant relationships. However, the  $\delta^{13}\text{C}$  of the total organic soil carbon, the residue fraction and the supernatant fraction were each inversely correlated to soil % clay content (significance  $P < 0.01$ ). This may reflect a chance selection of the more clay-rich soils from sites associated with  $\text{C}_3$ -type vegetation ( $^{13}\text{C}$ -depleted relative to  $\text{C}_4$ -type vegetation). There was some evidence that the  $\delta^{13}\text{C}$  difference between organic fractions was smallest for soils with highest clay content, perhaps because organic-microbial-clay interactions somehow promote greater stable-carbon isotope homogeneity of the organic fractions.

The tracer- $^{14}\text{C}$ -contaminated soils represent an interesting demonstration with respect to the acid hydrolysis methods. If, indeed, the contamination did not occur at the University of Arizona but from field experimentation or from a laboratory or storage environment where dust associated with  $^{14}\text{C}$  tracers might occur, we could verify the effectiveness of acid hydrolysis in removing this strong signal in whatever form it occurs. Two of the four surface soils that had  $^{14}\text{C}$  activities in excess of 300 pMC were acid-hydrolyzed. The  $^{14}\text{C}$  ages of residues were  $2900 \pm 50$  and  $2300 \pm 50$  BP. These are among the eight oldest residues (out of 29  $^{14}\text{C}$  measurements) from the surface soils. Unless these surface soils are really much older, the elevated ages of the residue suggest the tracer was in a soluble form that was effectively removed in the processing. Confirmation could be made by analyzing the  $^{14}\text{C}$  content of the hydrolyzate fraction, but this has yet to be done. The form of this tracer may be very soluble in order for it not to be retained in the residue fraction.

The 6N HCl hydrolysis of the cotton plants resulted in 74% of the plant carbon ending up in the residue fraction (76 and 71% for the Arizona FACE experiment  $\text{CO}_2$ -enriched and control samples, respectively). This suggests that the acid hydrolysis itself could be enhancing by a substantial amount the influence of recent carbon from plant residues on the residue pool. Numerous techniques have been used to separate the oldest soil carbon from recently added substrates, including humic-fulvic fractionations, acid hydrolysis, separation of soil into various particle sizes and fractionation of intact soil aggregates. No one technique can be expected to completely separate the fractions on an age basis because of differences in chemical recalcitrance, and the fact that much of the soil carbon is derived from microbial products that are protected from decomposition by physical as well as chemical stabilization (Paul and Clark 1989). The hydrolysis in 6N HCl has proven the simplest and most reproducible method for separating young from old carbon while giving meaningful separations of  $^{13}\text{C}$  and  $^{14}\text{C}$  in both field and laboratory experiments (Martel and Paul 1974). The primary



limitation of hydrolysis of plant residue has been that lignin will remain in the non-hydrolyzable pool even though it is obviously not old material, and this is confirmed in our hydrolysis of cotton fragments. However, this effect may be minimized in our methods because all recognizable plant fragments were floated and picked from the soils prior to hydrolysis. Results from soils of the FACE project seem to support this contention: acid hydrolysis of soils from CO<sub>2</sub>-enriched plots, where <sup>14</sup>C-depleted, newly added plant residue (depleted as a consequence of <sup>14</sup>C-“dead” CO<sub>2</sub> used to enrich the plots to 550 ppm, ca. 200 ppm above ambient) would be expected to produce greater <sup>14</sup>C ages than plant matter of the control plots, did not show <sup>14</sup>C differences relative to the control plots (Leavitt *et al.* 1994).

## CONCLUSION

Overall, our methods seem to be effective in separating younger, more easily decomposable carbon compounds from older, more recalcitrant carbon compounds. The ages of the acid-hydrolysis residue in all cases are older by an average of 1800 yr relative to the total carbon pool, and the procedure is therefore isolating a relatively recalcitrant pool. This old residue fraction is ca. 2.6‰ <sup>13</sup>C-depleted relative to total organic soil carbon, perhaps related to microbial effects. The floating and picking are effective in removing young carbon from the soil samples, without which substantial amounts of “young” carbon might make its way into the acid-hydrolysis residue fraction, as evidenced in the hydrolysis experiment with recent crop stubble. The dried masses and the isotopic composition of the floated and picked materials can also be determined to better define the pool of plant residue structural material.

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## **<sup>14</sup>C DATING AND SOIL ORGANIC MATTER DYNAMICS IN ARCTIC AND SUBARCTIC ECOSYSTEMS**

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**ABSTRACT.** The carbon content, pH and <sup>14</sup>C concentration of humic acids were determined for three soil series of Arctic and Subarctic ecosystems. The measured <sup>14</sup>C ages were interpreted in the light of an equilibrium model of humus formation and of mineralization processes in recent soils, and the coefficient of renovation,  $K_r$ , was calculated for humic acids. The comparison of  $K_r$  for series formed under different climatic conditions suggested that global warming could accelerate decomposition of soil organic matter and possibly increase productivity of ecosystems of the Arctic region.

### **INTRODUCTION**

The recent increased interest in the study of soil organic matter (SOM) dynamics in Arctic and Subarctic ecosystems is due mainly to environmental and global change issues. Radiocarbon analysis is the most important method used to investigate the rates of the exchange/cycling carbon processes. Yet these processes, in the soils of arctic regions, have received very little attention.

Thirteen tundra and north taiga soils, representing different climate conditions, vegetation types and geomorphic surfaces, were sampled according to their genetic horizons. This research was conducted as part of the Russian project, "Global Change of the Environment and Climate". I propose to define the renovation rate of organic carbon in the different fractions of the north ecosystem soils.

Determination of rates and coefficients of incorporation of fresh organic residue into humous substances is one of the principal tasks in research of soil biochemical processes. No less important an indicator of humus balance in soils is the coefficient of renovation of humus,  $K_r$ , which can be determined by measuring the natural concentration of <sup>14</sup>C in SOM and its fractions. This method is applied to SOM originally present in the soil and the resultant coefficient  $K_r$  reflects both biological mineralization and humus dissipation due to other processes such as erosional loss and leaching with solid solutions.

### **SITE DESCRIPTIONS**

#### **Arctic Soil, Svalbard Series, Norway (Sampled by A. E. Cherkinsky)**

1. Peaty-gleyic arctic soil (gelic Gleysol) (77°35'N, 20°56'E), Edgeya Island, Ekralhamna Cape marine terrace, elevation *ca.* 10 m, permafrost table, 31 cm. The soil consists of a mixture of weakly decomposed peat, mor humus, almost unchangeable sandy loam and rock fragments. The surface has a full cover of mosses with polar willow, bog saxifrage and arctic bell heather also present.
2. Rendzina raw humic arctic (calcic Arenosol) (77°27'N, 21°01'E) Edgeya Island marine terrace, elevation *ca.* 30 m, permafrost table, 75 cm. The soil consists of sand and shell fragments with mor humus. There is only sparse vegetation on this surface (*e.g.*, lichens, saxifrages, arctic bell heather, polar poppy). Near this profile the fossil rib of a whale was found which permitted the dating of the terrace (see Table 1).
3. Peat arctic soil (gelic Histosol). Profile is 200 m westward of previous soil in microdepression. Permafrost table, 26 cm. The full 30 cm of the profile consists of weakly decomposed peat; it is covered by mosses with species of cotton grass and polar willow.

4. Rendzina humic arctic (Rendzina) (78°42'N, 16°30'E), West Spitsbergen, marine terrace, elevation *ca.* 25 m, permafrost table, 49 cm. The soil consists of sandy loam, shell fragments and humus of mor or moder types. The surface is completely covered by mosses with polar willow, saxifrages and polar poppy also present.
5. Dry peaty arctic soil (Arenosol) (79°32'N, 13°21'E), small volcanic cones and hot springs at Bockfjorden in northern Spitsbergen, elevation *ca.* 40 m, permafrost table, 65 cm. The soil consists of peat, mor humus and a mixture of basal till (sandy loam) with basaltic fragments. The surface is covered by a turf of mosses with mountain avens, rock sedge, saxifrages and polar poppy also present.
6. Arctic podbur (Arenosol) (78°20'N, 17°06'E) (sampled by V. O. Targulian and A. V. Kulikov). Coloradofjella plateau, elevation 400 m, Central Spitsbergen, permafrost table, 24 cm. The soil consists of loamy sand, rock fragments, mor humus and scarce vegetation (lichens, mosses and saxifrages).

**South Tundra Soils, Mezen Series, Russia (66°25'N, 42°34'E) (Sampled by S. V. Goryachkin and A. E. Cherkinsky)**

1. Fe-humic podzol (Haplic Podzol) is on the top of a small moraine hill. The soil consists of loamy sand, raw humus (in upper horizons) and illuvial humus (in deeper horizons). The surface has sparse vegetation, consisting of mosses, lichens and graminaceous plants.
2. Humic Podzol (Humic Podzol) is at the foot of the same hill. The soil is the same structure, and differs only by more humus content in the illuvial horizon. The surface has more mosses with no lichens; dwarf birch appears.
3. Peat gleyic soil (Dystric Gleysol) is at a depression 500 m north of Fe-humic Podzol. The top 39 cm is medium decomposed peat, and gleyic horizons with illuvial humus are deeper. The surface is densely covered with dwarf birch and mosses with sedges.

**North Taiga Soils, Kuloy Series, Russia (Sampled by S. V. Goryachkin and A. E. Cherkinsky)**

1. Podzolic gleyic soil (Gleyic Podzolusol) (64°36'N, 42°55'E) is on the slope of a moraine hill. Loamy soil with a humus-illuvial horizon. The surface has spruce with feather mosses and dwarf shrubs.
2. Sod-calcareous soil (Rendzina) (65°17'N, 43°11'E) is on the top of the residual hill. Loamy soil with calcareous fragments and mor-moder humus. The surface has larch and spruce forest with grasses and shrubs.
3. Sod-calcareous leaching soil (Cambisol) is on a gentle slope 200 m north of previous soil. Loamy soil with moder-mull humus. Vegetation is the same.
4. Burozem raw humus (Eutric Cambisol) (65°03'N, 45°34'E) is on the top part of a slope. Red loam soil with mor humus. The surface has larch and spruce forest with dwarf shrubs, shrubs and feather mosses.

**METHODS**

With peat, litter and soil samples, all obvious fragments of roots and other unhumified organic material were discarded by handpicking. The peat and litters were then digested in 2.0 M HCl (at 96°C for 2 h), 0.5 M NaOH (at 96°C for 0.5 h) and 2.0 M HCl (at 96°C for 0.5 h). Samples were then washed reagent-free with distilled water after each digestion stage and dried.

Small roots and plant residues were discarded from the soil samples by flotation and were then washed calcium-ion-free in 0.1 M HCl. Humic acids were then separated with 0.1 M NaOH, repeat-

edly. Humic acid was precipitated from the separated solution by the addition of HCl or H<sub>2</sub>SO<sub>4</sub> to give a pH = 1–2. The humic acid precipitate was washed acid-free with distilled water and dried. All reactions were carried out at room temperature. The samples were converted to benzene using the standard technique of Gupta and Polach (1985).

To define the renovation rate of organic carbon, Cherkinsky and Brovkin (1993) suggest using the coefficient of renovation  $K_r$ , which is the integral figure of organic carbon renovation resulting both from biochemical reactions of mineralization and from its migration within the soil profile

$$K_r = \lambda \frac{A_{sn}}{(A_{on} - A_{sn}) \exp \lambda (y - 1950)} \times 100\% \text{ y}^{-1} \quad (1)$$

where  $y$  = the year of measurement of the reference standard activity ( $A_{on}$ ),  
 $\lambda = 1/8267 \text{ y}^{-1}$ , and  
 $A_{sn}$  = specific activity of the sample.

Percent of modern carbon (pMC) was calculated according to the definition of Stuiver and Polach (1977). All <sup>14</sup>C data are expressed at the 2-sigma ( $\sigma$ ) interval for overall analytical confidence.

Total organic carbon contents ( $C_{tot}$ ) in one soil mineral horizon were determined by wet oxidation with potassium dichromate and concentrated sulfur acid. In the organic horizons (e.g., peat, litter) organic carbon contents were determined by dry combustion. Values are expressed as weight percent of the dry sample.

Sample depth increments are quoted (in cm) relative to the soil surface at the time of sampling including organic horizons; pH levels were determined in water suspensions.

All <sup>14</sup>C measurements that are identified with IGAN coding were determined by liquid scintillation counting as applied routinely at the Institute of Geography Radiocarbon Laboratory.

## RESULTS AND CONCLUSION

Tables 1–3 show measured <sup>14</sup>C concentrations and related data. As the Tables show,  $K_r$  decreases with depth in the soil profile and from south to north as a consequence of the reduction in biochemical activity. The renovation rate of the surface horizons of Svalbard soils is 0.02–0.03% C yr<sup>-1</sup> in Arenosols and Gelic Histosols; this rate is 0.18% C yr<sup>-1</sup> in Calcic Arenosols. The maximum renovation rate is 0.27% C yr<sup>-1</sup> measured in Gelic Gleysols.

In the Russian European North (Mezen series, Arkhangelsk region), south tundra soils have much faster renovation rates than arctic soils: the minimum rate is 0.3% C yr<sup>-1</sup> in Haplic Podzols and the maximum is 1.7 in Humic Podzols for surface horizons.

North taiga soils (Kuloy series, Arkhangelsk region) have  $K_r$  of 3.7% C yr<sup>-1</sup> for Gleyic Podzoluvisols and 1.9% C yr<sup>-1</sup> for Rendzina surface horizons–litters. These bioclimate-induced differences among soil carbon renovation rates suggest that warming could accelerate decomposition of SOM, but, at the same time, it could increase productivity of ecosystems of the Arctic region, and consequently increase the store of soil carbon.

TABLE 1. Arctic Tundra Soil Profiles (Svalbard Series, Norway)

Lab code (IGAN-)	Soil order and type		Year sampled	Depth (cm)	OM type	<sup>14</sup> C results			C <sub>tot</sub> WT%	Soil pH <sub>H<sub>2</sub>O</sub>
	FAO*	Local				pMC	Conv. age†	K <sub>r</sub> , % yr <sup>-1</sup>		
988	Gelic Gleysols	Peaty- gleyic arctic soil	1988	0-4	HA	98.9 ± 1.5	85 ± 50	0.265	19.3	5.28
989				4-11	HA	92.6 ± 1.3	640 ± 90	0.124	14.3	5.62
987				11-21	HA	89.0 ± 1.0	960 ± 80	0.090	11.7	5.62
986				21-31	HA	70.9 ± 0.6	2840 ± 60	0.030	11.9	5.94
994	Calcic Arenosols	Rendzina raw humic arctic	1988	0-2	Bone	54.6 ± 0.9	5000 ± 250	--	--	--
996				0-2	HA	95.9 ± 1.0	350 ± 70	0.180	2.3	7.38
995				2-11	HA	84.7 ± 0.9	1370 ± 80	0.065	0.9	7.51
993	Gelic Histosols	Peat arctic soil	1988	0-6	Peat	87.7 ± 1.4	1090 ± 100	0.080	43.8	4.05
992				6-15	Peat	75.4 ± 0.8	2330 ± 70	0.037	42.1	4.64
991				15-26	Peat	65.1 ± 0.7	3550 ± 60	0.023	44.2	5.27
990				26-30	Peat	64.0 ± 0.7	3690 ± 60	0.022	43.5	5.80
1175	Rendzic Leptosols	Rendzina humic arctic	1988	0-3	HA	105.3 ± 0.9	--	0.604	4.8	7.23
1174				3-8	HA	95.7 ± 1.2	360 ± 70	0.177	2.6	7.47
1173				8-16	HA	87.2 ± 0.7	1130 ± 80	0.077	1.4	7.42
1250	Arenosols	Dry- peaty arctic soil	1989	0-5	HA	99.5 ± 1.2	--	0.282	20.4	5.80
1249				5-15	HA	83.4 ± 0.6	1500 ± 65	0.059	10.0	6.22
1248				18-28	HA	71.4 ± 0.6	2780 ± 80	0.030	7.5	7.20
1247				28-32	HA	66.3 ± 0.7	3400 ± 120	0.024	4.5	7.08
157	Arenosols	Podbur	1975	1-3	HA	63.9 ± 0.4	3700 ± 40	0.022	3.2	5.65

\*Relates to soil type locations and descriptions as provided in Site Descriptions section

†Conventional <sup>14</sup>C ages reported in yr BP ± 1 σ

TABLE 2. South Tundra Soil Profiles (Mezen Series, Russia)

Lab code (IGAN-)	Soil order and type		Year sampled	Depth (cm)	OM type	<sup>14</sup> C results			C <sub>tot</sub> WT%	Soil pH <sub>H<sub>2</sub>O</sub>
	FAO*	Local				pMC	Conv. age†	K <sub>r</sub> , % yr <sup>-1</sup>		
781	Haplic Podzol	Fe-Humic Podzol	1986	0-3	HA	99.6 ± 1.6	--	0.299	28.3	4.03
779				3-6	HA	93.7 ± 0.4	540 ± 30	0.142	6.5	4.21
778				15-22	HA	72.5 ± 0.4	2660 ± 30	0.032	1.8	4.70
782				15-22	FA	80.8 ± 1.1	1760 ± 130	0.050	--	--
795				22-27	HA	71.3 ± 0.6	2800 ± 50	0.030	1.6	4.72
777				22-27	FA	78.8 ± 0.7	1970 ± 70	0.045	--	--
752				Humic Podzol	Humic Podzol	1986	0-4	HA	115.7 ± 0.7	--
788	4-19	HA	78.3 ± 0.5				2020 ± 40	0.043	11.1	4.24
796	19-38	HA	72.9 ± 0.4				2610 ± 40	0.033	3.4	4.50
789	38-72	HA	69.6 ± 0.5				3000 ± 70	0.028	1.2	4.56
753	Dystric Gleysol	Peat- gleyic soil	1986	0-9	Peat	109.0 ± 0.5	--	0.941	40.7	4.05
751				33-39	Peat	53.4 ± 0.5	5190 ± 60	0.014	38.6	4.72
755				39-42	HA	56.2 ± 0.5	4770 ± 50	0.016	3.8	5.12
787				39-42	FA	54.8 ± 0.5	4980 ± 60	0.015	--	--
754				42-47	HA	56.3 ± 0.5	4750 ± 60	0.016	2.6	5.35
786				42-47	FA	71.1 ± 1.0	2820 ± 90	0.030	--	--
783				47-55	HA	61.9 ± 0.8	3960 ± 100	0.020	2.3	5.27
784				47-55	FA	63.8 ± 1.0	3710 ± 170	0.022	--	--

\*Relates to soil type locations and descriptions as provided in Site Descriptions section

†Conventional <sup>14</sup>C ages reported in yr BP ± 1 σ

TABLE 3. North Taiga Soil Profiles (Kuloy Series, Russia)

Lab code (IGAN-)	Soil order and type		Year sampled	Depth (cm)	OM type	<sup>14</sup> C results			C <sub>tot</sub> WT%	Soil pH <sub>H2O</sub>
	FAO*	Local				pMC	Conv. age†	K <sub>r</sub> , % yr <sup>-1</sup>		
665	Gleyic Podzo- luvisol	Podzolic gleyic soil	1984	0-6	Litter	126.9 ± 0.6	--	3.716	41.4	4.91
664				6-12	Litter	98.3 ± 0.4	140 ± 30	0.256	34.7	4.62
663				12-25	HA	79.7 ± 0.4	1880 ± 50	0.047	4.4	4.49
662				33-42	HA	57.6 ± 0.7	4760 ± 110	0.017	3.2	5.35
806	Rendzina	Sod- calcareous soil	1985	0-1	Litter	117.2 ± 0.8	--	1.896	28.9	6.33
805				1-5	HA	107.0 ± 0.4	--	0.779	7.5	5.88
804				5-10	HA	99.1 ± 0.4	--	0.283	2.4	5.71
803				10-17	HA	91.9 ± 0.4	650 ± 30	0.118	0.9	6.23
809	Cambisol	Sod- calcareous leached soil	1985	1-8	HA	106.8 ± 0.7	--	0.762	2.7	4.43
808				8-16	HA	93.2 ± 0.5	580 ± 50	0.135	1.4	4.95
807				16-31	HA	87.0 ± 0.7	1150 ± 100	0.077	0.8	4.79
1054	Eutric	Burozem	1986	--	Grass	126.1 ± 0.5	--	--	--	--
1053	Cambisol	raw humic		0-2	HA	115.6 ± 1.6	--	1.645	17.1	4.98
1052				2-8	HA	112.4 ± 0.8	--	1.274	3.0	4.78

\*Relates to soil type locations and descriptions as provided in Site Descriptions section

†Conventional <sup>14</sup>C ages reported in yr BP ± 1 σ

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National University.  
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## **<sup>14</sup>C MEASUREMENTS OF SOIL ORGANIC MATTER, SOIL CO<sub>2</sub> AND DISSOLVED ORGANIC CARBON (1987–1992)**

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**ABSTRACT.** For several undisturbed sites in Germany, <sup>14</sup>C data are reported for soil organic matter (SOM) (4 sites), soil CO<sub>2</sub> (10 sites) and dissolved organic carbon (DOC) (1 site). With the assumption of a fast degradable component (lifetime *ca.* 1 yr) and a slow degradable component (lifetime *ca.* 100 yr), a range between 0.6 and 1.6 mm yr<sup>-1</sup> has been determined for the downward migration rates of soil organic carbon at the sampling sites from the soil <sup>14</sup>C data. The soil CO<sub>2</sub> measurements show that in deciduous forests the fast degradable component is *ca.* 60% and the slow degradable component is *ca.* 40% of the SOM. In coniferous forests this ratio is reversed. The <sup>14</sup>C results for DOC could not be explained with the assumption of a first-order decay process. The removal of soil organic carbon by DOC is of minor importance for the estimation of carbon budgets for the investigated site.

### **INTRODUCTION**

We report <sup>14</sup>C data for soil organic matter (SOM), soil CO<sub>2</sub> and dissolved organic carbon (DOC) in seepage water, all derived from undisturbed forest soil. The samples were collected to study the turnover mechanism of soil organic carbon, which was considered have two dynamic components: a slow degradable component with a lifetime ( $\tau$ ) of *ca.* 100 yr, and a fast degradable component with  $\tau = 1$  yr (Dörr and Münnich 1986). The <sup>14</sup>C content of soil CO<sub>2</sub> and of DOC from the seepage water should reflect the microbial degradation of the solid organic material in the soil profile and therefore give more detailed information about these degradation processes.

The sampled <sup>14</sup>C content of solid SOM was determined as a function of depth. The profile data reported here (M1, M4, W14C) were measured in 1987. Earlier soil profile data are reported in Dörr and Münnich (1986, 1989). The <sup>14</sup>C of soil CO<sub>2</sub> was measured at several sites (M1..4, SK1, RO1..3, RU, WQ) at different times of the year in 1990 and 1991. The DOC samples were collected at one site (M3) during 1989–1991 and processed in 1991–1992.

### **SAMPLING SITES**

All sites are located in Germany: W14C is near Muenster (Westphalia) (51.6°N, 7.4°E). Sites M1, M2, and M3 are *ca.* 15 km south of Heidelberg in the upper Rhine River valley (49.2°N, 8.4°E). M1 is a mixed forest stand (75% beech, 25% spruce), with sandy soil (calcaric Regosol). Carbonate content in the topsoil is *ca.* 5%, and the soil pH is 7.7. It was previously a shifting sand dune and has been forested for 100 yr. M2 is a deciduous forest stand (beech, oak, maple) with a loamy soil layer (*ca.* 80 cm) over sandy sediments. The carbonate content in the topsoil is *ca.* 4%, and the soil pH is 8. Sited close to a former drainage ditch, this soil had been frequently flooded until the beginning of this century. M3 is a mixed forest stand (75% beech, 25% spruce) with no lower vegetation. The soil is sandy (podzolic brown earth); it contains no carbonates, and soil pH is 3.5. The site has been for-

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ested for *ca.* 300 yr; the humus layer is *ca.* 15 cm deep. M4 is *ca.* 25 km southeast of Heidelberg in the Odenwald hillside (49.2°N, 8.5°E). It is a coniferous forest stand (fir/Douglas fir) with loamy soil (calcaric Regosol). Carbonate content in the topsoil is <5% and soil pH is 5. The site has been forested for *ca.* 300 yr. SK1 is *ca.* 15 km northeast of Heidelberg in the Odenwald hillside (49.3°N, 8.6°E), in a coniferous forest stand (fir) with loamy soil. RO1, RO2 and RO3 are located 30 km southeast of Frankfurt and 90 km northeast of Heidelberg on a Main River terrace (49.6°N, 9.1°E). RO1 is a coniferous forest stand (60% spruce, 40% fir) with clay soil. RO2 is a coniferous forest stand (100% spruce) with sandy soil. RO3 is a mixed forest stand (70% deciduous (oak, birch, acacia), 30% spruce) with sandy soil. These sites have been uncultivated for at least the last 100 yr, with exception of RO3, which was cultivated until 40 yr ago.

RU is *ca.* 1 km north of Heidelberg (49.3°N, 8.4°E). It is cultivated, planted with a variety of vegetables. The soil is loamy. WQ is located in Sachsen, 40 km east of Hof and 50 km south of Plauen (50.1°N, 12.2°E). It is a lawn site over a gravelly soil.

## METHODS

The <sup>14</sup>C content in each profile was determined from soil cores taken (including the litter layer) with metal cylinders 10 cm in diameter and 12 cm long. The samples were cleaned of living roots in the laboratory. Carbonates and the soluble organic fraction were then removed by washing in 0.1 N HCl and 0.1 N NaOH. Soil organic carbon contents were determined from the volume of CO<sub>2</sub> produced by combustion, or by the loss of weight after heating the sample to 800°C. Some of the samples were oxidized to CO<sub>2</sub> by combustion of the organic material in a closed system under an oxygen pressure of *ca.* 10 bar. This method is described in Dörr, Kromer and Münnich (1989).

For soil CO<sub>2</sub> measurements, soil gases were pumped through 300–400 ml 4 N NaOH solution for several hours to absorb the CO<sub>2</sub>. *Ca.* 100–200 mMol CO<sub>2</sub> were recovered in each case, by sampling at 70 and 120 cm depth. Weber (1991) gives a detailed description of this sampling method.

We collected seepage water for DOC analysis using stainless-steel lysimeter plates that drained into plastic bottles. These were emptied weekly. Because of the low organic carbon concentrations, a minimum of 6 to 9 liters of water had to be collected over a sampling time of at least one year. We measured a “summer” (TDOC1) and a “winter” sample (TDOC4) from 5-cm depth, as well as samples from depths of 10 (TDOC2) and 50 cm (TDOC3). The samples were processed in two different ways: samples TDOC1 and TDOC4 were oxidized by UV radiation and the CO<sub>2</sub> produced was absorbed in 100 ml 4 N NaOH solution; samples TDOC2 and TDOC3 were dried and the residue was burned in O<sub>2</sub>. A more detailed description of these procedures is given in Tegen (1992). All <sup>14</sup>C measurements were carried out in the <sup>14</sup>C Laboratory at the Institute for Environmental Physics, University of Heidelberg.

## RESULTS AND DISCUSSION

The results for the measurements of soil profiles, soil CO<sub>2</sub>, and DOC are listed in Tables 1 through 3, respectively. Carbon isotope enrichments are calculated and reported in accordance with the definitions of Stuiver and Polach (1977), *viz.*,

$$\Delta^{14}\text{C} = \delta^{14}\text{C} - (2\delta^{13}\text{C} + 50) \cdot \left(1 + \frac{\delta^{14}\text{C}}{1000}\right) (\text{‰}) \quad (1)$$

where

$$\delta^{14}\text{C} = \left( \frac{\left( \frac{^{14}\text{C}}{^{12}\text{C}} \right)_{\text{sample}}}{0.95 \cdot \left( \frac{^{14}\text{C}}{^{12}\text{C}} \right)_{\text{standard}}} - 1 \right) \cdot 1000 (\text{‰}) \quad (2)$$

and

$$\delta^{13}\text{C} = \left( \frac{\left( \frac{^{13}\text{C}}{^{12}\text{C}} \right)_{\text{sample}}}{\left( \frac{^{13}\text{C}}{^{12}\text{C}} \right)_{\text{standard}}} - 1 \right) \cdot 1000 (\text{‰}) . \quad (3)$$

$\delta^{14}\text{C}$  is the deviation in per mil from the NIST HOxII,  $\delta^{14}\text{C}$  data are corrected in respect to a  $\delta^{13}\text{C}$  standard value of  $-25\text{‰}$  due to kinetic separation effects.

TABLE 1. Results of Soil Profile Measurements

Sampling site	Depth (cm)	Organic C (mMol cm <sup>-3</sup> )	$\delta^{13}\text{C}$ (‰)	$\Delta^{14}\text{C}$ (‰)	Date	Soil bulk density (g cm <sup>-3</sup> )
W14C	0-1	2.2	-30.5	236 ± 5	6/87	0.09
W14C	1-2	1.9	-28.4	262 ± 5	6/87	0.08
W14C	2-3	2.7	-28.5	275 ± 5	6/87	0.10
W14C	8-9	3.0	-27.5	59 ± 5	6/87	0.55
M1	0-1	1.9	-27.5	227 ± 6	7/87	0.2
M1	2-3	2.3	-27.4	218 ± 8	7/87	0.54
M1	3-4	2.1	-27.1	247 ± 6	7/87	0.57
M1	4-6	1.1	-26.8	174 ± 6	7/87	0.74
M1	6-8	0.65	-26.6	80 ± 6	7/87	0.9
M1	9-13	0.27	-26.8	40 ± 7	7/87	1.1
M4	0-0.5	2.4	-27.2	239 ± 7	7/87	0.20
M4	0.5-1	1.1	-27.8	215 ± 7	7/87	0.35
M4	1.5-3	1.2	-27.3	215 ± 7	7/87	0.82
M4	3-5	0.34	-26.6	206 ± 11	7/87	0.91
M4	5-7	1.0	-26.1	101 ± 5	7/87	0.95
M2	0-1	1.2	-27.0	143 ± 9	8/87	0.85
M2	1-2	1.2	-26.7	128 ± 9	8/87	0.87
M2	2-3	0.5	-27.1	159 ± 15	8/87	0.86
M2	3-4	1.7	-24.6	68 ± 6	8/87	0.86
M2	4-5	1.5	-24.2	55 ± 8	8/87	0.87

Against the background of possible global warming caused by rising atmospheric CO<sub>2</sub>, it is important to estimate the carbon fluxes between the atmosphere and the soil. Organic carbon is added to the soil by litter and removed by microbial decomposition of the SOM into both CO<sub>2</sub> and DOC. The global soil organic carbon reservoir is estimated at 1400 Gt. *Ca.* 60 Gt yr<sup>-1</sup> are added to the soil by litter and 60 Gt yr<sup>-1</sup> are removed as CO<sub>2</sub> (Macdonald and Sertorio 1989). Our investigations showed that the removal of organic carbon as DOC is only *ca.* 1% of that removed as CO<sub>2</sub> at our sampling site (Tegen 1992). Thus, we conclude that, for the soils investigated, the DOC flux is of minor importance for the

estimation of carbon budgets. However, the  $^{14}\text{C}$  data of DOC can help to describe the microbial processes in the soil that lead to the production of DOC. It cannot be determined whether removal of soil organic carbon by DOC production would be significant at other locations or on a global scale.

Given a scenario of global warming, the carbon fluxes between atmosphere and biosphere are no longer in steady state. Not only is vegetation likely to respond with enhanced litter production and therefore more production of SOM, the rate of degradation of soil organic material could be accelerated by enhanced microbial activity. In this context, it is important to investigate the time constants ( $\tau$ ) of this microbial degradation, which can be determined by comparing  $^{14}\text{C}$  measurements of soil organic carbon and soil  $\text{CO}_2$ .

Table 1 lists the results from the soil profile measurements. SOM is not homogenous but consists of at least two components (Dörr and Münnich 1986): a fast degradable component with a lifetime ( $\tau$ ) of  $\approx 1$  yr and a slow degradable component with  $\tau \approx 100$  yr. The  $^{14}\text{C}$  depth profiles show that organic material is younger at the top of the soil than at the bottom. From these  $^{14}\text{C}$  data, Dörr and Münnich (1989) determined for the downward migration rates of organic material a range between 0.6 and 1.6 mm  $\text{yr}^{-1}$  at the sampling sites M1–M4.

Table 2 lists the  $^{14}\text{C}$  data of soil  $\text{CO}_2$ . Included here are  $\text{CO}_2$  concentrations,  $\delta^{13}\text{C}$ , and  $\Delta^{14}\text{C}$  of soil  $\text{CO}_2$  and for atmospheric  $\text{CO}_2$ . With these  $\Delta^{14}\text{C}$  data, the  $\Delta^{14}\text{C}$  of soil respiration ( $\Delta^{14}\text{C}_{\text{soil respiration}}$ ) are calculated to reflect the actual  $\text{CO}_2$  emissions out of the soil, using

$$\Delta^{14}\text{C}_{\text{soil respiration}} = \frac{\text{CO}_{2 \text{ soil}} \cdot \Delta^{14}\text{C}_{\text{soil air}} - \text{CO}_{2 \text{ air}} \cdot \Delta^{14}\text{C}_{\text{air}}}{\text{CO}_{2 \text{ soil}} - \text{CO}_{2 \text{ air}}} - 8\text{‰} \quad (4)$$

The 8‰ has to be subtracted because of the kinetic separation due to molecular diffusion in the soil. These values range between  $-46$  and  $251\text{‰}$ . The microbial degradation of SOM into  $\text{CO}_2$  is assumed to be a first-order process. Using this assumption, Weber (1991) calculated the ratio of the fast and slow decomposable organic fraction with the  $\Delta^{14}\text{C}$  data listed in Table 2 in the analyzed soils. In deciduous forest soils, Weber (1991) found the short-lived fraction of organic matter to be  $63 \pm 17\%$  and the long-lived fraction to be  $37 \pm 17\%$ . In coniferous forest soils, the short-lived fraction of organic matter is  $37 \pm 24\%$  and the long-lived fraction is  $63 \pm 24\%$ .

TABLE 2. Results of Soil  $\text{CO}_2$  Measurements

Site	Sampling date	$\text{CO}_{2 \text{ soil}}$ (ppm)	$\text{CO}_{2 \text{ air}}$ (ppm)	$\delta^{13}\text{C}_{\text{soil}}$ (‰)	$\delta^{13}\text{C}_{\text{air}}$ (‰)	$\Delta^{14}\text{C}_{\text{air}}$ (‰)	$\Delta^{14}\text{C}_{\text{soil air}}$ (‰)	$\Delta^{14}\text{C}_{\text{soil respiration}}$ (‰)
M3	4/90	2663 ± 39	436 ± 65	-26.55 ± 0.05	-11 ± 2	137 ± 4	154 ± 8	150 ± 10
M2	4/90	6473 ± 165	436 ± 65	-23.13 ± 0.05	-11 ± 2	137 ± 4	143 ± 6	135 ± 6
M4	4/90	5324 ± 103	436 ± 65	-22.05 ± 0.05	-11 ± 2	137 ± 4	145 ± 6	137 ± 7
M1	5/90	3233 ± 33	436 ± 65	-21.4 ± 0.05	-11 ± 2	137 ± 4	168 ± 5	165 ± 6
M2	7/90	9930 ± 53	436 ± 65	-23.45 ± 0.05	-11 ± 2	137 ± 4	156 ± 6	149 ± 6
SK1	7/90	13,442 ± 4	436 ± 65	-26.71 ± 0.05	-11 ± 2	137 ± 4	164 ± 6	157 ± 6
M1	7/90	4271 ± 71	436 ± 65	-21.26 ± 0.05	-11 ± 2	137 ± 4	180 ± 6	176 ± 7
M3	7/90	5354 ± 432	436 ± 65	-21.35 ± 0.05	-11 ± 2	137 ± 4	158 ± 6	152 ± 7
M4	7/90	9758 ± 164	436 ± 65	-22.04 ± 0.05	-11 ± 2	102 ± 4	144 ± 6	136 ± 6
M4	1/91	4037 ± 376	436 ± 65	-23.99 ± 0.05	-11 ± 2	102 ± 4	134 ± 6	130 ± 7
M2	1/91	3831 ± 76	436 ± 65	-22.29 ± 0.05	-11 ± 2	102 ± 4	158 ± 7	157 ± 8
M1	1/91	1884 ± 37	436 ± 65	-20.82 ± 0.05	-11 ± 2	102 ± 4	162 ± 6	172 ± 9
M3	1/91	2171 ± 40	436 ± 65	-20.12 ± 0.05	-11 ± 2	102 ± 4	115 ± 5	111 ± 7
RO3	3/91	1378 ± 29	436 ± 65	-20.45 ± 0.05	-11 ± 2	102 ± 4	209 ± 6	251 ± 15
RO1	3/91	1315 ± 42	436 ± 65	-19.33 ± 0.05	-11 ± 2	102 ± 4	149 ± 6	165 ± 12
RO2	3/91	1222 ± 97	436 ± 65	-18.97 ± 0.05	-11 ± 2	102 ± 4	152 ± 6	172 ± 13
RU	4/91	10,078 ± 376	423 ± 32	-24.13 ± 0.05	-9 ± 2	120 ± 4	-32 ± 6	-46 ± 6
WQ	5/91	13,337 ± 667	411 ± 15	-23.32 ± 0.05	-9 ± 2	120 ± 4	32 ± 3	22 ± 3

Table 3 lists the  $\Delta^{14}\text{C}$  data for DOC which range between 29 and 105‰. Because of the long time interval between the sampling date and the date of analysis, the easily degradable component of DOC is probably removed from the samples and the data describe the slowly degradable component of DOC. Unlike the soil  $\text{CO}_2$ , the  $\Delta^{14}\text{C}$  data of DOC could *not* be described by a model that assumes DOC to be produced from the solid organic matter by a first-order decay process. The data could be explained by assuming that SOM is not degraded into DOC earlier than 20–30 yr after deposition (Tegen 1992).

TABLE 3. Results of DOC Measurements

Sample	Sampling date	DOC (mg liter <sup>-1</sup> )	$\delta^{13}\text{C}$ (‰)	$\Delta^{14}\text{C}$ (‰)	Date of analysis
TDOC1	6/89–10/89 5/90–10/90	80	-29.2	29 ± 27	6/91
TDOC4	10/89–5/90 10/90–5/91	30	-29.9	88 ± 25	12/91
TDOC2	6/89–7/90	40	-28.3	93 ± 11	9/91
TDOC3	6/89–7/90	50	-27.6	105 ± 8	4/92

## CONCLUSION

In undisturbed soils,  $^{14}\text{C}$  measurements can be used to determine turnover rates for soil organic carbon. Knowledge of such turnover rates is necessary to investigate the role of SOM in the global carbon budget and the impact of changing climatic conditions on soil organic carbon fluxes. Our results indicate that the type of vegetation cover affects the turnover rates of the carbon reservoirs, as coniferous forests appear to produce 50% more slow than fast degradable SOM, whereas deciduous forests produce 50% more fast than slow degradable SOM. These results need to be compared to results for different ecosystems. Unlike the soil  $\text{CO}_2$   $\Delta^{14}\text{C}$  data, the DOC  $\Delta^{14}\text{C}$  results cannot be explained by a first-order decay process but seem to indicate a time lag of several years between litter fall and production of non-volatile DOC. Although at our sampling site the contribution of DOC to the carbon budget seemed to be of minor importance, it remains to be investigated whether this holds true for other sites, specifically sites with different climatic conditions.

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## HISTORIC MEASUREMENTS OF RADIOCARBON IN NEW ZEALAND SOILS

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**ABSTRACT.** Extensive measurements of radiocarbon have been used in New Zealand since the mid-1960s to follow carbon (C) movement and turnover in soils. We present here unpublished radiocarbon (<sup>14</sup>C) measurements on a range of eight New Zealand soils with details of the sites, ecosystems, climates, soil descriptions and associated analytical data. An overview is also given of published <sup>14</sup>C measurements on soils, and the use of these measurements to model soil C turnover.

### INTRODUCTION

New Zealand stretches *ca.* 2000 km along a north-south axis, spanning from 34° to 47°S in the south-west Pacific. The country is more remote from its nearest neighbor than any other global landmass. This isolation coupled with a high degree of climatic, geological, soil and biological diversity compressed into just 270,000 km<sup>2</sup>, and the relative freedom from industrial pollution, provide unique opportunities for investigating surface-atmosphere exchange processes of global significance.

Furthermore, relatively undisturbed remnants of indigenous ecosystems of great antiquity are available for direct comparison with the introduced pastures and planted forests that replaced them over large areas, particularly of lowland New Zealand. It is against this background that in the early 1960s New Zealand nuclear and soil scientists recognized the opportunities for investigating these surface-atmosphere exchange processes (Rafter *et al.* 1965), provided by the then rapidly rising atmospheric concentration of radiocarbon from nuclear weapons tests that had commenced in 1954. In 1962, the New Zealand Radiocarbon Laboratory (NZRL), which was part of DSIR's Institute of Nuclear Sciences until July 1992, began analyzing anthropogenic <sup>14</sup>C in New Zealand soils. The first systematic mapping and classification of New Zealand soils was being concluded at this time (New Zealand Soil Bureau 1968), so that a representative range of well characterized sites and soils was immediately available for investigating soil dynamic processes, including rates of organic matter turnover. This research, using well-defined soil sequences to investigate the main soil forming factors (*e.g.*, Stevens and Walker 1970; Jackman 1964), soon revealed the dynamic character of soil organic matter (SOM), and illustrated the unique opportunities New Zealand offered for understanding and quantifying the processes of ecosystem development. Thus, the scene was set for using the <sup>14</sup>C enrichment of the biosphere through photosynthesis to trace the pathways, and measure the fluxes, of C in soil. Subsequently, through a program of extensive measurements of  $\delta^{13}\text{C}$  and  $\Delta^{14}\text{C}$  in New Zealand soils and plants that followed, it eventually became possible to model biogeochemical pathways for the movement and turnover of C in soil (Rafter and Stout 1970; O'Brien and Stout 1978; O'Brien *et al.* 1981; O'Brien 1984, 1986).

Our aims in this report are 1) to list unpublished  $\Delta^{14}\text{C}$  and  $\delta^{13}\text{C}$  measurements made on New Zealand soil samples from eight profiles that have been well described and analyzed for their chemical and physical properties, and 2) to review briefly research previously published that used anthropogenic <sup>14</sup>C to investigate the dynamics of soil organic C.

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## METHODS

### Sites and Soils

The eight sites (Fig. 1) and soils listed under "Summary of Unpublished  $^{14}\text{C}$  Measurements on New Zealand Soils" reflect the wide diversity of climates and soil types represented in New Zealand. Descriptions are given for each of the eight sites and soils listed. Analytical data for  $\Delta^{14}\text{C}$ ,  $\delta^{13}\text{C}$  and a range of soil chemical and physical properties are presented by horizon; designations for soil horizons follow the convention of Clayden and Hewitt (1989).

### Analytical Methods and Definitions

The C isotope results reported here were all obtained by stable-isotope mass spectrometry and by gas-counting techniques made on pretreated soils. The pretreatment comprises a hot-water wash, acid treatment (usually 2% phosphoric acid to remove carbonates), and water rinse. Values for  $^{13}\text{C}/^{12}\text{C}$  ratios and for  $^{14}\text{C}/(^{12}\text{C} + ^{13}\text{C})$  are expressed relative to standards after Craig (1953) and Stuiver and Polach (1977), respectively, by Equations (1)–(3).

$$\delta^{13}\text{C}(\text{‰}) = 1000 [(R_{13} / R_0) - 1] \quad (1)$$

$$\left(1 + \frac{\Delta^{14}\text{C}}{1000}\right) = \left(1 + \frac{\delta^{14}\text{C}}{1000}\right) \left(\frac{975}{(1000 + \delta^{13}\text{C})}\right)^2 \quad (2)$$

$$\delta^{14}\text{C}(\text{‰}) = 1000 [(R_{14} / R_{std}) - 1] \quad (3)$$

In these equations,  $R_{13}$  and  $R_0$  are the  $^{13}\text{C}/^{12}\text{C}$  ratios in the soil C samples and isotope standard (Pee-Dee Belemnite, or PDB), respectively;  $R_{14}/R_{std}$  is the  $^{14}\text{C}$  content of the sample per gram of C, decay-corrected to the time of sampling, relative to that for the NIST radiometric standard (0.95 oxalic acid, HOxI), decay-corrected back to 1950. By convention,  $\delta^{13}\text{C}$ ,  $\delta^{14}\text{C}$  and  $\Delta^{14}\text{C}$  are expressed in ‰. Since in most New Zealand ecosystems soil  $\delta^{13}\text{C}$  values are consistently close to  $-25\text{‰}$ , there is no significant numerical distinction between  $\Delta^{14}\text{C}$  and  $\delta^{14}\text{C}$ .

### Soil Chemical and Physical Analyses

The methods for preparing soil samples for analysis, and the analytical methods themselves, are fully described by Blakemore *et al.* (1987). Carbon contents (wt%) were determined by wet oxidation (Metson *et al.* 1979) prior to 1967, and subsequently by dry combustion (Blakemore *et al.* 1987). Although microbial biomass was not measured on the soil samples listed, measurements have been reported for some corresponding topsoils as follows: Carrick, Tima (Ross *et al.* 1980); Judgeford (Ross *et al.* 1990); Egmont (Ross 1992). The isotope, and soil chemical and physical data were largely retrieved from the NZRL archives, and the National Soils' Database, respectively.

## RESULTS

### Summary of Unpublished $^{14}\text{C}$ Measurements on New Zealand Soils

The locations of the sites for the eight soils listed are shown in Figure 1, together with sites for which  $\Delta^{14}\text{C}$  and  $\delta^{13}\text{C}$  measurements have already been published.

For each of the eight soils, we present two tabulations. The first contains site information, including location, climate, site, geology, land use, vegetation and soil classification, as well as a brief description of the soil by horizon. The second tabulation presents the isotope, soil chemical and physical analyses, along with explanatory notes as appropriate. All soil samples are identified by two laboratory codes, one for the isotope analyses designated "NZ", and the other, "SB", for the soil data. In

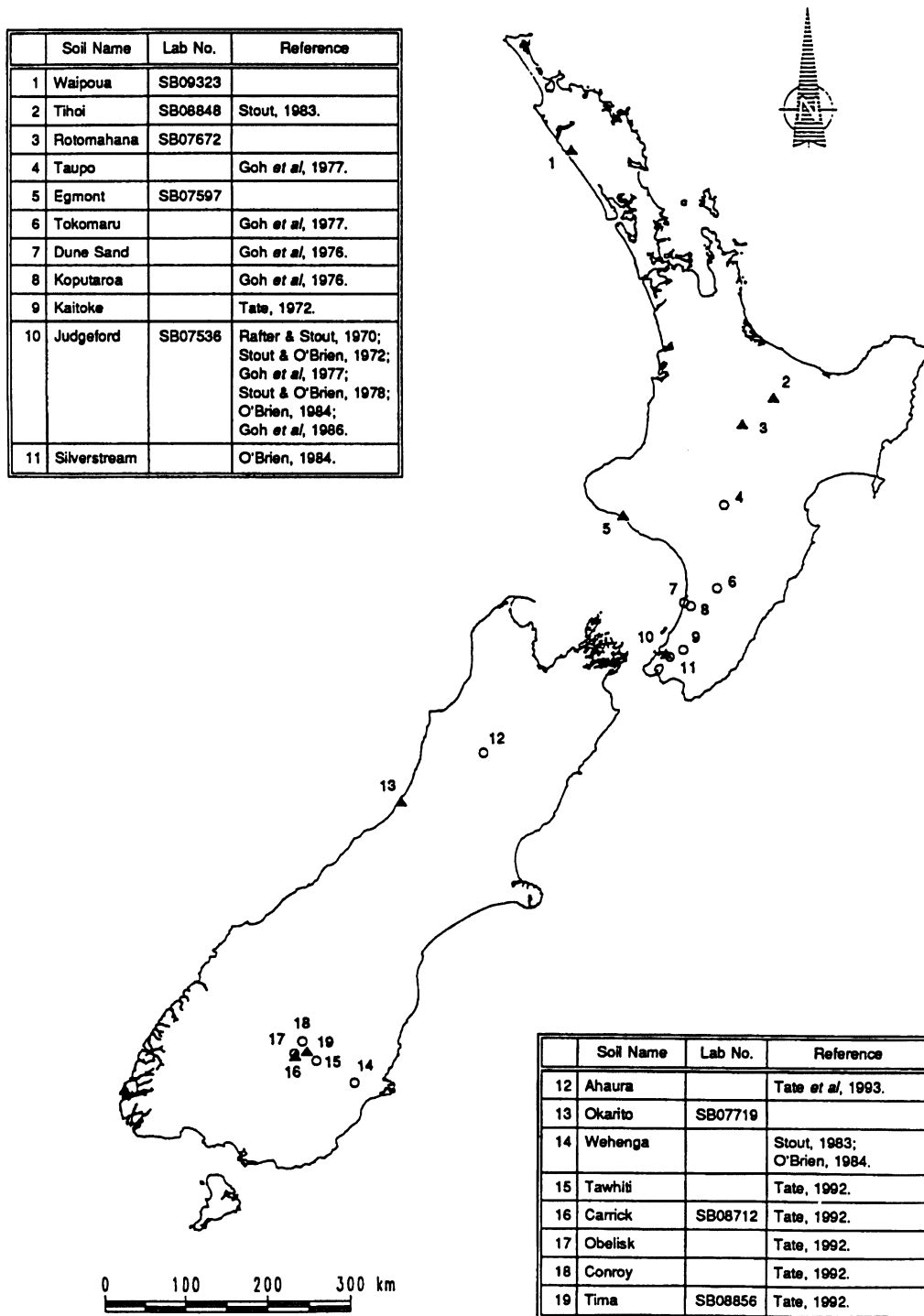


Fig. 1. Map of New Zealand (latitudes from 34 to 47°S) showing the location of North and South Island sites and soils (▲) described in this report. Sites where isotope data for soils has previously been published (○) are also shown.

most cases, the  $\Delta^{14}\text{C}$  and  $\delta^{13}\text{C}$  measurements were made on the same soil samples as for the chemical and physical analyses. The exception was the Tihoi soil (Fig. 1, site 2), where the C isotope analyses reported are for soil samples from a nearby site also under pasture. The horizons analyzed in the Rotomahana soil for  $\Delta^{14}\text{C}$  and  $\delta^{13}\text{C}$  straddle the boundary between the base of the contemporary soil, formed on volcanic mud erupted in 1886, and the soil beneath. Although many measurements of  $\Delta^{14}\text{C}$  and  $\delta^{13}\text{C}$  have been made, and reported, on Judgeford soils (see site summary in Fig. 1), the data presented here for a full profile has not been reported previously. Precisions for the  $\delta^{13}\text{C}$  isotope data are typically  $\pm 0.1\text{‰}$  (standard deviation).

#### OVERVIEW OF PUBLISHED $^{14}\text{C}$ MEASUREMENTS ON NEW ZEALAND SOILS

Several factors including climate, ecosystem and soil type, resource quality and soil biology, together regulate the turnover of SOM. The effects of some of these factors on organic matter turnover have been investigated in New Zealand soils using natural  $^{14}\text{C}$  enrichment.

##### Climate

Stout and O'Brien (1972) contrasted the  $^{14}\text{C}$  enrichment of the litter and soils in a warm temperate New Zealand kauri (*Agathis Australis*) forest (latitude  $36^\circ\text{S}$ ) with two topsoils in a tropical mixed kauri-broadleaved forest in New Hebrides ( $19^\circ\text{S}$ ). They concluded from the high level of  $^{14}\text{C}$  found in the two tropical topsoils, coupled with their relatively thin litter layers compared to the temperate forest, that C was cycling much more rapidly in the former. However, a highly productive temperate New Zealand topsoil was enriched with  $^{14}\text{C}$  to a similar extent as the two tropical topsoils.

More recently, the  $^{14}\text{C}$  measured in five profiles of a soil climosequence in native tussock grassland in South Island, New Zealand, first published by Stout and Rafter (1978), was used in conjunction with total soil C contents to investigate the possible effects of global warming on soil C turnover (Tate 1992). The climates across the sequence ranged from cold to warm temperate. A soil C turnover model (O'Brien 1984) indicated that turnover rates for three of the soils were in the range expected for New Zealand grassland soils. For two of the soils, however, much lower levels of  $^{14}\text{C}$  indicated unexpectedly slow turnover rates that were attributed to a memory effect from the former beech forest that grew on these sites in prehistoric times. Local site factors including intermittent waterlogging may also have impeded decomposition processes and affected the overall soil C balance (Tate *et al.* 1995).

##### Ecosystem Type

The types of vegetation and soil can have a strong influence on the pattern of  $^{14}\text{C}$  distribution in soils (Rafter and Stout 1970). Whereas  $\Delta^{14}\text{C}$  in pasture grasses reflected the composition of atmospheric  $\text{CO}_2$ , during the period of rapid change in the 1960s,  $\Delta^{14}\text{C}$  in beech leaves lagged behind that of the atmosphere. Rafter and Stout (1970) attributed this lower  $\Delta^{14}\text{C}$  to mixing of photosynthesized C during a two-year period of beech leaf growth.  $\Delta^{14}\text{C}$  in the beech litter was measured annually for seven years from 1953, and again from 1964 until 1967. Comparison of the soil profiles beneath these two vegetation types indicated a different distribution pattern of  $\Delta^{14}\text{C}$ . The upper soil horizon under the pasture was only slowly enriched with "bomb"  $^{14}\text{C}$ , and the subsoil had very low  $\Delta^{14}\text{C}$  values. By contrast, the litter horizon of the southern beech (near site 11, Fig. 1), and the deeper soil horizons, showed that appreciable  $^{14}\text{C}$  enrichment had occurred. Thus, C entering the beech profile was more mobile than that entering the pasture profile. In the latter, plant residues were decomposed upon entering the soil, and released more rapidly back to the atmosphere as  $\text{CO}_2$ . Rafter and Stout

(1970) presented  $^{13}\text{C}$  and  $^{14}\text{C}$  data for five different soil types, although not all horizons were analyzed, and little accompanying site and soil data were recorded.

In a comparison of two adjacent ecosystems on similar soil types near Wellington—an old-growth, lowland southern beech forest and a productive pasture—Tate (1972) found that  $^{14}\text{C}$  was again restricted to the topsoil under the pasture, but occurred throughout the soil profile (to 0.38 m depth) under the beech forest. The  $^{14}\text{C}$  in the soils and their chemically separated fractions (humic and fulvic acids) were used to confirm that soil polyphenols in the subsoil beneath the pasture were derived predominantly from the original beech forest. The  $^{14}\text{C}$  in the soil beneath this and a nearby beech forest are discussed in more detail elsewhere (Stout and O'Brien 1972; Stout, *et al.* 1976; O'Brien 1984).

More recently, Tate *et al.* (1993) used  $\Delta^{14}\text{C}$  in soil profiles beneath an old-growth southern beech forest together with soil chemical, physical and biological data to calculate C turnover rates. The accumulation of old C in these soils was attributed to the effects on soil C turnover of a long history of soil mixing by tree overturn in this ancient forest. This process is not observed in grassland soils.

### Soil Type

The age of SOM appears to be more closely related to soil type, and soil forming processes, than to climate (Stout *et al.* 1981). This was shown in investigations of a chronosequence of New Zealand soils developed on wind-blown sand. Soil ages along the chronosequence ranged from 20 to *ca.* 10,000 yr (Goh and Stout 1972; Goh *et al.* 1976). The mass of total organic C in the soil profiles and the carbon isotope composition of specific soil horizons were presented in these investigations. The rate of accumulation of soil organic C was rapid in the first 500 yr of soil formation, with a considerable range in the age of the SOM as a function of depth within a profile. The younger soils were more enriched with  $^{14}\text{C}$ , with topsoils more enriched than subsoils (Goh and Stout 1972). Goh *et al.* (1976) attempted to interpret  $^{14}\text{C}$  levels in the classical humus fractions of some of these soils (humic and fulvic acids, and humins) in terms of possible genetic relationships between them, but the outcome was mainly equivocal.

In a closely related study, Goh *et al.* (1977) found that the  $^{14}\text{C}$  levels in the humus fractions varied both within and among soil types, as well as between topsoils and subsoils. They studied a range of soil types from several sites in grasslands of mainly low to medium fertility. These soil types included a Fluvaquent, Typic Fragiaqualf, Typic Dystrochrept and Umbric Vitrandept (Soil Survey Staff 1992). It appeared that  $^{14}\text{C}$  levels were primarily determined by the stage of decomposition of the organic matter, rather than by soil type. These studies on soil humus fractions have added to the weight of evidence (O'Brien *et al.* 1981) indicating that these classical fractions have limited value in unraveling the complex biological pathways involved in SOM turnover. The effect of soil type on organic matter turnover in New Zealand is expressed most strongly in those soils in North Island containing short-range order minerals, *e.g.*, allophane (Jackman 1964). Current research (K. R. Tate, unpublished results) seeks to quantify the influence of allophane and ferrihydrite on organic matter turnover, and for this purpose, the distribution of "bomb"  $^{14}\text{C}$  in three volcanic ash soils is being investigated.

### Soil Biological Activity

Earthworms, both native and introduced, have an important influence on New Zealand soils by comminuting and incorporating plant residues, thereby accelerating organic matter turnover rates. Stout (1983) and O'Brien (1984) used soil  $^{14}\text{C}$  measurements on different soil types in attempts to

quantify the effect of earthworms at sites with known populations. In the absence of earthworms there was little evidence for downward movement of  $^{14}\text{C}$  in the soil and decomposition rates were slow. Stout (1983) reported total masses of soil organic C,  $^{14}\text{C}$  and  $^{13}\text{C}$  for several Tihoi (Site 2, Fig. 1) and Wehenga soil profiles without worms and with known worm populations. These studies showed that the presence of earthworms had increased topsoil organic matter contents, and accelerated decomposition including that of old C. O'Brien (1984) modeled the turnover of soil C using the profile distributions of "bomb"  $^{14}\text{C}$  in the soil profiles, and found that in the South Island Wehenga soil the presence of earthworms had caused organic matter turnover rates to increase five-fold.

### Modeling SOM Turnover

Much of the research in New Zealand on the use of "bomb"  $^{14}\text{C}$  to investigate soil C turnover has involved investigating Judgeford soils near Wellington. O'Brien and Stout (1978) developed a steady-state model to represent organic C turnover, and used "bomb"  $^{14}\text{C}$  and soil organic C measurements in Judgeford soil profiles sampled over a *ca.* 15-yr period to evaluate the model parameters: C input, decomposition time and downward diffusivity in the soil profile. Estimates of these model parameters have subsequently been made for five New Zealand pasture soils and a forest soil (O'Brien 1984), five soils in native tussock grassland (Tate 1992), and an old-growth southern beech forest (Tate *et al.* 1993). Evidence from detailed  $^{14}\text{C}$  and  $^{13}\text{C}$  measurements made on a soil core to the base of the Judgeford soil (Goh *et al.* 1984), and in a nearby soil (O'Brien 1986), supported the hypothesis in the model that the concentration of old (inert) C—possibly polymethylene C (Theng *et al.* 1992)—remains constant with depth in the profile.

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**Site 1: Location and Soil Description**

Soil name: Waipoua Clay		Lab no. SB09323	
Site, Soil Descriptions and Analytical Data			Date sampled: 19 Feb. 1972
Latitude:	35°39'S	Longitude:	173°34'
N.Z. genetic classification:	Brown granular clay	NZSC:	Acidic orthic granular soil
Survey:	North Auckland	FAO:	Humic acrisol
		Region:	Northland, North Island
Location:	Parliament grove, Waipoua forest, 1.2 km NW of the bridge at intersection of main road and forest HQ road		
Topdressing:	Nil	Elevation:	170 m
Annual rain:	1648 mm	Mean annual temp:	14°C
Landform:	Ridge	Slope:	10°
Microrelief:	Flat	Topsoil loss:	0%
Slope movement:	Not known	Land use:	Unused-natural
Drainage:	Moderately good		
Improvements:	Nil		
Vegetation:	Kauri forest, <i>Agathis australis</i> , <i>Podocarpus ferugineus</i>		
Parent materials:	Strongly weathered basalt, weathered andesitic		
Notes:	Pit to 31 cm. Auger 1 (50–61 cm), Auger 2 (61–92 cm), Auger 3 (61–92 cm). Augers 30 cm triangle apart. At –2.0 cm transition litter/mineral soil with many roots up to 2 cm. Also sampled at 61–92 cm yellowish-red, red, and dark brown (5YR 4/6, 2.5YR 4/6 and 7.5YR 4/4) silty clay; very firm; mod. coarse and med. nut breaking to fine and very fine blocky and nut structure; fine structure in above horizons may be inherited from the weathered rock.		
Horizon	Depth (cm)	Horizon description	
L	–22–19	Decomp. litter coated with reddish humus; Kauri leaves, bark, laminae of cones, twigs, male flowers; indistinct boundary.	
F	–19–15	Reddish-black (10R 2/1) peaty; to 2.5YR 2/2; within wiry mattress of f. roots; abund part decomp. litter as above; few gum frag.; ab. loose root nodules.	
H1	–15–10	Reddish-black (10R 2/1) peaty; very weak soil strength; weakly developed very fine crumb structure, v. abund. fibers; decaying monocot roots; root nodules; indistinct boundary.	
H2	–8–0	Dark reddish-brown (5YR 2/2) clay; strongly developed medium granular plus strongly developed fine granular structure; and very fine cast granules; 20% med nut; some gum fragments; distinct boundary.	
AB	0–15	Dark brown (7.5YR 3/2) clay; moderately firm soil strength; moderately developed coarse nut breaking to medium nut structure; to 7.5YR 4/2 and 2/2; also f. nut and v.f. block struct.; rootless thru' peds; indistinct boundary.	
Bt1	15–31	Dark brown (7.5YR 3/2) clay; moderately firm soil strength; moderately developed coarse nut breaking to fine blocky structure; indistinct boundary.	
Bt2	31–50	Brown to dark brown (7.5YR 4/2) clay; moderately firm soil strength; strongly developed medium blocky plus strongly developed very fine granular structure; to 7.5YR 4/4; friable when disturbed; also v. fine block; indistinct boundary.	
BC1	50–61	Brown to dark brown (7.5YR 4/4) clay; moderately firm soil strength; moderately developed medium nut breaking to fine blocky structure; also 7.5YR 5/8–8/6 gritty clay loam weathering P.M.; friable when disturbed; also v. fine block; indistinct boundary.	
BC2	61–91	Reddish-brown (5YR 4/4) silty clay loam; very firm soil strength; moderately developed very coarse nut breaking to fine blocky structure; to 7.5YR 5/6; also fine and very fine block structure.	

## Site 1: Measurements

S.B. lab no.	N.Z. lab no.	Horizon desig.	Sample depth (cm)	Isotopes		Chemical Properties								
				$\delta^{14}\text{C}$ ‰	$\delta^{13}\text{C}$ ‰	C %	N %	C/N	CEC† me %	pH (H <sub>2</sub> O) dry soil	pH (H <sub>2</sub> O) moist soil	Total P mg %	Org. P mg %	Inorg. P mg %
9323A		L	-22-19			50.0	0.83	60	79.2		4.1	39		
9323B	1439	F	-19-15	18.3 ± 7.0	-25.2	49.0	1.21	40	130.5		4.1	40		
9323C	1440	H1	-15-10	-4.9 ± 6.9	-25.2	50.0	1.29	39	125.5		3.5	40		
9323D	1443	H2	-8-0	-17.0 ± 7.0	-24.0	19.0	0.66	29	57.7		3.8			
9323E	1557	AB	0-15	-31.8 ± 6.9	-25.7	7.8	0.32	24	34.9		4.3			
9323F	1573	Bt1	15-31	-35.1 ± 6.8	-26.5	4.5	0.22	20	26.2		4.8			
9323G	1574	Bt2	31-50	-72.2 ± 6.7	-25.8	3.1	0.14	22	20.8		4.7			
9323H		BC1	50-61			1.7	0.07	24	16.2		4.7			
9323I		BC2	61-92			0.9	0.03	*	15.1		4.9			
9323J		BC2	61-92			1.2	0.04	*	14.1		4.7			

S.B. lab no.	N.Z. lab no.	Horizon desig.	Sample depth (cm)	Physical-Mineralogical Properties					Clay minerals (topsoil only)	Comments
				Dry bulk density t/m <sup>3</sup>	Stones (% v/v)	% Sand 2.0-0.02 mm	% Silt 0.02-0.002 mm	% Clay <0.002 mm		
9323A		L		0.04					Predominantly kaolin  Small amounts of vermiculite, quartz	L, F, H litter horizon bulk densities (italics) are approximate, est. from means for litters under southern beech and mixed podocarp/broadleaf forest.  Bulk densities (italics) for AB, Bt horizons estimated from 5 brown granular clay soils at similar depths.  AB mean = 0.94 std. dev. = 0.17 Bt mean = 0.93 std. dev. = 0.15 Blank spaces in tables indicate no available data. *C/N ratio not calculated when N < 0.05. †CEC = cation exchange capacity; me % = c mol(+) kg <sup>-1</sup>
9323B	1439	F		0.08						
9323C	1440	H1		0.08						
9323D	1443	H2		0.08						
9323E	1557	AB		0.94						
9323F	1573	Bt1		0.93						
9323G	1574	Bt2		0.93						
9323H		BC1								
9323I		BC2								
9323J		BC2								

## Site 2: Location and Soil Description

Soil name: Tihoi Loamy Sand

Lab no. SB08848

Site, Soil Descriptions and Analytical Data

Date sampled: 27 Mar. 1973

Latitude:	38°35'S	Longitude:	175°57'		
N.Z. genetic classification:	Podzolized yellow-brown pumice soil	NZSC:	Humose orthic podzol	FAO:	Orthic podzol
Survey:	Soils of Taupo region	Region:	Taupo, North Island		
Location:	Lomond Road, 1 km east of obsidian cutting, cutting of old scoria quarry.				
Topdressing:	Not known				
Annual rain:	2000 mm	Elevation:	610 m	Mean annual temp:	12°C
Landform:	In rolling country				
Microrelief:	Flat	Slope:	4°	Aspect:	360°
Slope movement:	Not known	Topsoil loss:	0%		
Drainage:	Well	Land Use:	Rough grazing		
Improvements:	Oversown, ploughed				
Vegetation:	Browntop, Yorkshire fog, Thistles, White clover				
Parent materials:	Taupo pumice overlying weathered rhyolitic tephra				
Notes:	Erosion - nil				



**Site 2: Location and Soil Description(Continued)**

Horizon	Depth (cm)	Horizon description
Ap	0–9	Black (10YR 2/1) loamy sand; very weak soil strength; weakly developed fine granular plus weakly developed fine crumb structure; abundant live roots; tending to 10YR 3/2; few fine lapilli; distinct irregular boundary.
Ea	11–9	Greyish brown (10YR 5/2) fine sand; moderately weak soil strength; weakly developed fine crumb plus weakly developed fine granular structure; many medium and fine lapilli; distinct smooth boundary.
Bh	21–37	Dark reddish brown (5YR 3/4) loamy sand; moderately weak soil strength; weakly developed fine nut plus weakly developed fine granular structure; few medium and fine lapilli; distinct smooth boundary.
Bs	40–50	Strong brown (7.5YR 5/6) gritty sand; moderately firm soil strength; massive; distinct smooth boundary
2Cu1	52–78	Yellowish brown (10YR 5/8) to 7.5YR 5/8 (and much darker grey rhyolite) pumice gravel; loose soil strength; single grain; Taupo lapilli; distinct irregular boundary.
3Cu2	80–105	Light yellowish brown (2.5Y 6/4) gritty sand; loose soil strength; massive breaking to single grain; pumice gravel; firm in situ; sharp smooth boundary.
3Cu3	105–107	Grey (5Y 5/1) loamy sand; moderately firm soil strength; massive; (Rotongaio Ash); sharp smooth boundary.
3Cu4	109–119	Yellowish brown (10YR 5/4) gritty sandy loam; moderately weak soil strength; weakly developed granular structure; greasy.

**Site 2: Measurements**

S.B. lab no.	N.Z. lab no.	Horizon desig.	Sample depth (cm)	Isotopes		Chemical Properties								
				$\Delta^{14}\text{C}$ ‰	$\delta^{13}\text{C}$ ‰	C %	N %	C/N	CEC† me %	pH (H <sub>2</sub> O) dry soil	pH (H <sub>2</sub> O) moist soil	Total P mg %	Org. P mg %	Inorg. P mg %
8848A	5986	Ap	0–9	142.8 ± 4.4	-26.6	4.6	0.30	15	13.5		6.0	57	29	28
8848B	5987	Ea	11–19	58.6 ± 5.8	-25.9	4.1	0.31	13	12.3		6.1	63	22	41
8848C	5988	Bh	21–37	0.4 ± 7.2	-25.9	2.8	0.16	18	13.1		6.2	43	21	22
8848D		Bs	40–50			2.0	0.11	18	8.6		6.1	39	12	27
8848E		2Cu1	52–78			0.8	0.04	*	4.9		6.4	40	2	38
8848F		3Cu2	80–105			0.7	0.03	*	3.7		6.5	37	1	36
8848G		3Cu3	105–107			0.8	0.08	10	7.2		6.6	23	6	17
8848H		3Cu4	109–119			1.2	0.05	24	7.2		6.4	34	20	14

S.B. lab no.	N.Z. lab no.	Horizon desig.	Sample depth (cm)	Physical–Mineralogical Properties					Clay minerals (topsoil only)	Comments
				Dry bulk density t/m <sup>3</sup>	Stones (% v/v)	% Sand 2.0–0.02 mm	% Silt 0.02–0.002 mm	% Clay <0.002 mm		
8848A	5989	Ap	0–9	0.89		80	18	2	Predominantly: Volcanic glass (Allophane at depth)	$\Delta^{14}\text{C}$ and $\delta^{13}\text{C}$ analyses of soil horizons are from a Tihoi Soil at a nearby site under pasture, altitude 600 m. Horizons, depths corresponding to these C isotope analyses: Ap 0–8 cm, Bsh 8–27 cm, Bs 27–44 cm. Blank spaces in tables indicate no available data. *C/N ratio not calculated where N < 0.05. †CEC = cation exchange capacity; me % = c mol(+) kg <sup>-1</sup>
8848B	5987	Ea	11–19	0.72		75	22	3		
8848C		Bh	21–37	0.90		89	7	4		
8848D	5988	Bs	40–50	1.17		89	7	4		
8848E		2Cu1	52–78	0.91		93	6	1	Small am'ts of: smectite and kaolin	
8848F		3Cu2	80–105			92	7	1		
8848G		3Cu3	105–107			71	19	10		
8848H		3Cu4	109–119			73	16	11		

**Site 3: Location and Soil Description**

Soil name: Rotomahana Sandy Loam		Lab no. SB07672	
Site, Soil Descriptions and Analytical Data		Date sampled: 22 Sept. 1960	
Latitude:	38°17'S	Longitude:	176°23'
N.Z. genetic classification:	Recent soil	NZSC:	Typic tephric recent soil
Survey:	1962 conference site	Region:	Rotorua, North Island
Location:	Waimangu Rd, 0.4 km west along road from tearooms, east side of road reserve		
Topdressing:	Not known	Elevation:	460 m
Annual Rain:	1270 mm	Mean annual temp:	13°C
Landform:	Ridge	Slope:	5°
Microrelief:	Flat	Topsoil loss:	Not known
Slope movement:	Not known	Land use:	Unused natural
Drainage:	Well	Aspect:	270°
Improvements:	Nil		
Vegetation:	Bracken fern, mahoe, coprosma, kamahi, lupin		
Parent Materials:	Rotomahana mud, hydrothermally altered rhyolitic ejecta from Lake Rotomahana's 1886 eruption		
Notes:			

Horizon	Depth (cm)	Horizon description
L	-2-1	Litter; mainly bracken remains.
H	-1-0	Brown organic matter.
Ah1	0-3	Black (10YR 2/1) sandy loam; moderately firm soil strength; moderately developed fine granular structure; many live roots; sharp boundary.
Ah2	3-8	Weak red (2.5YR 5/2) silt loam; moderately weak soil strength; moderately developed fine granular plus medium nut structure; many live roots; diffuse boundary.
Cu	8-74	Light grey (5Y 7/2) bands; pale olive grey (5Y 6/2) bands; silt loam.
2bAh	74-82	Black (10YR 2/1) fine sandy loam; moderately firm soil strength; weakly developed fine granular structure;
2bAB	82-92	Very dark brown (10YR 2/2) fine sandy loam; moderately weak soil strength; moderately developed fine granular plus medium nut structure
3bCu	92-98+	Loose soil strength; single grain; pumice travel
2bAB	82-92	Very dark brown (10YR 2/2) fine sandy loam; moderately weak soil strength; moderately developed fine granular plus medium nut structure.
3bCu	92-98+	Loose soil strength; single grain; pumice gravel.

**Site 3: Measurements**

S.B. lab no.	N.Z. lab no.	Horizon desig.	Sample depth (cm)	Isotopes		Chemical Properties								
				$\Delta^{14}\text{C}$ ‰	$\delta^{13}\text{C}$ ‰	C %	N %	C/N	CEC† me %	pH (H <sub>2</sub> O) dry soil	pH (H <sub>2</sub> O) moist soil	Total P mg %	Org. P mg %	Inorg. P mg %
		L												
		H												
7672A		Ah1	0-3			9.9	0.54	18	32.8	5.7	5.8	67	36	31
7672B		Ah2	3-8			1.3	0.07	19	11.3	5.5	5.8			
7672C	5729	Cu	30-61	-436.9 ± 13.9	-26.8	0.2	0.02	*	14.0	6.2	6.4	29	0	29
7672D	5727	2bAh	74-79	-33.8 ± 3.8	-25.8	3.6	0.21	17	18.3	6.1	5.4			
7672E	5728	2bAB	84-91	-94.9 ± 5.3	-24.8	3.4	0.14	24	12.9	6.2	5.5			
7672F		Ah	0-8			1.9	0.12	16	11.1	5.7	5.9			

## Site 3: Measurements (Continued)

S.B. lab no.	N.Z. lab no.	Horizon desig.	Sample depth (cm)	Physical-Mineralogical Properties					Clay minerals (topsoil only)	Comments
				Dry bulk density t/m <sup>3</sup>	Stones (% v/v)	% Sand 2.0-0.02 mm	% Silt 0.02-0.002 mm	% Clay <0.002 mm		
		L								
		H							Predominantly: smectite  Small amounts of: mica, allophane, kaolin  7672F was a bulk sample from 2-10 cm of the topsoil. Dry bulk density figures for 7672A,B were assumed from the measured value for 7672F. Blank spaces in tables indicate no available data. *C/N ratio not calculated where N < 0.05. †CEC = cation exchange capacity; me % = c mol(+) kg	
7672A		Ah1	0-3	0.69		61	25	12		
7672B		Ah2	3-8	0.69						
7672C	5729	Cu	30-61	1.20		50	36	13		
7672D	5727	2bAh	74-79	0.69						
7672E	5728	2bAB	84-91							
7672F		Ah	0-8	0.69						

## Site 5: Location and Soil Description

Soil name: Egmont Black Loam		Lab no: SB07597	
Site, Soil Descriptions and Analytical Data			Date sampled: 1 Mar. 1960
Latitude:	39°37'S	Longitude:	174°18'
N.Z. genetic classification:	Yellow brown loam	NZSC:	Typic Orthic allophanic soil
Survey:	N.Z. soils	Region:	Hawera, North Island
Location:	Rear of Whareroa Social Hall, Whareroa Rd.		
Topdressing:	Fertilized	Elevation:	91 m
Annual rain:	1020 mm	Mean annual temp:	14°C
Landform:	Rolling country	Slope:	Aspect:
Microrelief:	Flat	Topsoil loss:	Not known
Slope movement:	Nil	Land use:	Long-term grass
Drainage:	Well		
Improvements:	Ploughed		
Vegetation:	Cocksfoot, sweet vernal, ryegrass		
Parent materials:	Fine andesitic ash		
Notes:	Mount Taranaki (Egmont) last erupted in AD 1755		
Horizon	Depth (cm)	Horizon description	
Ap	0-20	Black (10YR 2/1) loam; moderately weak soil strength; moderately developed fine nut structure; no mottles; abundant live roots; distinct irregular boundary.	
Bw1	20-46	Brown (10YR 5/3) loam; very weak soil strength; weakly developed medium nut breaking to crumb structure; no mottles; common live roots; some small pieces of weathered pumice; distinct wavy boundary.	
Bw2	46-66	Yellowish brown (10YR 5/4) silt loam; moderately firm soil strength; massive plus weakly developed fine blocky structure; no mottles; common live roots; some small pieces of weathered pumice; indistinct boundary.	
BC	66-70+	Dark yellowish brown (10YR 4/4) silt loam; very firm soil strength; massive; no mottles; few live roots; some small pieces of weathered pumice.	

## Site 5: Measurements

S.B. lab no.	N.Z. lab no.	Horizon desig.	Sample depth (cm)	Isotopes		Chemical Properties								
				$\Delta^{14}\text{C}$ ‰	$\delta^{13}\text{C}$ ‰	C %	N %	C/N	CEC† me %	pH (H <sub>2</sub> O) dry soil	pH (H <sub>2</sub> O) moist soil	Total P mg %	Org. P mg %	Inorg. P mg %
7597A		Ap	0-8			12.3	0.93	13	36.9	5.7	6.0	256	136	120
7597B	2345	Ap	8-15	43.2 ± 7.3	-26.2	8.7	0.71	12	31.2	6.2	6.0	238	125	113
7597C	2346	Bw1	20-36	-97.5 ± 4.5	-24.7	3.6	0.40	9	19.1	6.3	6.4	248	107	141
7597D	2347	Bw2	46-56	-176.5 ± 6.4	-25.5	1.7	0.20	9	13.2	6.4	6.4	167	57	110
7597E	2348	BC	74-89	-457.5 ± 5.9	-25.0	1.1	0.15	7	14.2	6.4	6.4	127	50	77

## Site 5: Measurements (Continued)

S.B. lab no.	N.Z. lab no.	Horizon desig.	Sample depth (cm)	Physical-Mineralogical Properties					Clay minerals (topsoil only)	Comments
				Dry bulk density t/m <sup>3</sup>	Stones (% v/v)	% Sand 2.0-0.02 mm	% Silt 0.02-0.002 mm	% Clay <0.002 mm		
7597A		Ap	0-8	0.66		55	23	22	Predominantly: Allophanes, volcanic glass, HIV Small amounts of: halloysite	Blank spaces in tables indicate no available data. †CEC = cation exchange capacity; me % = c mol(+) kg <sup>-1</sup>
7597B	2345	Ap	8-15	0.82		56	26	18		
7597C	2346	Bw1	20-36	0.74		65	26	9		
7597D	2347	Bw2	46-56	0.83		67	21	12		
7597E	2348	BC	74-89	0.85						

## Site 10: Location and Soil Description

Soil name: Judgeford Silt Loam

Lab no. SB07536

## Site, Soil Descriptions and Analytical Data

Date sampled: 7 Sept. 1959

Latitude:	41°07'S	Longitude:	174°57'		
N.Z. genetic classification:	Yellow-brown earth	NZSC:	Pallic firm brown soil	FAO:	Dystric cambisol
Survey:	Paekakariki	Region:	Hutt County, North Island		
Location:	Judgeford, Abbots farm - 2.4 km east of Pauatahanui				
Topdressing:	Not known	Elevation:	60 m	Mean annual temp:	13°C
Annual rain:	1145 mm				
Landform:	Ridge of hill in rolling country				
Microrelief:	Flat	Slope:	5° convex crest	Aspect:	270°
Slope movement:	Creep mantle	Topsoil loss:	Not known		
Drainage:	Moderately good	Land use:	Long-term grass		
Improvements:	Ploughed				
Vegetation:	Browntop, Yorkshire fog, Cocksfoot, Bracken fern				
Parent materials:	Moderately weathered loess from greywacke plus volcanic ash				
Notes:					
Horizon	Depth (cm)	Horizon description			
Ap1	0-8	Dark greyish brown (10YR 4/2) silt loam; moderately weak soil strength; moderately developed fine granular structure; no mottles; many live roots; many fine cast granules; indistinct boundary.			
Ap2	8-23	Brown to dark brown (10YR 4/3) silt loam; moderately weak soil strength; moderately developed fine nut structure; no mottles; many live roots; some fine cast granules; indistinct boundary.			
Bw	23-43	Dark yellowish brown (10YR 4/4) silt loam; moderately firm soil strength; moderately developed fine nut structure; no mottles; many live roots; distinct boundary.			
Bw(f)	43-53	Dark yellowish brown (10YR 4/4) & light olive brown (2.5Y 5/4) silt loam; moderately firm soil strength; weakly developed fine blocky breaking to massive structure; many coarse dark brown (7.5YR 3/2) mottles; no live roots; indistinct boundary.			
BC(f)	53-74	Light olive brown (2.5Y 5/4) silt loam; moderately firm soil strength; weakly developed medium blocky structure; many fine strong brown (7.5YR 5/6) mottles; no live roots; indistinct boundary.			
Cu	74-80+	Light olive brown (2.5Y 5/4) silt loam; moderately firm soil strength; massive; no mottles; no live roots.			

## Site 10: Measurements

S.B. lab no.	N.Z. lab no.	Horizon desig.	Sample depth (cm)	Isotopes		Chemical Properties								
				$\Delta^{14}\text{C}$ ‰	$\delta^{13}\text{C}$ ‰	C %	N %	C/N	CEC* me %	pH (H <sub>2</sub> O) dry soil	pH (H <sub>2</sub> O) moist soil	Total P mg %	Org. P mg %	Inorg. P mg %
7536A	2352	Ap1	0-8	-34.9 ± 6.8	-27.5	7.1	0.48	15	19.9	4.5	5.9	49	36	13
7536B	2353	Ap2	8-20	-44.0 ± 6.8	-27.1	4.2	0.31	14	15.5	5.4	5.9	35	22	13
7536C	2354	Bw	25-43	-140.2 ± 6.4	-26.0	1.9	0.12	16	10.5	5.4	5.9	24	13	11
7536D	2355	Bw(f)	46-53	-350.0 ± 6.6	-26.1	1.0	0.08	13	8.5	5.6	5.9	28	16	12
7536E	2356	BC(f)	58-74	-457.1 ± 6.1	-24.9	0.7	0.07	10	9.4	5.5	5.6	33	18	15
7536F		Cu	76-94			0.7	0.06	12	6.0	5.3	5.8	45	23	22

S.B. lab no.	N.Z. lab no.	Horizon desig.	Sample depth (cm)	Physical-Mineralogical Properties					Clay minerals (topsoil only)	Comments
				Dry bulk density t/m <sup>3</sup>	Stones (% v/v)	% Sand 2.0-0.02 MM	% Silt 0.02-0.002 MM	% Clay <0.002 mm		
7536A	2352	Ap1	0-8	0.94		31	33	23	Predominantly: vermiculite  Small amounts of: mica-vermiculite	Blank spaces in tables indicate no available data. *CEC = cation exchange capacity; me % = c mol(+) kg <sup>-1</sup>
7536B	2353	Ap2	8-20	1.05		32	36	32		
7536C	2354	Bw	25-43	1.31		39	33	22		
7536D	2355	Bw(f)	46-53	1.38		41	32	27		
7536E	2356	BC(f)	58-74	1.42		39	34	27		
7536F		Cu	76-94							

## Site 13: Location and Soil Description

Soil name: Okarito Peaty Loam		Lab no. SB07719		
Site, Soil Descriptions and Analytical Data				Date sampled: 7 Feb. 1961
Latitude:	42°43'S	Longitude:	170°59'	
N.Z. genetic classification:	Gley Podzol	NZSC:	Peaty-silt-mantled Perch-gley Podzol	FAO: Gleyic podzol
Survey:	1962 Conference site	Region:	Westland, South Island	
Location:	Aerodrome Terrace, Hokotika, 155 m east of south runway			
Topdressing:	Nil			
Annual rain:	2795 mm	Elevation:	30 m	Mean annual temp: 11°C
Landform:	Terrace in coastal system	Aspect:		Slope:
Microrelief:	Flat	Topsoil loss:	Not known	
Slope movement:	Not known	Land use:	Unused natural	
Drainage:	Moderately well			
Improvements:	Nil			
Vegetation:	Blechnum minor, Sedge, Lycopodium, Umbrella fern, Moss			
Parent materials:	Silty alluvium or loess over greywacke, schist, granite gravels			
Notes:				
Horizon	Depth (cm)	Horizon description		
Oh1	0-10	Dark reddish brown (5YR 3/2) peaty loam; no mottles; sharp smooth boundary.		
Oh2	10-30	Dark reddish brown (5YR 3/2) peaty silt loam; slightly sticky; no mottles; distinct boundary.		
Er	30-56	Grey (5Y 5/1) silt loam; very firm soil strength; massive; no mottles; many fine live roots; distinct boundary.		
bAh	56-84	Dark brown (10YR 3/3) fine sandy loam; moderately firm soil strength; massive; no mottles; few rounded gravels; indistinct boundary.		

**Site 13: Location and Soil Description (Continued)**

bEr1	84-97	Weak red (2.5YR 4/2) loamy very fine sand; very firm soil strength; massive; no mottles; indistinct boundary.
bEr2	97-102	Grey (5Y 5/1) loamy very fine sand; moderately firm soil strength; massive; no mottles; indistinct boundary.
2bBh	102-112	Dark brown (10YR 3/3) gravelly sand; loose soil strength; no mottles; many strongly weathered subangular stones.
2bBfm/Cu	112-127	Dark reddish brown (2.5YR 3/4) strongly cemented; no mottles; continuous iron-pan; sandwiching loose gravelly sand. single grain; no mottles; many stones.
3bCu	127-130+	Gravelly sand; loose soil strength; single grain; no mottles; many stones.

**Site 13: Measurements**

S.B. lab no.	N.Z. lab no.	Horizon desig.	Sample depth (cm)	Isotopes		Chemical Properties								
				$\Delta^{14}\text{C}$ ‰	$\delta^{13}\text{C}$ ‰	C %	N %	C/N	CEC* me %	pH (H <sub>2</sub> O) dry soil	pH (H <sub>2</sub> O) moist soil	Total P mg %	Org. P mg %	Inorg. P mg %
7719A		Oh1	0-8			30.8	1.28	24	66.4	4.1	4.4	58	47	11
7719B	2361	Oh2	13-30	-24.7 ± 6.9	-27.6	19.7	0.59	33	30.8	4.1	4.7	21	10	11
7719C	2362	Er	33-48	-317.0 ± 5.9	-27.9	5.2	0.12	43	15.3	4.7	5.1	14	7	7
7719D	2363	bAh	56-84	-646.6 ± 3.9	-28.0	8.9	0.19	47	27.5	4.9	4.9	36	30	6
7719E	2364	bEr	84-97	-667.8 ± 4.7	-29.2	4.3	0.13	33	15.6	4.8	4.8	24	18	6
7719F	2365	2bBh	102-112	-442.4 ± 5.4	-27.8	4.0	0.09	44	21.2	4.8	5.0	38	9	29

S.B. lab no.	N.Z. lab no.	Horizon desig.	Sample depth (cm)	Physical-Mineralogical Properties					Clay minerals (topsoil only)	Comments
				Dry bulk density t/m <sup>3</sup>	Stones (% v/v)	% Sand 2.0-0.02 mm	% Silt 0.02-0.002 mm	% Clay <0.002 mm		
7719A		Oh1	0-8	0.38					Predominantly: quartz, mica Small amounts of: mica-vermiculite, vermiculite, kaolin	Dry bulk density figures for 7719A, B were estimated from 12 Oh horizons with similar carbon contents at similar depths. Mean = 0.38 Std Dev = 0.17 Blank spaces in tables indicate no available data. *CEC = cation exchange capacity; me % = c mol(+) kg <sup>-1</sup>
7719B	2361	Oh2	13-30	0.38		49	43	8		
7719C	2362	Er	33-48	0.91		56	34	10		
7719D	2363	bAh	56-84	0.76		60	28	12		
7719E	2364	bEr	84-97	1.03		63	31	6		
7719F	2365	2bBh	102-112		20	80	15	5		

**Site 16: Location and Soil Description**

Soil name: Carrick Fine Sandy Loam

Lab no. SB08712

Site, Soil Descriptions and Analytical Data

Date sampled: 19 Nov. 1971

Latitude:	45°27'S	Longitude:	169°13'
N.Z. genetic classification:	Yellow-brown earth	N.Z.S.C.:	Mottled acid brown soil
Survey:	Tussock Grassland Study	FAO:	Dystric cambisol
Location:	Top of Waikaia Bush Rd, 370 m below crossing of Boulder Ck branch of Shingle Creek, 6 m from SW side of Rd.		
Topdressing:	Nil		
Annual rain:	1400 mm	Elevation:	1460 m
Landform:	Hill country	Mean annual temp:	2°C
Microrelief:	Flat	Slope:	10°
		Aspect:	45°

**Site 16: Location and Soil Description (Continued)**

Slope movement:	Not known	Topsoil loss:	Not known
Drainage:	Moderately well	Land use:	Rough grazing
Improvements:	Nil		
Vegetation:	<i>Chionochloa rigida</i> and <i>macra</i> , <i>Poa colensoi</i> , Open tussock		
Parent materials:	Moderately weathered Schist		
Notes:	A ? on the "Soil Name" indicates the series name for this profile is tentative, however, all other data is considered to be valid. Hor 3 10YR 4/3. Hor 4 5Y 5/2–6/2 (Gley areas)		

Horizon	Depth (cm)	Horizon description
Ah1	0–1	Very dark greyish brown (10YR 3/2) silt loam; moderately developed crumb structure; abundant live roots; distinct boundary.
Ah2	1–4	Brown to dark brown (10YR 4/3) silt loam; moderately developed crumb structure; many live roots; indistinct boundary.
Bw	4–9	Yellowish brown (10YR 5/4) silt loam; weakly developed nut plus moderately developed crumb structure; few stones; few live roots; distinct boundary.
Bw(f)	9–20	Light olive brown (2.5Y 5/4) silt loam; weakly developed medium nut structure; faint strong brown (7.5YR 5/6) mottles; weakly weathered schist stones; indistinct boundary.
BC	20–36	Light olive brown (2.5Y 5/4) fine sandy loam; weakly developed blocky structure; yellowish brown (10YR 5/6) coatings; abundant stones.

**Site 16: Measurements**

S.B. lab no.	N.Z. lab no.	Horizon desig.	Sample depth (cm)	Isotopes		Chemical Properties									
				$\Delta^{14}\text{C}$ ‰	$\delta^{13}\text{C}$ ‰	C %	N %	C/N	CEC* me %	pH (H <sub>2</sub> O) dry soil	pH (H <sub>2</sub> O) moist soil	Total P mg %	Org. P mg %	Inorg. P mg %	
		Ah1	0–1												
8712A	4570	Ah2	1–4	85.0 ± 8.6	–25.7	4.9	0.32	15	19.5		4.6	88	65	23	
8712B	4571	Bw	4–9	–16.8 ± 3.9	–25.8	3.7	0.21	18	18.8		4.6	71	52	19	
8712C	4572	Bw(f)	9–20	–79.2 ± 3.8	–25.8	2.9	0.19	15	18.1		4.7	61	45	16	
8712D	4573	BC	20–36	–117.2 ± 3.7	–25.9	2.3	0.16	14	17.5		4.8	57	44	13	

S.B. lab no.	N.Z. lab no.	Horizon desig.	Sample depth (cm)	Physical–Mineralogical Properties						Clay minerals (topsoil only)	Comments
				Dry bulk density (t/m <sup>3</sup> )	Stones (% v/v)	% Sand 2.0–0.02 (mm)	% Silt 0.02–0.002 (mm)	% Clay <0.002 (mm)			
		Ah1	0–1								
8712A	4570	Ah2	1–4	0.79						Predominantly: mica-vermiculite	Blank spaces in tables indicate no available data. *CEC = cation exchange capacity; me % = c mol(+) kg <sup>–1</sup>
8712B	4571	Bw	4–9	0.88							
8712C	4572	Bw(f)	9–20	1.03							
8712D	4573	BC	20–36	1.19							

## Site 19: Location and Soil Description

Soil name: Tima Silt Loam		Lab no. SB08856	
Site, Soil Descriptions and Analytical Data			Date sampled: 29 Mar. 1973
Latitude:	45°24'S	Longitude:	169°25'
N.Z. genetic classification:	Yellow-grey earth	NZSC:	Typic Laminar Pallic soil
Survey:	Tussock Grasslands	FAO:	Dystric Cambisol
Location:	Top of Knobby Range Rd. 20 km NE of Roxburgh; Roadside reserve		
Topdressing:	Nil		
Annual rain:	625 mm	Elevation:	750 m
Landform:	Ridge	Mean annual temp:	8°C
Microrelief:	Flat	Slope:	0° concave
Slope movement:	Not known	Topsoil Loss:	Not known
Drainage:	Moderately well	Land use:	Rough grazing
Improvements:	Nil		
Vegetation:	Lowland tussock grassland, <i>Festuca novae-zelandiae</i> , <i>Chionocloa rigida</i> , <i>Poa colensoi</i> .		
Parent materials:	Weakly weathered schist loess over schist bedrock.		
Notes:	A ? on the "Soil name" indicates that the series name for this profile is tentative; however, all other data is considered to be valid. Fragipan+drainage semi-impermeable. Erosion slight; topsoil loss in past, nil now. Microfeatures = exhumed tor landscape. Hor 1 few casts. Hor 2 many abundant casts. Hor 3 +5y5/2 with some nut structure, common casts. Hor 4 +2.54/4, very few casts, few fine Fe mottles, rare clay skin. Hor 5 +5y6/2 matcol.		
Horizon	Depth (cm)	Horizon description	
Ah1	0-13	Dark greyish brown (10YR 4/2) fine sandy loam; moderately developed medium nut plus crumb structure; many live roots; indistinct boundary.	
Ah2	13-22	Dark greyish brown (10YR 4/2) fine sandy loam; moderately developed medium plus coarse nut structure; common live roots; indistinct boundary.	
Bw(f)	22-32	Dark greyish brown (2.5Y 4/2) fine sandy loam; weakly developed moderately developed coarse blocky structure; many medium faint dark yellowish brown (10YR 4/4) mottles; few live roots; indistinct boundary.	
BCx	32-50	Olive grey (5Y 5/2) fine sandy loam; weakly developed coarse prismatic plus moderately developed coarse blocky structure; few fine mottles; few live roots; distinct boundary.	
Cu	50-70	Pale yellow (5Y 7/3) sandy loam; massive.	

## Site 19: Measurements

S.B. lab no.	N.Z. lab no.	Horizon desig.	Sample depth (cm)	Isotopes					Chemical Properties					
				$\Delta^{14}\text{C}$ ‰	$\delta^{13}\text{C}$ ‰	C %	N %	C/N	CEC† me %	pH (H <sub>2</sub> O) dry soil	pH (H <sub>2</sub> O) moist soil	Total P mg %	Org. P mg %	Inorg. P mg %
8856A	4533	Ah1	0-13	145.0 ± 4.4	-26.2	2.3	0.19	12	11.1		5.9	89	38	51
8856B	4534	Ah2	13-22	14.8 ± 3.5	-26.3	1.7	0.15	11	11.4		6.2	81	42	39
8856C	4535	Bw(f)	22-32	-46.9 ± 5.3	-24.8	1.0	0.09	11	9.5		6.1	54	31	23
8856D	4536	BCx	32-50	-264.4 ± 6.7	-26.2	0.6	0.06	10	9.8		6.1	53	26	27
8856E	4537	Cu	50-70	-378.8 ± 6.3	-26.0	0.2	0.02	*	5.1		6.2	67	8	59



**Site 19: Measurements (Continued)**

S.B. lab no.	N.Z. lab no.	Horizon desig.	Sample depth (cm)	Physical-Mineralogical Properties					Clay minerals (topsoil only)	Comments
				Dry bulk density t/m <sup>3</sup>	Stones (% v/v)	% Sand 2.0-0.02 mm	% Silt 0.02-0.002 mm	% Clay <0.002 mm		
8856A	4533	Ah1	0-13	1.12	0	61	20	19	Predominantly: mica  Small amounts of: mica-vermiculite, mica-HIV, kaolin	Blank spaces in tables indicate no available data.  *C/N ratio not calculated where N < 0.05. †CEC = cation exchange capacity; me % = c mol(+) kg <sup>-1</sup>
8856B	4534	Ah2	13-22	1.25	0	59	22	19		
8856C	4535	Bw(f)	22-32	1.45	0	56	24	20		
8856D	4536	BCx	32-50	1.69	0	58	21	21		
8856E	4537	Cu	50-70	1.47	0	72	24	4		

## THE DATABASE

### File Structure and Data Validity

The unique identifier for each record on the database is the **Laboratory Number**. The information on file for each sample is as concise as possible, but in order to allow the maximum degree of flexibility when searching the database, each record has an average of at least 20 indexes. The database presently consists of 36 fields ranging in size from 1 to 230 characters, plus a text field containing up to 8000 characters (Fig. 1). Each record requires only *ca.* 1 kbyte of disk storage space.

LAB. No.	: <b>Lab. Number</b>	SUBMITTERS:	<b>Submitter</b>
FIELD No.	: <b>Field Number</b>	UPDATED:	95.09.25 (YY.MM.DD)
COLLECTORS	: <b>Collectors</b>		
MATERIAL	: <b>Material</b>	COLLECTION DATE:	<b>col. date</b> (YY.MM.DD)
TAXA	: <b>Taxa (Genus Species); identified by</b>		
ENCL MAT'L	: <b>Enclosing material</b>		
LOCALITY	: <b>Locality</b>		
COORDINATES	: <b>Latitude</b> <b>N</b>	<b>Longitude</b> <b>W</b>	UTM GRID No.: 99 999999 9999999
ELEVATION	: <b>Elevation</b> <b>m</b>	NTS No.: 999 Z/99	AGE (NORM.): <b>Normalized to -25%</b>
AGE (UNCOR.)	: <b>Uncorrected Age ± 2σ</b>	$\delta^{13}\text{C} = $ <b>99.99‰</b>	AGE (CORR.): <b>Corrected to 0.0%</b>
SIGNIFICANCE	: <b>Significance</b>		
ASSOC. DATES	: <b>Associated Dates</b>		
ADDITIONAL INFORMATION	: <b>Additional Information</b>		
REFERENCES	: <b>References 19xx</b> <b>References</b> <b>References</b> <b>References</b> <b>References</b> <b>References</b> <b>References</b>		
*****			
Dec. Latitude	: <b>00.0000°N</b> , Dec. Longitude	: <b>000.0000°W</b>	
COMMENTS	: <b>by the Submitter - upto 8k characters of free-form text</b>		

Fig. 1. Data record format. **Data fields** are shaded; **indexed words** are bolded.

Data verification is the responsibility of the submitter. The Laboratory initiates the addition of new records to the file, but once on file, existing data is modified only with the express knowledge and consent of the submitter. Although the database has been developed as a scientific tool for the research community, its completeness and overall utility depend largely upon altruism, *i.e.*, the clientele's ongoing commitment to add and validate data.

### Data Searches and Retrievals

Selected fields such as the **submitter's name**, **locality**, coordinates (**latitude** and **longitude**), sample **material**, **taxon** and **significance** have been designated as KEY fields, and entries within these fields have been selectively indexed to facilitate searches, although non-index searches can be processed on a character string in any field. Because of the speed of the searches, the database is typically interrogated in an interactive mode. A request can be simple—for example, "all dates on paleosols" (requires one KEY; select **material** = paleosol); a more complex selection could be "only those dates on peat in Nova Scotia related to Holocene deglaciation" (requires four KEYS; select **material** = peat, **locality** = Nova Scotia; **significance** = deglaciation; and **age** = <10 ka).

## GEOLOGICAL SURVEY OF CANADA SOIL DATABASE

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**ABSTRACT.** The Geological Survey of Canada (GSC) has developed, over the past decade, a user-oriented database, Date Locator File, of Canadian samples dated by the  $^{14}\text{C}$  technique. This database presently contains >3500 soil and soil-related dates. The primary category in this suite of dates is peat, as a large portion of the Canadian landscape is covered with this type of organic soil. The data is available *gratis* to all researchers in a large variety of formats from simple lists to complex tables for inclusion in publications. The site localities can also be plotted on base maps suitable for publication. The database is actively augmented on an ongoing basis, but to continue to be relevant, it depends largely on the altruism of the scientific community.

### INTRODUCTION

Like most scientific disciplines, Quaternary research has undergone an information explosion in the last few decades. Since the inception of the radiocarbon dating technique more than 45 years ago, the number of samples dated by this technique has steadily increased, especially since the advent of accelerator mass spectrometry (AMS) dating. The large quantity of dates now available has made it increasingly difficult for Quaternary researchers to locate and manage the existing information.

More than a decade ago the GSC Radiocarbon Dating Laboratory developed a database, Date Locator File, which was designed specifically to address the requirements of the laboratory's scientific clientele. We have been using this database to manage the samples dated at the GSC and published in our annual date lists. The utility of this computerized information (database) is only now becoming evident as regional and global syntheses are developed.

### THE GSC DATE LOCATOR FILE

The Date Locator File is a computerized database of selected information on published  $^{14}\text{C}$ -dated samples. Because the database has been developed to be of use to the Laboratory's clientele, it has been based on the Geological Survey of Canada Radiocarbon Age Data Form and contains primarily field parameters, rather than laboratory variables. The file does not attempt to mimic  $^{14}\text{C}$  date lists or other types of reports, but instead provides the most pertinent information in as concise a format as possible, yet allowing for extensive descriptive text. When a date of interest is identified by a search of the database, the user can consult both the references and the submitter to obtain additional information on the sample.

### HISTORICAL DEVELOPMENT

In 1982, an interactive proto-database was developed on a mainframe computer using the Scientific Information Retrieval (SIR) database management system, but the increasing availability of microcomputers and associated database programs provided an alternative mode for developing the database. By September 1983, a dBASE prototype of the database was installed on a VICTOR 9000 microcomputer; early in 1985, the database was transferred to the database program CARDBOX-Plus because the database involves primarily text management and does not require the capability for calculations. For the novice user, this program has a very short learning curve and incorporates all the capabilities required to manage textual information. Data entry and verification as well as enhancement of the database capabilities have continued to the present.

The few search limitations that exist are primarily acceptable ones. The more specific a request, the greater the likelihood that records will not be included because of missing data. As with all databases, the search terms must match exactly, although wildcards can be used when there is a known variability in the spelling of a word, *e.g.*, "paleosol" and "palaeosol". If a term of interest occurs in a text field, this text field can be searched, although this is seldom done because of the additional time required to process the request, especially on slower processors (*e.g.*, 80286). In most cases, the required selection can be roughly obtained using KEYS such as coordinates and then a text-search made on the reduced data set.

#### **DATA REPORTS**

Once a retrieval has been made, the information can be printed or transferred in a wide variety of formats, including the complete data record, and tables or lists of data for publication. For example, the most pertinent data can be printed onto index cards for the client's reference file. Standard formats have been developed of the commonly required information, but customized reports can be designed to fulfil almost any requirement. After a search has been made, the data can be sorted and then transferred directly to a word processor for incorporation into a manuscript. This facility eliminates the necessity for retyping the information, and proofreading and editing the tables or lists.

#### **DATA TRANSFERS**

The master Date Locator File is maintained at the GSC, but extracted subsets of the database, in a variety of formats (*e.g.* ASCII, CSV, tab-delimited), can be transmitted to other locations either electronically or on diskette for the use on compatible microcomputers, or converted to be used on non-DOS machines. After this type of transfer, the maintenance of the data subset is the responsibility of the user. To avoid a multiplicity of databases that may not correspond to the master file, the wholesale transfer of the database is not usually undertaken. Periodic upgrades can be requested from the GSC and reciprocal data-sharing arrangements are encouraged between the GSC and active users.

#### **PLOTTING**

The master file contains the site coordinates, both as the submitter provided them and decimal degrees. The retrieval of subsets of data with decimal degrees facilitates the plotting of site locations on graphics or in Geographic Information Systems (GIS) applications. As an example of this type of plotting, Figure 2 shows the distribution of all dated peat samples in Canada on the database. In addition, the complete UTM coordinates are available on file, and can be used for plotting overlays to verify the original site locations, and thus the latitude and longitude provided by the submitter.

#### **SOIL DATABASE**

Because the Date Locator File has been developed as a tool for Quaternary researchers working in Canada, it includes dates on soil-related materials as old as 40 ka BP. The materials on this database include organics associated with soils and paleosols (including paleosols developed on, and organics related to tephra of various ages), and organic soils (peat) that cover a very large part of the Canadian landscape are a key component of this database. The database is presently comprised of 4350 records on samples from Canada and adjoining states and countries. Because peat is an important entity within the Canadian landmass an Atlas of Peatland Development During the Holocene has been developed (Dyke 1995a). The dating of basal peats as a minimum age estimate of deglaciation in various regions of Canada has indicated that the development of peatlands has occurred during

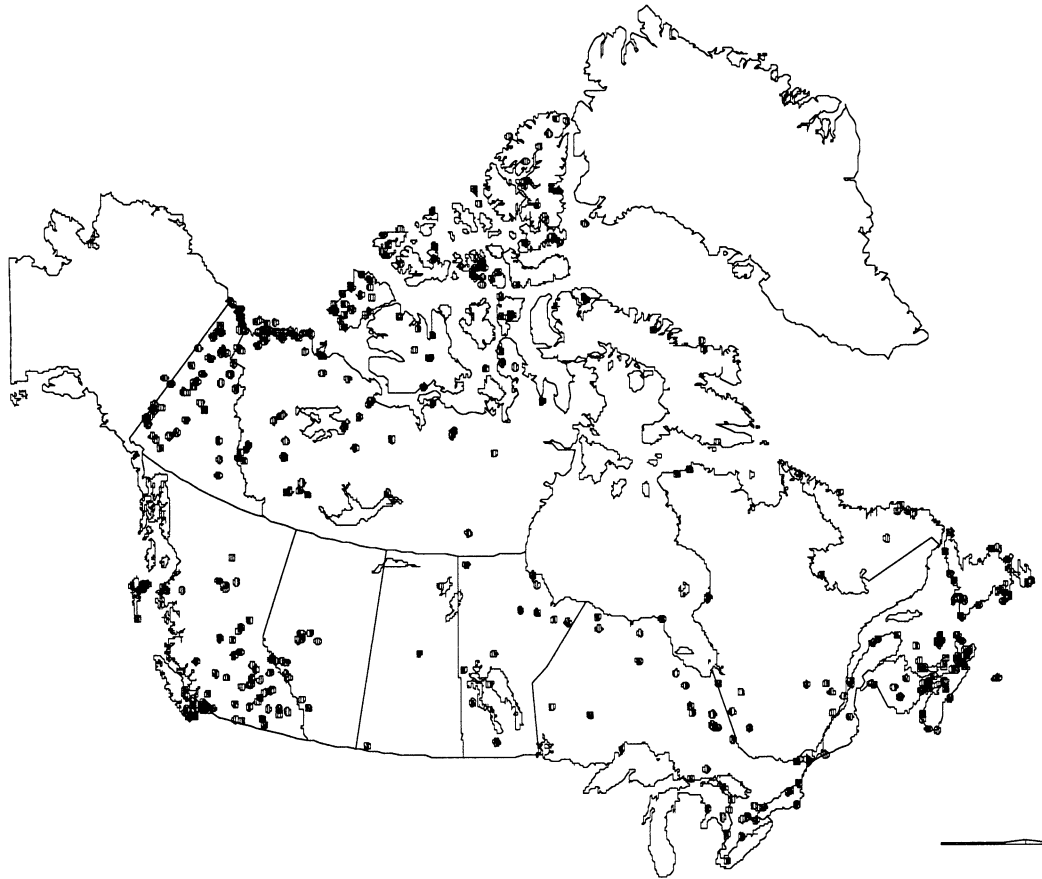


Fig. 2. Dated peat samples in Canada

the Early to Late Holocene and follows a regional pattern of initiation (*cf.* Dyke 1995b for detailed treatment of this data).

In addition to the foregoing uses of the database, the GSC has initiated a project dealing with the distribution and stratigraphy of peatlands (I. Kettles, personal communication 1995) to identify paleo-environmental changes, and geochemical pathways of heavy metals and anthropogenic contaminants, thereby providing a basis for mapping and characterizing Canadian peat deposits. Many of the Provincial Geological Surveys and Departments of Mines have also initiated programs to ascertain the distribution and thickness of their peatland resources. Dr. Kettle's project will also assess the release of both CO<sub>2</sub> and methane from Canadian peatlands, as it is anticipated that global warming will have a marked worldwide effect on peatlands by increasing the release of "greenhouse" gases from the oxidation of existing peats. On the other hand, global warming, in favorable environments, may cause an increase in the productivity of some peatlands (see Watson *et al.* 1990 for an extensive discussion).

**DISTRIBUTION**

Because this database has been developed for the benefit of the scientific community at large, and the information is in the public domain, copies of this database can be obtained *gratis* as a DOS Open File on diskette from the GSC by writing to:

Dr. J.-S. Vincent, Director  
 Terrain Sciences Division  
 Geological Survey of Canada  
 601 Booth St.  
 OTTAWA, Ontario K1A 0E8  
 Canada

**ACKNOWLEDGMENTS**

The development of this database is primarily a result of the conscientious work of a large number of summer students over the past decade; to all, my sincerest thanks. We at the GSC would also like to acknowledge our appreciation for the kind generosity of Dr. P. J. H. Richard, University of Montreal, who has allowed his database of samples from Québec to be incorporated into the GSC Date Locator File. Our thanks to Dr. A. S. Dyke, who kindly provided many non-GSC dates to augment the soils database as a result of his projects on peatland development during the Holocene.

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## RADIOCARBON DATING OF SOILS: DATABASE CONTRIBUTION BY BONN AND HAMBURG

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**ABSTRACT.** We present a compilation of  $^{14}\text{C}$  soil dates measured at the University of Hamburg through 1984 (HAM-1597).

### INTRODUCTION

The inherent problems in determining the “age of a soil” were described by Scharpenseel and Becker-Heidmann (1992). The dating procedure applied in the production of  $^{14}\text{C}$  dates of soil profiles and samples listed below was described by Scharpenseel, Pietig and Tamers (1968) and Scharpenseel and Pietig (1970). For reasons of brevity, we report only the oldest  $^{14}\text{C}$  age of the data set indicated for the layers, thin layers or horizons of the  $^{14}\text{C}$ -dated soil profiles. We present an annotated bibliography as well as date lists of our work. We also include unpublished soil dates measured in our laboratory, and, following this contribution, Peter Becker-Heidmann reports continuing dates from HAM-1600 onward.

### DATE LISTS IN RADIOCARBON: BONN I–VII AND HAMBURG I–IV\*

Material dated	Lab code(s)	Age (yr BP)
<b>University of Bonn, Natural Radiocarbon Measurements I (Scharpenseel, Pietig and Tamers 1968)</b>		
<i>Soil and Soil Profile Dates</i>		
<i>Germany</i>		
Hapludoll, Söllingen A	B 26–31	$\leq 4800 \pm 100$
Hapludoll, Söllingen B	B 33–40	$\leq 4060 \pm 80$
Hapludoll, Söllingen C	B 98–104	$\leq 5300 \pm 80$
Hapludoll, Söllingen D	B 106–113	$\leq 5550 \pm 80$
Haplaquoll, Hildesheim A	B 114–120	$\leq 3130 \pm 70$
Haplaquoll, Hildesheim A	B 121–128	$\leq 4000 \pm 80$
Fossil Chernozem below Hapludalf, Soest II	B 3	$4000 \pm 80$
Fossil Chernozem below Hapludalf, Soest I	B 4	$4170 \pm 80$
Fossil Chernozem in dark-brown steppe soil, Wallertheim	B 22	$2560 \pm 60$
Buried Eutrochrept below Allerød trachyt blanket	B 96	$9130 \pm 100$
Udoll, Ostholsteen A, Grossenbrode	B 156	$\leq 1850 \pm 70$
Udoll, Ostholsteen B, Grossenbrode	B 161–165	$\leq 1390 \pm 70$
Plaggept, Greven (Albachtensch, Marktesch, etc.)	B 9–13	$\leq 1300 \pm 80$
Plaggept, Greven, Albachtensch, whole profile	B 43–48	$\leq 1220 \pm 80$
Plaggept, Rheine	B 49–54	$\leq 1260 \pm 60$
Plaggept, Lengerich	B 129–135	$\leq 1190 \pm 70$
(B 135, 80 cm deep $3960 \pm 80$ )		
Spodosol, Sennesand	B 14	$930 \pm 80$
Spodosol, Irrel	B 19	$810 \pm 50$
Spodosols, Darlaten A and B	B 20, 21	$\leq 1220 \pm 60$

\*From 1968–1974 our laboratory was located in Bonn, and our laboratory code designation, as reported in *Radiocarbon*, was BONN. In 1976, our laboratory numbers changed to HAM- to reflect our relocation to Hamburg. (Lab codes in this table are abbreviated to “B” and “H”.) This compilation of our work represents 27 years of soil dating from all over the world.



Material dated	Lab code(s)	Age (yr BP)
Spodosols, Scherpenseel A and B	B 90, 91	≤ 2960 ± 70
Spodosol, Wilsede	B 41	1140 ± 60
Spodosol, Obrehaverbeck	B 42	940 ± 50
Spodosols, Flaesheim	B 15–17	≤ 2420 ± 80
Hapludalf, Frimmersdorf	B 92–95	≤ 1880 ± 80
Half bog soils, Fibrist, Kalkarer Moor I	B 82–85	≤ 7790 ± 110
Half bog soils, Fibrist, Kalkarer Moor II	B 86–89	≤ 3160 ± 50

#### University of Bonn, Natural Radiocarbon Measurements II (Scharpenseel, Pietig and Tamers 1969)

##### Germany

##### Rendolls

Tangelrendsina, Kramer (German Alps)	B 318–322	≤ 4180 ± 70
Moderrendsina Krottenkopf (German Alps)	B 324	600 ± 50

##### Udalfs, argillic horizon probably former A horizon of Mollisol:

Parabrown earth, Eltville	B 326–331	≤ 4940 ± 80
Parabrown earth, Inden	B 334–342	≤ 4170 ± 70
Brown earth, Haaren-Sintfeld	B 355–358	≤ 1580 ± 50
Parachernozem, Fellbach	B 372–379	≤ 2730 ± 70
Parachernozem, Fellbach, brickpit	B 380–384	≤ 4150 ± 50

##### Plaggepts:

Southeast of Rietberg	B 343–345	≤ 1200 ± 70
Brede near Rietberg	B 348	720 ± 70
Hoffeld	B 349–350	≤ 1130 ± 70
Sinnesche Brede	B 351	1540 ± 60
Am Hohen Lande	B 352	810 ± 70
Krax bei Neuenkirchen	B 353	900 ± 60

Modern bomb-carbon samples BONN 172–200 and BONN 303–317  
(bomb C curve on cereals, beets, winter rape, wine, 1956–1967)

#### University of Bonn, Natural Radiocarbon Measurements III (Scharpenseel and Pietig 1970)

##### Udolls, Vertisols, Fossil A-horizon of Paleosol-Mollisol in Argillic Horizon of Hapludalf

##### Germany

Parabrown earth Lantershofen	B 403–409	≤ 5530 ± 90
Ochtendung (below trachytic pumice)	B 411–416	≤ 10,580 ± 100
Muddersheim, Thineland	B 417–421	≤ 3700 ± 60
Quarry “Schäferkalkwerke”	B 422–431	≤ 25,000 ± 700
Buried soil organic matter (SOM), Eddersheim	B 448	8300 ± 120
Humus containing sand with charcoal (fireplace), Amalienhof	B 608a	2530 ± 70
Buried humus, Heiligensee Forest, Berlin	B 609	760 ± 60
Bone collagen in paleosol below trachytic tuff, Michelsberg	B 763	10,800 ± 100

##### Bohemia, Czech Republic

Argiudoll, Kozojedy, Jicin District	B 437–440	≤ 4150 ± 90
Agiudoll, Smince, Uradec, Kralové District	B 441	4020 ± 60
Hapludoll, Brazdim, Prahoviphod, Tilery District	B 442–444	≤ 3430 ± 65
Vertisol (Smonitza), Prunevor, Choumtov District	B 445–447	≤ 6370 ± 65
Hapludoll, Chernozem, Zozelice, Königgrätz District	B 485–487	≤ 1460 ± 110
Aquoll, Zozelice II, Königgrätz District	B 488–490	≤ 1950 ± 70

##### Moravia, Czech Republic

Argiudoll, Brnicko, Olmütz District	B 491, 495	≤ 4055 ± 80
Udoll, sandy loess, Moravia	B 496–499	≤ 3610 ± 75
Udoll, Chernozem, Bilorice	B 500, 600–603	≤ 2450 ± 70
Vertic Udoll (Vertisol-like Chernozem), Tegel, Pole, Brünn	B 604–607	≤ 4070 ± 70

Material dated	Lab code(s)	Age (yr BP)
<i>Russia</i>		
Hapludoll, Vermudoll, Chernozem, Orel	B 455–457	≤ 4720 ± 60
Udoll, Chernozem, Charkov	B 460–462	≤ 5920 ± 140
Udoll, Chernozem, Zaparorskje	B 464–466	≤ 3270 ± 80
Udoll, Chestnut soil, Askania Nova	B 468–470	≤ 2710 ± 80
<i>Tunisia</i>		
Vertisol, Beja (deepest humus layer)	B 433	2920 ± 40
Vertisol, Zouarine, Ebba Ksour	B 434	3680 ± 65
<i>Finland</i>		
Sandy humus 75 cm deep, Kevo, North Finlandia	B 449	2350 ± 70
<i>Spitzbergen</i>		
Fossil A horizon, 55 cm deep, Hohenstaufen Plateau, Barents I	B 432	3040 ± 80
<i>Germany</i>		
SOM fractions		
Chernozem, Söllingen, total organic matter	B 6A	2100 ± 80
Chernozem, Söllingen, humic acid extract only	B 6B	2240 ± 80
Spodosol, Scherpenseel, brown humic acid fraction	B 138	2060 ± 60
Spodosol, Scherpenseel, gray humic acid fraction	B 139	1720 ± 60
Spodosol, Scherpenseel, rim of gravel pit	B 366	2930 ± 40
Spodosol, Scherpenseel, hymatomelanic acid fraction	B 367	1580 ± 80
Spodosol, Scherpenseel, brown humic acid fraction	B 368	2530 ± 60
Spodosol, Scherpenseel, gray humic acid fraction	B 369	2980 ± 70
Spodosol, Scherpenseel, humin fraction	B 370	2850 ± 70
Histosol, Kalkarer Moor, fulvic acid fraction	B 360	4270 ± 80
Histosol, Kalkarer Moor, hymatomelanic acid fraction	B 361	4510 ± 80
Histosol, Kalkarer Moor, brown humic acid fraction	B 362	5380 ± 80
Histosol, Kalkarer Moor, gray humic acid fraction	B 363	5970 ± 40
Histosol, Kalkarer Moor, humin fraction	B 364	3490 ± 70
Histosol, Kalkarer Moor, humus coal fraction	B 365	4460 ± 80
Aquoll, pseudogley-Chernozem, Adlum, fulvic acid fraction	B 397	1800 ± 60
Aquoll, pseudogley-Chernozem, hymatomelanic acid fraction	B 398	1390 ± 70
Aquoll, pseudogley-Chernozem, brown and gray humic acid fraction	B 399	4890 ± 50
Aquoll, pseudogley-Chernozem, humin fraction	B 401	2980 ± 70
Aquoll, pseudogley-Chernozem, humus coal fraction	B 402	2810 ± 60

#### University of Bonn, Natural Radiocarbon Measurements IV (Scharpenseel and Pietig 1971)

##### Soil Profiles

##### Hungary

Udalf, Chernozem, Erd, southeast Budapest	B 611–615	≤ 9680 ± 100
Udalf, Chernozem in sand-loess, Balatonföldvár, south bank of Lake Balaton	B 625–627	≤ 4690 ± 60
Udalf, Chernozem in fine sandy loess, Koszarhegy	B 633–636	≤ 4575 ± 60
Udalf, meadow soil, Boconad, east-northeast Budapest	B 616–620	≤ 5260 ± 50
Eutrochrept, Brown earth in loess, Kapoly	B 628–632	≤ 3990 ± 70
Hapludalf, Nagyrécsce, southeast Budapest (below 112 cm, <sup>14</sup> C age jumps to 16,750 ± 290)	B 621–624	≤ 2870 ± 115
Natrustalf, Hortobagy, southwest Debrecen, Rusta Plain	B 648–651	≤ 10,080 ± 160
Histosol, bog soil, Nadasdladany, northeast of Lake Balaton	B 637–647	≤ 9300 ± 340

##### Russia

Udoll, deep Chernozem, Orel (240 cm)	B 458	12,470 ± 360
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Material dated	Lab code(s)	Age (yr BP)
<i>Ireland</i>		
Plaggept, Donoure, Ardfield	B 660	480 ± 50
Plaggept, Cahesetrant, Dingle	B 661–663	≤ 2135 ± 50
<i>Australia</i>		
Ustoll, Krasnozem, Wollongbar	B 664	1400 ± 60
Ustoll, Krasnozem, Babbınbar	B 679–680, 766	≤ 6010 ± 100
Ustoll, Krasnozem, Beechmont	B 681, 767	≤ 3850 ± 360
Krasnozem, Binjour on lateritic plateau	B 682 and 768	≤ 1780 ± 70
Krasnozem, Gurgena on lateritic plateau	B 683 and 769	≤ 570 ± 70
Krasnozem, Coulston Lakes I, valley plain	B 684 and 770	≤ 950 ± 50
Krasnozem, Coulston Lakes II, valley plain	B 685 and 771	≤ 980 ± 50
Krasnozem, Maleny, dissected plateau, 15 cm	B 685	150 ± 50
Krasnozem, Memerambi, dissected plateau	B 687 and 772	4000 ± 150
<i>Argentina</i>		
Vertisol, Entre Rios, Concepción del Uruguay	B 804–813	≤ 11,160 ± 150
<i>Germany</i>		
Humod, Hauset/Hergenrath	B 652–656	≤ 2240 ± 50
Placorthod, Schliffkopfhaus	B 859–861	≤ 2280 ± 60
Andosol, Brown earth in trachytic ash, 200 m south of Andernach-Kruft road	B 818–822	≤ 4470 ± 70
Andosol, Brown earth in trachytic ash, Neuwied basin	B 823–828	≤ 4210 ± 80
Inceptisol, Brown earth in trachytic ash, Niedermendig	B 829	3990 ± 100
Mardelle with peat-carbon, Pirmasens	B 1132	900 ± 60
<i>Czech Republic</i>		
Udalf, Chernozem (buried), Sedlec	B 843–845	≤ 12,480 ± 120
Udalf, Chernozem (not buried), Sedlec	B 843–845	≤ 5910 ± 60
Udalf, Chernozem (buried) underlying Holocene Chernozem	B 847	25,730 ± 550
Udalf, Chernozem, Chabry (Holocene)	B 848–849	≤ 5810 ± 60
Udalf, Chernozem, Chabry (Pleistocene)	B 850–853	≤ 17,520 ± 540
Udalf, Chernozem, Chabry (some locations, deepest point)	B 854	25,630 ± 710
<i>Italy</i>		
Fossil steppe soil, buried, Vintschgau, Bolzano	B 864	5270 ± 60
<i>SOM Fractions</i>		
<i>Germany</i>		
Udoll, Chernozem, Söllingen, fulvic acids	B 670	104.3 ± 0.5 pMC
Same soil, brown- and gray humic acids	B 671	1560 ± 70
Same soil, humin and humus coal	B 672	2275 ± 60
Michelsberg, fulvic acids	B 673	4310 ± 210
Same soil, brown and gray humic acids	B 674	7600 ± 220
Same soil, humins	B 675	6930 ± 80
Same soil, humus coal	B 676	6830 ± 100
SOM of Udalf, Chernozem, Söllingen, different centrifugal gravity		
500 rpm	B 831	2000 ± 50
2000 rpm	B 832	1870 ± 70
3000 rpm	B 833	1680 ± 50
4000 rpm	B 834	1820 ± 100
5000 rpm	B 835	1770 ± 60
5400 rpm	B 836	1780 ± 50

Material dated	Lab code(s)	Age (yr BP)
Hapludalf, Inden, fossil A horizon in B <sub>t</sub> (argillic horizon), varying texture fractions		
>60 μ φ	B 1133	3170 ± 80
60–2 μ	B 1134	3450 ± 80
2–1 μ	B 1135	3280 ± 80
1–0.5 μ	B 1136	2790 ± 70
0.5–0.25 μ	B 1137	2500 ± 70
<i>Subhydrous Soils, Gytja</i>		
<i>Germany</i>		
Schalkenmeerer Maar, 0–230 cm	B 781–802	≤ 4600 ± 70
Lake of Selent, gytja, 0–560 cm	B 882–908	≤ 6800 ± 150

#### University of Bonn, Natural Radiocarbon Measurements V (Scharpenseel and Pietig 1973a)

##### *Soil Profiles*

##### *Israel*

Xeralf, Hamra, below dune cover, Tel Aviv–Haifa highway, corner of Richlon Street	B 688–691	≤ 14,740 ± 200
Xeralf, same location, foot of slope, no continuous dune cover	B 692–695	≤ 10,470 ± 130
Xeralf, same location, emerging into recent soil, no dune cover	B 696–699	≤ 11,860 ± 150
Xeralf, Hamra, in dune material with lime concretions (Curcar), Wingate Institute of Athletics, near Tel Aviv–Haifa highway	B 701–706	≤ 17,920 ± 180
Xeralf, calcinated root in Hamra, street to Ecron	B 709	16,930 ± 240
Curcar–Hamra sequence, Rehovot, corner of Main Street and Batia Markov	B 711	14,920 ± 230
Aqualf, Nazas, Jashresh	B 712, 713	≤ 2960 ± 220
<i>Soil Associations on Limestone</i>		
Xeroll on soft limestone, Mitzpe Mesua	B 742, 743	≤ 1500 ± 50
Xerochrept, calcareous brown earth on harder limestone, near Mitzpe Mesua	B 744	2040 ± 60
Rhodustalf–Terra Rossa on hard limestone, Mattah	B 745	2420 ± 70
Xeralf with recalcification (Husmas soil), Agricultural School, Kanot	B 748, 749	≤ 5050 ± 160
Xeroll, Burozem, overlying Xeralf with recalcification (Husmas), Kibbutz Ruchama		
Xeroll	B 751, 752	≤ 9000 ± 200
Underlying recalcified Xeralf	B 753–755	≤ 13,400 ± 190
Fossil clay below recalcified Xeralf	B 757	19,920 ± 340
Dark brown soil in calcareous dune sand, chesnut-like soil, Mafkiim, south of Ashkalon	B 750	4760 ± 80
Palexeroll near Shuval, road from Beer Shewa to Tel Aviv	B 760–762	≤ 15,470 ± 230
Paleorthid in loess, Eshel Hanassi, near Beer Sheva	B 758–759	≤ 4020 ± 220
Xerert, west Plain of Barkai, road from Afula to Hedra	B 715–718	≤ 1850 ± 70
Xerert, Valley of Jesrael	B 719–723	≤ 2760 ± 80
Xerert, Valley of Jesrael, drainage ditch	B 724–728	≤ 7440 ± 80
Xerert, El Hamma	B 729–734	≤ 19,430 ± 350
Xerert, near Kefar Manachem Kibbutz	B 735–741	≤ 16,100 ± 270
Xerert along Syrian Quarantine Station and Jordan flow into Lake Genezareth	B 773–776	≤ 2670 ± 100

##### *Bulgaria*

Vertic Albaqualf, Glavatsi	B 1071–1074	≤ 8050 ± 80
Udic Haplustoll, leached Chernozem, near Gorni Dubnik	B 1075–1079	≤ 11,100 ± 90

Material dated	Lab code(s)	Age (yr BP)
Typic Caciustoll, calcareous Chernozem, northwest Pleven	B 1080–1085	≤ 5760 ± 90
Paleustalf, Gray Forest soil, 12 km south of Pleven	B 1086–1092	≤ 18,920 ± 340
Udic Haplustalf, Gray Forest soil, Kozlevo–Shoumen	B 1093–1097	≤ 3370 ± 100
Udertic Paleustalf, degraded Cinnamon Forest soil, near Bourgas	B 1098–1104	≤ 14,150 ± 240
Vertic Albaqualf, Cinnamonic, podzolized Planosol, Badeshte, Thracian plain	B 1105–1108	≤ 9850 ± 240
Pellustert, Smonitsa-Vertisol, Sredets, Thracian Plain	B 1108–1114	≤ 16,140 ± 460
Chromic Luvisol, Cinnamonic Forest soil, Koren	B 1115–1119	≤ 8480 ± 140
<i>Sardinia</i>		
Xerert (aquic), Plane de Cuga, Ittiri, southwest Sassari	B 1154–1157	≤ 570 ± 50
Chromoxerert, river terrace, Rio Mannu di S. Vero	B 1141–1164	≤ 3870 ± 130
Pelloxerert, Arziadas, Tuvoi	B 1167–1174	5430 ± 100
Chromoxerert, Monastir, 20 km north of Cagliari	B 1175–1178	≤ 2270 ± 70
Pellustert, Nurallo	B 1180–1187	≤ 3220 ± 80
<i>Sicily</i>		
Chromoxerert, Scalilli near Corleone	B 1326–1331	≤ 3030 ± 90
Pelloxerert, Plana di Scala, Corleone	B 1332–1338	≤ 3670 ± 100
Pelloxerert, Azienda Sporacia, Farm, Università di Palermo	B 1339–1351	≤ 5470 ± 120
Pelloxerert, Azienda Sporacia, Farm, Università di Palermo	B 1352–1363	≤ 15,160 ± 370
<i>Romania</i>		
Humic horizon underlying Danube alluvium, near Bucharest	B 1379–1385	≤ 8070 ± 130
<i>Germany</i>		
Aqualfic Fragiorthod, Amelsbüren (below 160 cm, 15,170 ± 230)	B 1363–1369	≤ 1980 ± 80
Placorthod, Grindenschwarzwald, Gernsbach	B 1371–1377	≤ 2550 ± 70

#### University of Bonn, Natural Radiocarbon Measurements VI (Scharpenseel and Pietig 1973b)

##### Soil Profiles

##### Spain

Xerert, La Rinconada (Seville), Casas vacas	B 1388–1392	≤ 6470 ± 130
Xerert, Carmona, km 10.5 Carmona-Arahal Street	B 1393–1397	≤ 6650 ± 120
Xerert, Los Palacios, Torbiscal Farm	B 1398–1406	≤ 8850 ± 130
Xerert, El Arahal, Estrella Farm, 40 km east of Seville	B 1407–1413	≤ 3480 ± 100
Xerert, Carmona, La Motilla Chica Farm	B 1417–1423	≤ 3440 ± 290

##### Portugal

Xerert, Black Barros, Beja	B 1425–1432	≤ 3070 ± 140
Vertic Xerochrept, Safara-Camauros Farm	B 1433–1437	≤ 2430 ± 70
Xerert, Safara field	B 1438–1442	≤ 2230 ± 190
Red Xerert, Salvada-Beja	B 1443–1446	≤ 1380 ± 70
Roman grain silo in Xerert, Beja-Serpa	B 1447	5150 ± 100
Dark red Xerert, Montes Velhos Aljustrel	B 1452–1457	≤ 2240 ± 80
Red-brown Xerert, Terra Grande de Lisboa, Tapaia da Ajuda, university campus near Lisbon	B 1458–1463	≤ 3170 ± 80

##### Australia

Ustert, Lillimur, Kaniva District, Victoria, Gilgai mound	B 1466–1485	≤ 5880 ± 180
Ustert, Lillimur, Kaniva District, Victoria, Gilgai depression	B 1486–1507	≤ 2280 ± 150
Ustert, Miram, Kaniva District, Victoria, Gilgai mound	B 1508–1527	≤ 8530 ± 250
Ustert, Miram, Kaniva District, Victoria, Gilgai depression	B 1528–1548	≤ 8450 ± 260

Material dated	Lab code(s)	Age (yr BP)
<i>Germany</i>		
Fossil A horizon, Eberspoint, 10 km west of Freising	B 1464	6160 ± 90
Fossil A horizon, terrace brown earth, 5 km south of Siegenburg, Abens Valley, north Bavaria	B 1648	2270 ± 70
Charcoal from different fireplaces	B 1648, 1649	≤ 1460 ± 70
Hapludalf in flood loam, lower terrace of the Rhine River	B 1652	5080 ± 110
Fossil A horizon below Hapludalf, same location	B 1653	8230 ± 470
Bituminous coating on gravel in younger Isar terrace, Ascholding, Wolfratshausen Basin	B 1657	28,320 ± 470
Umbrept (Plaggept?), Würmian basal moraine of Isar foreland glacier, Unterbuchen, Bavaria	B 1669–1672	≤ 3800 ± 80
Root in Maintag gravel pit, Upper Franconia	B 1700	1810 ± 70
Fossil A horizon in Maintag gravel pit, Upper Franconia	B 1801	7980 ± 110
Root, vertical in sediment, Maintag gravel pit	B 1802	4360 ± 90
Fossil A horizon, covered by Pleistocene terrace material, Kärlich, Rhineland	B 1659	30,450 ± 1270
Humus in silty A horizon, below tuffaceous material, north slope of Bausenberg, Lengsdorf, Rhineland	B 1699	22,360 ± 510
Fossil A horizon, gravelly gley, Gammelsbach valley, North Eberbach, Odenwald	B 1815	1350 ± 110
<i>Soil Fractions</i>		
Fossil A horizon, underlying Allerød trachytic tuff, charcoal only	B 1681	11,550 ± 160
Same location, particle size fraction > 0.2 mm	B 1681	7570 ± 190
Same location, particle size fraction 63–2 μ	B 1684	10,950 ± 150
SOM from continuous extraction, successive fractions (1st extraction: 0.1N H <sub>2</sub> SO <sub>4</sub> ; 2nd extraction: 0.15 M Na <sub>4</sub> P <sub>2</sub> O <sub>7</sub> )		
Udalf Aseler Holz:	B 1809	1030 ± 100
	B 1810	4130 ± 270
	B 1811	4970 ± 80
Spodosol (Humod) gravel pit Weber, Scherpenseel (Dutch border):	B 1688	1400 ± 140
	B 1689	1160 ± 70
	B 1691	1460 ± 80
	B 1692	1350 ± 110
	B 1693	1510 ± 130
	B 1697	1290 ± 70
<i>Subhydrous Soils</i>		
Gyttja, bottom of Schalkenmehrer Maar, Eifel I	B 994–1005	≤ 12,130 ± 140
Gyttja, bottom of Schalkenmehrer Maar, Eifel II	B 1007–1025	≤ 12,160 ± 130
Gyttja, bottom of Schalkenmehrer Maar, Eifel IV	B 1026–1045	≤ 12,130 ± 140
Gyttja, bottom of Schalkenmehrer Maar, Eifel V	B 1046–1064	≤ 3840 ± 80
Gyttja, bottom of Lake of Selent, Holstein I	B 869–880, 1122–1125	≤ 1670 ± 100
2 m below end of case lot (sampling instrument)		10,170 ± 140
Gyttja, bottom of Lake of Selent, Holstein III	B 911–932, 963–964	≤ 24,830 ± 970
Gyttja, bottom of Lake of Selent, Holstein IV	B 933–959	≤ 17,390 ± 460
On-shore profile, Lake of Selent, Holstein IV (opposite Profile IV)	B 1127–1131	≤ 2670 ± 70
Gyttja, bottom of Lake of Selent, Holstein V	B 967–976	≤ 14,180 ± 670
Gyttja, bottom of Lake of Selent, Holstein VI	B 977–993	≤ 10,080 ± 520
at 340 cm depth		30,930 ± 1150

Material dated	Lab code(s)	Age (yr BP)
<b>University of Bonn, Natural Radiocarbon Measurements VII (Scharpenseel and Pietig 1974)</b>		
<i>Germany</i>		
<i>Subhydrous Soils</i>		
Gyttja, bottom of Lake Laach, Eifel I	B 1572–1590	≤ 23,010 ± 460
Gyttja, bottom of Lake Laach, Eifel II	B 1591–1607	≤ 20,500 ± 380
Gyttja, bottom of Lake Laach, Eifel III	B 1609–1622	≤ 22,070 ± 440
Gyttja, bottom of Lake Laach, Eifel IV	B 1623–1636	≤ 10,700 ± 140
Gyttja, bottom of Lake Laach, Eifel V	B 1637–1643	≤ 20,130 ± 300
Gyttja, bottom of Meerfelder Maar, Eifel I	B 1818–1832	≤ 6800 ± 100
Gyttja, bottom of Meerfelder Maar, Eifel II	B 1838–1838	≤ 9140 ± 120
Gyttja, bottom of Meerfelder Maar, Eifel III	B 1839–1849	≤ 7380 ± 90
Gyttja, bottom of Meerfelder Maar, Eifel IV	B 1850–1859	≤ 5550 ± 90
Gyttja, bottom of Meerfelder Maar, Eifel V	B 1860–1867	≤ 3520 ± 80
Gyttja, bottom of Meerfelder Maar, Eifel VI	B 1868–1876	≤ 4050 ± 80
Gyttja, bottom of Pulvermaar, Eifel I	B 1877–1891	≤ 8040 ± 120
Gyttja, bottom of Pulvermaar, Eifel II	B 1892–1903	≤ 6720 ± 110
Gyttja, bottom of Pulvermaar, Eifel III	B 1904–1916	≤ 6420 ± 180
Gyttja, bottom of Pulvermaar, Eifel IV	B 1917–1930	≤ 4980 ± 80
Gyttja, bottom of Pulvermaar, Eifel VI	B 1931–1946	≤ 5170 ± 180
<b>Hamburg University, Radiocarbon Dates I (Scharpenseel, Pietig and Schiffmann 1976)</b>		
<i>Germany</i>		
<i>Soil samples and soil profile samples</i>		
Argillic horizon of Hapludalf, Friesdorf near Bonn	H 1	5010 ± 280
South side of same pit, Friesdorf	H 2	3620 ± 70
Aquept, alluvial loess, Isar terrace, Landshut/Ergolding	H 4–7	≤ 10,880 ± 140
Udoll high terrace of Isar, Landshut/Ergolding	H 8–10	≤ 3190 ± 80
Hapludalf, high terrace of Isar, Landshut/Ergolding	H 11–13	≤ 3320 ± 70
Koislhof, lower to upper nether terrace, Isar River	H 14–25	≤ 10,680 ± 140
North Bavaria, north of Danube, humic colluvium on old Riss moraine north of Landsberg	H 26–31	≤ 4340 ± 70
Mollisol in steep bank of Lech River, Kaufering	H 32–36	≤ 4410 ± 80
Fossil organic matter in tuffaceous limestone, Neuenried, near sources of Mindel River	H 37–43	≤ 4580 ± 90
Fossil argillic horizon between terrace gravel and loess loam, Fellheim brick factory, Iller River	H 44–46	≤ 4340 ± 130
Eutochrept, Hohentrüdigen on Dogger (Jurassic)	H 47–49	≤ 15,730 ± 410
Vertisol on Tertiary deposits in the Ries, Maihingen	H 50–55	≤ 11,820 ± 170
Aquic Vertisol on younger Ries lake deposits, Pfäfflingen	H 56–60	≤ 10,470 ± 130
Aquic Vertisol, Ries lake deposits Wechingen	H 61, 62	≤ 3860 ± 80
Vertic Brown earth (Inceptisol), Ries lake deposits, Munningen	H 63, 64	≤ 4840 ± 60
Folist, Nether Moor, Koislhof, near Landshut	H 64	10,960 ± 180
Udalf Smonica/Chernozem, Lötzeiler, Rheinhessen	H 66, 67	≤ 5430 ± 90
	H 68,69	≤ 2580 ± 70
Mollisol/Hapludalf, Dorla I/2, Fritzlar-Gudensberg	H 70	3300 ± 70
Mollisol/Hapludalf, Lohne I, north of village of Lohne	H 71	2040 ± 60
Mollisol/Hapludalf, Wehren I/2, Wehren-Kirchberg	H 72	2890 ± 70
Mollisol/Hapludalf, Wehren I/3, Wehren-Kirchberg	H 73	4740 ± 80
Mollisol Heuchelheim, overlying degraded Chernozem	H 74	3660 ± 90
Colluvium overlying degraded Chernozem, Bleichen II	H 75	6480 ± 80
Loess on solifluction debris, south of Höingen, Vogelsberg	H 76	10,550 ± 130
Humus in solifluction debris, 300 m east of Taufstein, Vogelsberg	H 77, 78	≤ 5360 ± 80

Material dated	Lab code(s)	Age (yr BP)
High flood loam, Main River, Kelsterbach-Lerchenberg, Kiesgrube Schmidt	H 79, 80	≤ 7900 ± 100
Fossil epipedon, on Allerød trachyte, Buchslag, plain south of Main River	H 81	10,000 ± 210
Colluvial loam, Hochheim, Hattersheim, valley sediments	H 82–93	≤ 6970 ± 100
Colluvium, Waldeck (Freienhagen, Ippinghausen)	H 94, 95	≤ 820 ± 110
Spodosol, "Raseneisengley", Black Forest, Altglashütten	H 96–101	≤ 3120 ± 90
Spodosol, Black Forest, between Breitnau and Hollertal	H 109–112	≤ 1120 ± 60
Charcoal and fossil Brown earth, Trescher, Black Forest	H 113–117	≤ 1040 ± 50
Low moor peat, charcoal, paleosol on Riss boulder marl, Altenerding, Bavaria	H 118–122	≤ 30,340 ± 1130
Cliff wall at Baltic Sea coastline, Heiligenhafen	H 123–127	≤ 4020 ± 90
Fossil Chernozem (Udalf), outskirts of Homberg, Kassel County	H 128	5650 ± 80
Fossil Chernozem (Udalf), Worms I, near town of Worms	H 129	3530 ± 70
Bones, wood, sediments believed from Roman times, Xanten	H 130–133	≤ 3150 ± 70
Humic soils near Dutch border, organic matter from pluggen or deep ploughing, total samples and 6 N HCl hydrolysis residue		
Walbecker Heide	H 134, 135	≤ 9580 ± 100
Maasaue near Velden	H 136, 137	≤ 2070 ± 90
Haus Beerenbruck	H 138, 139	≤ 1860 ± 90
Schandelah	H 140, 141	≤ 1440 ± 60
Walbeck	H 142, 143	≤ 1880 ± 130
Subhydrous soil samples, Baldeney lake, Essen-Werden, south shore	H 183–188	≤ 12,160 ± 270
Clay pit Kaerlich, wood sample underlying Allerød and Holocene soil	H 189	29,600 ± 1150
<i>Wales</i>		
Placorthods, Hiraethog, Denbighmors, Denbigshire	H 103–108	≤ 5810 ± 150
Peny Gwrydd Pass, Snowdonia National Park, Caernarvonshire	H 105–106	≤ 1770 ± 80
<i>Austria</i>		
Low moor (Histosol), Neumarkt, Am Wallersee, Salzburg	H 144–145	≤ 3770 ± 70
<i>Former USSR</i>		
Predkaokadzye Chernozem (Ustoll), south Rostov	H 146	11,330 ± 980
Chernozem, lower terrace of Don River, from krotovinas	H 147	5250 ± 150
Meadow Chernozem in Azow system	H 148	6510 ± 260
Dark Gray Forest soil, Zhiguli	H 149	5490 ± 280
Pelehymorphic Chernozem, Zhiguli, krotovina	H 150–151	≤ 4750 ± 80
Chernozem Privolzhye Upland, Karlinsky Far, Ulyanovsk	H 152	5390 ± 120
Chernozem near Solod Meadow, Ulyanovsk Agricultural Institute campus	H 153	5550 ± 140
Gray Forest soil, Vysokogorsky, Tatar SSR	H 154	7890 ± 680
Chernozem, Tour I, 10th International Congress on Soil Science, Moscow	H 155–156	≤ 4540 ± 80
<i>Tunisia</i>		
Paleosol in pasture, Ain Oktor, Korbous	H 157	2250 ± 60
Fossil horizons, Ferme Korba, pasture experiment	H 158–160	≤ 2300 ± 100
Fossil horizons near Ferme Korba, pasture experiment	H 161–162	≤ 2470 ± 70
Fossil horizons, Ferme Ennasser near Bir Bou Rekba	H 163–164	≤ 4230 ± 60
Fossil horizon near Enfida, km 52, road to Kairouan	H 165	4510 ± 80
Fossil horizon, Medjerdah alluvium near Ghardimaou bridge	H 166–167	≤ 8000 ± 180
Fossil horizons in Medjerdah alluvium, near Bou Salem	H 168–171	≤ 7800 ± 160
Fossil horizons, Medjerdah River, Bou Huertma River	H 172–174	≤ 2400 ± 120
Pale-Xerochrept, Sol Brun à Croûte (crust faintly developed), Medjerdah Valley, hill site	H 175–182	≤ 27,160 ± 1090



Material dated	Lab code(s)	Age (yr BP)
<b>Hamburg University, Radiocarbon Dates II (Scharpenseel and Schiffmann 1977)</b>		
<i>Argentina</i>		
Argiudoll, Brunizem, Rafaela	H 231–237	≤ 480 ± 80
Argiudoll, Brunizem, Esperanza	H 238–244	≤ 2030 ± 80
Argiudoll, slightly planosolic Brunizem, Angel Gallardo	H 246–252	≤ 2780 ± 80
Argiudoll, Profile B9, Villa Concepción del Tío	H 253–257	≤ 1870 ± 90
<i>Tunisia</i>		
Paleosol in Bou Huertma alluvium	H 258–259	≤ 4930 ± 80
<i>Germany</i>		
Mud in old river bed of Ems River, on top of low moor, Rietberg	H 280–281	≤ 6680 ± 90
Ochrept, Holzkirchen, near Munich-Salzburg highway	H 631–634	≤ 2510 ± 50
Humic acid samples of Lower Saxonian soils	H 260–279	≤ 19,800 ± 710
SOM fractions, Spodosol, Scherpenseel, near Dutch border	H 282–285	≤ 5410 ± 90
SOM fractions, Udoll, Aseler Wald, near Hildesheim	H 286–297	≤ 3160 ± 70
SOM fraction, paleosol of loess, below trachytic tuff of Allerød volcanism	H 298–310	≤ 11,360 ± 150
SOM fractions, vertic, clayey Histosol, Koislhof, lower terrace of Isar River	H 311–328	≤ 13,140 ± 200
SOM fractions after repeated 6 HCl hydrolysis, Udoll from Asel clay pit near Hildesheim	H 623–630, 801	≤ 3260 ± 100
SOM fractions after repeated 6 HCl hydrolysis, histic Udoll, Ergolding near Landshut	H 762–788, 800	≤ 6110 ± 90
Sea level–coast line study based on peat dating, North Sea shore	H 765–776	≤ 6130 ± 240
Humus in Elbe River alluvium, Billwerder-Allermöhe, south Hamburg, measurement of deposition date	H 791–799	≤ 3370 ± 100
<b>Hamburg University, Radiocarbon Dates III (Scharpenseel, Schiffmann and Hintze 1984)</b>		
<i>Germany</i>		
Histice Hapludoll, 5 km south of Söllingen, 0–90 cm, at 5-cm intervals		
Carbonate-free soil	B 2255–2272	≤ 6210 ± 90
6 N HCl hydrolysis residue	B 2401–433	≤ 6370 ± 80
6N HCl hydrolyzate	B 2400–2428	≤ 3240 ± 70
Acid from carbonate destruction	B 2438–2450	≤ 3570 ± 70
Typic Hapludoll, Söllingen, near old windmill, 0–75 cm, at 5-cm intervals, carbonate-free soil		
6 N HCl hydrolysis residue	B 2476, 2478, 2484–2492, 2500, 2502, 2504	≤ 3260 ± 60
6 N HCl hydrolyzate	B 75, 2477, 2483–2491, 2499, 2501, 2503	≤ 40 ± 60
Acid from carbonate destruction	B 2459–2469	≤ 240 ± 90
Eutochrept, near Hohentrüdingen, Jurassic Dogger, near Nördlinger Ries crater, taken in 5-cm intervals, 5 to 105 cm depth		
Haplaquept on Isar River terrace, near Landshut, Ergolding, Bavaria, taken in 5-cm intervals, 0 to 90 cm depth	H 655–672	≤ 5560 ± 80
Humic matter, coastline levee along Baltic coast, near Heiligenhafen		
Elbe River marsh series, Allermöhe, Vier and Marschlande, south of Hamburg	B 2367–2385	≤ 6240 ± 110
(H 835, paleosol, 7420 ± 110)	H 826–841	≤ 3970 ± 80

Material dated	Lab code(s)	Age (yr BP)
Peaty material, underlying valley of Elbe River, sampled in three cross-sections, east and west of Hamburg	H 1393–1406	≤ 8140 ± 100
<i>Australia</i> (see also B 664 to 772, Scharpenseel and Pietig (1973b: 258–263))		
Vertisol (Chromustert), Chinchilla	H 674–702	≤ 9850 ± 170
Vertisol (Chromustert), Paget	H 734–758	≤ 11,570 ± 210
Eutrustox (Krasnozem) Gabbinbar (H 731, 200–220 cm, 7420 ± 110)	H 719–726	≤ 1810 ± 80
Eutrustox (Krasnozem), Beechmont (subtropical rainforest)	H 703–711	≤ 2020 ± 70

### Hamburg University, Radiocarbon Dates IV (Scharpenseel, Schiffmann and Becker 1984)

#### Soil Samples and Profiles

##### Tunisia

Fossil gyttja, northwest Degache, Chott el Rharsa	H 1029	2420 ± 70
Paleoargid, near Algerian border, underlying fringe of dunes	H 1030	22,730 ± 400
Paleosol in terrace, Oued Lakarit	H 1031	8050 ± 100
Buried Argixeroll, 12 km from Ksour Essaf	H 1032	3470 ± 70
Tirsoid Xerert, Enfida, Station, Amélioration des Parcours	H 1033	4550 ± 80
Paleroll, 18 km from Tadjerouine toward Le Kef	H 1034	7960 ± 110
Saem profile fossil horizon 180 cm deep	H 1035	8520 ± 180
Dates of paleosols from perhumid to Saharian climate, 20 km west of Nefta	H 1222–1223	≤ 10,260 ± 120
North rim of Chott Djerid, 13 km from Nefta	H 1224–1226	≤ 4330 ± 90
Humic layer, gravel terrace, rim of Chott	H 1227	1950 ± 60
Paleosol, street bridge G P 16, Kebili to Gabes, 62 km west of Gabes	H 1229	920 ± 80
Polyphasic steppe soil, bank of Oued Ersifa, 25 km from Matmata	H 1233–1236	≤ 6420 ± 130
Paleosol in bank of Oued, 5 km north of Remada, near GP 19	H 1237, 1239	≤ 5200 ± 160
Paleosols in Oued Tatahouine, profile north of Foum Tatahouine	H 1240, 1247	≤ 13,490 ± 220
Cut in sediments 300 m southwest of Matmata-Toujane Street	H 1248–1251	≤ 13,530 ± 370
Sequence of paleosols, south of St. M 201 Gafsa-Moulares, cut in bank of Oued Melah	H 1264–1274	≤ 5520 ± 80
Polyphasic paleosol, west El Frouch, Djebel Chambi, east of road to Serept	H 1275–1279	≤ 7270 ± 90
Red relict soil in rock crevices, Sta. Bordj Chambi, Djebel Chambi	H 1283	4080 ± 80
Polyphasic paleosol, ca. 800 m from H 1275–1279	H 1285–1292	≤ 4880 ± 80
Cut in bank of Oued Bou Hamid, foot of Djebel Semmama	H 1295, 1304	≤ 3070 ± 90
Bank of Oued Bou Hamid, profile 200 m downstream from H 1295–1304	H 1305–1311	≤ 6860 ± 100
	(H 1311 300 cm	14,530 ± 250)
Transition to terrace at base of previous profile	H 1312	9920 ± 120
Organic matter in Oued Bou Hamid terrace, opposite bank to profile H 1305–1312	H 1313–1318	≤ 4670 ± 90
Duplex Vertisol, northwest Jendouba, north of street to Chamtou, before Satisfoura	H 1319–1324	≤ 6760 ± 90
Polyphasic paleosol, bank of Oued Ogla, 2 km west of GP 17, Le Kef-Tadjerouine, north bank	H 1326–1330	≤ 5550 ± 80
Mejerdah alluvium, east Tebourba	H 1334–1340	≤ 5850 ± 90
Alluvium of Oued Miliane, north of street Pont du Fahs-Smindja, 10 km from Pont du Fahs	H 1341–1346	≤ 3350 ± 90
Paleosol in Wadi north of GP 3, Kairouan to Sbeitla	H 1347–1352	≤ 4030 ± 90
Cut in alluvium of Oued Melize, south of GP 6, Jendouba to Ghardimaou, near bridge	H 1358–1364	≤ 11,020 ± 130
West of GP 1, Tunis-Sfax, 84 km from Sousse, near crossing to Hammamet Road cut	H 1365–1367	≤ 780 ± 80

Material dated	Lab code(s)	Age (yr BP)
Paleosol in bank of Oued Guilene, east of GP 12, Haffouz to Maktar, near bridge	H 1369	1040 ± 80
Paleosol in bank of Oued Hatab, south of GP 4, Maktar to Tebessa, 300 m before crossing with MC 71	H 1371–1377	≤ 8080 ± 130
Paleosol in Bank of Medjerdah River, 500 m east of Ghardimaou Bridge (H 1381, 100 m west 15,000 ± 210)	H 1378–1381	≤ 3820 ± 80
<i>Sudan</i>		
<i>Vertisol profiles from Gezira</i>		
Profile southwest of Wad Shawer	H 1407–1420	≤ 5570 ± 100
Profile Selemme Hum Dalik Minor, Wad Mahmoud Major	H 1424–1434	≤ 6300 ± 90
Vertisol 400 m west of Sara Omeir Minor	H 1440–1454	≤ 10,370 ± 150
Vertisol, Madina Block 15	H 1455–1464	≤ 4390 ± 120
Vertisol, 1 km west of Meheiriba	H 1473–1487	≤ 5980 ± 170
Vertisol, Qoz er Ruheid	H 1488–1497	≤ 4940 ± 250
Vertisol, in terrace of White Nile, southeast rim of Tureina	H 1500–1506	≤ 5340 ± 100
Vertisol, 1 km west of Buweika	H 1511–1515	≤ 4310 ± 100
Vertisol, 8 km west of Mesou, direction of Secondary School, Laota Block	H 1521–1524	≤ 5560 ± 100
Vertisol, 3 km northeast of Esh Shaval	H 1530–1538	≤ 5930 ± 120
Vertisol, 3 km north of Tamsul	H 1539–1547	≤ 5320 ± 110
Vertisol, entic Pellustert, Hosh series, Ghabsaneblock	H 1012–1016	≤ 3360 ± 90
Vertisol, entic Chromustert, Wad Medani, fallow plot in Gezira Research Station	H 1017–1021	≤ 1740 ± 80
<i>Argentina</i>		
Argiudoll, soil from rolling pampa near Pergamino	H 1178	2810 ± 70
Typic Argiudoll, loess, Rojas series	H 1179	2220 ± 70
Typic Hapludoll, sandy loess, Segui series	H 1180	1650 ± 60
Pelludert, intersection of La Paz Highway and Feliciano Street, San Gustavo	H 1204–1208	≤ 4440 ± 110
Pelludert, Facultad de Agricultura, Universidad Nacional del Litoral (UNL) near Parana	H 1209–1212	≤ 2180 ± 90
Argiudoll, Facultad de Agricultura, UNL near Parana	H 1213	108 ± 1% pMC
Pelludert, 10 km southwest of General Campos, near main street	H 1214, 1550–1554, 1666	≤ 4140 ± 90
Argiudoll, Oro Verde 2, 1.25 km northwest of INTA Experimental Station, Parana, Campo anexo	H 1556	1910 ± 60
Argiudoll, Facultad de Agricultura, UNL near Parana, Febré 2	H 1549	1110 ± 70

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  - $^{14}\text{C}$  dates due to profile scan reflecting preferential link of C to clay minerals by clay-organic complexation

## APPENDIX 2: PREVIOUSLY UNPUBLISHED SOIL DATES

Lab code	Site	Age (yr BP)
<i>Four samples from Mosbruch, base of Histosol</i>		
HAM 1557	Mosbruch 1	11,760 ± 435
HAM 1558	Mosbruch 2	11,580 ± 500
HAM 1559	Mosbruch 3	12,200 ± 500
HAM 1560	Mosbruch 4	11,980 ± 435
HAM 1566	Fossil argillic horizon, Tunisia, 50–60 cm, sand dunes 4 km east of Algerian border, south Tozeur	430 ± 200
HAM 1568	Sample of Vertisol, Argentina, San Gustavo, 130–150 cm	5600 ± 100
<i>Channel cast, Waldsee</i>		
HAM 1570	Waldsee 1, 99–104 cm, lightly decomposed peat	3440 ± 80
HAM 1571	Waldsee 2, 199–204 cm, strongly decomposing peat	4200 ± 80
HAM 1573	Waldsee 4, 209–219 cm, fossil stem material, Alnus	4630 ± 80
HAM 1572	Waldsee 3, 295–300 cm fine detrital gyttja and plant remains	20,600 ± 350
<i>Relics found in soil profile</i>		
HAM 1574	Knodl 1, floating wood pieces	9220 ± 100
HAM 1575	Knodl 2, floating wood pieces	25,500 ± 900
HAM 1576	Grapel, pine wood at base of low moor	8700 ± 160
<i>Peat and gyttja samples of palynologically tested profile, Moor (peat) Plidutscha, Canton Graubünden, Switzerland</i>		
HAM 1587	VM 7.3 A, peat, 24–35 cm	2710 ± 85
HAM 1588	VM 7.2 A, peat, 50–60 cm	910 ± 140
HAM 1589	VM 7.1 A, peat, 77–87 cm	620 ± 150
HAM 1590	VM 3.1 B, peat with gyttja, 181–192 cm	2810 ± 120
HAM 1591	VM 3.1 A, gyttja with peat, 195–207 cm	3380 ± 120
HAM 1592	VM 4.2, gyttja, 229–242 cm	3820 ± 150
HAM 1593	VM 4.1, gyttja, 256–270 cm	4990 ± 140
HAM 1594	VM 5.3, gyttja, 280–292 cm	5130 ± 145
HAM 1595	VM 6.2 A, clayey gyttja, 382–390 cm	9050 ± 250
HAM 1597	Sediment from Hamburg harbor basin	1730 ± 80



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## HAMBURG RADIOCARBON THIN LAYER SOILS DATABASE

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**ABSTRACT.** We report here the remainder of the Hamburg University dates on thin soil layers (HAM 1652–3129).

### INTRODUCTION

Soils and especially soil organic matter (SOM) have been objects of radiocarbon dating since the early days of the technique (Scharpenseel and Becker-Heidmann 1992). The first major objective, to determine the “age” or starting point of the development of a specific soil, is still open to methodological questions. On the other hand, natural  $^{14}\text{C}$  measurements have been successful in providing information on the dynamics of SOM. Chemical and physical fractions of SOM have been “dated” separately to study the mechanisms of turnover, humification and stabilization. Interest in pedogenesis and the processes of soil development motivated the dating of several or all horizons of a soil profile. In the 1970s, with the increase in global environmental research and computer modeling of the major nutrient cycles, demand grew for more detailed information on soil carbon inventory and fluxes. SOM represents the second largest organic pool in the global carbon cycle, comprising 1200 to 1500 Pg of C in the  $12.8 \times 10^9$  ha of ice-free terrestrial soils and 300–500 Pg of C in the  $6.3 \times 10^8$  ha of wetlands (Scharpenseel and Becker-Heidmann 1994b).

In 1981 we started sampling and dating entire soil profiles as thin layers. Trying to cover the important soil classes, we have now completed  $^{14}\text{C}$  and  $\delta^{13}\text{C}$  measurements on 26 profiles and set up a database in dBASE IV. Here, we present these data together with available additional information according to the specifications of the planned International Radiocarbon Soils Data Base (IRSDB) (Becker-Heidmann 1996). Our intention is to provide the modeling community with test and reference soil data. Useful examples of applications were first published by Harrison *et al.* (1993) and Becker-Heidmann *et al.* (1995).

### METHODS

The soil samples were taken as successive complete 2-cm thin layers from the top to the bottom of the profile, using trowel, meter and water level to control depth and horizontal plane, as described by Becker-Heidmann and Scharpenseel (1986). After air drying and transport to the laboratory in Hamburg, roots and stones were removed. Each sample was carefully homogenized, followed by sieving to 2 mm and drying at 105°C, before subsamples were taken for the different analyses. We measured pH in a 0.01 M  $\text{CaCl}_2$  solution, and tested for the presence of  $\text{CaCO}_3$ .

Organic and inorganic carbon content were measured conductometrically using a Wösthoff apparatus and were separated by temperature (Becker-Heidmann 1990). For the determination of  $\delta^{13}\text{C}$ , the samples were combusted in an oxygen stream. The  $\text{CO}_2$  was purified in a vacuum line and trapped. Details on the preparation line with computer-controlled cooling traps are given in Becker-Heidmann (1989). The measurements were carried out using a high-precision isotope mass spectrometer (Finnigan MAT 250). The natural  $^{14}\text{C}$  concentrations were determined as described by Becker-Heidmann (1989, 1990), *via* combustion and conversion of the sample carbon to strontium carbonate, benzene synthesis and measurement by liquid scintillation spectrometry. Some samples with very low carbon content were augmented with  $\text{CO}_2$  free of  $^{14}\text{C}$  (Scharpenseel and Pietig 1970).

**SOIL DATABASE**

At present, the database contains the measurement results of pH, carbon content,  $\delta^{13}\text{C}$  and  $^{14}\text{C}$  activity of 26 complete profiles. Additionally, the conventional  $^{14}\text{C}$  age (Stuiver and Polach 1977) is given here for organic matter of  $\text{C}_3$  plant origin, *i.e.*, corrected for isotope discrimination to a nominal  $\delta^{13}\text{C}$  value of  $-25\text{‰}$  PDB. Rice (*Oryza sativa* L.), for example, is a  $\text{C}_3$  plant. For carbonates and  $\text{C}_4$ -plant-derived organic matter (*e.g.*, sorghum and tropical grasses) the  $^{14}\text{C}$  age is calculated without isotope correction.

We also list here all available information on the soil profiles relevant for further use in modeling SOM dynamics. The profile description is kept brief; where the detailed results have already been published, reference is made to the corresponding literature.

The profiles are grouped according to their hydrological, climatic and management regime and their location.

- A Terrestrial soils
  - A1 Humid climate
    - A1a Forest soils
      - Ohlendorf Forest, Profile A
      - Ohlendorf Forest, Profile B
      - Wohldorf Forest
      - Timmendorf Forest
      - Trittau Forest
    - A1b Agricultural soils
      - Klein Altendorf
      - Savarit, Profile 1
      - Savarit, Profile 2
  - A2 Mediterranean Climate
    - Akko
    - Qedma
  - A3 Semiarid Climate
    - Patancheru, Profile P
    - Patancheru, Profile R
- B Wetland soils
  - Los Baños
  - Pangil
  - Pao
  - Bugallon
  - Tiaong, Profile L
  - Tiaong, Profile H
  - San Dionisio, Profile T
  - Namtou Hsien
  - Pingtung
  - Chum Pae
  - Klong Luang
  - Tachiat, Profile 1
  - Tachiat, Profile 2
  - Tonsang

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## OHLENDORF FOREST, PROFILE A

### Profile-Related Data

Location (longitude, latitude):	52°17'N, 9°42'E
Location (country, next city or village):	Germany, Hiddestorf south of Hannover
Soil order and type (FAO classification):	Stagnic Luvisol
Soil order and type (local classification):	Pseudogley-Parabraunerde
Parent material:	Würmian loess
Mean annual temperature:	8.5°C
Annual rainfall:	620 mm
Vegetation and land use:	120-yr-old beech stand ( <i>Fagus sylvatica</i> L.) with single <i>Carpinus betulus</i> L. and oaks ( <i>Quercus robur</i> L.), farmer's woodland
Site description:	67.0 m above sea level (asl), plane
Date of sampling:	1982
Date of measurement:	1983 and 1984
Collector:	Becker-Heidmann
Submitter:	Becker-Heidmann
Submitter's comment:	See literature refs.
Literature references:	Becker-Heidmann (1989); Becker-Heidmann and Scharpenseel (1986); Neue (1980)

## Description of Profile A at Ohlendorf Forest

Depth (cm)	Horizon	Description
-4 to -1	L	Litter
-1 to 0	Of	
0-2	Ah1	Brown (10YR 5/3) <sup>1</sup> , silt, roots
2-4	Ah2	Silt loam
4-8	Ae	White (10YR 8/2), silt, few roots
8-15	Bhs	Humus enriched, light gray (10YR 7/2), silt loam, frequent roots
15-40	Al1	Clay eluviated, Lessivé, light gray (10YR 7/2), silt loam, few to frequent roots
40-50	Al2	Pale brown (10YR 7/3), silt loam
50-60	SwBt1	Clay enriched, pale brown (10YR 7/4), silt loam, few coarse roots, clay cutanes, few manganese concretions
60-70	SwBt2	Pale brown (10YR 7/4), silt loam
70-78	SdBt	Water impeding, clay enriched, pale brown (10YR 7/4), stark lehmiger Schluff, manganese concretions

TABLE 1. Ohlendorf Forest, Profile A

Lab code	Depth (cm)	pH	C	$\delta^{13}\text{C}$	$^{14}\text{C}$ activity	$^{14}\text{C}$ age
HAM-1652	0-2	--	5.05	-28.0	113.33 $\pm$ 0.74	Modern
HAM-1654	2-4	3.6	2.23	-27.4	99.20 $\pm$ 1.02	30 $\pm$ 80
HAM-1655	4-6	3.5	0.50	-26.8	99.79 $\pm$ 1.03	30 $\pm$ 80
HAM-1656	6-8	3.7	0.53	-27.1	94.50 $\pm$ 0.99	420 $\pm$ 80
HAM-1657	8-10	3.6	1.02	-27.0	94.76 $\pm$ 0.99	400 $\pm$ 80
HAM-1658	10-12	3.7	0.76	-27.0	96.48 $\pm$ 1.00	260 $\pm$ 80
HAM-1659	12-15	3.8	1.01	-26.6	94.07 $\pm$ 0.99	470 $\pm$ 90
HAM-1660	15-18	3.8	0.72	-27.0	94.45 $\pm$ 0.98	430 $\pm$ 80
HAM-1661	18-20	3.8	0.67	-26.7	96.66 $\pm$ 1.00	250 $\pm$ 80
HAM-1662	20-22	3.8	0.53	-26.7	96.70 $\pm$ 0.87	240 $\pm$ 70
HAM-1663	22-24	3.8	0.60	-26.6	96.48 $\pm$ 0.86	260 $\pm$ 70
HAM-1664	24-26	3.8	0.47	-26.8	99.15 $\pm$ 0.89	40 $\pm$ 70
HAM-1665	26-28	3.8	0.50	-27.0	93.69 $\pm$ 0.63	490 $\pm$ 50
HAM-1666	28-30	3.8	0.53	-26.4	93.61 $\pm$ 0.98	510 $\pm$ 80
HAM-1667	30-32	3.8	0.44	-27.0	94.09 $\pm$ 0.85	460 $\pm$ 70
HAM-1668	32-34	3.8	0.45	-26.0	91.62 $\pm$ 0.84	690 $\pm$ 70
HAM-1669	34-36	3.7	0.41	-26.6	94.31 $\pm$ 0.85	440 $\pm$ 70
HAM-1670	36-38	3.8	0.33	-26.3	96.32 $\pm$ 0.97	280 $\pm$ 80
HAM-1671	38-40	3.7	0.44	-25.8	108.67 $\pm$ 0.70	Modern
HAM-1672	40-42	3.8	0.36	-26.7	92.39 $\pm$ 0.63	610 $\pm$ 60
HAM-1673	42-44	3.7	0.37	-26.4	94.42 $\pm$ 0.64	440 $\pm$ 50
HAM-1674	44-46	3.8	0.24	-26.3	93.94 $\pm$ 0.64	480 $\pm$ 50
HAM-1675	46-48	3.8	0.27	-26.4	89.76 $\pm$ 0.94	850 $\pm$ 80
HAM-1676	48-50	3.7	0.25	-26.4	90.30 $\pm$ 0.94	800 $\pm$ 80
HAM-1677	50-52	3.7	0.22	-26.3	86.90 $\pm$ 0.92	1110 $\pm$ 90
HAM-1678	52-54	3.8	0.19	-26.0	86.62 $\pm$ 0.90	1140 $\pm$ 80
HAM-1679	54-56	3.7	0.20	-25.9	83.52 $\pm$ 0.88	1430 $\pm$ 90

<sup>1</sup>For YR soil color codes, see *Munsell Soil Color Charts* (1975).

TABLE 1. Ohlendorf Forest, Profile A (Continued)

Lab code	Depth (cm)	pH	C	δ <sup>13</sup> C	<sup>14</sup> C activity	<sup>14</sup> C age
HAM-1680	56–58	3.7	0.14	-25.7	85.31 ± 0.60	1260 ± 60
HAM-1681	58–60	3.7	0.18	-26.0	80.49 ± 0.85	1730 ± 90
HAM-1682	60–62	3.7	0.14	-25.6	84.28 ± 0.60	1360 ± 60
HAM-1683	62–64	3.7	0.16	-25.6	83.54 ± 0.60	1440 ± 60
HAM-1684	64–66	3.7	0.14	-25.5	77.54 ± 0.58	2030 ± 60
HAM-1685	66–68	3.8	0.12	-25.8	70.48 ± 0.76	2800 ± 90
HAM-1686	68–70	3.9	0.12	-26.0	67.25 ± 0.74	3170 ± 90
HAM-1687	70–72	3.8	0.09	-25.5	84.66 ± 0.60	1330 ± 60
HAM-1688	72–74	3.9	0.11	-25.5	66.06 ± 0.72	3320 ± 90
HAM-1689	74–76	3.9	0.11	-25.6	68.85 ± 0.75	2990 ± 90
HAM-1690	76–78	3.8	0.58	-25.6	79.61 ± 0.58	1820 ± 60

## Bulk Density (Neue 1980: Table 16, p. 177)

Depth (cm)	Bulk density (g cm <sup>-3</sup> )
0–15	0.88
15–33	1.39
33–50	1.34
50–65	1.47
65–80	1.56
80–100	1.60
100–120	1.65
120–138	1.67
138–150	1.61
>150	1.89

**OHLENDORF FOREST, PROFILE B****Profile-Related Data**

Location (longitude, latitude):	52°17'N, 9°42'E
Location (country, next city or village):	Germany, Hiddestorf, south of Hannover
Soil order and type (FAO classification):	Stagnic Luvisol
Soil order and type (local classification):	Parabraunerde-Pseudogley
Parent material:	Würmian loess
Mean annual temperature:	8.5°C
Annual rainfall:	620 mm
Vegetation and land use:	120-yr-old beech stand ( <i>Fagus sylvatica</i> L.) with single <i>Carpinus betulus</i> L. and oaks ( <i>Quercus robur</i> L.), farmer's woodland
Site description:	66.2 m asl, plane, ca. 20 m southeast of Profile A
Date of sampling:	1983
Date of measurement:	1984 and 1985
Collector:	Becker-Heidmann
Submitter:	Becker-Heidmann
Submitter's comment:	See literature references
Literature references:	Becker-Heidmann (1989); Becker-Heidmann and Scharpenseel (1986); Neue (1980)



**Description of Profile B at Ohlendorf Forest**

Depth (cm)	Horizon	Description
-4 to 2	L	Litter
-2 to 0	Of	Fermented litter
0-5	Ah	Dark brown (10YR 4/3), silt loam, frequent roots
5-10	Ae	Pale brown (10YR 6/3), silt, few roots
10-20	Bhs	Light yellowish brown (10YR 6/4), silt loam, frequent roots
20-38	Al1	Clay eluviated, Lessivé, brownish yellow (10YR 6/6), silt, frequent roots
38-44	Al2	Silt
44-58	SwAl	Light yellowish brown (10YR 6/4), pale mottles, silt, few roots, esp. at the lower border of the horizon
58-84	SdBt	Yellowish brown (10YR 5/6) with dark mottles (clay lentils), silty clay loam, in the upper part horizontal root veins, partly filled with Al material, clay cutanes, few manganese concretions
84-90	BtSd1	Yellowish brown (10YR 5/6), more frequently mottled, silt loam, increasing concretions
90-102	BtSd2	Yellowish brown (10YR 5/8), silt loam
102-140	BtSd3	Yellowish brown (10YR 5/6), few roots, partly dead, gray lentil with ferrous coating, gravels partly with manganese coating, silt loam
140+	IIC	Yellowish brown (10YR 5/6) to brown (10YR 5/3) mottled, gravelled loam

TABLE 2. Ohlendorf Forest, Profile B

Lab code	Depth (cm)	pH	C	$\delta^{13}\text{C}$	$^{14}\text{C}$ activity	$^{14}\text{C}$ age
HAM-1910	0-3	3.8	5.42	-26.7	107.22 ± 0.72	Modern
HAM-1911	3-5	3.6	5.17	-26.4	100.23 ± 0.71	Modern
HAM-1912	5-7	3.8	2.10	-26.1	99.52 ± 0.68	20 ± 60
HAM-1913	7-10	3.9	1.01	-26.5	96.91 ± 0.70	230 ± 60
HAM-1914	10-12	3.9	1.45	-26.4	95.48 ± 0.67	350 ± 60
HAM-1915	12-14	4.0	1.28	-26.5	98.37 ± 0.68	110 ± 60
HAM-1916	14-16	4.1	1.41	-26.6	96.48 ± 0.67	260 ± 60
HAM-1917	16-18	3.8	1.02	-26.3	97.74 ± 0.67	160 ± 60
HAM-1918	18-20	3.8	1.36	-26.3	95.82 ± 0.67	320 ± 60
HAM-1919	20-22	3.8	0.73	-26.0	97.09 ± 0.67	220 ± 60
HAM-1920	22-24	3.8	0.59	-26.0	95.00 ± 0.74	400 ± 60
HAM-1921	24-26	3.8	0.58	-26.3	95.18 ± 0.66	380 ± 60
HAM-1922	26-28	4.0	0.39	-25.9	95.51 ± 0.67	360 ± 60
HAM-1923	28-30	3.9	0.40	-25.5	93.20 ± 0.66	560 ± 60
HAM-1924	30-32	3.8	0.32	-25.5	90.69 ± 0.66	780 ± 60
HAM-1925	32-34	3.8	0.31	-25.2	91.03 ± 0.65	750 ± 60
HAM-1926	34-36	3.8	0.34	-25.3	89.03 ± 0.64	930 ± 60
HAM-1927	36-38	3.8	0.16	-25.1	96.47 ± 0.67	290 ± 60
HAM-1928	38-40	3.9	0.20	-25.1	87.29 ± 0.63	1090 ± 60
HAM-1929	40-42	3.8	0.23	-25.0	86.49 ± 0.63	1170 ± 60
HAM-1930	42-44	3.9	0.16	-25.3	84.25 ± 0.62	1370 ± 60
HAM-1931	44-46	3.9	0.12	-25.1	94.57 ± 0.67	450 ± 60
HAM-1932	46-48	3.8	0.15	-25.2	86.61 ± 0.63	1150 ± 60
HAM-1933	48-50	3.8	0.12	-25.1	87.39 ± 0.64	1080 ± 60

TABLE 2. Ohlendorf Forest, Profile B (Continued)

Lab code	Depth (cm)	pH	C	δ <sup>13</sup> C	<sup>14</sup> C activity	<sup>14</sup> C age
HAM-1934	50-52	3.8	0.13	-25.3	74.58 ± 0.59	2350 ± 60
HAM-1935	52-54	3.7	0.14	-25.2	71.09 ± 0.57	2740 ± 70
HAM-1936	54-56	3.5	0.15	-25.0	82.75 ± 0.62	1520 ± 60
HAM-1937	56-58	3.5	0.17	-24.6	95.76 ± 0.67	350 ± 60
HAM-1938	58-60	3.5	0.17	-24.7	85.45 ± 0.59	1270 ± 60
HAM-1939	60-62	3.5	0.20	-24.4	87.76 ± 0.64	1060 ± 60
HAM-1940	62-64	3.6	0.18	-24.4	88.21 ± 0.64	1020 ± 60
HAM-1941	64-66	3.6	0.19	-24.3	75.85 ± 0.59	2230 ± 60
HAM-1942	66-68	3.7	0.15	-24.7	90.02 ± 0.65	850 ± 60
HAM-1943	68-70	3.8	0.17	-24.8	82.29 ± 0.68	1570 ± 70
HAM-1944	70-72	3.6	0.16	-24.5	84.23 ± 0.68	1390 ± 60
HAM-1945	72-74	3.8	0.13	-24.5	78.79 ± 0.66	1920 ± 70
HAM-1946	74-76	4.0	0.11	-24.4	83.34 ± 0.67	1470 ± 70
HAM-1947	76-78	3.9	0.10	-24.4	72.82 ± 0.64	2560 ± 70
HAM-1948	78-80	4.1	0.09	-24.5	85.76 ± 0.68	1240 ± 60
HAM-1949	80-82	4.1	0.08	-24.4	--	--
HAM-1950	82-84	4.2	0.07	-24.5	111.16 ± 0.80	Modern
HAM-1951	84-86	4.0	0.10	-24.2	83.09 ± 0.68	1500 ± 70
HAM-1952	86-88	4.3	0.08	-24.6	88.24 ± 0.80	1010 ± 70
HAM-1953	88-90	4.2	0.09	-24.5	113.45 ± 0.80	Modern
HAM-1954	90-93	4.5	0.08	-24.1	93.04 ± 0.73	590 ± 60
HAM-1955	93-96	4.3	0.09	-24.4	85.60 ± 0.68	1260 ± 60
HAM-1956	96-99	4.6	0.08	-24.1	73.02 ± 0.63	2540 ± 70
HAM-1957	99-102	4.6	0.07	-24.7	123.09 ± 0.84	Modern
HAM-1958	102-105	4.7	0.07	-24.6	95.13 ± 0.72	410 ± 60
HAM-1959	105-108	4.7	0.06	-24.4	96.23 ± 0.73	320 ± 60
HAM-1960	108-111	4.9	0.09	-24.5	92.65 ± 0.71	620 ± 60
HAM-1961	111-114	4.9	0.08	-24.2	71.27 ± 0.70	2730 ± 80
HAM-1962	114-117	5.1	0.07	-24.5	81.21 ± 0.85	1680 ± 80
HAM-1963	117-120	5.1	0.11	-24.0	75.63 ± 0.87	2260 ± 90
HAM-1964	120-123	5.7	0.11	-25.4	81.23 ± 0.67	1660 ± 70
HAM-1965	123-126	5.7	0.07	-24.7	73.77 ± 0.63	2450 ± 70
HAM-1966	126-129	5.8	0.06	-25.7	85.46 ± 0.68	1250 ± 60
HAM-1967	129-132	6.2	0.07	-25.4	76.29 ± 0.64	2170 ± 70
HAM-1968	132-135	6.0	0.06	-25.6	73.61 ± 0.63	2450 ± 70
HAM-1969	135-138	6.0	0.06	-25.6	84.84 ± 0.68	1310 ± 60
HAM-1970	138-140	5.9	0.06	-25.0	84.48 ± 0.79	1350 ± 80
HAM-1971	140-142	5.8	0.06	-25.2	77.71 ± 0.73	2020 ± 80
HAM-1972	142-144	6.0	0.06	-24.9	96.02 ± 0.90	330 ± 80
HAM-1973	144-146	5.9	0.10	-26.4	98.01 ± 0.74	140 ± 60

## Bulk Density (Neue 1980: Table 17, p. 178)

Depth (cm)	Bulk density (g cm <sup>-3</sup> )
0-5	0.92
5-10	1.23
10-20	1.30
20-37	1.37

Bulk Density (Neue 1980: Table 17, p. 178) (Continued)

Depth (cm)	Bulk density (g cm <sup>-3</sup> )
37–50	1.48
50–70	1.52
70–110	1.59
110–130	1.66
130–144	1.72
144–164	1.85

**WOHLDORF FOREST****Profile-Related Data**

Location (longitude, latitude):	53°43'N, 10°9'E
Location (country, next city or village):	Germany, Hamburg
Soil order and type (FAO classification):	Stagnic Luvisol
Soil order and type (local classification):	Podsolige Braunerde above Parabraunerde-Pseudogley
Parent material:	Weichselian boulder cover sand above Saalean boulder loam
Mean annual temperature:	8.5°C
Annual rainfall:	741 mm
Vegetation and land use:	100-yr beech stand, forest, nature reserve
Site description:	31.1 m asl, top of terminal moraine, plane
Date of sampling:	1982
Date of measurement:	1983
Collector:	Becker-Heidmann
Submitter:	Becker-Heidmann
Submitter's comment:	See literature references
Literature references:	Becker-Heidmann (1989); Becker-Heidmann and Scharpenseel (1986); Scharpenseel and Becker-Heidmann (1994b)

**Description of the Profile at Wohldorf Forest**

Depth (cm)	Horizon	Description
–8 to 5	L	Litter
–5 to 3	Of1	Leaf structure visible
–3 to 1	Of2	Macerated litter
–1 to 0	Oh	
0–3	Ah	Grayish brown (10YR 5/2), loamy sand, very frequent roots
3–5	Ae	Light gray (10YR 7/2), loamy sand, few roots
5–11	Bhs	Very pale brown (10YR 7/3), sandy loam, very frequent roots
11–23	Bv	Very pale brown (10YR 7/3), sandy loam, very frequent roots
23–40	IISwAl	Clay eluviated (Lessivé), white (10YR 8/2), sandy loam, few roots
40–50	IISdBt1	Clay enriched, water impeding, very pale brown (10YR 8/3), sandy loam, few roots
50–63	IISdBt2	Very pale brown (10YR 8/4), sandy loam

63–73	IISdBt3	Very pale brown (10YR 8/4), silt loam, roots in leading channels
73–88	IISdBt4	Very pale brown (10YR 8/4), loam, roots in leading channels
88–100	IISdBt5	Very pale brown (10YR 7/4), mottled, sandy loam, roots in leading channels
100–110+	IIICv	Very pale brown (10YR 7/4), loamy sand, gravels, bands, iron enriched

TABLE 3. Wohldorf Forest

Lab code	Depth (cm)	pH	C	$\delta^{13}\text{C}$	<sup>14</sup> C activity	<sup>14</sup> C age
HAM-1750	-8 to -5	--	40.36	-30.4	--	--
HAM-1751	-5 to -4	--	27.38	-30.2	129.23 ± 0.87	Modern
HAM-1752	-4 to -3	--	29.33	-30.0	142.47 ± 0.92	Modern
HAM-1753	-3 to -1	--	25.89	-27.4	145.06 ± 0.94	Modern
HAM-1754	-1 to 0	--	15.77	-27.1	107.05 ± 0.77	Modern
HAM-1755	0–3	3.0	3.46	-28.6	103.26 ± 0.88	Modern
HAM-1756	3–5	3.4	0.72	-28.1	113.27 ± 0.95	Modern
HAM-1757	5–7	3.7	1.36	-27.5	107.18 ± 1.03	Modern
HAM-1758	7–9	3.7	1.26	-27.1	105.00 ± 1.01	Modern
HAM-1759	9–11	3.8	1.46	-27.8	102.44 ± 0.99	Modern
HAM-1760	11–13	3.9	0.75	-27.3	97.93 ± 0.85	130 ± 70
HAM-1761	13–15	4.0	0.88	-27.3	116.07 ± 1.01	Modern
HAM-1762	15–17	3.9	0.99	-27.4	95.51 ± 0.83	330 ± 70
HAM-1763	17–19	4.1	1.13	-27.1	95.37 ± 0.83	350 ± 70
HAM-1764	19–21	4.1	0.45	-26.9	96.69 ± 0.84	240 ± 70
HAM-1765	21–23	4.1	0.26	-27.1	95.08 ± 0.83	370 ± 70
HAM-1766	23–25	4.1	0.78	-27.5	106.75 ± 1.03	Modern
HAM-1767	25–27	4.2	0.47	-27.3	125.62 ± 1.17	Modern
HAM-1768	27–29	4.2	0.46	-27.2	97.96 ± 0.85	130 ± 70
HAM-1769	29–31	4.2	1.29	-27.4	94.84 ± 0.83	390 ± 70
HAM-1770	31–33	4.2	0.53	-27.2	93.50 ± 0.82	510 ± 70
HAM-1771	33–35	4.3	0.21	-27.0	99.24 ± 1.09	30 ± 90
HAM-1772	35–37	4.2	0.38	-27.1	101.49 ± 1.00	Modern
HAM-1773	37–40	4.2	0.16	-26.7	114.98 ± 1.10	Modern
HAM-1774	40–42	4.2	0.16	-26.4	119.28 ± 1.14	Modern
HAM-1775	42–44	4.1	0.14	-26.4	110.81 ± 1.05	Modern
HAM-1776	44–46	4.1	0.17	-25.7	89.87 ± 0.80	850 ± 70
HAM-1777	46–48	4.0	0.16	-26.2	88.57 ± 1.10	960 ± 100
HAM-1778	48–50	4.0	0.15	-25.9	103.67 ± 1.00	Modern
HAM-1779	50–52	3.9	0.15	-25.8	94.06 ± 0.91	480 ± 80
HAM-1780	52–55	3.9	0.10	-25.7	87.14 ± 0.78	1090 ± 70
HAM-1781	55–58	3.8	0.15	-25.5	89.13 ± 0.79	920 ± 70
HAM-1782	58–60	3.9	0.07	-26.1	78.49 ± 0.72	1930 ± 70
HAM-1783	60–63	3.9	0.11	-26.1	94.90 ± 0.92	400 ± 80
HAM-1784	63–65	3.8	0.12	-25.7	94.67 ± 0.92	430 ± 80
HAM-1785	65–67	3.8	0.19	-25.7	83.23 ± 0.76	1460 ± 70
HAM-1786	67–69	3.8	0.10	-25.4	82.14 ± 0.76	1570 ± 70
HAM-1787	69–71	3.8	0.14	-25.4	86.24 ± 0.78	1180 ± 70
HAM-1788	71–73	3.8	0.10	-25.7	92.33 ± 0.92	630 ± 80
HAM-1789	73–75	3.8	0.09	-25.9	85.78 ± 0.85	1220 ± 80
HAM-1790	75–77	3.8	0.09	-25.7	85.11 ± 0.85	1280 ± 80
HAM-1791	77–80	3.8	0.07	-26.1	79.68 ± 0.80	1810 ± 80
HAM-1792	80–82	3.8	0.10	-25.7	77.75 ± 0.79	2010 ± 80
HAM-1793	82–84	3.7	0.09	-25.3	74.73 ± 0.86	2340 ± 90

TABLE 3. Wohldorf Forest (Continued)

Lab code	Depth (cm)	pH	C	$\delta^{13}\text{C}$	$^{14}\text{C}$ activity	$^{14}\text{C}$ age
HAM-1794	84–86	3.8	0.09	-26.2	76.78 $\pm$ 0.71	2100 $\pm$ 80
HAM-1795	86–88	3.8	0.08	-26.3	81.92 $\pm$ 0.75	1580 $\pm$ 70
HAM-1796	88–90	3.7	0.06	-26.1	77.29 $\pm$ 0.73	2050 $\pm$ 80
HAM-1797	90–92	3.7	0.05	-25.4	74.92 $\pm$ 0.70	2310 $\pm$ 80
HAM-1798	92–94	3.6	0.07	-25.0	85.60 $\pm$ 0.84	1250 $\pm$ 90
HAM-1799	94–96	3.7	0.06	-25.6	--	--
HAM-1800	96–98	3.7	0.06	-24.9	78.95 $\pm$ 0.73	1900 $\pm$ 70
HAM-1801	98–100	3.7	0.04	-26.8	82.78 $\pm$ 0.87	1490 $\pm$ 80
HAM-1802	100–102	3.7	0.06	-26.2	99.16 $\pm$ 0.88	50 $\pm$ 70
HAM-1803	102–104	3.8	0.05	-25.7	94.10 $\pm$ 0.79	480 $\pm$ 70
HAM-1804	104–106	3.8	0.04	-27.6	84.71 $\pm$ 0.72	1290 $\pm$ 70
HAM-1805	106–108	3.9	0.04	-28.8	79.20 $\pm$ 0.93	1810 $\pm$ 90
HAM-1806	108–110	4.0	0.03	-27.4	80.96 $\pm$ 1.14	1660 $\pm$ 110

**TIMMENDORF FOREST****Profile-Related Data**

Location (longitude, latitude):	54°0'N, 10°47'E
Location (country, next city or village):	Germany, Timmendorf
Soil order and type (FAO classification):	Stagnic Luvisol
Soil order and type (local classification):	Pseudogley-Parabraunerde
Parent material:	Weichselian loam
Mean annual temperature:	8.2°C
Annual rainfall:	600 mm
Vegetation and land use:	110-yr-old beech ( <i>Fagus sylvatica</i> L.), nature reserve
Site description:	2.5 m asl, plane
Date of sampling:	1982
Date of measurement:	1984
Collector:	Becker-Heidmann
Submitter:	Becker-Heidmann
Submitter's comment:	See literature references
Lab comment:	--
Literature references:	Becker-Heidmann (1989)

**Description of the Profile at Timmendorf Forest**

Depth (cm)	Horizon	Description
-4 to -3	L	Litter
-3 to 0	Of	Leaf structure visible
0–10	Ah1	Black (10YR 2/1), sandy loam, very frequent roots
10–18	Ah2	Dark brown (10YR 3/3), silty clay loam, frequent roots
18–30	AhA1	Dark brown (10YR 3/3), sandy loam, frequent roots
30–38	Al1	Clay eluviated (Lessivé), brown (10YR 4/3), sandy loam, gravels, single vertical roots
38–56	Al2	Brown (10YR 4/3), sandy loam
56–74	SwBt	Clay enriched, dark yellowish brown (10YR 4/4), mottled, clay loam, gravels, horizontal roots, iron and manganese concretions, weathered rock material, mica, single earthworms ( <i>L. terrestris</i> )

74-110	(II)SdBt	Water impeding, clay enriched, dark yellowish brown (10YR 4/6) and yellowish brown (10YR 5/4), sandy loam, few gravels, old horizontal root channels, clay cutanes clearly visible
110+	IIC	Boulder marl

TABLE 4. Timmendorf Forest

Lab code	Depth (cm)	pH	C	δ <sup>13</sup> C	<sup>14</sup> C activity	<sup>14</sup> C age
HAM-1860	0-2	3.5	4.77	-26.8	119.51 ± 0.73	Modern
HAM-1861	2-4	3.5	4.53	-26.7	117.05 ± 0.73	Modern
HAM-1862	4-6	3.5	3.37	-26.6	110.35 ± 0.70	Modern
HAM-1863	6-8	3.4	2.76	-26.5	103.61 ± 0.68	Modern
HAM-1864	8-10	3.5	1.47	-26.5	100.21 ± 0.56	Modern
HAM-1865	10-12	3.6	3.21	-26.4	98.72 ± 0.63	80 ± 50
HAM-1866	12-14	3.7	1.77	-26.2	95.70 ± 0.62	330 ± 50
HAM-1867	14-16	3.7	1.61	-26.7	93.51 ± 0.62	510 ± 50
HAM-1868	16-18	3.7	2.61	-26.3	93.98 ± 0.62	480 ± 50
HAM-1869	18-20	3.8	1.06	-26.2	95.77 ± 0.61	330 ± 50
HAM-1870	20-22	3.7	0.87	-25.0	94.12 ± 0.62	490 ± 50
HAM-1871	22-24	3.9	0.87	-26.0	92.16 ± 0.61	640 ± 50
HAM-1872	24-26	4.0	0.68	-26.2	91.92 ± 0.60	660 ± 50
HAM-1873	26-28	3.8	1.10	-25.9	91.46 ± 0.61	700 ± 50
HAM-1874	28-30	3.9	0.67	-25.9	91.35 ± 0.61	710 ± 50
HAM-1875	30-32	4.0	1.04	-25.9	90.11 ± 0.60	820 ± 50
HAM-1876	32-34	4.0	0.90	-26.2	90.56 ± 0.61	780 ± 50
HAM-1877	34-36	4.0	1.21	-26.0	88.28 ± 0.60	990 ± 50
HAM-1878	36-38	4.2	0.43	-25.7	91.52 ± 0.63	700 ± 60
HAM-1879	38-40	4.0	0.69	-25.6	87.70 ± 0.59	1040 ± 50
HAM-1880	40-42	4.1	0.24	-25.5	87.35 ± 0.59	1080 ± 50
HAM-1881	42-44	4.1	0.39	-25.6	87.01 ± 0.59	1110 ± 60
HAM-1882	44-46	4.1	0.25	-25.2	85.43 ± 0.59	1260 ± 60
HAM-1883	46-48	4.0	0.41	-25.5	84.41 ± 0.61	1350 ± 60
HAM-1884	48-50	4.2	0.13	-25.3	88.72 ± 0.60	960 ± 50
HAM-1885	50-52	4.2	0.20	-25.6	83.69 ± 0.58	1420 ± 60
HAM-1886	52-54	4.2	0.10	-25.2	81.48 ± 0.57	1640 ± 60
HAM-1887	54-56	4.0	0.37	-25.2	88.19 ± 0.60	1010 ± 50
HAM-1888	56-58	4.1	0.18	-25.4	82.28 ± 0.57	1560 ± 60
HAM-1889	58-60	4.2	0.13	-25.3	89.60 ± 0.60	880 ± 50
HAM-1890	60-62	4.0	0.20	-25.3	85.38 ± 0.59	1260 ± 60
HAM-1891	62-64	4.1	0.39	-25.5	83.13 ± 0.58	1480 ± 60
HAM-1892	64-66	3.8	0.42	-24.7	79.73 ± 0.58	1820 ± 60
HAM-1893	66-68	3.9	0.24	-25.1	78.81 ± 0.58	1910 ± 60
HAM-1894	68-70	4.0	0.26	-25.1	76.41 ± 0.55	2160 ± 60
HAM-1895	70-72	4.1	0.16	-25.0	75.48 ± 0.55	2260 ± 60
HAM-1896	72-74	4.4	0.15	-25.1	71.87 ± 0.53	2650 ± 60
HAM-1897	74-76	5.0	0.15	-25.1	71.20 ± 0.53	2730 ± 60
HAM-1898	76-78	5.1	0.10	-24.9	75.28 ± 0.54	2280 ± 60
HAM-1899	78-80	5.0	0.11	-24.8	77.80 ± 0.56	2020 ± 60
HAM-1900	80-82	5.0	0.18	-24.9	80.02 ± 0.56	1790 ± 60
HAM-1901	82-84	5.1	0.18	-24.8	83.46 ± 0.57	1460 ± 60

**TRITTAU FOREST****Profile-Related Data**

Location (longitude, latitude):	53°37'N, 10°28'E
Location (country, next city or village):	Germany, Trittau (Hahnheide)
Soil order and type (FAO classification):	Cambic Podzol
Soil order and type (local classification):	Schwach podsolierte Braunerde above Pseudogley-Parabraunerde
Parent material:	Weichselian boulder cover sand above Weichselian or late Saalean boulder loam (Fuhlsbüttel moraine)
Mean annual temperature:	8.1°C
Annual rainfall:	742 mm
Vegetation and land use:	110-yr-old beech stand, forest
Site description:	70 m asl, slope
Date of sampling:	1982
Date of measurement:	1983/1984
Collector:	Becker-Heidmann
Submitter:	Becker-Heidmann
Submitter's comment:	The profile seemed to be disturbed
Lab comment:	--
Literature references:	Neue (1980)

**Description of the Profile at Trittau Forest:**

Depth (cm)	Horizon	Description
-5 to -3	L	Litter
-3 to -2	Of	Fungus
-2 to 0	Oh	Many roots
0-4	Aeh	Silty loamy sand, gray (10YR 5/1), few roots, undulating border
4-7	Bhs	Dark brown (7.5YR 3/2), many fine roots
7-20	Bv1	Yellowish brown (10YR 5/8), many roots
20-32	Bv2	Weathered
32-44	SwBv	
44-54	IISdBt1	Loamy sand, brownish yellow (10YR 6/6), very few roots, single vertical roots
54+	IISdBt2	Dark brown (10YR 4/6), few roots, manganese concretions

TABLE 5. Trittau Forest

Lab code	Depth (cm)	pH	C	$\delta^{13}\text{C}$	$^{14}\text{C}$ activity	$^{14}\text{C}$ age
HAM-1704	0-1	3.1	4.61	-27.6	96.85 ± 0.61	215 ± 50
HAM-1705	1-4	3.1	4.05	-27.4	96.78 ± 0.69	220 ± 60
HAM-1708	4-7	3.3	2.75	-26.9	100.61 ± 0.61	Modern
HAM-1709	7-10	3.5	0.99	-26.7	99.86 ± 0.61	Modern
HAM-1710	10-12	3.7	1.02	-26.8	98.81 ± 0.61	65 ± 50
HAM-1711	12-14	3.9	1.11	-26.6	98.58 ± 0.61	90 ± 50
HAM-1712	14-16	4.1	0.72	-26.5	97.82 ± 0.61	155 ± 50
HAM-1713	16-18	4.0	1.08	-26.5	94.50 ± 0.60	430 ± 50
HAM-1714	18-20	4.1	1.02	-26.4	96.36 ± 0.61	275 ± 50
HAM-1715	20-22	4.1	0.76	-26.4	94.85 ± 0.60	400 ± 50

TABLE 5. Trittau Forest (Continued)

Lab code	Depth (cm)	pH	C	δ <sup>13</sup> C	<sup>14</sup> C activity	<sup>14</sup> C age
HAM-1716	22–24	4.1	0.77	-26.4	96.64 ± 0.60	255 ± 50
HAM-1717	24–26	4.1	0.66	-26.3	93.69 ± 0.60	500 ± 50
HAM-1718	26–28	4.1	0.54	-26.2	95.26 ± 0.60	370 ± 50
HAM-1719	28–30	4.1	0.44	-26.0	97.95 ± 0.61	150 ± 50
HAM-1720	30–32	4.1	0.38	-26.0	95.66 ± 0.60	340 ± 50
HAM-1721	32–34	4.1	0.30	-25.6	105.25 ± 0.63	Modern
HAM-1722	34–36	4.1	0.21	-25.3	97.56 ± 0.85	190 ± 70
HAM-1723	36–38	4.0	0.17	-25.0	97.24 ± 0.61	225 ± 50
HAM-1724	38–40	4.0	0.15	-25.1	94.07 ± 0.59	490 ± 50
HAM-1725	40–42	4.0	0.13	-24.6	94.65 ± 0.60	450 ± 50
HAM-1726	42–44	3.9	0.12	-25.0	122.70 ± 0.69	Modern
HAM-1727	44–46	3.9	0.10	-24.9	101.57 ± 0.62	Modern
HAM-1728	46–48	3.8	0.10	-24.8	102.90 ± 0.63	Modern
HAM-1729	48–50	3.8	0.08	-24.2	93.99 ± 0.69	510 ± 60
HAM-1730	50–52	3.7	0.08	-24.3	98.96 ± 0.61	95 ± 50
HAM-1731	52–54	3.7	0.07	-24.3	123.23 ± 1.38	Modern
HAM-1732	54–56	3.7	0.08	-24.3	117.03 ± 2.02	Modern
HAM-1733	56–58	3.7	0.08	-24.4	115.97 ± 1.37	Modern
HAM-1734	58–60	3.7	0.07	-24.2	124.35 ± 1.93	Modern

## Bulk density (Neue 1980: Table 24, p. 185)

Depth (cm)	Bulk density (g cm <sup>-3</sup> )
0–3	1.13
3–5	1.29
5–9	1.30
9–20	1.34
20–38	1.67
38–48	1.72
48–68	1.81

**KLEIN ALTENDORF****Profile-Related Data**

Location (longitude, latitude):	50°38'N, 6°59'E
Location (country, next city or village):	Germany, Klein Altendorf south of Bonn
Soil order and type (FAO classification):	Haplic Luvisol
Soil order and type (local classification):	Parabraunerde
Parent material:	Würmian loess
Mean annual temperature:	8.9°C
Annual rainfall:	625 mm
Vegetation and land use:	Test area for fruit production, 1956–1981 apples, 1981–1982 cherries
Site description:	177.5 m asl, plane
Date of sampling:	1982
Date of measurement:	1984
Collector:	Becker-Heidmann



Submitter:	Becker-Heidmann
Submitter's comment:	See literature references
Lab comment:	--
Literature references:	Becker-Heidmann (1989); Scharpenseel and Becker-Heidmann (1994b)

**Description of the Profile at Klein Altendorf Experimental Station**

Depth (cm)	Horizon	Description
0–14	Ap1	Light yellowish brown (10YR 6/4), loamy silt, frequent roots
14–24	Ap2	Very frequent fine roots, loamy silt
24–40	ApA1	Clay eluviated, light yellowish brown (10YR 6/4), loamy silt, few roots
40–54	A11	Clay eluviated, very pale brown (10YR 7/4), loamy silt, very few roots
54–68	A12	Very pale brown (10YR 7/4), loamy silt
68–78	Bt1	Clay enriched, brownish yellow (10YR 6/6), loamy silt, no roots
78–92+	Bt2	Bright yellowish brown (10YR 6/6), loamy silt

TABLE 6. Klein Altendorf

Lab code	Depth (cm)	pH	C	$\delta^{13}\text{C}$	$^{14}\text{C}$ activity	$^{14}\text{C}$ age
HAM-1810	0–2	6.5	0.70	-25.0	87.25 ± 0.66	1090 ± 60
HAM-1811	2–4	6.6	0.82	-25.2	86.61 ± 0.66	1150 ± 60
HAM-1812	4–6	6.4	0.70	-25.0	89.04 ± 0.67	930 ± 60
HAM-1813	6–8	6.2	0.87	-25.7	85.77 ± 0.66	1220 ± 60
HAM-1814	8–10	6.0	0.88	-24.9	88.40 ± 0.66	990 ± 60
HAM-1815	10–12	5.9	0.97	-25.0	88.72 ± 0.66	960 ± 60
HAM-1816	12–14	5.8	1.00	-25.9	89.02 ± 0.84	920 ± 80
HAM-1817	14–16	5.8	1.11	-25.1	94.35 ± 0.69	470 ± 60
HAM-1818	16–18	6.0	1.01	-24.9	87.49 ± 0.66	1080 ± 60
HAM-1819	18–20	6.2	1.19	-25.7	95.23 ± 0.62	380 ± 50
HAM-1820	20–22	6.1	1.16	-25.7	96.43 ± 0.67	280 ± 60
HAM-1821	22–24	6.0	1.17	-26.0	96.30 ± 0.67	290 ± 60
HAM-1822	24–26	6.0	0.92	-24.7	89.99 ± 0.65	850 ± 60
HAM-1823	26–28	6.1	0.81	-25.4	89.09 ± 0.63	920 ± 60
HAM-1824	28–30	6.2	0.74	-24.5	90.43 ± 0.65	820 ± 60
HAM-1825	30–32	6.3	0.58	-23.9	89.50 ± 0.64	910 ± 60
HAM-1826	32–34	6.3	0.44	-24.2	87.90 ± 0.62	1050 ± 60
HAM-1827	34–36	6.4	0.43	-24.1	89.98 ± 0.65	860 ± 60
HAM-1828	36–38	6.3	0.41	-24.1	87.49 ± 0.64	1090 ± 60
HAM-1829	38–40	6.1	0.38	-24.0	87.17 ± 0.63	1120 ± 60
HAM-1830	40–42	6.1	0.37	-24.6	84.50 ± 0.62	1360 ± 60
HAM-1831	42–44	6.3	0.38	-24.7	81.79 ± 0.62	1620 ± 60
HAM-1832	44–46	6.2	0.37	-23.5	79.80 ± 0.63	1840 ± 60
HAM-1833	46–48	6.3	0.36	-23.3	81.17 ± 0.61	1700 ± 60
HAM-1834	48–50	6.4	0.36	-24.3	80.51 ± 0.61	1750 ± 60
HAM-1835	50–52	6.5	0.36	-24.9	79.67 ± 0.61	1830 ± 60
HAM-1836	52–54	6.2	0.35	-23.8	78.80 ± 0.60	1930 ± 60
HAM-1837	54–56	6.2	0.36	-24.0	79.38 ± 0.75	1870 ± 80
HAM-1838	56–58	6.5	0.36	-23.8	74.43 ± 0.59	2390 ± 60
HAM-1839	58–60	6.5	0.36	-23.9	72.15 ± 0.57	2640 ± 60
HAM-1840	60–62	6.5	0.39	-24.1	68.25 ± 0.56	3080 ± 70

TABLE 6. Klein Altendorf (Continued)

Lab code	Depth (cm)	pH	C	$\delta^{13}\text{C}$	<sup>14</sup> C activity	<sup>14</sup> C age
HAM-1841	62–64	6.6	0.35	-23.9	67.36 ± 0.55	3190 ± 70
HAM-1842	64–66	6.3	0.35	-23.6	66.26 ± 0.55	3330 ± 70
HAM-1843	66–68	6.3	0.34	-23.8	65.00 ± 0.54	3480 ± 70
HAM-1844	68–70	6.6	0.38	-23.8	60.60 ± 0.53	4040 ± 70
HAM-1845	70–72	6.6	0.38	-24.2	59.93 ± 0.53	4130 ± 70
HAM-1846	72–74	6.4	0.39	-24.9	58.72 ± 0.53	4280 ± 70
HAM-1847	74–76	6.5	0.38	-24.9	59.30 ± 0.52	4200 ± 70
HAM-1848	76–78	6.6	0.39	-24.6	62.88 ± 0.56	3730 ± 70
HAM-1849	78–80	6.3	0.33	-24.5	64.05 ± 0.54	3590 ± 70
HAM-1850	80–82	6.4	0.31	-24.8	63.50 ± 0.54	3650 ± 70
HAM-1851	82–84	6.4	0.26	-24.6	60.48 ± 0.58	4050 ± 80
HAM-1852	84–86	6.6	0.44	-24.6	60.40 ± 0.53	4060 ± 70
HAM-1853	86–88	6.7	0.44	-24.6	60.55 ± 0.52	4040 ± 70
HAM-1854	88–90	6.6	0.35	-24.6	59.60 ± 0.52	4160 ± 70
HAM-1855	90–92	6.5	0.35	-24.7	62.33 ± 0.53	3800 ± 70

**SAVARIT, PROFILE 1****Profile-Related Data**

Location (longitude, latitude):	46°07'N, 0°50'W
Location (country, next city or village):	France, Savarit, 60 km southwest of Niort in Poitou-Charentes province
Soil order and type (FAO classification):	Rendzic Leptosol
Soil order and type (USDA classification):	Lithic Rendoll, clayey skeletal mesic udic
Soil order and type (local classification):	Groies
Parent material:	Malm
Mean annual temperature:	15°C
Annual rainfall:	790 mm
Vegetation and land use:	Wheat, sunflower, currently experimental site for studies on biological nitrogen fixation (BNF)
Site description:	24 m asl, slight slope, top of a toposequence
Date of sampling:	1990
Date of measurement:	1990
Collector:	Becker-Heidmann
Submitter:	Becker-Heidmann

**Submitter's comment:**

Both Savarit soil profiles contain large amounts of carbonate. Therefore, after measuring the pH of the total samples, carbon,  $\delta^{13}\text{C}$  and <sup>14</sup>C activity were measured on the inorganic and organic fraction of the samples separately. The high carbon content, *ca.* 3–4% throughout the profile, supports good crop production. The amounts of organic and inorganic carbon are nearly equal except in the uppermost 2 cm. The  $\delta^{13}\text{C}$  values of organic and inorganic carbon are very distinguishable, with *ca.* -22‰ for organic and -2‰ for inorganic C. We detected no substantial exchange between carbonate- and Humus-C. The humus is well decomposed throughout the whole profile, consistently with high pH. The <sup>14</sup>C activity of the organic carbon is rather low (<100 pmC), which reflects good decomposition of the humus. The <sup>14</sup>C activity of the carbonate is low, corresponding to a high <sup>14</sup>C age between 8000 and 11,000 BP. The reason for its great variation over the soil profile is not clear.

Literature references: Becker-Heidmann (1992); Drachenberg (1992)

**Description of the Profile at Savarit 1**

Depth (cm)	Horizon	Description
0–14	Ap	Silty loam, dark brown, 10 YR 3/3, calcareous, gravels
14–20	R	Clayey silt, brown 10 YR 4/3, calcareous, gravels
20+	C	

TABLE 7. Savarit, Profile 1

Lab code	Depth (cm)	pH	C <sub>org</sub>	δ <sup>13</sup> C <sub>org</sub>	<sup>14</sup> C <sub>org</sub> activity	<sup>14</sup> C <sub>org</sub> age	C <sub>in</sub>	δ <sup>13</sup> C <sub>in</sub>	<sup>14</sup> C <sub>in</sub> activity	<sup>14</sup> C <sub>in</sub> age
HAM-3035	0–2	7.4	2.83	-22.3	96.10 ± 0.56	365 ± 50	3.81	-2.1	39.96 ± 0.40	7370 ± 80
HAM-3036	2–4	7.5	3.37	-20.8	95.35 ± 0.57	450 ± 50	3.52	-2.3	28.53 ± 0.36	10,080 ± 110
HAM-3037	4–6	7.4	3.22	-21.0	95.46 ± 0.56	435 ± 50	3.67	-2.4	23.21 ± 0.35	11,730 ± 130
HAM-3038	6–8	7.4	3.54	-22.2	95.02 ± 0.58	460 ± 50	3.40	-2.8	35.95 ± 0.38	8220 ± 90
HAM-3039	8–10	7.4	3.48	-21.6	95.06 ± 0.56	460 ± 50	3.26	-2.5	33.66 ± 0.52	8750 ± 130
HAM-3040	10–12	7.3	3.37	-21.7	95.58 ± 0.56	415 ± 50	3.45	-2.7	31.64 ± 0.37	9240 ± 100
HAM-3041	12–14	7.4	3.19	-21.0	95.39 ± 0.56	445 ± 50	3.64	-2.5	31.52 ± 0.37	9270 ± 100
HAM-3042	14–16	7.4	3.38	-20.1	93.59 ± 0.55	610 ± 50	3.59	-2.7	25.13 ± 0.35	11,090 ± 120
HAM-3043	16–18	7.4	2.99	-21.5	91.65 ± 0.55	760 ± 50	3.57	-2.3	24.67 ± 0.35	11,240 ± 120

**SAVARIT, PROFILE 2****Profile-Related Data**

Same as Profile 1, except:

Site description:

Bottom of the toposequence

Submitter's comment:

The values of pH, organic carbon, δ<sup>13</sup>C and <sup>14</sup>C are similar to those of Profile 1. The carbonate content is higher, and even more striking; the variation of the values is relatively high. The δ<sup>13</sup>C value is generally *ca.* 2‰ higher. The δ<sup>13</sup>C depth distribution of the organic carbon shows an unexpected rise from -20‰ to -16‰ at *ca.* 8 cm. The <sup>14</sup>C activity of the carbonate fraction changes several times between 20 and 30 pMC; the corresponding <sup>14</sup>C age differs between 10,000 and 15,000 BP within the lower half of the soil profile. The reason for this irregularity is not clear.

**Description of Profile at Savarit 2**

Depth (cm)	Horizon	Description
0–18	Ap	Silty loam, brown 10 YR 4/3, calcareous, many fine roots, gravels
18–30	R	Silty loam, brown 10 YR 4/3, calcareous, some fine roots, gravels
30+	C	

TABLE 8. Savarit, Profile 2

Lab code	Depth (cm)	pH	C <sub>org</sub>	δ <sup>13</sup> C <sub>org</sub>	<sup>14</sup> C <sub>org</sub> activity	<sup>14</sup> C <sub>org</sub> age	C <sub>in</sub>	δ <sup>13</sup> C <sub>in</sub>	<sup>14</sup> C <sub>in</sub> activity	<sup>14</sup> C <sub>in</sub> age
HAM-3044	0–2	7.4	3.00	-20.6	95.32 ± 0.57	455 ± 50	5.28	-1.9	16.83 ± 0.33	14,310 ± 170
HAM-3045	2–4	7.5	3.18	-19.5	94.24 ± 0.56	565 ± 50	5.09	-1.4	16.10 ± 0.33	14,670 ± 170
HAM-3046	4–6	7.4	3.36	-20.1	95.87 ± 0.56	420 ± 50	5.00	-1.5	20.11 ± 0.34	12,880 ± 150
HAM-3047	6–8	7.4	3.24	-19.8	96.71 ± 0.56	355 ± 50	5.22	-1.7	21.50 ± 0.34	12,350 ± 140
HAM-3048	8–10	7.5	3.33	-16.6	97.90 ± 0.57	305 ± 50	5.15	-1.1	26.31 ± 0.35	10,730 ± 120
HAM-3049	10–12	7.3	3.23	-19.6	93.15 ± 0.56	660 ± 50	5.02	-1.6	18.28 ± 0.33	13,650 ± 160
HAM-3050	12–14	7.3	3.41	-21.4	95.39 ± 0.56	440 ± 50	4.93	-1.5	26.77 ± 0.36	10,590 ± 120
HAM-3051	14–16	7.4	3.48	-20.3	92.57 ± 0.56	700 ± 50	5.10	-1.6	15.66 ± 0.32	14,890 ± 180

TABLE 8. Savarit, Profile 2 (Continued)

Lab code	Depth (cm)	pH	C <sub>org</sub>	δ <sup>13</sup> C <sub>org</sub>	<sup>14</sup> C <sub>org</sub> activity	<sup>14</sup> C <sub>org</sub> age	C <sub>in</sub>	δ <sup>13</sup> C <sub>in</sub>	<sup>14</sup> C <sub>in</sub> activity	<sup>14</sup> C <sub>in</sub> age
HAM-3052	16–18	7.4	2.99	-20.6	97.58 ± 0.56	265 ± 50	5.54	-1.1	28.61 ± 0.36	10,050 ± 110
HAM-3053	18–20	7.5	2.36	-19.9	95.23 ± 0.56	475 ± 50	5.57	-0.8	19.29 ± 0.34	13,220 ± 150
HAM-3054	20–22	7.5	2.21	-17.7	93.45 ± 0.55	660 ± 50	5.61	-0.5	22.86 ± 0.34	11,850 ± 130
HAM-3055	22–24	7.5	1.94	-19.4	92.59 ± 0.55	710 ± 50	5.36	-0.7	20.84 ± 0.34	12,600 ± 140

**AKKO****Profile-Related Data**

Location (longitude, latitude):	32°54'N, 35°15'E
Location (country, next city or village):	Israel, Akko
Soil order and type (USDA classification):	Typic Pelloxerert, fine, mixed (calcareous), thermic
Soil order and type (local classification):	Grumusol
Mean annual temperature:	19°C
Annual rainfall:	626 mm
Vegetation and land use:	Noncultivated

**Site description:**

The profile was located on site GR-4 of the Western Galilee Experimental Farm in the coastal plain near Akko, in the north of Israel. The soil had been studied before by Yaalon and Kalmar (1972, 1978) regarding its pedoturbation by cracking and swelling processes.

Date of sampling:	1985
Date of measurement:	1986/1987
Collector:	Dov Kalmar, Dan Yaalon
Submitter:	Dov Kalmar

**Lab comment:**

This Vertisol has a pH close to 8 throughout the profile. Accordingly, the carbonate content is on average as high as the organic carbon content, almost exceeding the latter in the bottom of the soil, and only less in the A1 horizon. The δ<sup>13</sup>C value of the organic matter corresponds to the C<sub>3</sub>-type crops grown here, with -28‰ at the soil surface, and also the shift with depth of ca. 3‰ is in the expected range for terrestrial soils (cf. Becker-Heidmann 1989; Bertram 1986). The δ<sup>13</sup>C value of the inorganic carbon ca. -13‰ is low compared to primary carbonate with 0‰. As in the other carbonate-containing profiles, an exchange between organic and inorganic matter has occurred within the whole profile. The shift, when related to the absolute δ<sup>13</sup>C values of the organic fraction, is comparable. Therefore, the same mechanism can be assumed. The exchange is also obvious when comparing the <sup>14</sup>C activity values. The carbonate of this soil is assumed to belong to the original sediment from which the soil developed, i.e., it should have a very low <sup>14</sup>C activity corresponding to a high <sup>14</sup>C age. Instead, it follows a depth curve similar to that of the <sup>14</sup>C activity of the organic carbon, at a lower level, ending up with a <sup>14</sup>C activity of ca. 20 pMC, corresponding to a <sup>14</sup>C age of 14,000 BP. The <sup>14</sup>C activities of the organic fraction as well as of the carbonate carbon fraction decrease distinctively with depth, and there is no discontinuity in the depth curve. So far, a pedoturbation, as expected for a Vertisol, could not be found.

Literature references: Becker-Heidmann (1990); Yaalon and Kalmar (1972)

**Description of the Profile at Akko**

Depth (cm)	Horizon	Description
0–15	A	Heavy clay, very dark grayish brown, very small carbonate nodules, many fine and medium roots
15–40	B1	Heavy clay, very dark grayish brown, very small carbonate nodules, many fine roots

40–100	B21	Heavy clay, very dark grayish brown, very small carbonate nodules, many slickensides, very few fine roots
100–140	B22	Heavy clay, very dark grayish brown, very small carbonate nodules, many well-expressed slickensides, very few fine roots
140–187+	B23	Heavy clay, very dark grayish brown, some carbonate nodules, many slickensides, no roots

TABLE 9. Akko

Lab code	Depth (cm)	pH	C <sub>org</sub>	δ <sup>13</sup> C <sub>org</sub>	<sup>14</sup> C <sub>org</sub> act.	<sup>14</sup> C <sub>org</sub> age	C <sub>in</sub>	δ <sup>13</sup> C <sub>in</sub>	<sup>14</sup> C <sub>in</sub> act.	<sup>14</sup> C <sub>in</sub> age
HAM-2501	0–2	7.4	2.04	-26.5	111.58 ± 0.87	Modern	0.17	-15.5	88.68 ± 0.70	970 ± 60
HAM-2502	2–4	7.6	1.39	-25.9	83.94 ± 0.67	1390 ± 60	0.24	-13.4	--	--
HAM-2503	4–6	7.7	1.13	-26.1	105.74 ± 0.77	Modern	0.04	-12.2	69.73 ± 0.77	2900 ± 100
HAM-2504	6–8	7.6	0.96	-24.7	106.44 ± 0.58	Modern	0.17	-15.6	65.45 ± 0.92	3410 ± 80
HAM-2505	8–10	7.5	0.72	-25.5	104.51 ± 0.76	Modern	0.30	-13.5	70.76 ± 0.49	2780 ± 60
HAM-2506	10–12	7.7	0.65	-24.6	87.63 ± 0.55	1070 ± 50	0.32	-12.4	66.22 ± 0.89	3310 ± 70
HAM-2507	12–15	7.7	0.67	-25.1	105.83 ± 0.63	Modern	0.51	-13.0	58.85 ± 0.89	4260 ± 80
HAM-2508	15–18	7.6	0.78	-24.7	96.49 ± 1.02	290 ± 90	0.20	-13.6	51.09 ± 0.61	5390 ± 70
HAM-2509	18–21	7.6	0.71	-25.2	96.83 ± 1.23	260 ± 100	0.16	-13.4	58.27 ± 1.56	4340 ± 150
HAM-2510	21–24	7.8	0.59	-24.2	97.92 ± 0.73	180 ± 60	0.32	-12.8	72.65 ± 0.58	2570 ± 60
HAM-2511	24–27	7.7	0.63	-24.2	96.49 ± 0.88	300 ± 70	0.26	-13.2	62.42 ± 0.90	3790 ± 80
HAM-2512	27–30	7.7	0.57	-24.4	84.92 ± 0.65	1320 ± 60	0.28	-13.7	60.69 ± 0.88	4010 ± 80
HAM-2513	30–33	8.0	0.57	-24.6	95.21 ± 0.72	400 ± 60	0.25	-12.7	51.78 ± 0.82	5290 ± 90
HAM-2514	33–36	7.9	0.60	-24.0	81.53 ± 5.19	1660 ± 510	0.24	-14.8	55.24 ± 0.90	4770 ± 90
HAM-2515	36–39	7.8	0.53	-23.3	84.34 ± 0.70	1400 ± 70	0.31	-11.5	54.65 ± 0.59	4850 ± 90
HAM-2516	39–42	7.8	0.51	-23.9	80.40 ± 0.73	1770 ± 70	0.23	-13.1	42.60 ± 0.78	6850 ± 100
HAM-2517	42–45	7.8	0.51	-23.6	79.40 ± 0.66	1880 ± 70	0.30	-13.3	45.84 ± 0.81	6270 ± 100
HAM-2518	45–48	7.9	0.41	-23.8	--	--	0.28	-14.7	60.26 ± 0.52	4070 ± 70
HAM-2519	48–51	7.9	0.55	-23.7	78.06 ± 0.65	2010 ± 70	0.12	-12.1	46.90 ± 0.68	6080 ± 80
HAM-2520	51–55	7.9	0.36	-24.2	68.45 ± 0.53	3060 ± 60	0.28	-13.1	58.35 ± 0.45	4330 ± 60
HAM-2521	55–59	7.9	0.40	-23.1	78.90 ± 0.94	1930 ± 100	0.24	-12.4	59.59 ± 0.66	4160 ± 90
HAM-2522	59–63	7.9	0.55	-24.7	77.88 ± 0.65	2010 ± 70	0.19	-13.3	59.13 ± 0.62	4220 ± 80
HAM-2523	63–67	7.5	0.41	-24.4	75.70 ± 0.65	2250 ± 70	0.25	-12.1	43.86 ± 1.34	6620 ± 170
HAM-2524	67–71	7.9	0.37	-23.7	76.83 ± 0.67	2140 ± 70	0.34	-14.1	--	--
HAM-2525	71–75	7.8	0.39	-22.4	70.31 ± 0.66	2870 ± 80	0.35	-12.3	44.91 ± 0.84	6430 ± 100
HAM-2526	75–79	8.0	0.30	-24.4	69.98 ± 0.61	2880 ± 70	0.50	-13.4	54.43 ± 0.54	4890 ± 80
HAM-2527	79–83	7.9	0.37	-24.5	69.22 ± 0.61	2960 ± 70	0.30	-13.1	52.52 ± 0.60	5170 ± 90
HAM-2528	83–87	7.9	0.31	-23.9	72.65 ± 0.53	2580 ± 60	0.43	-13.1	55.18 ± 0.60	4780 ± 90
HAM-2529	87–91	8.0	0.33	-24.5	67.08 ± 0.62	3220 ± 80	0.41	-12.9	52.07 ± 0.59	5240 ± 90
HAM-2530	91–95	8.0	0.29	-23.1	70.16 ± 0.80	2880 ± 90	0.47	-13.1	51.57 ± 0.43	5320 ± 70
HAM-2531	95–99	8.0	0.33	-24.3	66.14 ± 0.68	3330 ± 80	0.33	-13.1	49.07 ± 0.82	5720 ± 90
HAM-2532	99–103	8.0	0.38	-23.1	61.18 ± 2.14	3980 ± 280	0.28	-13.3	51.24 ± 0.45	5370 ± 70
HAM-2533	103–107	7.8	0.38	-24.1	62.37 ± 0.87	3810 ± 110	0.33	-12.3	50.79 ± 1.14	5440 ± 180
HAM-2534	107–111	7.8	0.28	-22.3	63.30 ± 0.67	3720 ± 90	0.26	-12.0	50.70 ± 0.63	5460 ± 100
HAM-2535	111–115	7.9	0.36	-23.7	70.83 ± 0.67	2790 ± 80	0.37	-13.0	48.71 ± 0.55	5780 ± 90
HAM-2536	115–119	7.7	0.39	-23.3	63.99 ± 0.46	3610 ± 60	0.27	-12.7	52.43 ± 0.64	5190 ± 100
HAM-2537	119–123	8.2	0.31	-22.8	60.31 ± 0.50	4100 ± 90	0.31	-12.0	47.88 ± 0.58	5920 ± 100
HAM-2538	123–127	7.9	0.41	-21.8	61.37 ± 0.56	3970 ± 70	0.25	-12.3	49.16 ± 0.54	5700 ± 90
HAM-2539	127–131	8.2	0.28	-22.9	57.68 ± 0.58	4450 ± 80	0.40	-11.5	48.53 ± 0.57	5810 ± 90
HAM-2540	131–135	7.9	0.35	-21.8	56.38 ± 0.61	4660 ± 90	0.45	-13.1	45.29 ± 0.41	6360 ± 70
HAM-2541	135–139	8.0	0.34	-23.2	53.95 ± 0.64	4990 ± 100	0.43	-12.8	45.00 ± 0.61	6410 ± 110
HAM-2542	139–143	8.1	0.33	-23.2	59.52 ± 0.55	4200 ± 80	0.46	-11.9	31.97 ± 0.80	9160 ± 140
HAM-2543	143–147	8.2	0.27	-23.1	55.97 ± 0.56	4690 ± 80	0.59	-11.1	28.98 ± 0.60	9950 ± 110
HAM-2544	147–151	8.2	0.24	-23.0	59.40 ± 0.47	4220 ± 60	0.50	-12.1	30.28 ± 0.69	9600 ± 80
HAM-2545	157–163	8.1	0.32	-23.1	47.61 ± 0.53	5990 ± 90	0.49	-11.5	18.91 ± 0.64	13,380 ± 180
HAM-2546	172–176	8.2	0.21	-22.8	42.24 ± 0.40	6960 ± 80	0.58	-11.4	19.79 ± 0.48	13,010 ± 200
HAM-2547	183–187	8.1	0.15	-22.2	41.66 ± 0.50	7080 ± 100	0.64	-11.4	18.41 ± 0.34	13,590 ± 150

## Bulk density (Yaalon and Kalmar 1972)

Depth (cm)	Bulk density(g cm <sup>-3</sup> )
0–15	1.25
15–100	1.31
100–140	1.36
140–185	1.37

## QEDMA

## Profile-Related Data

Location (longitude, latitude):	31°41'N, 34°47'E
Location (country, next city or village):	Israel, Qedma near Qiriat Gat southwest of Jerusalem
Soil order and type (FAO classification):	Calcic Vertisol
Soil order and type (USDA classification):	Typic Pelloxerert, fine, mixed (calcareous), thermic
Soil order and type (local classification):	Grumusol
Parent material:	Aeolian clay
Mean annual temperature:	19°C
Annual rainfall:	465 mm
Vegetation and land use:	Fallow, before cotton (irrigated)
Site description:	100 m asl
The profile was located at a cement mining pit. Therefore, the soil was sampled down to 9 m, giving the chance to study the differences of soil and sediment.	
Date of sampling:	1986
Date of measurement:	1987/1988
Collectors:	Becker-Heidmann, Scharpenseel, Yaalon
Submitter:	Becker-Heidmann, Scharpenseel, Yaalon

## Submitter's comment:

The pH is nearly the same in value and depth curve as in the Akko profile. The organic carbon content is generally extremely low (< 0.5%), whereas the carbonate carbon content is comparably high, 2.5% in the topsoil and between 0.5 and 1% in layer C. The lowest sediment layer, Cca, again has 1.5%. Although the  $\delta^{13}\text{C}$  of the inorganic carbon is nearly homogeneous through the profile, the  $\delta^{13}\text{C}$  value of the organic carbon shows a great variability and an increasing tendency down to 140 cm. It can be concluded that decomposition processes, increasing the  $\delta^{13}\text{C}$  value of the soil remaining residue fraction, are considerable, but that pedoturbation has disturbed the expected linearly increasing depth curve. The inorganic carbon is obviously not influenced by the actual  $\delta^{13}\text{C}$  of the organic matter. The constant  $\delta^{13}\text{C}$  value of  $-10\text{‰}$  throughout the profile indicates that the exchange process between organic and inorganic carbon occurred a long time ago. This interpretation is supported by the <sup>14</sup>C activity of the inorganic carbon, which is very low, *ca.* 40%, corresponding to a <sup>14</sup>C age of nearly 8000 BP. Only the topmost surface layers seem to take part in a recent exchange process. The <sup>14</sup>C activity depth distribution curves of both organic and inorganic carbon show only a very slight decrease down to a depth of 90 cm, which is the maximum depth of observed cracks, and decrease more steeply in the lower part of the profile. In this soil, in contrast to the Akko soil, a distinctive self-mulching effect can therefore be recognized from the <sup>14</sup>C activity curves. The increase in  $\delta^{13}\text{C}$  can be explained by the comparison of transport and mulching speeds: Peloturbation lasts 2000 yr and is slower than organic carbon transport (Yaalon, personal communication).

Literature references: Becker-Heidmann (1990)

## Description of the Profile at Qedma:

Depth (cm)	Horizon	Description
0–8	A11	Heavy clay, very dark grayish brown
8–26	A12	Heavy clay, very dark grayish brown

26–56	B1	Dry and heavy clay, carbonate nodules at 52–56 cm
56–112	B21	Heavy clay, very dark grayish brown many well expressed slickensides, polyeder, Mn concretions, cracks down to 100–112 cm
112–140	B22	Heavy clay, very dark grayish brown, many slickensides
140–250	B23	Slickensides, carbonate nodules at 250 cm
250–750	C	Bedrock
750–900	Cca	Calcic horizon, bedrock

TABLE 10. Qedma

Lab code	Depth		pH	C <sub>org</sub>	δ <sup>13</sup> C <sub>org</sub>	<sup>14</sup> C <sub>org</sub> act.	<sup>14</sup> C <sub>org</sub> age	C <sub>in</sub>	δ <sup>13</sup> C <sub>in</sub>	<sup>14</sup> C <sub>in</sub> act.	<sup>14</sup> C <sub>in</sub> age
	(cm)										
HAM-2552	0–2	8.1	0.35	-25.0	93.99 ± 0.49	495 ± 40	2.30	-8.9	22.31 ± 0.29	12,050 ± 100	
HAM-2553	2–4	8.1	0.31	-20.7	75.50 ± 1.31	2330 ± 140	2.58	-9.4	41.03 ± 0.55	7160 ± 110	
HAM-2554	4–6	8.0	0.29	-20.2	106.24 ± 0.61	Modern	2.62	-11.9	37.64 ± 0.32	7850 ± 70	
HAM-2555	6–8	8.1	0.30	-19.6	106.09 ± 0.52	Modern	2.56	--	35.16 ± 0.32	8400 ± 70	
HAM-2556	8–10	8.2	0.36	-21.2	94.08 ± 0.78	550 ± 70	2.61	-9.8	33.57 ± 0.78	8770 ± 120	
HAM-2557	10–12	8.2	0.37	-20.3	110.32 ± 0.61	Modern	2.26	-9.7	43.21 ± 0.34	6740 ± 60	
HAM-2558	12–14	8.1	0.37	-23.8	105.41 ± 0.52	Modern	2.68	-9.8	36.05 ± 0.32	8200 ± 70	
HAM-2559	14–16	8.0	0.38	-19.6	101.64 ± 0.77	Modern	2.46	-9.6	35.55 ± 0.32	8310 ± 70	
HAM-2560	16–18	8.1	0.41	-19.8	101.64 ± 0.51	Modern	2.37	-9.7	36.40 ± 0.32	8120 ± 70	
HAM-2561	18–20	8.0	0.40	-18.9	102.99 ± 0.60	Modern	2.36	-9.9	33.63 ± 0.38	8750 ± 90	
HAM-2562	20–22	8.0	0.36	-23.5	103.77 ± 0.59	Modern	2.47	-9.7	38.53 ± 0.40	7660 ± 80	
HAM-2563	22–24	8.1	0.31	-21.2	103.44 ± 0.58	Modern	2.50	-9.5	46.21 ± 0.59	6200 ± 100	
HAM-2564	24–26	8.0	0.34	-21.8	103.82 ± 0.61	Modern	2.38	-8.8	40.32 ± 0.40	7300 ± 80	
HAM-2565	26–28	7.9	0.32	-22.2	80.31 ± 0.48	1805 ± 45	2.16	-9.4	38.52 ± 0.33	7660 ± 70	
HAM-2566	28–30	7.8	0.32	-21.1	100.26 ± 0.83	Modern	2.37	-9.6	42.59 ± 0.56	6860 ± 110	
HAM-2567	30–32	8.2	0.31	-22.7	90.37 ± 0.66	850 ± 60	2.22	-9.7	38.95 ± 0.33	7570 ± 70	
HAM-2568	32–36	8.0	0.31	-18.7	90.31 ± 0.57	920 ± 50	2.39	-9.4	37.71 ± 0.40	7830 ± 80	
HAM-2569	36–40	7.8	0.30	-17.6	92.88 ± 0.61	710 ± 50	2.46	-9.9	37.67 ± 0.84	7840 ± 170	
HAM-2570	40–44	7.8	0.29	-16.6	86.43 ± 0.47	1310 ± 40	2.39	-9.9	36.82 ± 0.39	8030 ± 80	
HAM-2571	44–48	8.0	0.30	-19.3	89.35 ± 0.47	995 ± 40	2.56	-9.7	35.83 ± 0.39	8240 ± 80	
HAM-2572	48–52	8.2	0.27	-22.4	92.29 ± 0.48	685 ± 40	2.54	-10.3	36.80 ± 0.51	8030 ± 110	
HAM-2573	52–56	8.1	0.20	-18.0	86.24 ± 0.65	1300 ± 60	2.68	-9.8	37.07 ± 0.72	7970 ± 150	
HAM-2574	56–60	7.9	0.27	-16.4	--	--	2.47	-9.6	35.97 ± 0.32	8210 ± 70	
HAM-2575	60–64	8.1	0.28	-16.3	83.54 ± 0.52	1585 ± 50	2.42	-9.4	34.22 ± 0.32	8610 ± 70	
HAM-2576	64–68	8.1	0.15	-16.8	79.04 ± 0.73	2020 ± 70	2.60	-9.6	31.45 ± 0.43	9290 ± 110	
HAM-2577	68–72	8.3	0.24	-16.0	81.84 ± 0.67	1760 ± 70	2.78	-9.5	29.73 ± 0.81	9740 ± 150	
HAM-2578	72–76	8.2	0.25	-17.3	81.78 ± 0.52	1740 ± 50	2.43	-9.5	37.71 ± 0.38	7830 ± 80	
HAM-2579	76–80	8.3	0.25	-16.4	79.72 ± 0.50	1960 ± 50	2.51	-9.6	30.36 ± 0.31	9580 ± 80	
HAM-2580	80–84	8.3	0.18	-17.1	77.91 ± 0.69	2130 ± 70	2.61	-9.7	34.13 ± 0.79	8640 ± 120	
HAM-2581	84–88	8.1	0.22	-14.8	78.34 ± 0.62	2130 ± 60	2.48	-9.5	30.28 ± 0.37	9600 ± 100	
HAM-2582	88–92	8.3	0.16	-16.0	77.32 ± 0.68	2210 ± 70	2.51	-9.5	30.40 ± 0.54	9570 ± 140	
HAM-2583	92–96	8.3	0.20	-15.6	77.34 ± 0.72	2220 ± 70	2.52	-9.3	27.08 ± 0.50	10,490 ± 150	
HAM-2584	96–100	8.3	0.23	-18.0	74.12 ± 0.46	2520 ± 50	2.44	-9.5	--	--	
HAM-2585	100–104	8.4	0.17	-20.3	71.32 ± 0.63	2790 ± 70	2.58	-8.9	--	--	
HAM-2586	104–108	8.5	0.23	-18.0	65.78 ± 0.47	3480 ± 60	2.54	-8.7	20.58 ± 0.34	12,700 ± 130	
HAM-2587	108–112	8.5	0.20	-13.0	56.45 ± 1.75	4790 ± 240	2.56	-8.8	17.16 ± 0.47	14,160 ± 230	
HAM-2588	112–116	8.2	0.19	-15.6	43.09 ± 0.56	6920 ± 110	2.63	-8.7	15.70 ± 0.32	14,870 ± 160	
HAM-2589	116–120	8.4	0.14	-14.2	44.64 ± 0.57	6650 ± 100	2.45	-9.1	18.33 ± 0.28	13,630 ± 120	
HAM-2590	120–124	8.4	0.18	-13.8	51.25 ± 0.65	5550 ± 100	2.66	-8.6	12.14 ± 0.45	16,940 ± 290	
HAM-2591	124–128	8.3	0.16	-14.3	41.87 ± 1.29	7170 ± 250	2.49	-9.5	10.96 ± 0.48	17,760 ± 380	
HAM-2592	128–132	8.5	0.11	-12.1	37.85 ± 0.39	8010 ± 80	2.22	-7.6	6.32 ± 0.57	22,180 ± 450	
HAM-2593	132–136	8.5	0.18	-13.5	43.82 ± 0.94	6810 ± 110	2.23	-8.6	7.89 ± 0.39	20,400 ± 280	
HAM-2594	136–140	8.3	0.15	-12.0	44.13 ± 1.59	6780 ± 290	2.20	-9.1	24.33 ± 0.29	11,350 ± 90	
HAM-2595	140–144	8.3	0.18	-12.0	21.95 ± 0.35	12,390 ± 120	2.18	-8.4	5.31 ± 0.97	23,580 ± 1370	
HAM-2596	144–148	8.3	0.17	-12.6	39.80 ± 0.47	7600 ± 90	1.93	-8.9	7.30 ± 0.43	21,020 ± 500	
HAM-2597	148–152	8.3	0.15	-13.4	17.07 ± 0.33	14,390 ± 150	2.01	-8.6	5.87 ± 0.29	22,780 ± 390	
HAM-2598	152–156	8.3	0.20	-14.3	--	--	1.79	-7.9	6.51 ± 0.36	21,940 ± 430	
HAM-2599	156–160	8.4	0.16	-13.2	34.59 ± 0.49	8720 ± 110	1.82	-8.6	6.09 ± 0.42	22,480 ± 550	
HAM-2600	160–164	--	0.23	-13.2	21.49 ± 3.00	12,540 ± 1030	1.82	-8.7	5.26 ± 0.55	23,660 ± 610	

TABLE 10. Qedma (Continued)

Lab code	Depth (cm)	pH	C <sub>org</sub>	δ <sup>13</sup> C <sub>org</sub>	<sup>14</sup> C <sub>org</sub> act.	<sup>14</sup> C <sub>org</sub> age	C <sub>in</sub>	δ <sup>13</sup> C <sub>in</sub>	<sup>14</sup> C <sub>in</sub> act.	<sup>14</sup> C <sub>in</sub> age
HAM-2601	164–168	8.3	0.26	-13.0	26.58 ± 0.35	10,840 ± 110	1.77	-8.8	4.25 ± 0.29	25,370 ± 540
HAM-2602	168–172	8.4	0.36	-13.4	33.14 ± 0.52	9060 ± 130	1.67	-9.2	5.61 ± 0.30	23,140 ± 430
HAM-2603	172–176	8.4	0.09	-14.1	36.48 ± 1.01	8280 ± 220	1.90	-9.8	6.72 ± 0.43	21,690 ± 530
HAM-2604	176–180	8.4	0.14	-13.0	28.79 ± 0.43	10,200 ± 120	1.55	-9.1	6.75 ± 0.25	21,650 ± 290
HAM-2605	180–184	8.3	0.10	-14.0	25.79 ± 0.48	11,060 ± 150	1.97	-8.8	4.09 ± 0.45	25,680 ± 820
HAM-2606	184–188	8.4	0.12	-13.7	23.31 ± 0.49	11,888 ± 170	1.93	-8.8	--	--
HAM-2607	188–192	8.4	0.21	-13.9	23.63 ± 0.49	11,770 ± 170	1.76	-9.6	3.46 ± 0.42	27,020 ± 1080
HAM-2608	192–196	8.3	0.18	-14.0	22.68 ± 0.34	12,100 ± 120	1.64	-10.4	3.71 ± 0.42	26,460 ± 980
HAM-2609	196–200	8.4	0.26	-15.0	18.85 ± 0.47	13,570 ± 200	1.45	-10.0	3.06 ± 0.42	28,010 ± 1100
HAM-2610	200–210	8.2	0.21	-16.1	21.68 ± 0.28	12,420 ± 100	1.56	-10.2	1.76 ± 0.23	32,450 ± 1040
HAM-2611	210–220	8.3	0.23	-16.3	--	--	1.47	-11.3	--	--
HAM-2612	220–230	8.4	0.15	-15.3	21.54 ± 0.82	12,490 ± 300	1.48	-11.1	--	--
HAM-2613	230–240	8.3	0.08	-14.9	21.20 ± 0.71	12,420 ± 270	1.58	-10.8	--	--
HAM-2614	240–250	8.3	0.17	-13.8	30.89 ± 0.73	9620 ± 190	1.84	-10.4	1.77 ± 0.41	32,410 ± 1920

The texture is characterized by 21.7% sand, 25.4% silt and 52.9% clay (Andresen 1987). The clay fraction consists mostly of montmorillonite (*ca.* 57%), kaolinite (*ca.* 21%) and illite (*ca.* 13%), besides 3% each of calcite, free oxides and quartz (Gal *et al.* 1974).

## PATANCHERU, PROFILE P

### Profile-Related Data

Location (longitude, latitude):	17°35'N, 78°17'E
Location (country, next city or village):	India, Patancheru near Hyderabad (ICRISAT)
Soil order and type (FAO classification):	Pellic Vertisol
Soil order and type (USDA classification):	Typic Pellustert, very fine, montmorillonitic, calcareous, isohyperthermic
Soil order and type (local classification):	Kasireddipalli Series
Parent material:	Basaltic alluvium
Mean annual temperature:	25.8°C
Annual rainfall:	760 mm
Vegetation and land use:	Sorghum, pulses, safflower
Site description:	540 m asl

The profile is on the ICRISAT farm, *ca.* 500 m southeast of the Rhodustalf profile. Like this profile, it had been prepared for the International Benchmark Sites Network for Agrotechnology Transfer (IBSNAT). A comparable site nearby was later used within the decomposition studies with <sup>14</sup>C labeled groundnut straw by Singer (1993). The vertisols are very homogeneous over the ICRISAT farm area.

Date of sampling:	1983
Date of measurement:	1988/1989
Collector:	Becker-Heidmann
Submitter:	Becker-Heidmann
Submitter's comment:	

The Vertisol differs from the Alfisol in some ways; it contains more clay and silt, but has no argillic horizon. The pH is 0.5–2 higher, because CaCO<sub>3</sub> content is about as high as the organic carbon content in the topsoil and even higher in the subsoil. Inorganic carbon is unhomogeneously distributed, increasing slightly from the top to the AC–C boundary and decreasing in horizon C with depth. Organic C decreases only slightly in the Ap, remains constant *ca.* 0.4% from A1 through A4, and decreases in AC down to 0.2% in C. With <sup>14</sup>C activity generally below 80% NBS, there is very little recent carbon in the soil. As usual, the <sup>14</sup>C activity decreases in Ap and below A14, reaching values of 40% comparable to the Alfisol. The δ<sup>13</sup>C value of organic



carbon is higher than expected for the C<sub>3</sub> plants grown in this soil, and the  $\delta^{13}\text{C}$  value of the carbonate is lower than primary (marine) carbonate. Therefore, an exchange of organic and inorganic carbon in the soil via  $\text{HCO}_3^-$  in solution/dissolution processes is most probable. The decrease of the  $^{14}\text{C}$  activity and the increase of  $^{14}\text{C}$  age, respectively, of organic carbon is generally very low, noteworthy only in the top 10 cm as an indicator of decomposition, and below 90 cm, which corresponds to the maximum depth of cracks due to the self-mulching effect. The typical swelling and shrinking cracks as well as the slickensides in the Vertisol were found down to a depth of 60–90 cm, corresponding to a constant  $^{14}\text{C}$  age, while the  $^{14}\text{C}$  age rises below 90 cm depth. The increase of the  $\delta^{13}\text{C}$  value of the inorganic carbon to  $-2\%$  within the AC means that it consists mainly of primary carbonate here, not influenced by biogenic processes.

Comment on both the Alfisol (R) and the Vertisol (P) profile:

The results of the two ICRISAT soils, concerning decomposition and fixation of organic matter, correspond not only to the abovementioned results of decomposition experiments by Singer (1993), but also to biomass determinations (organic C, Chloroform and fumigize method) by Sahrawat (personal communication). He found more biomass in the Alfisol than in the Vertisol, *i.e.*, the decomposition is more intensive in the Alfisol than in the Vertisol.

Literature references:

Becker-Heidmann (1990); Murthy (1982)

### Description of Profile P at Patancheru

Depth (cm)	Horizon	Description
0–20	Ap	Very dark grayish brown (10YR 3/2), common roots, lime
20–40	A12	Very dark grayish brown (10YR 3/2), common roots, lime
40–60	A13	Very dark gray (10YR 3/1), very few roots, lime concretions
60–90	A14	Very dark gray (10YR 3/1), very few roots, lime concretions
90–130	AC	Very dark gray (10YR 3/1), darkbrown (10YR 3/3) mottles
130–180+	C	Yellowish to olive brown (2.5Y 5/4), basaltic alluvium

TABLE 11. Patancheru, Profile P

Lab code	Depth									
	(cm)	pH	C <sub>org</sub>	$\delta^{13}\text{C}_{\text{org}}$	$^{14}\text{C}_{\text{org}}$ act.	$^{14}\text{C}_{\text{org}}$ age	C <sub>in</sub>	$\delta^{13}\text{C}_{\text{in}}$	$^{14}\text{C}_{\text{in}}$ act.	$^{14}\text{C}_{\text{in}}$ age
HAM-2054	0–2	7.4	0.53	-15.5	81.81 ± 0.53	1615 ± 50	0.45	-6.7	67.86 ± 0.65	3110 ± 80
HAM-2055	2–4	7.4	0.57	-15.2	70.30 ± 0.53	2830 ± 60	0.38	-7.5	70.50 ± 0.66	2810 ± 80
HAM-2056	4–6	7.4	0.52	-14.9	73.57 ± 0.56	2470 ± 60	0.45	-7.0	68.52 ± 0.66	3040 ± 80
HAM-2057	6–8	7.4	0.46	-14.8	74.10 ± 0.50	2410 ± 50	0.32	-6.7	67.50 ± 0.65	3160 ± 80
HAM-2058	8–10	7.4	0.50	-14.9	73.64 ± 0.50	2460 ± 60	0.48	-6.7	66.38 ± 0.64	3290 ± 80
HAM-2059	10–12	7.4	0.52	-14.3	71.86 ± 0.50	2650 ± 50	0.37	-6.4	64.80 ± 1.15	3490 ± 140
HAM-2060	12–14	7.4	0.44	-14.7	72.62 ± 0.50	2570 ± 60	0.42	-6.8	--	--
HAM-2061	14–16	7.4	0.53	-14.7	71.72 ± 0.51	2670 ± 50	0.46	-6.9	64.35 ± 0.64	3540 ± 80
HAM-2062	16–18	7.4	0.43	-14.8	72.13 ± 0.51	2620 ± 60	0.53	-7.0	64.69 ± 0.64	3500 ± 80
HAM-2063	18–20	7.4	0.44	-14.8	63.08 ± 0.47	3700 ± 60	0.58	-6.9	70.94 ± 0.70	2760 ± 80
HAM-2064	20–22	7.4	0.45	-14.8	70.34 ± 0.49	2830 ± 60	0.54	-7.2	65.13 ± 0.63	3440 ± 80
HAM-2065	22–24	7.4	0.46	-14.9	73.50 ± 0.50	2470 ± 60	0.35	-7.4	45.55 ± 0.61	6320 ± 110
HAM-2066	24–26	7.4	0.45	-14.6	32.20 ± 0.38	9100 ± 90	0.50	-6.9	64.04 ± 0.51	3580 ± 60
HAM-2067	26–28	7.5	0.47	-14.6	68.59 ± 0.49	3030 ± 60	0.45	-7.3	64.90 ± 0.48	3470 ± 60
HAM-2068	28–30	7.5	0.46	-14.6	68.13 ± 0.49	3080 ± 60	0.51	-6.7	63.01 ± 0.69	3710 ± 90
HAM-2069	30–32	7.5	0.46	-14.4	69.88 ± 0.49	2880 ± 60	0.42	-7.0	63.88 ± 0.63	3600 ± 80
HAM-2070	32–34	7.5	0.46	-14.8	64.70 ± 0.47	3500 ± 60	0.34	-7.4	64.38 ± 0.63	3540 ± 80
HAM-2071	34–36	7.4	0.47	-14.6	68.85 ± 0.49	3000 ± 60	0.42	-6.9	64.42 ± 0.65	3530 ± 80
HAM-2072	36–38	7.6	0.44	-14.5	74.24 ± 1.08	2390 ± 120	0.50	-6.8	61.09 ± 0.46	3960 ± 60
HAM-2073	38–40	7.6	0.48	-14.5	71.45 ± 0.50	2700 ± 60	0.34	-7.2	62.32 ± 0.63	3800 ± 80
HAM-2074	40–42	7.6	0.46	-14.4	75.49 ± 0.51	2260 ± 50	0.48	-6.5	60.79 ± 0.46	4000 ± 60
HAM-2075	42–44	7.6	0.48	-14.5	73.13 ± 0.60	2510 ± 70	0.38	-6.8	45.47 ± 0.70	6330 ± 120
HAM-2076	44–46	7.6	0.48	-14.7	75.56 ± 0.68	2250 ± 70	0.46	-6.6	59.66 ± 0.62	4150 ± 80
HAM-2077	46–48	7.6	0.48	-14.5	68.33 ± 0.65	3060 ± 80	0.50	-6.8	63.10 ± 0.63	3700 ± 80

TABLE 11. Patancheru, Profile P (Continued)

Lab code	Depth (cm)	pH	C <sub>org</sub>	δ <sup>13</sup> C <sub>org</sub>	<sup>14</sup> C <sub>org</sub> act.	<sup>14</sup> C <sub>org</sub> age	C <sub>in</sub>	δ <sup>13</sup> C <sub>in</sub>	<sup>14</sup> C <sub>in</sub> act.	<sup>14</sup> C <sub>in</sub> age
HAM-2078	48–50	7.6	0.48	-14.5	74.30 ± 0.48	2390 ± 50	0.44	-6.7	61.39 ± 0.62	3920 ± 80
HAM-2079	50–52	7.7	0.46	-14.4	28.47 ± 0.50	10,090 ± 140	0.36	-6.9	59.51 ± 0.61	4170 ± 80
HAM-2080	52–54	7.7	0.44	-14.6	68.24 ± 1.26	3070 ± 150	0.41	-6.9	10.23 ± 0.46	18,310 ± 360
HAM-2081	54–56	7.7	0.46	-14.4	77.24 ± 0.69	2070 ± 70	0.51	-7.0	54.86 ± 0.60	4820 ± 90
HAM-2082	56–58	7.7	0.42	-14.7	81.61 ± 0.61	1630 ± 60	0.55	-6.9	58.33 ± 0.62	4330 ± 90
HAM-2083	58–60	7.7	0.43	-14.3	--	--	0.61	-7.7	--	--
HAM-2084	60–62	7.7	0.46	-14.9	83.54 ± 0.61	1440 ± 60	0.43	-7.4	61.64 ± 0.63	3890 ± 80
HAM-2085	62–64	7.8	0.47	-14.3	76.47 ± 0.95	2160 ± 100	0.52	-6.9	60.14 ± 0.63	4080 ± 80
HAM-2086	64–66	7.8	0.46	-14.3	71.33 ± 0.62	2710 ± 70	0.37	-6.9	57.69 ± 0.61	4420 ± 90
HAM-2087	66–68	7.7	0.46	-14.2	75.82 ± 2.37	2220 ± 250	0.54	-6.8	23.79 ± 0.48	11,530 ± 160
HAM-2088	68–70	7.7	0.45	-14.3	57.40 ± 0.62	4460 ± 90	0.57	-6.7	61.88 ± 0.63	3860 ± 80
HAM-2089	70–72	7.7	0.42	-14.3	30.85 ± 0.52	9450 ± 140	0.62	-6.7	48.47 ± 0.58	5820 ± 100
HAM-2090	72–74	7.8	0.44	-14.2	68.68 ± 0.65	3020 ± 80	0.61	-6.5	59.16 ± 0.62	4220 ± 80
HAM-2091	74–76	7.9	0.46	-14.4	67.73 ± 3.01	3130 ± 360	0.42	-6.7	57.22 ± 0.46	4480 ± 70
HAM-2092	76–78	7.8	0.48	-14.2	65.26 ± 0.50	3430 ± 60	0.40	-6.7	61.24 ± 0.77	3940 ± 100
HAM-2093	78–80	7.8	0.44	-14.2	70.69 ± 0.93	2790 ± 110	0.61	-6.3	58.11 ± 0.61	4360 ± 80
HAM-2094	80–82	7.8	0.47	-14.0	66.09 ± 0.64	3330 ± 80	0.48	-6.3	57.23 ± 0.62	4480 ± 90
HAM-2095	82–84	7.9	0.46	-14.7	75.97 ± 0.68	2210 ± 70	0.43	-7.0	60.39 ± 0.62	4050 ± 80
HAM-2096	84–86	7.9	0.46	-14.0	70.78 ± 0.66	2780 ± 80	0.63	-6.3	55.57 ± 0.61	4720 ± 90
HAM-2097	86–88	8.0	0.44	-14.2	47.85 ± 0.42	5920 ± 70	0.44	-6.4	23.35 ± 0.74	11,680 ± 250
HAM-2098	88–90	7.9	0.43	-14.2	67.08 ± 0.66	3210 ± 80	0.42	-6.8	55.92 ± 0.60	4670 ± 90
HAM-2099	90–92	7.9	0.44	-14.4	60.17 ± 0.53	4080 ± 70	0.42	-6.4	52.32 ± 3.68	5200 ± 560
HAM-2100	92–94	7.9	0.39	-14.2	66.97 ± 0.65	3220 ± 80	0.63	6.1	53.25 ± 0.60	5060 ± 90
HAM-2101	94–96	8.0	0.40	-14.0	63.48 ± 0.65	3650 ± 80	0.50	-5.8	48.39 ± 0.64	5830 ± 110
HAM-2102	96–98	8.0	0.38	-14.0	46.04 ± 0.57	6230 ± 100	0.59	5.7	50.48 ± 0.59	5490 ± 90
HAM-2103	98–100	7.9	0.46	-14.3	74.66 ± 0.67	2350 ± 70	0.40	-5.9	53.12 ± 0.60	5080 ± 90
HAM-2104	100–102	8.1	0.35	-14.0	60.49 ± 0.80	4040 ± 110	0.53	-5.0	47.87 ± 0.58	5920 ± 100
HAM-2105	102–104	8.0	0.35	-13.8	--	--	0.41	-5.4	--	--
HAM-2106	104–106	8.0	0.34	-13.4	60.74 ± 0.56	4000 ± 70	0.66	-4.5	42.91 ± 0.41	6800 ± 80
HAM-2107	106–108	8.0	0.34	-13.6	72.92 ± 0.83	2540 ± 90	0.51	-4.9	44.32 ± 0.57	6540 ± 100
HAM-2108	108–110	8.1	0.30	-13.3	36.61 ± 1.60	8070 ± 350	0.78	-4.2	38.72 ± 0.54	7620 ± 110
HAM-2109	110–112	8.0	0.33	-13.9	71.89 ± 0.67	2650 ± 70	0.56	-4.9	44.66 ± 0.57	6480 ± 100
HAM-2110	112–114	8.0	0.32	-13.8	68.01 ± 0.75	3100 ± 90	0.50	-5.2	41.70 ± 0.55	7030 ± 110
HAM-2111	114–116	8.0	0.27	-13.2	59.11 ± 0.83	4220 ± 110	0.50	-4.9	37.61 ± 0.54	7860 ± 120
HAM-2112	116–118	8.0	0.29	-14.5	47.68 ± 0.58	5950 ± 100	0.48	-4.7	2.83 ± 0.40	28,640 ± 1150
HAM-2113	118–120	8.0	0.27	-13.5	70.86 ± 0.86	2770 ± 100	0.50	-4.4	41.38 ± 0.55	7090 ± 110
HAM-2114	120–122	8.0	0.22	-13.2	63.48 ± 0.77	3650 ± 100	0.46	-4.6	38.99 ± 0.58	7570 ± 120
HAM-2115	122–124	8.1	0.20	-12.4	58.69 ± 0.46	4280 ± 60	0.68	-3.9	29.59 ± 0.51	9780 ± 140
HAM-2116	124–126	8.0	0.20	-13.3	71.37 ± 0.57	2710 ± 60	0.82	-3.4	40.91 ± 0.55	7180 ± 110
HAM-2117	126–128	8.1	0.22	-11.1	61.34 ± 0.86	3930 ± 110	1.04	-2.3	30.68 ± 0.51	9490 ± 130
HAM-2118	128–130	8.0	0.20	-11.3	51.09 ± 0.49	5390 ± 80	1.26	-2.2	22.36 ± 0.49	12,030 ± 180
HAM-2119	130–132	8.0	0.22	-13.0	68.19 ± 0.96	3080 ± 110	0.52	-3.5	33.92 ± 0.52	8690 ± 120
HAM-2120	132–134	8.1	0.18	-12.0	67.84 ± 0.90	3120 ± 110	0.61	-3.3	30.72 ± 0.50	9480 ± 130
HAM-2121	134–136	8.0	0.23	-12.8	40.05 ± 0.55	7350 ± 110	0.53	-3.6	28.27 ± 0.50	10,150 ± 140
HAM-2122	136–138	8.1	0.21	-12.6	63.23 ± 0.63	3680 ± 80	0.46	-3.5	27.42 ± 0.49	10,390 ± 140
HAM-2123	138–140	8.1	0.18	-12.4	66.13 ± 0.64	3320 ± 80	0.58	-3.3	30.10 ± 0.50	9640 ± 140
HAM-2124	140–142	8.0	0.21	-14.1	70.20 ± 0.87	2840 ± 100	0.50	-3.3	30.38 ± 0.51	9570 ± 140
HAM-2125	142–144	8.0	0.19	-11.5	--	--	0.95	-2.7	--	--
HAM-2126	144–146	8.1	0.18	-11.5	--	--	0.59	-3.1	29.54 ± 0.50	9800 ± 140
HAM-2127	146–148	8.1	0.19	-14.0	68.37 ± 0.67	3050 ± 80	0.67	-3.6	22.27 ± 0.47	12,070 ± 170
HAM-2128	148–150	8.0	0.21	-11.6	71.00 ± 1.00	2750 ± 110	0.77	-3.3	26.37 ± 0.49	10,710 ± 150
HAM-2129	150–152	8.1	0.21	-12.5	--	--	0.68	-2.4	20.39 ± 0.46	12,770 ± 180
HAM-2130	152–154	8.1	0.10	-11.4	58.97 ± 0.63	4240 ± 90	0.61	-2.5	19.10 ± 0.47	13,300 ± 200
HAM-2131	154–156	8.1	0.17	-11.4	65.34 ± 0.67	3420 ± 80	0.42	-3.0	28.37 ± 0.51	10,120 ± 140
HAM-2132	156–158	8.1	0.17	-11.2	65.77 ± 0.83	3370 ± 100	0.61	-2.6	21.54 ± 0.48	12,330 ± 180
HAM-2133	158–160	8.1	0.18	-12.4	60.56 ± 0.48	4030 ± 60	0.35	-3.4	20.06 ± 0.47	12,900 ± 190

TABLE 11. Patancheru, Profile P (Continued)

Lab code	Depth (cm)	pH	C <sub>org</sub>	δ <sup>13</sup> C <sub>org</sub>	<sup>14</sup> C <sub>org</sub> act.	<sup>14</sup> C <sub>org</sub> age	C <sub>in</sub>	δ <sup>13</sup> C <sub>in</sub>	<sup>14</sup> C <sub>in</sub> act.	<sup>14</sup> C <sub>in</sub> age
HAM-2134	160–162	8.1	0.17	-11.2	61.33 ± 0.66	3930 ± 90	0.33	-2.9	18.60 ± 0.47	13,510 ± 200
HAM-2135	162–164	8.1	0.12	-13.4	63.81 ± 0.65	3610 ± 80	0.42	-3.2	27.06 ± 0.50	10,500 ± 150
HAM-2136	164–166	8.1	0.16	-12.2	58.98 ± 0.90	4240 ± 120	0.36	-2.9	21.30 ± 0.47	12,420 ± 180
HAM-2137	166–168	8.1	0.15	-12.4	70.77 ± 1.06	2780 ± 120	0.41	-2.8	24.63 ± 0.49	11,260 ± 160
HAM-2138	168–170	8.1	0.17	-12.3	62.27 ± 0.89	3810 ± 120	0.17	-2.8	21.19 ± 0.48	12,460 ± 180
HAM-2139	170–172	8.0	0.19	-11.7	66.38 ± 0.91	3290 ± 110	0.35	-3.2	24.08 ± 0.48	11,440 ± 160
HAM-2140	172–174	8.1	0.15	-10.4	45.01 ± 0.64	6410 ± 110	0.62	-1.9	11.29 ± 0.44	17,520 ± 310
HAM-2141	174–176	8.0	0.16	-11.4	71.86 ± 0.96	2650 ± 110	0.60	-2.1	18.22 ± 0.47	13,680 ± 210
HAM-2142	176–178	8.1	0.16	-12.4	61.40 ± 0.70	3920 ± 90	0.37	-3.1	26.36 ± 0.49	10,710 ± 150
HAM-2143	178–180	8.0	0.19	-13.5	74.36 ± 0.71	2380 ± 80	0.50	-2.5	22.99 ± 0.48	11,810 ± 170
HAM-2144	180–182	8.1	0.15	-12.7	68.05 ± 1.06	3090 ± 130	0.30	-3.0	24.86 ± 0.48	11,180 ± 160
HAM-2145	182–184	8.0	0.16	-12.5	66.20 ± 1.02	3310 ± 120	0.40	-3.1	19.08 ± 0.46	13,310 ± 200
HAM-2146	184–186	8.1	0.15	-13.0	68.34 ± 0.95	3060 ± 110	0.29	-3.0	22.85 ± 0.48	11,860 ± 170

Bulk density: 1–1.2 g cm<sup>-3</sup>, according to VERMA (1990), cited in Singer (1993).

## PATANCHERU, PROFILE R

### Profile-Related Data

Location (longitude, latitude):	17°35'N, 78°17'E
Location (country, next city or village):	India, Patancheru near Hyderabad (ICRISAT)
Soil order and type (FAO classification):	Chromic Luvisol
Soil order and type (USDA classification):	Udic Rhodustalf, clayey-skeletal, mixed, nonacid, isohyperthermic
Soil order and type (local classification):	Patancheru series
Parent material:	Weathered granite-gneiss
Mean annual temperature:	25.8°C
Annual rainfall:	760 mm
Vegetation and land use:	Rainfed Sorghum, maize, pulses
Site description:	540 m asl

The sampling area is located within the experimental farm of the Institute for Crops Research of the Semi-Arid Tropics (ICRISAT). The mean soil temperature is 1° higher than the air temperature. On both soils also decomposition experiments with <sup>14</sup>C labeled straw have been conducted (SINGER 1993). The chosen soil profile had been prepared for the International Benchmark Sites Network for Agrotechnology Transfer (IBSNAT).

Date of sampling:	1983
Date of measurement:	1984/1985
Collector:	Becker-Heidmann
Submitter:	Becker-Heidmann
Submitter's comment:	

The C content of the Rhodustalf profile is very low. Only the topmost 2-cm layer and the bottom of the A12 horizon shows a maximum value of 0.8%, probably due to the recently settled grass vegetation. The site had been fallow since 1973, when ICRISAT was founded. The decomposition of SOM then probably led to the decrease of C content from the bottom to the top of the formerly plowed horizon. In the B horizon the C content is decreasing with depth in the normal manner. The increase beneath the B, in the Cv, is according to increasing CaCO<sub>3</sub> content.

Concerning the natural <sup>14</sup>C activity, four zones can be distinguished in the Patancheru profile. The <sup>14</sup>C activity is equal to the atmospheric bomb carbon level only in the topmost 2-cm layer. The <sup>14</sup>C activity is nearly

uniform, as expected, but <120 pMC below the first 4 cm of the plowed horizon. This indicates low annual C input combined with rapid turnover, in accordance with the low C content of the soil. This interpretation is strongly supported by experiments of Singer (1993) who found 76% of incorporated <sup>14</sup>C-labeled groundnut straw decomposed in a nearby Alfisol after 30 weeks. The  $\delta^{13}\text{C}$  value decreases from -15‰ to -18‰.  $\delta^{13}\text{C}$  usually is not only more negative but also increases with depth in the topsoil, correlating with C<sub>3</sub> plants and the increasing decomposition stage of organic matter with depth (Stout, Goh and Rafter (1981); O'Brien and Stout (1978); Becker-Heidmann and Scharpenseel (1986)). The reason for a different curve in this profile is most probably a recent change in cultivation from pulses (C<sub>3</sub> plants) to sorghum (C<sub>4</sub>), as could be clarified by measurements of  $\delta^{13}\text{C}$  of the vegetation presently found there (Becker-Heidmann 1989, Table 5.1). Some of the fresh organic substances with higher  $\delta^{13}\text{C}$  values move downwards and are added to the older organic matter, which originated in the C<sub>3</sub> crops. Below Ap and A1,  $\delta^{13}\text{C}$  shows a normal pattern. From A12 to B21t horizons, the <sup>14</sup>C activity decreases continuously until it is ca. 60 pMC, corresponding to a rapidly decreasing rejuvenation by translocated carbon. Accordingly, increasing  $\delta^{13}\text{C}$  shows a rise in more decomposed organic matter with depth. In the B22t horizon, then, the tendencies of both <sup>14</sup>C activity and  $\delta^{13}\text{C}$  inflect coincidentally with the beginning of the decline of the clay content. We also found the same effect in Alfisols from temperate zone forests, e.g., at Wohldorf and Ohlendorf Forest (see above) (Becker-Heidmann and Scharpenseel 1986).

Because of the low precipitation at Patancheru, little young organic matter enters and moves downwards into the profile. But with decreasing amounts of clay and, consequently, of clay-complexated old organic matter, the influence of young organic matter grows with depth. Greenland (1971) and Theng (1979) found 98% of the carbon of tropical red soils (C content 1.7%) bound to clay minerals.

Literature references:

Becker-Heidmann (1989); Murthy *et al.* (1982)

#### Description of Profile R at Patancheru

Depth (cm)	Horizon	Description
0–10	Ap	Yellowish red (5YR 5/6), many fine roots
10–20	A12	Reddish brown (5YR 4/4), few fine roots
20–30	B1	Reddish brown (2.5YR 4/4), few fine roots
30–48	B21t	Dark reddish brown (2.5YR 3/4), few fine roots
48–102	B22t	Dark reddish brown (2.5YR 3/4), very few roots
102–146	BC	Reddish (2.5YR 5/4) to yellowish brown (10YR 6/4), iron concretions
146–160+	C	Yellowish brown (10YR 6/4), weathered granite-gneiss

TABLE 12. Patancheru, Profile R

Lab code	Depth (cm)	pH	C	$\delta^{13}\text{C}$	<sup>14</sup> C activity	<sup>14</sup> C age*
HAM-1975	0–2	6.7	0.72	-14.9	114.88 ± 0.71	Modern
HAM-1976	2–4	6.3	0.42	-16.2	101.16 ± 0.66	Modern
HAM-1977	4–6	6.6	0.45	-16.4	107.37 ± 0.67	Modern
HAM-1978	6–8	6.5	0.43	-16.8	105.48 ± 0.67	Modern
HAM-1979	8–10	6.4	0.48	-16.8	107.44 ± 0.68	Modern
HAM-1980	10–12	6.2	0.42	-17.3	104.38 ± 0.66	Modern
HAM-1981	12–14	6.0	0.44	-17.8	99.60 ± 0.64	150 ± 50
HAM-1982	14–16	5.9	0.56	-17.6	97.42 ± 0.63	330 ± 50
HAM-1983	16–18	5.8	0.59	-17.8	95.09 ± 0.62	520 ± 50
HAM-1984	18–20	5.8	0.65	-17.6	92.09 ± 0.61	780 ± 50
HAM-1985	20–22	5.7	0.60	-18.0	90.52 ± 0.61	910 ± 50
HAM-1986	22–24	5.8	0.67	-17.2	89.25 ± 0.60	1040 ± 50
HAM-1987	24–26	5.8	0.58	-17.3	88.94 ± 0.60	1070 ± 50
HAM-1988	26–28	5.8	0.55	-17.4	87.57 ± 0.60	1190 ± 60

TABLE 12. Patancheru, Profile R (Continued)

Lab code	Depth (cm)	pH	C	$\delta^{13}\text{C}$	$^{14}\text{C}$ activity	$^{14}\text{C}$ age*
HAM-1989	28–30	5.9	0.55	-16.9	86.81 ± 0.59	1270 ± 50
HAM-1990	30–32	5.9	0.52	-16.7	86.16 ± 0.59	1330 ± 60
HAM-1991	32–34	6.3	0.53	-16.9	83.74 ± 0.62	1560 ± 60
HAM-1992	34–36	5.9	0.50	-16.9	82.79 ± 0.58	1650 ± 60
HAM-1993	36–38	6.2	0.47	-16.1	78.71 ± 0.56	2070 ± 60
HAM-1994	38–40	5.9	0.48	-16.1	77.76 ± 0.56	2170 ± 60
HAM-1995	40–42	5.9	0.52	-15.2	77.37 ± 0.55	2220 ± 60
HAM-1996	42–44	5.8	0.46	-15.5	73.21 ± 0.55	2660 ± 60
HAM-1997	44–46	5.8	0.51	-15.5	69.79 ± 0.53	3040 ± 60
HAM-1998	46–48	5.7	0.53	-15.4	65.51 ± 0.52	3550 ± 60
HAM-1999	48–50	5.8	0.51	-15.4	65.54 ± 0.52	3550 ± 60
HAM-2000	50–52	5.7	0.45	-15.5	67.06 ± 0.52	3360 ± 60
HAM-2001	52–54	5.8	0.54	-15.3	71.06 ± 0.53	2900 ± 60
HAM-2002	54–56	5.7	0.33	-15.7	62.38 ± 0.51	3940 ± 70
HAM-2003	56–58	5.7	0.40	-15.2	67.52 ± 0.52	3310 ± 60
HAM-2004	58–60	5.7	0.44	-15.3	64.39 ± 0.51	3690 ± 60
HAM-2005	60–62	5.7	0.36	-15.1	63.97 ± 0.60	3750 ± 80
HAM-2006	62–64	5.7	0.40	-15.1	61.99 ± 0.59	4000 ± 80
HAM-2007	64–66	5.8	0.43	-14.9	63.38 ± 0.60	3660 ± 80
HAM-2008	66–68	5.7	0.33	-14.9	61.02 ± 0.58	3970 ± 80
HAM-2009	68–70	5.7	0.31	-15.0	60.79 ± 0.58	4160 ± 80
HAM-2010	70–72	5.8	0.32	-15.4	57.88 ± 0.57	4550 ± 80
HAM-2011	72–74	5.8	0.33	-15.7	60.61 ± 0.58	4170 ± 80
HAM-2012	74–76	6.0	0.49	-15.2	66.44 ± 0.60	3440 ± 70
HAM-2013	76–78	5.8	0.33	-16.0	62.50 ± 0.60	3920 ± 80
HAM-2014	78–80	5.8	0.33	-16.4	64.72 ± 0.60	3630 ± 70
HAM-2015	80–82	5.7	0.26	-17.0	63.37 ± 0.60	3790 ± 80
HAM-2016	82–84	5.7	0.26	-16.4	66.00 ± 0.60	3480 ± 70
HAM-2017	84–86	5.7	0.29	-15.8	62.46 ± 0.59	3930 ± 80
HAM-2018	86–88	5.8	0.31	-16.9	65.92 ± 0.60	3480 ± 70
HAM-2019	88–90	5.8	0.22	-17.1	66.74 ± 0.61	3380 ± 70
HAM-2020	90–92	5.8	0.32	-16.0	67.69 ± 0.61	3280 ± 70
HAM-2021	92–94	5.8	0.29	-16.7	67.25 ± 0.61	3320 ± 70
HAM-2022	94–96	5.8	0.24	-17.3	67.28 ± 0.61	3310 ± 70
HAM-2023	96–98	5.8	0.28	-17.7	65.62 ± 0.60	3500 ± 70
HAM-2024	98–100	5.8	0.25	-17.9	65.74 ± 0.60	3480 ± 70
HAM-2025	100–102	5.8	0.33	-17.7	66.69 ± 0.60	3370 ± 70
HAM-2026	102–104	5.8	0.24	-17.6	64.73 ± 0.59	3610 ± 70
HAM-2027	104–106	5.8	0.23	-17.5	67.97 ± 0.61	3220 ± 70
HAM-2028	106–108	5.8	0.27	-18.1	65.98 ± 0.60	3450 ± 70
HAM-2029	108–110	5.8	0.22	-18.1	67.34 ± 0.61	3290 ± 70
HAM-2030	110–112	5.8	0.30	-17.1	71.70 ± 0.62	2800 ± 70
HAM-2031	112–114	5.8	0.24	-17.5	67.06 ± 0.60	3330 ± 70
HAM-2032	114–116	5.8	0.25	-17.3	66.88 ± 0.60	3360 ± 70
HAM-2033	116–118	5.8	0.28	-17.4	66.53 ± 0.61	3400 ± 70
HAM-2034	118–120	6.0	0.19	-18.1	67.31 ± 0.61	3290 ± 70
HAM-2035	120–122	5.8	0.28	-17.2	65.50 ± 0.60	3530 ± 70
HAM-2036	122–124	6.4	0.24	-17.5	66.30 ± 0.60	3420 ± 70
HAM-2037	124–126	6.8	0.20	-16.9	65.49 ± 0.60	3530 ± 70

TABLE 12. Patancheru, Profile R (Continued)

Lab code	Depth (cm)	pH	C	δ <sup>13</sup> C	<sup>14</sup> C activity	<sup>14</sup> C age*
HAM-2038	126–128	6.7	0.19	-17.2	65.03 ± 0.60	3580 ± 70
HAM-2039	128–130	7.1	0.17	-16.1	59.79 ± 0.61	4280 ± 80
HAM-2040	130–132	7.0	0.23	-15.7	58.14 ± 0.57	4510 ± 80
HAM-2041	132–134	7.2	0.24	-15.2	57.10 ± 0.56	4660 ± 80
HAM-2042	134–136	6.6	0.23	-17.2	65.68 ± 0.60	3500 ± 70
HAM-2043	136–138	6.6	0.19	-16.9	64.81 ± 0.59	3620 ± 70
HAM-2044	138–140	6.9	0.25	-15.3	60.28 ± 0.58	4220 ± 80
HAM-2045	140–142	7.1	0.24	-15.0	59.68 ± 0.57	4150 ± 80
HAM-2046	142–144	7.1	0.23	-15.4	60.60 ± 0.58	4180 ± 80
HAM-2047	144–146	7.2	0.27	-13.1	55.91 ± 0.56	4670 ± 80
HAM-2048	146–148	7.1	0.33	-12.8	54.04 ± 0.55	4940 ± 80
HAM-2049	148–150	7.2	0.41	-10.8	46.80 ± 0.61	6100 ± 110
HAM-2050	150–152	6.9	0.29	-12.1	50.57 ± 0.54	5480 ± 90
HAM-2051	152–154	7.1	0.40	-11.3	50.22 ± 0.54	5530 ± 90
HAM-2052	154–156	7.0	0.34	-11.8	49.44 ± 0.56	5660 ± 90
HAM-2053	156–158	7.1	0.38	-10.9	46.77 ± 0.53	6100 ± 90

\*No isotope correction

Bulk density: 1.5–1.56 g cm<sup>-3</sup>, according to VERMA (1990), cited in Singer (1993).

## LOS BAÑOS

### Profile-Related Data

Location (longitude, latitude):	14°10'N, 121°16'E
Location (country, next city or village):	Philippines, Los Baños, Luzon, IRRI
Soil order and type (USDA classification):	Aeric Tropaquept, fine, mixed, nonacid/noncalcareous, isohyperthermic
Soil order and type (local classification):	Maahas clay series
Parent material:	Mainly volcanic tuffa
Mean annual temperature:	26°C
Annual rainfall:	2150 mm
Vegetation and land use:	Rice (former coconut)
Site description:	21 m asl

The Aeric Tropaquept on the IRRI farm at Los Baños was chosen, because a site of this series has also been used for decomposition experiments with <sup>14</sup>C-labeled rice straw (Martin 1985; Neue and Scharpenseel 1987). The soil was sampled after harvest, before puddling. In earlier geological periods the area was below the sea level of the nearby laguna.

Date of sampling:	1983
Date of measurement:	1985
Collector:	Becker-Heidmann
Submitter:	Becker-Heidmann
Submitter's comment:	

Because of intensive rice cultivation, the Los Baños profile has a low organic C content and, consequently, a high <sup>14</sup>C activity level in the topsoil. The <sup>14</sup>C activity is uniform down to 8 cm, with a value of 121 pMC; below, in the main rooty zone, there is a maximum. After puddling, the <sup>14</sup>C activity should be the same everywhere within the Apg. The <sup>14</sup>C activity of the carbon input to the soil has been decreasing from one season to the next because of the continuing decrease of the <sup>14</sup>C activity of the atmosphere since the atomic bomb

test ban treaty. Therefore, at the time of sampling, after harvest and before puddling, we expected in the main rooty zone between 10 and 14 cm a lower  $^{14}\text{C}$  activity than elsewhere in the Apg. But the uppermost 8 cm showed a lower  $^{14}\text{C}$  activity. This may be interpreted as a rapid translocation of the freshly decomposed material to the surface. This interpretation is supported by the results of decomposition experiments with  $^{14}\text{C}$ -labeled rice straw in the same soil (Becker-Heidmann *et al.* 1985).

The uniform value of  $\delta^{13}\text{C}$  in the entire Ap1g horizon indicates regular homogenization by puddling, whereas the Ap2g seems to be rarely puddled. Below the Apg horizon, the  $^{14}\text{C}$  activity decreases to ca. 100 pMC; the  $^{14}\text{C}$  increases. The portion of soluble, percolating organic substances is obviously too low to cause a considerable rejuvenation in the subsoil. The steep slope of the  $^{14}\text{C}$  activity and the  $\delta^{13}\text{C}$  curve at the plow pan, together with the low C content and maximum clay concentration at this layer, indicates a strong fixation of highly metabolized and aged humic substances to the clay. The percolating younger material passes through to the bottom, as can be concluded from both the  $\delta^{13}\text{C}$  inflection and the high  $^{14}\text{C}$  activity values in the whole basal soil.

Spikes of high-concentration bomb carbon can be found in the lower part of the  $^{14}\text{C}$  activity depth distribution curve. These are located exactly at the transition layers of adjacent horizons, where there are wetter conditions than in the middle of the horizons, because drainage is impeded by texture changes. Thus, percolating organic matter cannot be bound permanently to clay in these layers, which would raise the  $^{14}\text{C}$  age. Another explanation could be that fine roots would concentrate in these layers and, because of their high turnover rate, permanently introduce modern C. However, the amount of fresh root material necessary for such a shift in  $^{14}\text{C}$  activity would also significantly alter  $\delta^{13}\text{C}$ , which has not been observed.

Literature references:

Becker-Heidmann and Scharpenseel (1989); Scharpenseel and Becker-Heidmann (1993); Neue *et al.* (1990); Scharpenseel and Becker-Heidmann (1994b); Martin (1985); Scharpenseel, Pfeiffer and Becker-Heidmann (1995a, 1995b)

#### Description of the Profile at Los Baños:

Depth (cm)	Horizon	Description
0–14	Ap1g	Black (10YR 2/1), fine roots
14–22	Ap2g	
22–36	IIA3	Black (10YR 2/1), yellowish brown (10YR 5/6) mottles, larger aggregates with reddish brown coating (Fe), little fine roots
36–50	IIB1	Yellowish brown, black tongues from IIA3
50–76	IIB2	Yellowish brown, green-blue tongues from IIB2g
76–90	IIB2g	Green-blue
90+	IIIR	Green-blue, large brown mottles (tuffa)

TABLE 13. Los Baños

Lab code	Depth(cm)	pH	C	$\delta^{13}\text{C}$	$^{14}\text{C}$ activity	$^{14}\text{C}$ age
HAM-2147	0–2	6.0	2.02	–22.8	121.80 ± 0.73	Modern
HAM-2148	2–4	5.8	2.26	–22.9	121.80 ± 0.72	Modern
HAM-2149	4–6	5.6	2.08	–23.0	121.40 ± 0.72	Modern
HAM-2150	6–8	5.7	1.90	–22.8	121.35 ± 0.77	Modern
HAM-2151	8–10	5.8	1.89	–22.9	123.65 ± 0.76	Modern
HAM-2152	10–12	6.1	1.82	–22.7	124.60 ± 0.73	Modern
HAM-2153	12–14	6.1	1.65	–22.8	123.20 ± 0.73	Modern
HAM-2154	14–16	6.1	2.10	–22.3	121.36 ± 0.72	Modern
HAM-2155	16–18	6.4	1.65	–22.0	121.36 ± 0.76	Modern
HAM-2156	18–20	6.4	1.36	–21.5	118.48 ± 0.71	Modern

TABLE 13. Los Baños (Continued)

Lab code	Depth(cm)	pH	C	δ <sup>13</sup> C	<sup>14</sup> C activity	<sup>14</sup> C age
HAM-2157	20–22	6.2	1.41	-20.8	116.08 ± 0.70	Modern
HAM-2158	22–24	6.3	0.99	-19.0	109.27 ± 0.67	Modern
HAM-2159	24–26	6.3	0.93	-17.7	106.52 ± 0.67	Modern
HAM-2160	26–28	6.5	0.80	-16.8	104.76 ± 0.66	Modern
HAM-2161	28–30	6.4	0.63	-16.1	101.61 ± 0.65	Modern
HAM-2162	30–32	6.7	0.64	-16.1	101.11 ± 0.65	Modern
HAM-2163	32–34	6.5	0.70	-16.1	102.45 ± 0.65	Modern
HAM-2164	34–36	6.6	0.52	-16.2	111.29 ± 0.68	Modern
HAM-2165	36–38	6.6	0.55	-15.9	106.19 ± 0.66	Modern
HAM-2166	38–40	6.5	0.51	-15.8	101.62 ± 0.65	Modern
HAM-2167	40–42	6.5	0.45	-15.7	103.74 ± 0.66	Modern
HAM-2168	42–44	6.5	0.45	-15.8	108.65 ± 0.67	Modern
HAM-2169	44–46	6.5	0.42	-15.7	104.21 ± 0.68	Modern
HAM-2170	46–48	6.5	0.36	-15.8	102.45 ± 0.65	Modern
HAM-2171	48–50	6.5	0.35	-15.9	119.38 ± 0.72	Modern
HAM-2172	50–52	6.5	0.30	-16.0	104.08 ± 0.66	Modern
HAM-2173	52–54	6.5	0.30	-16.0	102.87 ± 0.72	Modern
HAM-2174	54–56	6.4	0.29	-16.0	102.52 ± 0.65	Modern
HAM-2175	56–58	6.6	0.28	-16.0	109.69 ± 0.68	Modern
HAM-2176	58–60	6.5	0.26	-16.2	101.43 ± 0.65	Modern
HAM-2177	60–62	6.5	0.24	-16.1	101.51 ± 0.65	Modern
HAM-2178	62–64	6.3	0.24	-16.3	100.03 ± 0.64	Modern
HAM-2179	64–66	6.6	0.23	-16.4	102.30 ± 0.66	Modern
HAM-2180	66–68	6.5	0.23	-16.6	102.01 ± 0.65	Modern
HAM-2181	68–70	6.4	0.21	-16.5	103.46 ± 0.65	Modern
HAM-2182	70–72	6.4	0.22	-16.6	99.78 ± 0.64	150 ± 50
HAM-2183	72–74	6.5	0.22	-16.8	103.00 ± 0.65	Modern
HAM-2184	74–76	6.4	0.19	-16.9	99.89 ± 0.69	140 ± 60
HAM-2185	76–78	6.5	0.19	-17.3	107.81 ± 0.67	Modern
HAM-2186	78–80	6.7	0.20	-17.3	109.08 ± 0.86	Modern
HAM-2187	80–82	6.7	0.20	-17.0	102.14 ± 0.72	Modern

Bulk density: (Martin 1985) wet (not directly applicable for our data, as our soil was sampled during dry season)

Depth (cm)	Bulk density (g cm <sup>-3</sup> )
0–5	1.08
5–10	1.13
10–15	1.22
15–20	1.37

### PANGIL

#### Profile-Related Data

Location (longitude, latitude):	14°24'N, 121°28'E
Location (country, next city or village):	Philippines, Pangil, Luzon
Soil order and type (FAO classification):	Eutric Gleysol
Soil order and type (USDA classification):	Aeric Tropaquept, fine, kaolinitic, nonacid/noncalcareous, isohyperthermic



Soil order and type (local classification): Bay clay series  
 Parent material: Alluvium (unspecified)  
 Mean annual temperature: 26°C  
 Annual rainfall: 2150 mm  
 Vegetation and land use: Rice (since >10 yr)  
 Site description: < 5 m asl, flat

The soil is on the site of an experimental station of IRRI. It has been used for lowland rice, but should be prepared for an upland rice experiment shortly after the sampling. The soil is known to suffer from phosphorus-deficiency. The profile was located within a plot that has never been fertilized.

Date of sampling: 1983  
 Date of measurement: 1986/1987  
 Collector: Becker-Heidmann  
 Submitter: Becker-Heidmann

Submitter's comment:

The  $\delta^{13}\text{C}$  value is *ca.* -22‰ in Apg, a value that has been found in several other tropical rice soils (see below). The increase of  $\delta^{13}\text{C}$  with depth due to decomposition is rather small. The  $^{14}\text{C}$  activity is the same as the  $^{14}\text{C}$  activity of the atmosphere in the topsoil. An inhibited decomposition in topsoil can be inferred from the slight increase of  $\delta^{13}\text{C}$  combined with the high C content. This interpretation fits with the known phosphorus deficiency and the low pH in this alluvial soil. It is also known that the farmers had not continuously grown rice in this field because of the low productivity. The  $^{14}\text{C}$  activity decreases very steeply below the plow pan, indicating again the low decomposition but also a low infiltration rate of fresh organic matter, *i.e.*, an effective hardpan.

Lab comment: --

Literature references: Becker-Heidmann and Scharpenseel (1992a); Scharpenseel and Becker-Heidmann (1989)

### Description of Profile at Pangil

Depth (cm)	Horizon	Description
0-16	Apg	Dark yellowish brown (10YR 3/4)
16-20	ABcn	Manganese concretions
20-60	Bg1	Grayish brown (10YR 3/2), red mottles (increasing to the bottom)
60+	Bg2	Lateral water seepage and groundwater table at 70+1

TABLE 14. Pangil

Lab code	Depth (cm)	pH	C	$\delta^{13}\text{C}$	$^{14}\text{C}$ activity	$^{14}\text{C}$ age
HAM-2365	0-2	4.8	2.45	-21.8	122.06 ± 0.84	Modern
HAM-2366	2-4	4.6	2.33	-22.0	122.46 ± 0.87	Modern
HAM-2367	4-6	4.6	2.35	-22.1	122.92 ± 0.84	Modern
HAM-2368	6-8	4.7	2.32	-22.2	122.12 ± 0.84	Modern
HAM-2369	8-10	4.8	2.20	-22.1	124.56 ± 0.85	Modern
HAM-2370	10-12	4.9	1.88	-22.3	121.16 ± 0.93	Modern
HAM-2371	12-14	5.2	1.62	-22.5	119.73 ± 0.88	Modern
HAM-2372	14-16	5.7	1.37	-22.3	113.03 ± 0.80	Modern
HAM-2373	16-18	6.1	0.90	-21.4	104.53 ± 0.76	Modern
HAM-2374	18-20	6.0	0.62	-20.8	96.84 ± 0.73	330 ± 60
HAM-2375	20-22	6.0	0.48	-21.2	90.24 ± 0.70	890 ± 60
HAM-2376	22-24	--	--	--	--	--
HAM-2377	24-26	6.0	0.49	-20.1	89.33 ± 0.70	980 ± 60
HAM-2378	26-28	6.1	0.47	-21.4	86.33 ± 0.65	1240 ± 60

TABLE 14. Pangi (Continued)

Lab code	Depth (cm)	pH	C	$\delta^{13}\text{C}$	$^{14}\text{C}$ activity	$^{14}\text{C}$ age
HAM-2379	28–30	6.0	0.47	-21.2	$83.75 \pm 0.61$	$1490 \pm 50$
HAM-2380	30–32	6.0	0.41	-20.7	$84.85 \pm 0.73$	$1390 \pm 70$
HAM-2381	32–34	6.1	0.42	-21.4	$83.78 \pm 0.60$	$1480 \pm 50$
HAM-2382	34–36	6.0	0.36	-20.5	$83.94 \pm 0.65$	$1480 \pm 60$
HAM-2383	36–38	6.1	0.38	-20.5	$82.35 \pm 0.60$	$1630 \pm 50$
HAM-2384	38–40	6.1	0.37	-20.7	$82.63 \pm 0.67$	$1600 \pm 70$
HAM-2385	40–42	5.9	0.32	-20.5	$83.43 \pm 0.68$	$1530 \pm 70$
HAM-2386	42–44	5.9	0.35	-20.1	$83.75 \pm 0.68$	$1500 \pm 70$
HAM-2387	44–46	6.1	0.48	-19.7	$91.87 \pm 0.71$	$770 \pm 60$
HAM-2388	46–48	6.0	0.30	-19.8	$83.59 \pm 0.67$	$1520 \pm 70$
HAM-2389	48–50	6.2	0.37	-20.8	$82.38 \pm 0.68$	$1630 \pm 70$
HAM-2390	50–52	6.1	0.33	-21.1	$84.19 \pm 0.66$	$1440 \pm 60$
HAM-2391	52–54	6.3	0.35	-21.1	$81.28 \pm 0.65$	$1730 \pm 60$
HAM-2392	54–56	5.8	0.31	-21.5	$79.75 \pm 0.64$	$1870 \pm 70$
HAM-2393	56–58	5.9	0.31	-21.5	$78.95 \pm 0.66$	$1960 \pm 70$
HAM-2394	58–60	6.1	0.32	-21.9	$80.57 \pm 0.65$	$1790 \pm 70$
HAM-2395	60–62	5.9	0.28	-21.9	$80.68 \pm 0.64$	$1770 \pm 60$
HAM-2396	62–64	5.7	0.32	-21.0	$77.60 \pm 0.63$	$2100 \pm 70$
HAM-2397	64–66	5.8	0.32	-21.7	$80.65 \pm 0.64$	$1780 \pm 60$
HAM-2398	66–68	5.6	0.32	-22.9	$76.58 \pm 0.77$	$2180 \pm 80$
HAM-2399	68–70	5.6	0.31	-21.8	$50.92 \pm 0.69$	$5470 \pm 60$

**PAO****Profile-Related Data**

Location (longitude, latitude):	16° N, 120°16'E
Location (country, next city or village):	Philippines, Pao, Pangasinan, Luzon
Soil order and type (USDA classification):	Aeric Tropaquept, fine silty over clayey, mixed, non-calcareous, isohyperthermic
Soil order and type (local classification):	San Manuel clay loam series
Mean annual temperature:	28°C
Annual rainfall:	2250 mm
Vegetation and land use:	Rice (rainy season) / Mungbeans (dry season, planned)
Site description:	--
Date of sampling:	1983
Date of measurement:	1990
Collector:	Becker-Heidmann
Submitter:	Becker-Heidmann
Submitter's comment:	

The organic carbon content is low, *ca.* 1% in the Apg and <0.5% in the subsoil. The  $\delta^{13}\text{C}$  value as well as the increase of  $\delta^{13}\text{C}$  from the surface through the B1 is comparatively high. This might be due to methane formation. The  $^{14}\text{C}$  activity in the Ap1g is only slight lower than the  $^{14}\text{C}$  activity of the atmosphere, indicating a low decomposition activity. The comparatively high activity in the subsoil, its very slow decrease, and especially the single very high value corresponding to a low  $\delta^{13}\text{C}$  at the B1–B2cn boundary, indicate a high percolation rate.

Lab comment:

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Literature references:

Becker-Heidmann (1990); Scharpenseel and Becker-Heidmann (1994a); Scharpenseel and Becker-Heidmann (1994b)

**Description of the Profile at Pao:**

Depth (cm)	Horizon	Description
0–10	Ap1g	Puddled zone, 8–10 “traffic pan”
10–19	Ap2g	Seldomly puddled zone
19–50	B1	
50–83	B2cn	Iron and manganese concretions
83+	B2g	Grayish blue

TABLE 15. Pao

Lab code	Depth (cm)	pH	C	$\delta^{13}\text{C}$	$^{14}\text{C}$ activity	$^{14}\text{C}$ age*
HAM-2330	0–2	7.0	0.98	-18.7	114.02 ± 0.80	Modern
HAM-2331	2–4	7.1	0.97	-18.2	114.37 ± 0.81	Modern
HAM-2332	4–6	7.0	0.99	-18.4	114.80 ± 0.81	Modern
HAM-2333	6–8	7.1	1.02	-17.9	117.49 ± 0.82	Modern
HAM-2334	8–10	7.0	0.95	-17.7	114.26 ± 0.62	Modern
HAM-2335	10–12	7.0	0.93	-17.2	112.39 ± 0.62	Modern
HAM-2336	12–14	7.2	0.86	-16.2	111.48 ± 0.03	Modern
HAM-2337	14–16	6.9	0.71	-14.9	104.57 ± 0.77	Modern
HAM-2338	16–19	6.9	0.64	-15.2	100.37 ± 0.75	130 ± 60
HAM-2339	19–22	6.9	0.59	-14.5	97.86 ± 0.75	340 ± 60
HAM-2340	22–25	6.9	0.49	-14.3	93.28 ± 0.79	730 ± 70
HAM-2341	25–28	6.9	0.52	-14.3	95.40 ± 0.74	550 ± 70
HAM-2342	28–30	7.1	0.49	-13.7	97.24 ± 0.76	410 ± 70
HAM-2343	30–32	6.9	0.47	-14.0	95.33 ± 0.74	560 ± 70
HAM-2344	32–34	6.9	0.44	-15.3	95.08 ± 0.57	560 ± 50
HAM-2345	34–36	6.9	0.35	-14.6	95.35 ± 0.73	550 ± 70
HAM-2346	36–38	6.8	0.42	-14.0	93.85 ± 0.75	690 ± 70
HAM-2347	38–40	6.8	0.34	-14.5	92.82 ± 0.75	770 ± 70
HAM-2348	40–42	6.8	0.36	-14.0	93.26 ± 0.74	740 ± 70
HAM-2349	42–44	7.0	0.36	-13.5	93.58 ± 0.57	720 ± 50
HAM-2350	44–47	6.7	0.32	-13.5	94.06 ± 0.74	680 ± 70
HAM-2351	47–50	6.9	0.29	-15.1	128.23 ± 0.87	Modern
HAM-2352	50–53	6.9	0.28	-15.7	93.71 ± 0.73	670 ± 70
HAM-2353	53–56	6.8	0.29	-13.8	92.44 ± 0.72	810 ± 70
HAM-2354	56–59	6.8	0.26	-14.3	91.99 ± 0.73	840 ± 70
HAM-2355	59–62	6.6	0.25	-14.9	93.03 ± 0.74	740 ± 70
HAM-2356	62–65	6.5	0.24	-14.6	90.99 ± 0.92	930 ± 70
HAM-2357	65–68	6.6	0.25	-14.6	91.93 ± 0.55	840 ± 50
HAM-2358	68–71	6.6	0.27	-14.4	91.32 ± 0.73	900 ± 70
HAM-2359	71–74	6.8	0.27	-14.6	88.98 ± 0.73	1110 ± 70
HAM-2360	74–77	6.5	0.27	-14.8	97.78 ± 0.75	350 ± 70
HAM-2361	77–80	6.5	0.27	-14.8	90.10 ± 0.55	1000 ± 50
HAM-2362	80–83	6.3	0.29	-14.6	89.65 ± 0.72	1050 ± 70
HAM-2363	83–86	7.3	0.31	-14.6	88.88 ± 0.72	1120 ± 70
HAM-2364	86–88	6.5	0.33	-14.6	87.64 ± 0.71	1230 ± 70

\* $\delta^{13}\text{C}$  correction applied, as the high  $\delta^{13}\text{C}$  here results not from  $\text{C}_4$  plants but from methane production.

## BUGALLON

## Profile-Related Data

Location (longitude, latitude): 15°55'N, 120°15'E  
 Location (country, next city or village): Philippines, Bugallon, Pangasinan, Luzon  
 Soil order and type (USDA classification): Haplic Hydraquent, fine loamy to sandy, mixed, non-calcareous, isohyperthermic  
 Parent material: Alluvium  
 Mean annual temperature: 28°C  
 Annual rainfall: 2250 mm  
 Vegetation and land use: Grasses; rice until 3 yr before sampling

## Site description:

The soil profile at Bugallon is located near Pao, ca. 700 m west of the road from Tarlac to Dagupan City. The area belongs to the western part of the Agno River basin between the foothills of the Zambales Mountains in the west and the floodplains of the Agno River. The surface relief is the result of alternate sedimentation and erosion processes during the development of the river beds. The soil has been studied by Hauptenthal *et al.* (1979) with regard to Zn deficiency and by Scharpenseel *et al.* (1981) with regard to Cr- and Ni-toxicity and the possible interaction with the Zn-deficiency. A P deficiency has also been noticed. The soil has developed from ultrabasic, ophiolitic and basic alluvium of the nearby Zambales hills.

Date of sampling: 1983  
 Date of measurement: 1987  
 Collector: Becker-Heidmann  
 Submitter: Becker-Heidmann  
 Submitter's comment:

The Bugallon soil was sampled only down to 30 cm, because artesian groundwater rapidly filled the profile. The carbon content decreases steeply at the plow pan, from 2% in the topsoil to 0.5% in the subsoil.

The  $\delta^{13}\text{C}$  value increases very steeply from a usual value of  $-22\text{‰}$  at the surface to  $-14\text{‰}$  in B2g. Here we have an indication of a decomposition producing mainly methane.

Lab comment: --

Literature references: Becker-Heidmann (1990); Scharpenseel and Becker-Heidmann (1994b)

## Description of the Profile at Bugallon:

Depth (cm)	Horizon	Description
0-12	Ap	Black, rooty to very rooty, silty clay
12-45+	B2g	Olive brown to black, little rooty, clay loam
45		Groundwater table from artesian activity

TABLE 16. Bugallon

Lab code	Depth (cm)	pH	C	$\delta^{13}\text{C}$	<sup>14</sup> C activity	<sup>14</sup> C age
HAM-2315	0-2	7.7	2.17	-22.0	116.19 ± 0.63	Modern
HAM-2316	2-4	7.4	2.22	-21.2	116.69 ± 0.64	Modern
HAM-2317	4-6	7.1	2.18	-21.6	109.07 ± 0.71	Modern
HAM-2318	6-8	7.4	1.96	-21.4	114.76 ± 0.62	Modern
HAM-2319	8-10	7.5	1.89	-20.9	111.01 ± 0.66	Modern
HAM-2320	10-12	7.3	1.39	-20.4	109.62 ± 0.60	Modern
HAM-2321	12-14	7.6	1.04	-17.4	104.88 ± 0.61	Modern
HAM-2322	14-16	7.4	0.79	-14.5	100.96 ± 0.65	Modern

TABLE 16. Bugallon (Continued)

Lab code	Depth (cm)	pH	C	$\delta^{13}\text{C}$	$^{14}\text{C}$ activity	$^{14}\text{C}$ age
HAM-2323	16–18	6.7	0.68	–13.5	95.71 $\pm$ 0.81	540 $\pm$ 70
HAM-2324	18–20	6.9	0.62	–13.4	95.43 $\pm$ 0.67	560 $\pm$ 60
HAM-2325	20–22	7.2	0.46	–13.3	95.10 $\pm$ 0.56	595 $\pm$ 45
HAM-2326	22–24	6.7	0.75	–13.8	67.74 $\pm$ 0.80	3310 $\pm$ 90
HAM-2327	24–26	6.9	0.76	–13.9	65.75 $\pm$ 0.79	3550 $\pm$ 90
HAM-2328	26–28	6.5	0.71	–13.5	66.54 $\pm$ 0.78	3460 $\pm$ 90
HAM-2329	28–30	6.6	0.52	–13.4	65.18 $\pm$ 0.78	3630 $\pm$ 90

**TIAONG, PROFILE L****Profile-Related Data**

Location (longitude, latitude):	13°59'N, 121°21'E
Location (country, next city or village):	Philippines, Tiaong, Luzon
Soil order and type (USDA classification):	Haplic Hydraquent, coarse silty, mixed, calcareous, isohyperthermic
Parent material:	Volcanic ash alluvium from Mt. Banahaw
Mean annual temperature:	26°C
Annual rainfall:	2150 mm
Vegetation and land use:	Rice (grasses at sampling time)
Site description:	43 asl

The profile is the lowest one of a toposequence, investigated earlier by Scharpenseel (1978) and Scharpenseel *et al.* (1981). It is similar to the Bugallon profile, but contains much more plant debris and organic matter. It contains a large number of different species of snails, some of them down to 60 cm depth. Big roots, probably from bamboo, were found in 50 to 60 cm depth (not within the sampling area). Artesian water filled the profile up to 30 cm depth, when the sampling reached 60 cm. The local problems with rice cultivation result from a pronounced Zn deficiency. At the time of sampling the soil was fallow and covered by grasses.

Date of sampling:	1983
Date of measurement:	1990
Collector:	Becker-Heidmann
Submitter:	Becker-Heidmann
Submitter's comment:	

The lower profile of the Tiaong toposequence is characterized by a high pH of 7.5 to 8 throughout the profile and the highest organic carbon content of all studied Philippine rice soils. It contains also a great deal of carbonate, >3% in the topsoil and even 2% in most parts of the subsoil. The  $\delta^{13}\text{C}$  value of the organic matter increases with depth within the Apg, as expected, and decreases below the plow pan in the subsoil. As the  $\delta^{13}\text{C}$  value of the carbonate lies between the  $\delta^{13}\text{C}$  value of primary carbonates and the  $\delta^{13}\text{C}$  of fresh  $\text{C}_3$  plant material, it is obvious that organic and inorganic carbon have exchanged in this profile. The parallel depth distribution curves of organic and inorganic  $\delta^{13}\text{C}$  suggest that this exchange is permanently occurring.

Lab comment:	--
Literature references:	Becker-Heidmann (1990); Scharpenseel and Becker-Heidmann 1994b

**Description of Profile L at Tiaong**

Depth (cm)	Horizon	Description
0–16	Ap1g	Dark brown, very rooty, fibers of bamboo
16–22	Ap2g	Dark brown, very rooty

22–58 B21g Bluish green, rooty  
58–64+ B22g Green blue, snails

TABLE 17. Tiaong, Profile L

Lab code	Depth (cm)	pH	C <sub>org</sub>	δ <sup>13</sup> C <sub>org</sub>	<sup>14</sup> C <sub>org</sub> act.	<sup>14</sup> C <sub>org</sub> age	C <sub>in</sub>	δ <sup>13</sup> C <sub>in</sub>	<sup>14</sup> C <sub>in</sub> act.	<sup>14</sup> C <sub>in</sub> age
HAM-2188	0–2	7.9	9.27	-21.6	113.69 ± 0.81	Modern	3.29	-8.7	97.76 ± 0.76	180 ± 60
HAM-2189	2–4	7.9	8.58	-22.2	112.32 ± 0.80	Modern	3.41	-8.2	99.51 ± 0.75	40 ± 60
HAM-2190	4–6	8.0	8.89	-21.7	112.55 ± 0.62	Modern	3.57	-7.1	91.85 ± 0.73	680 ± 70
HAM-2191	6–8	8.1	8.52	-18.7	118.42 ± 0.83	Modern	3.23	-4.8	90.89 ± 0.55	765 ± 50
HAM-2192	8–10	8.2	7.98	-17.9	115.16 ± 0.81	Modern	3.14	-5.3	97.42 ± 0.75	210 ± 60
HAM-2193	10–12	8.0	7.74	-18.8	116.48 ± 0.82	Modern	3.28	-5.1	115.66 ± 0.82	Modern
HAM-2194	12–14	7.7	6.47	-17.2	118.42 ± 0.82	Modern	3.37	-7.5	99.98 ± 0.76	Modern
HAM-2195	14–16	8.0	6.65	-17.8	94.20 ± 0.74	600 ± 70	3.47	-5.7	96.63 ± 0.74	280 ± 60
HAM-2196	16–18	7.8	6.13	-17.4	118.32 ± 0.64	Modern	3.87	-5.6	95.39 ± 0.57	380 ± 50
HAM-2197	18–20	7.8	5.94	-16.6	112.87 ± 0.80	Modern	3.25	-5.4	97.21 ± 0.75	230 ± 70
HAM-2198	20–22	8.0	5.53	-19.0	113.74 ± 0.83	Modern	2.93	-5.9	86.10 ± 0.72	1200 ± 70
HAM-2199	22–24	8.0	2.01	-19.0	104.13 ± 0.77	Modern	2.65	-6.5	90.54 ± --	800 ± 80
HAM-2200	24–26	7.6	4.34	-18.9	99.03 ± 0.58	180 ± 50	2.32	-7.5	81.95 ± 0.53	1600 ± 50
HAM-2201	26–28	7.7	4.08	-19.3	95.68 ± 0.75	450 ± 60	2.54	-7.5	81.96 ± 0.70	1600 ± 70
HAM-2202	28–30	7.6	3.37	-19.4	94.28 ± 0.74	560 ± 60	2.75	-7.1	77.76 ± 0.70	2020 ± 70
HAM-2203	30–32	7.5	4.88	-19.8	93.41 ± 0.74	630 ± 70	2.38	-8.4	81.62 ± 0.69	1630 ± 70
HAM-2204	32–34	7.6	4.00	-18.9	91.10 ± 0.75	850 ± 70	2.88	-8.5	78.06 ± 0.70	1990 ± 70
HAM-2205	34–36	7.5	3.31	-19.1	91.49 ± 0.75	810 ± 70	3.01	-8.3	80.84 ± 0.70	1710 ± 70
HAM-2206	36–38	7.5	3.60	-19.1	89.14 ± 0.55	1020 ± 50	2.71	-8.4	80.40 ± 0.70	1750 ± 70
HAM-2207	38–40	7.4	4.17	-19.4	93.43 ± 0.74	640 ± 70	2.42	-8.9	81.26 ± 0.70	1670 ± 70
HAM-2208	40–42	7.5	3.50	-19.6	91.59 ± 0.73	790 ± 70	2.85	-8.5	82.16 ± 0.57	1580 ± 60
HAM-2209	42–44	7.4	4.30	-19.2	92.16 ± --	750 ± 70	2.60	-10.9	78.80 ± --	1910 ± 60
HAM-2210	44–46	7.6	2.93	-20.3	90.53 ± 0.72	880 ± 70	2.40	-9.2	80.70 ± 0.69	1720 ± 70
HAM-2211	46–48	7.5	2.85	-20.4	91.52 ± 0.73	790 ± 70	2.71	-9.2	80.87 ± 0.69	1710 ± 70
HAM-2212	48–50	7.5	2.95	-20.2	93.12 ± 0.73	650 ± 70	2.60	-8.9	97.67 ± 0.60	190 ± 60
HAM-2213	50–52	7.4	2.58	-20.8	118.09 ± 0.78	Modern	2.67	-9.4	81.23 ± 0.69	1670 ± 70
HAM-2214	52–54	7.5	2.37	-20.5	93.71 ± 0.73	590 ± 70	2.28	--	80.39 ± 0.59	1750 ± 60
HAM-2215	54–56	7.6	2.05	-22.0	95.17 ± 0.73	450 ± 60	2.16	-8.7	80.26 ± 0.68	1770 ± 70
HAM-2216	56–58	7.3	1.79	-21.8	101.11 ± 0.76	Modern	1.79	-9.1	86.55 ± 0.54	1160 ± 50
HAM-2217	58–60	7.4	0.93	-22.4	95.79 ± 0.80	390 ± 70	1.53	-9.3	82.44 ± 0.69	1550 ± 70
HAM-2218	60–62	8.0	1.19	-23.3	89.49 ± 0.71	920 ± 70	0.75	-9.8	81.63 ± 0.69	1630 ± 70
HAM-2219	62–64	7.3	0.72	-24.7	--	--	0.46	-10.6	--	--
HAM-2220	64–66	7.3	1.02	-25.2	99.90 ± 0.58	5 ± 50	0.27	-14.7	85.81 ± 0.71	1230 ± 70

**TIAONG, PROFILE H****Profile-Related Data**

Location (longitude, latitude): 13°59'N, 121°21'E  
 Location (country, next city or village): Philippines, Tiaong, Luzon  
 Soil order and type (USDA classification): Typic Haplaquoll, fine silty, mixed, noncalcareous, isohyperthermic  
 Parent material: Volcanic ash from nearby Mt. Banahaw  
 Mean annual temperature: 26°C  
 Annual rainfall: 2150 mm  
 Vegetation and land use: Banana, coconut  
 Site description: 45 m asl

This profile was located at the top of the toposequence at Tiaong, ca. 200 m apart from Profile L in a small banana and coconut grove, and was characterized by an epiaquic water regime.

Date of sampling: 1983

Date of measurement: 1985  
 Collector: Becker-Heidmann  
 Submitter: Becker-Heidmann  
 Submitter's comment:

The soil had not been plowed; thus, its A11 horizon shows a maximum of the  $^{14}\text{C}$  activity between 6 and 8 cm below surface, in the main rooty zone, and a constant decrease below that. In the A12 horizon the  $^{14}\text{C}$  activity varies *ca.* 110 pMC, and in the A12 and B2g horizons there are only a few layers where  $^{14}\text{C}$  activity is < 100 pMC. This means that a continuous admixture of fresh organic matter had occurred throughout the whole profile. Because of the wet conditions combined with nonintensive cultivation, an accumulation of organic matter is probable. With decreasing clay content,  $\delta^{13}\text{C}$  decreases with depth and  $^{14}\text{C}$  activity increases slightly. At the transition zone between the A12 and the water-saturated B2g horizon, the redox potential seasonally changes with the groundwater level, as proven by a strong enrichment of Mn in the layers of the  $^{14}\text{C}$  activity peaks. These conditions prevent fixation and aging of organic substances and lead to the "modern"  $^{14}\text{C}$  activity peak.

The fact that organic substances with high  $^{14}\text{C}$  activity ("bomb radiocarbon", *i.e.*, built after the beginning of thermonuclear testing) are found in the subsoil can be interpreted as intact bioturbation within the total profile depth of this Mollisol. Measurements of the  $^{14}\text{C}$  activity of earthworms from a Mollisol showing the "bomb carbon" in worms of all depths, support this hypothesis (Scharpenseel *et al.* 1986). Recent carbon can be introduced also in clay-organic complexes of all depths in this clayey and silty soil. As a comparison, the age of the total organic matter of a Mollisol from Inner Mongolia in China (Becker-Heidmann, Liu and Scharpenseel 1988) increases with  $69.3 \text{ yr cm}^{-1}$  depth, which corresponds to a very slow soil development *via* an extended pararendzina phase. Here  $\delta^{13}\text{C}$  does not deviate from the value of fresh plant material throughout the profile, indicating early protection against further degradation. The reason for the slow soil development is most probably the climatic regime, with temperatures of  $-20^\circ\text{C}$  and <25 mm rainfall in January and *ca.*  $18^\circ\text{C}$  and between 25 and 50 mm rainfall in June, rather than the influence of the geological conditions.

Literature references: Becker-Heidmann and Scharpenseel (1989); Scharpenseel and Becker-Heidmann (1992b); Scharpenseel *et al.* (1989)

#### Description of the Profile at Tiaong (H):

Depth (cm)	Horizon	Description
0–20	A11	Black, rooty, big earthworms
20–36	A12	Slightly rooty
36–60	B2	Grayish brown, reddish brown mottles, no roots
60		Groundwater table

TABLE 18. Tiaong (H)

Lab code	Depth (cm)	pH	C	$\delta^{13}\text{C}$	$^{14}\text{C}$ activity	$^{14}\text{C}$ age
HAM-2222	0–2	6.4	3.74	-20.2	$118.43 \pm 0.82$	Modern
HAM-2223	2–4	6.3	3.98	-20.1	$119.23 \pm 0.82$	Modern
HAM-2224	4–6	6.7	3.47	-19.7	$118.32 \pm 0.82$	Modern
HAM-2225	6–8	6.9	3.44	-19.5	$120.96 \pm 0.83$	Modern
HAM-2226	8–10	7.0	2.71	-19.3	$115.75 \pm 0.81$	Modern
HAM-2227	10–12	6.9	2.67	-19.3	$116.54 \pm 0.82$	Modern
HAM-2228	12–14	7.2	2.42	-18.7	$111.57 \pm 0.79$	Modern
HAM-2229	14–16	7.2	2.36	-18.5	$110.72 \pm 0.79$	Modern
HAM-2230	16–18	7.1	1.86	-17.8	$107.92 \pm 0.78$	Modern
HAM-2231	18–20	7.2	1.75	-16.5	$103.15 \pm 0.76$	Modern

TABLE 18. Tiaong (H) (Continued)

Lab code	Depth (cm)	pH	C	δ <sup>13</sup> C	<sup>14</sup> C activity	<sup>14</sup> C age
HAM-2232	20–22	7.0	1.53	-16.2	102.72 ± 0.75	Modern
HAM-2233	22–24	7.0	1.31	-16.3	101.55 ± 0.75	Modern
HAM-2234	24–26	7.1	1.27	-15.8	98.37 ± 0.74	280 ± 60
HAM-2235	26–28	7.0	1.16	-15.8	101.18 ± 0.75	Modern
HAM-2236	28–30	7.1	0.92	-16.0	101.16 ± 0.75	Modern
HAM-2237	30–32	7.1	0.80	-16.5	100.03 ± 0.74	Modern
HAM-2238	32–34	7.0	0.63	-16.8	98.97 ± 0.74	220 ± 60
HAM-2239	34–36	6.9	0.59	-17.3	100.63 ± 0.75	Modern
HAM-2240	36–38	6.9	0.50	-17.8	107.59 ± 0.77	Modern
HAM-2241	38–40	6.9	0.47	-18.1	107.08 ± 0.77	Modern
HAM-2242	40–42	7.0	0.36	-18.3	100.06 ± 0.74	Modern
HAM-2243	42–44	7.1	0.41	-18.4	99.74 ± 0.74	130 ± 60
HAM-2244	44–46	6.9	0.40	-18.7	96.80 ± 0.75	360 ± 60
HAM-2245	46–48	6.8	0.31	-15.9	103.86 ± 0.76	Modern
HAM-2246	48–50	6.9	0.31	-16.8	100.24 ± 0.74	Modern
HAM-2247	50–52	6.9	0.30	-19.6	102.84 ± 0.77	Modern
HAM-2248	52–54	6.6	0.30	-18.9	101.55 ± 0.79	Modern

**SAN DIONISIO, PROFILE T****Profile-Related Data**

Location (longitude, latitude):	11°32'N, 123°07'E
Location (country, next city or village):	Philippines, San Dionisio, Panay
Soil order and type (USDA classification):	Typic Paleudult, coarse loamy over clayey, mixed, oxidic, acid, isohyperthermic
Parent material:	Colluvium above estuarine clayey alluvium
Mean annual temperature:	26°C
Annual rainfall:	3000 mm
Vegetation and land use:	Rice, presently fallow

**Site description:**

The profile was located in the northernmost part of the island Panay, near the village San Dionisio. The area is part of a former river delta system. At San Dionisio, the former sediment contains pure kaolinite, which is now being mined for pottery. It is covered by colluvium of 300 cm depth from a nearby mountain with a high iron content. Therefore, the soil suffers from iron toxicity and is not very suitable for rice production. Some of the stones found below 100 cm have been dated to >16,000 yr (V. Toquillo, personal communication).

Date of sampling:	1983
Date of measurement:	1985
Collector:	Becker-Heidmann, V. P. Singh
Submitter:	Becker-Heidmann
Submitter's comment:	

This profile has developed under tidal influence in a river estuary, where sedimentation and mangrove growing caused high contents of clay as well as organic matter. Microbial reduction of sulfates from seawater and anaerobic decomposition of the organic matter led to a decrease of the pH value and subsequently, a reduction of the minerals of the sediment to kaolinite. Later, the iron-rich colluvium of the nearby hill covered this acid sulfate soil. The present situation is characterized by the reduction of Fe<sub>3+</sub> to Fe<sub>2+</sub> under low pH and, simultaneously, the oxidation of SOM with a release of Fe<sub>2+</sub> in toxic concentrations under the anaerobic conditions of rice cultivation.



An unexpectedly high value of  $\delta^{13}\text{C}$  in and its decrease with depth may be explained by the field lying fallow and subsequent covering by tropical grasses, among which are probably some  $\text{C}_4$  species. This is supported by the  $^{14}\text{C}$  activity being nearly as high as the atmospheric level and indicating a continued high C input to the soil. Organic matter in the puddling zone is dominated by bomb carbon of the sampling year concentration (ca. 120 pMC), which indicates a high turnover rate. The high  $^{14}\text{C}$  activity in the topsoil, together with the low carbon content, show also that the decomposition and humification is very low in this soil. Especially in the layers where  $\delta^{13}\text{C}$  is constant, no biological activity was detected by IRRI's Dept. of Microbiology (V. P. Singh, personal communication 1983). The low biological activity is probably caused by the large percentage of kaolinite with a low cation exchange capacity responsible for a weak dispersion of the soil.

The soil below B1 shows a constant  $\delta^{13}\text{C}$  value with a continuous and rapid decrease in  $^{14}\text{C}$  activity and a correspondingly increasing  $^{14}\text{C}$  age. The steep increase of the  $^{14}\text{C}$  age up to ca. 3000 yr already at 80 cm depth can be explained by the high clay content of ca. 50%. We conclude that organic matter in the lower part of the profile is well protected against further metabolization or admixture of young material.

Literature references: Becker-Heidmann and Scharpenseel (1989)

#### Description of the Profile at San Dionisio:

Depth (cm)	Horizon	Description
0-20	Apg	Brown-gray, very rooty
20-36	B1	Light brown-gray, reddish mottles (iron), no roots
36-70	B2	Light reddish brown
70+	B2	Light reddish brown, intense red mottles, alluvium rich in iron

TABLE 19. San Dionisio, Profile T

Lab code	Depth(cm)	pH	C	$\delta^{13}\text{C}$	$^{14}\text{C}$ activity	$^{14}\text{C}$ age
HAM-2249	0-2	3.9	1.48	-17.4	117.58 ± 0.82	Modern
HAM-2250	2-4	3.7	1.07	-18.3	121.42 ± 0.84	Modern
HAM-2251	4-6	3.7	0.99	-19.1	116.57 ± 0.82	Modern
HAM-2252	6-8	3.7	0.92	-18.8	121.56 ± 0.84	Modern
HAM-2253	8-10	3.7	0.83	-18.5	120.42 ± 0.83	Modern
HAM-2254	10-12	3.8	0.74	-18.8	120.00 ± 0.83	Modern
HAM-2255	12-14	3.6	0.69	-18.8	109.65 ± 0.80	Modern
HAM-2256	14-16	4.2	0.60	-19.3	105.48 ± 0.83	Modern
HAM-2257	16-18	3.5	0.60	-19.2	102.21 ± 0.76	Modern
HAM-2258	18-20	4.0	0.57	-19.4	100.03 ± 0.74	Modern
HAM-2259	20-22	4.0	0.58	-19.6	99.00 ± 0.75	170 ± 60
HAM-2260	22-24	3.4	0.48	-20.0	99.99 ± 0.74	80 ± 60
HAM-2261	24-26	3.7	0.50	-20.9	106.95 ± 0.60	Modern
HAM-2262	26-28	3.5	0.53	-22.1	93.50 ± 0.72	590 ± 60
HAM-2263	28-30	3.5	0.49	-23.0	94.03 ± 0.78	530 ± 70
HAM-2264	30-32	3.5	0.48	-22.9	102.15 ± 0.74	Modern
HAM-2265	32-34	3.6	0.49	-22.7	92.09 ± 0.66	700 ± 60
HAM-2266	34-36	3.7	0.47	-22.7	93.17 ± 0.62	600 ± 50
HAM-2267	36-38	3.5	0.48	-22.3	90.09 ± 0.60	880 ± 50
HAM-2268	38-40	3.7	0.47	-22.3	88.38 ± 0.60	1040 ± 50
HAM-2269	40-42	4.2	0.55	-22.3	89.53 ± 0.60	930 ± 50
HAM-2270	42-44	3.8	0.55	-22.4	87.70 ± 0.60	1100 ± 50
HAM-2271	44-46	3.6	0.55	-22.7	80.96 ± 0.57	1730 ± 60
HAM-2272	46-48	3.8	0.50	-22.3	83.36 ± 0.67	1510 ± 70
HAM-2273	48-50	3.7	0.54	-22.3	83.44 ± 0.61	1500 ± 60
HAM-2274	50-60	3.6	0.56	-22.3	81.21 ± 0.57	1720 ± 60

TABLE 19. San Dionisio, Profile T (Continued)

Lab code	Depth(cm)	pH	C	δ <sup>13</sup> C	<sup>14</sup> C activity	<sup>14</sup> C age
HAM-2275	60–70	3.6	0.57	-22.1	73.41 ± 0.59	2530 ± 60
HAM-2276	70–80	3.9	0.54	-22.0	70.23 ± 0.53	2890 ± 60
HAM-2277	80–90	3.6	0.57	-20.2	60.81 ± 0.50	4070 ± 70
HAM-2278	90–110	3.4	0.63	-19.3	52.87 ± 0.47	5210 ± 70
HAM-2279	110–130	3.7	0.53	-20.4	60.76 ± 0.58	4080 ± 80
HAM-2280	130–150	3.9	0.58	-21.0	47.38 ± 0.53	6070 ± 90
HAM-2281	150–170	3.7	0.44	-17.7	41.56 ± 0.60	7170 ± 120
HAM-2282	170–190	3.7	0.59	-16.1	31.02 ± 0.39	9550 ± 100
HAM-2283	190–210	3.9	0.51	-15.6	30.29 ± 0.39	9750 ± 100
HAM-2284	210–240	3.3	0.50	-15.7	45.63 ± 0.52	6450 ± 90
HAM-2285	240–270	3.2	0.73	-12.6	19.58 ± 0.35	13,300 ± 150
HAM-2286	270–285	3.3	0.31	-15.3	23.69 ± 0.52	11,730 ± 180
HAM-2287	285–300	3.1	0.28	-14.9	25.19 ± 0.37	11,240 ± 120
HAM-2288	300–315	3.9	0.46	-13.7	25.86 ± 0.55	11,050 ± 170
HAM-2289	315–330	3.4	0.66	-13.4	13.53 ± 0.35	16,260 ± 210
HAM-2290	330–340	3.7	1.85	-12.2	9.70 ± 0.39	18,950 ± 330
HAM-2291	340–350	3.7	1.31	-12.6	8.67 ± 0.32	19,850 ± 300
HAM-2292	350–360	3.6	1.21	-12.4	8.03 ± 0.32	20,460 ± 320
HAM-2293	360–370	4.0	0.80	-12.9	8.14 ± 0.32	20,350 ± 310
HAM-2294	370–380	3.3	0.64	-14.1	8.77 ± 0.32	19,730 ± 290
HAM-2295	380–390	3.9	0.74	-14.1	7.17 ± 0.39	21,350 ± 430
HAM-2296	390–410	3.6	5.06	-15.2	2.88 ± 0.37	28,660 ± 1040
HAM-2297	410–430	3.8	4.98	-14.4	4.06 ± 0.38	25,910 ± 750
HAM-2298	430–450	4.2	2.07	-14.4	4.46 ± 0.38	25,150 ± 680
HAM-2299	450–470	3.8	4.14	-14.6	4.08 ± 0.49	25,870 ± 960
HAM-2300	470–490	3.1	1.89	-13.6	4.91 ± 0.38	24,400 ± 620
HAM-2301	490–510	3.8	1.36	-15.8	4.10 ± 0.38	25,810 ± 740

**NAMTOU HSIEN****Profile-Related Data**

Location (longitude, latitude):	24°08'N, 120°41'E
Location (country, next city or village):	Republic of China, Namtou Hsien near Taichung
Soil order and type (USDA classification):	Typic Fluvaquent, fine to moderate
Parent material:	Sandstone and shales alluvium
Mean annual temperature:	22°C
Annual rainfall:	2250 mm
Vegetation and land use:	Rice
Site description:	

The soil developed in a relatively new deposit above an old soil, which also was used for rice.

Date of sampling:	1984
Date of measurement:	1989
Collector:	Becker-Heidmann, H. Y. Guo
Submitter:	Becker-Heidmann, H. Y. Guo
Submitter's comment:	

The δ<sup>13</sup>C value was low in the Ap1, -26‰, corresponding to fresh plant material from rice. We observed a steep increase within the Ap2; in the subsoil the δ<sup>13</sup>C value was *ca.* -16‰ PDB. As the fossil soil below was also used for growing rice and not for, *e.g.*, sugar cane, this increase can easily be explained by decomposi-

tion processes involving larger amounts of CH<sub>4</sub>. The <sup>14</sup>C activity value of only 108 pMC in the Ap1 strongly supports the hypothesis of a high decomposition rate. The curve in the subsoil shows the normal pattern. Literature references: Becker-Heidmann (1990); Scharpenseel and Becker-Heidmann (1994b)

#### Description of the Profile at Namtjou Hsien:

Depth (cm)	Horizon	Description
0–10	Ap1	Very dark gray brown, clay loam
10–20	Ap2	Dark gray brown
20–40	IIfAp	Silty clay
40–50	IIAB	Gray brown
50–70	IIB21	Gray brown
70+	IIB22	Gray brown

TABLE 20. Namtsou Hsien

Lab code	Depth(cm)	pH	C	δ <sup>13</sup> C	<sup>14</sup> C activity	<sup>14</sup> C age
HAM-2661	0–2	4.5	1.60	-26.3	107.10 ± 0.79	Modern
HAM-2662	2–4	4.6	1.64	-25.9	108.09 ± 0.80	Modern
HAM-2663	4–6	4.8	1.50	-25.4	106.69 ± 0.80	Modern
HAM-2664	6–8	4.9	1.57	-25.9	105.77 ± 0.78	Modern
HAM-2665	8–10	5.4	1.47	-25.8	107.50 ± 0.61	Modern
HAM-2666	10–12	5.8	1.51	-25.0	106.58 ± 0.79	Modern
HAM-2667	12–14	5.9	1.32	-25.6	107.00 ± 0.79	Modern
HAM-2668	14–16	6.1	1.05	-24.0	104.81 ± 0.79	Modern
HAM-2669	16–18	6.0	0.85	-21.7	100.11 ± 0.76	Modern
HAM-2670	18–20	6.2	0.87	-20.6	98.93 ± 0.58	160 ± 50
HAM-2671	20–22	6.3	0.86	-19.3	94.12 ± 0.75	580 ± 70
HAM-2672	22–24	6.3	0.84	-17.0	92.71 ± 0.74	740 ± 70
HAM-2673	24–26	6.3	0.89	-16.1	93.00 ± 0.74	730 ± 70
HAM-2674	26–28	6.3	0.88	-16.1	91.49 ± 0.73	860 ± 70
HAM-2675	28–30	6.1	1.02	-16.4	93.90 ± 0.57	645 ± 50
HAM-2676	30–32	6.2	1.07	-15.7	93.96 ± 0.75	650 ± 70
HAM-2677	32–34	6.1	1.11	-15.6	91.02 ± 0.73	910 ± 70
HAM-2678	34–36	6.2	1.32	-15.7	90.78 ± 0.74	930 ± 70
HAM-2679	36–38	6.1	1.48	-15.7	92.82 ± 0.75	750 ± 70
HAM-2680	38–40	6.2	1.72	-14.8	89.78 ± 0.73	1030 ± 70
HAM-2681	40–42	6.2	1.75	-15.2	90.24 ± 0.56	985 ± 50
HAM-2682	42–44	6.1	1.20	-14.8	89.62 ± 0.73	1050 ± 70
HAM-2683	44–46	6.1	1.17	-14.7	86.96 ± 0.73	1290 ± 70
HAM-2684	46–48	6.1	1.13	-14.8	86.16 ± 0.72	1360 ± 70
HAM-2685	48–50	6.2	1.04	-15.3	86.32 ± 0.54	1340 ± 50
HAM-2686	50–60	6.2	0.97	-15.4	84.71 ± 0.71	1490 ± 70
HAM-2687	60–70	6.1	0.78	-16.0	81.63 ± 0.84	1780 ± 80
HAM-2688	70–80	5.9	0.73	-16.1	81.36 ± 0.95	1800 ± 100

#### PINGTUNG

##### Profile-Related Data

Location (longitude, latitude): 22°40'N, 120°29'E

Location (country, next city or village): Republic of China, experimental station of Pingtung near Kaohsiung  
 Soil order and type (USDA classification): Typic Fluvaquent  
 Soil order and type (local classification): --  
 Parent material: Slate alluvium  
 Mean annual temperature: 24°C  
 Annual rainfall: 2500 mm  
 Vegetation and land use: 2× rice + 1× beans per year for 20 yr  
 Site description: --  
 Date of sampling: 1984  
 Date of measurement: 1989  
 Collector: Becker-Heidmann, Guo  
 Submitter: Becker-Heidmann, Guo  
 Submitter's comment:

The carbon content is rather low, with 1.2% near the surface and <0.3% in the subsoil. The  $\delta^{13}\text{C}$  value shows the normal pattern of increasing with depth within the topsoil. Its shift is not as pronounced as in the Taichung soil. Therefore, methane production might be not as high as in the Taichung profile. The <sup>14</sup>C activity is generally comparable in value and curve to the Taichung soil. This indicates that the strength of decomposition is also comparable. A considerable difference in the depth distribution pattern can be observed at the boundaries of the horizons of the subsoil. High to extremely high <sup>14</sup>C activity shows that considerable amounts of fresh soluble organic substances have been percolating down through the profile. In spite of the low organic matter content of the soil and of losses by percolation, the productivity of this soil is rather high. In a soil fertility study of 130 soils of Kaohsiung-Pingtung region, Chang *et al.* (1983) showed that the morphological characteristics of the subsoil created by illuviation, deposition, reduction, oxidation, and ground-water behavior has no response to rice production and surface soil fertility.

Lab comment: --

Literature references: Becker-Heidmann and Scharpenseel (1992a)

#### Description of the Profile at Pingtung:

Depth (cm)	Horizon	Description
0–16	Ap1	Brown-gray, brown mottles, rooty
16–26	Ap2	Brown-gray to ochre, many reddish mottles
26–44	B1	Gray-brown, light brown mottles, silty
44–80	B21	Gray-brown to brown
80+	B22	

TABLE 21. Pingtung

Lab code	Depth (cm)	pH	C	$\delta^{13}\text{C}$	<sup>14</sup> C activity	<sup>14</sup> C age
HAM-2689	0–2	4.6	1.20	-22.1	108.49 ± 0.80	Modern
HAM-2690	2–4	4.7	1.18	-23.0	111.46 ± 0.62	Modern
HAM-2691	4–6	4.7	1.15	-22.9	112.36 ± 0.81	Modern
HAM-2692	6–8	4.7	1.08	-23.1	110.76 ± 0.80	Modern
HAM-2693	8–10	4.6	0.37	-23.1	97.09 ± 0.74	270 ± 60
HAM-2694	10–12	4.8	0.87	-22.6	105.98 ± 0.78	Modern
HAM-2695	12–14	5.1	1.00	-22.5	106.69 ± 0.78	Modern
HAM-2696	14–16	5.4	0.83	-22.5	102.46 ± 0.77	Modern
HAM-2697	16–18	5.5	0.75	-21.9	66.60 ± 0.66	3320 ± 80
HAM-2698	18–20	5.8	0.59	-21.3	91.36 ± 0.74	790 ± 70
HAM-2699	20–22	5.8	0.48	-20.2	89.55 ± 0.55	965 ± 50

TABLE 21. Pingtung (Continued)

Lab code	Depth (cm)	pH	C	$\delta^{13}\text{C}$	$^{14}\text{C}$ activity	$^{14}\text{C}$ age
HAM-2700	22–24	6.3	0.36	–19.5	87.89 ± 0.72	1130 ± 70
HAM-2701	24–26	--	1.16	–19.1	105.55 ± 0.78	Modern
HAM-2702	26–28	6.1	0.44	–18.3	84.74 ± 0.73	1440 ± 70
HAM-2703	28–30	6.1	0.36	–18.6	82.97 ± 0.71	1600 ± 70
HAM-2704	30–32	6.1	0.41	–17.6	82.77 ± 0.65	1640 ± 60
HAM-2705	32–34	6.2	0.43	–17.6	92.09 ± 0.75	780 ± 70
HAM-2706	34–36	6.2	0.44	–17.2	100.39 ± 1.26	Modern
HAM-2707	36–38	6.1	0.45	–17.3	84.71 ± 0.54	1460 ± 50
HAM-2708	38–40	6.1	0.39	–17.7	84.04 ± 0.71	1520 ± 70
HAM-2709	40–42	6.1	0.31	–17.7	81.72 ± 0.70	1740 ± 70
HAM-2710	42–44	6.0	0.31	–18.6	135.60 ± 0.88	Modern
HAM-2711	44–46	6.2	0.29	–18.8	91.77 ± 0.73	790 ± 70
HAM-2712	46–48	6.2	0.28	–18.6	77.52 ± 0.52	2150 ± 50
HAM-2713	48–50	6.4	0.27	–18.9	76.37 ± 0.69	2260 ± 70
HAM-2714	50–60	6.1	0.25	–19.2	67.99 ± 0.91	3190 ± 110
HAM-2715	60–70	6.1	0.18	–19.3	62.46 ± 0.63	3870 ± 80
HAM-2716	70–80	6.2	0.16	–19.2	61.91 ± 0.63	3940 ± 80
HAM-2717	80–90	6.2	0.16	–18.8	70.35 ± 0.50	2930 ± 60
HAM-2718	90–100	6.2	0.19	–19.3	49.78 ± 0.59	5700 ± 100

**CHUM PAE****Profile-Related Data**

Location (longitude, latitude):	16°33'N, 102°02'E
Location (country, next city or village):	Thailand, rice experimental station at Chum Pae near Khon Kaen
Soil order and type (USDA classification):	Aeric Paleaquult, loamy, mixed, nonacid, isohyperthermic
Soil order and type (local classification):	Roi Et series
Mean annual temperature:	28°C
Annual rainfall:	1100 mm
Vegetation and land use:	Rice
Site description:	The Chum Pae rice experimental station is located at the depressed part of the Korat plateau in Northeast Thailand in an undulating region with limited rainfall. The groundwater table is below 300 cm during the peak of the dry season. At the time of sampling, the soil was dry down to 170 cm.
Date of sampling:	1984
Date of measurement:	1986
Collector:	Becker-Heidmann
Submitter:	Becker-Heidmann
Submitter's comment:	The carbon content is very low, not only in the subsoil, but especially in the topsoil and shows no difference between topsoil and subsoil. $\delta^{13}\text{C}$ is rather high but not unusual for tropical rice soils. The increase with depth from –21 to –18‰ is lower than in the other studied soils, indicating a weak decomposition. The fact that the $^{14}\text{C}$ activity is as high as the atmospheric one in the plowed horizon also indicates low decomposition. Decomposition is slow in this puddled soil with low and imbalanced nutrient supply, high bulk density and low biological diversity and activity, as was shown also by decomposition experiments with $^{14}\text{C}$ -labeled rice straw, which were conducted in the same field after the samples for this study were taken (Snitwongse

*et al.* 1988). During the first year, 55–60% of the straw was lost. The half-life of the resistant part of the added organic matter was 13.5 yr in the submerged planted soil followed by dry fallow. After 3 yr, 30% was found within 50 cm depth.

Literature references:

Becker-Heidmann (1990); Scharpenseel *et al.* (1989);  
Scharpenseel and Becker-Heidmann (1993);  
Scharpenseel and Becker-Heidmann (1994b)

#### Description of the Profile at Chum Pae Station

Depth (cm)	Horizon	Description
0–12	Ap1g	Light yellowish brown (10YR 6/4), silty, aggregate surfaces covered with iron
12–20	Ap2g	Dark yellowish brown (10YR 4/4), puddling zone
20–38	B1	Dark grayish brown (10YR 4/2), silty clay loam, hard black concretions (manganese) with red covering (iron), soft yellowish red concretions (iron)
38–48	B21tg	Dark grayish brown(10YR 4/2), clay loam, probably clay migration, manganese and iron concretions
48–74	B22tg	Dark grayish brown, clay loam, probably clay migration, concretions
74–92+	B3g	Dark grayish brown, clay loam, concretions

TABLE 22. Chum Pae

Lab code	Depth (cm)	pH	C	$\delta^{13}\text{C}$	<sup>14</sup> C activity	<sup>14</sup> C age
HAM-2401	0–2	4.2	0.93	-20.4	116.72 ± 0.71	Modern
HAM-2402	2–4	4.4	0.20	-20.7	118.20 ± 0.71	Modern
HAM-2403	4–6	4.5	0.33	-19.8	118.07 ± 0.71	Modern
HAM-2404	6–8	4.5	0.48	-19.5	118.67 ± 0.71	Modern
HAM-2405	8–10	4.8	0.48	-19.6	119.37 ± 0.72	Modern
HAM-2406	10–12	5.0	0.29	-20.1	116.12 ± 0.70	Modern
HAM-2407	12–14	5.7	0.43	-19.4	101.65 ± 0.84	Modern
HAM-2408	14–16	6.2	0.33	-19.0	96.78 ± 0.73	360 ± 60
HAM-2409	16–18	5.0	0.32	-18.6	92.07 ± 0.61	770 ± 50
HAM-2410	18–20	6.7	0.37	-18.6	102.50 ± 0.65	Modern
HAM-2411	20–23	6.5	0.40	-18.5	95.15 ± 0.73	510 ± 60
HAM-2412	23–24	6.0	0.42	-18.5	93.85 ± 0.72	610 ± 60
HAM-2413	24–26	5.8	0.41	-18.3	99.37 ± 0.64	160 ± 50
HAM-2414	26–28	5.9	0.25	-18.6	98.19 ± 0.63	250 ± 50
HAM-2415	28–30	5.8	0.28	-18.2	94.94 ± 0.72	530 ± 60
HAM-2416	30–32	5.8	0.26	-18.2	93.79 ± 0.72	630 ± 60
HAM-2417	32–34	5.5	0.41	-18.1	94.14 ± 0.73	600 ± 60
HAM-2418	34–36	5.4	0.45	-18.3	93.32 ± 0.72	660 ± 60
HAM-2419	36–38	5.7	0.24	-18.4	98.01 ± 0.64	270 ± 50
HAM-2420	38–40	5.9	0.36	-18.5	92.26 ± 0.72	750 ± 60
HAM-2421	40–42	5.6	0.23	-18.4	93.30 ± 0.72	660 ± 60
HAM-2422	42–44	5.9	0.19	-18.8	92.47 ± 0.71	730 ± 60
HAM-2423	44–46	5.0	0.39	-18.5	95.39 ± 0.63	480 ± 50
HAM-2424	46–48	5.3	0.25	-18.6	96.90 ± 0.63	360 ± 50
HAM-2425	48–50	5.1	0.33	-18.8	93.76 ± 0.62	620 ± 50
HAM-2426	50–52	6.0	0.35	-19.0	95.70 ± 0.73	450 ± 60
HAM-2427	52–54	4.9	0.33	-18.8	93.88 ± 0.62	610 ± 50

TABLE 22. Chum Pae (Continued)

Lab code	Depth (cm)	pH	C	$\delta^{13}\text{C}$	$^{14}\text{C}$ activity	$^{14}\text{C}$ age
HAM-2428	54-56	5.3	0.25	-19.0	87.74 ± 0.70	1150 ± 60
HAM-2429	56-58	5.3	0.24	-19.1	87.08 ± 0.69	1210 ± 60
HAM-2430	58-60	5.1	0.30	-19.4	88.19 ± 0.69	1100 ± 60
HAM-2431	60-62	5.0	0.23	-19.3	89.90 ± 0.60	950 ± 50
HAM-2432	62-64	5.4	0.12	-19.8	90.40 ± 0.60	890 ± 50
HAM-2433	64-66	5.1	0.30	-19.9	86.62 ± 0.69	1240 ± 60
HAM-2434	66-68	5.0	0.22	-19.9	91.60 ± 0.61	790 ± 50
HAM-2435	68-70	5.1	0.22	-20.3	88.19 ± 0.75	1090 ± 70
HAM-2436	70-72	5.3	0.25	-20.1	86.10 ± 0.72	1280 ± 70
HAM-2437	72-74	5.2	0.28	-20.4	89.61 ± 0.61	960 ± 60
HAM-2438	74-76	5.8	0.19	-20.6	89.28 ± 0.60	980 ± 50
HAM-2439	76-78	5.4	0.25	-20.5	88.66 ± 0.60	1040 ± 50
HAM-2440	78-80	5.8	0.25	-20.8	85.36 ± 0.69	1340 ± 70
HAM-2441	80-82	5.9	0.18	-20.9	85.86 ± 0.79	1290 ± 70
HAM-2442	82-84	5.8	0.25	-20.9	87.42 ± 0.60	1150 ± 60
HAM-2443	84-86	5.5	0.16	-21.2	90.01 ± 0.62	910 ± 60
HAM-2444	86-88	5.7	0.18	-21.2	87.71 ± 0.60	1110 ± 60
HAM-2466	88-90	6.2	0.23	-21.4	88.77 ± 0.60	1020 ± 60
HAM-2467	90-92	5.8	0.16	-21.3	83.98 ± 0.68	1460 ± 70

**KLONG LUANG****Profile-Related Data**

Location (longitude, latitude):	14°05'N, 100°44'E
Location (country, next city or village):	Thailand, Klong Luang near Bangkok
Soil order and type (USDA classification):	Sulfic Tropeaquept, loamy, mixed, acid, isohyperthermic
Soil order and type (local classification):	Rangsit series
Parent material:	River alluvium
Mean annual temperature:	27°C
Annual rainfall:	1300 mm
Vegetation and land use:	Rice
Site description:	

The soil, located at Klong Luang north of Bangkok, is representative of the acid sulfate soils that cover *ca.* 8000 km<sup>2</sup> in the Bangkok Plain. These soils were studied in detail by van Breemen (1976); a similar profile of Rangsit series is described in his book. By <sup>14</sup>C dating of peat material, van der Kevie (1972) estimated the formation of the tidal marsh deposits at *ca.* 3100 yr ago.

Date of sampling:	1984
Date of measurement:	1986
Collector:	S. Pongpan, P. Snitwongse
Submitter:	P. Snitwongse, Becker-Heidmann
Submitter's comment:	

The pH is very low throughout the profile due to the high sulfate content. The carbon content is only 1.3% in the topsoil, but does not decrease much to the subsoil. The  $\delta^{13}\text{C}$  value near the surface is the normal one for tropical rice soils (*cf.* the other soils in this study), but the increase with depth in the topsoil of this profile is the lowest. The <sup>14</sup>C activity in this soil is one of the lowest of all studied rice soils. Together with the small increase of  $\delta^{13}\text{C}$ , it suggests that there has been very little input of organic matter. Otherwise, the <sup>14</sup>C age of the subsoil could not have reached the age of the sediments, mentioned above in the soil description (*cf.* van der Kevie 1972).

Literature references: Becker-Heidmann (1990)

**Description of the Profile at Klong Luang:**

Depth (cm)	Horizon	Description
0–10	Ap1g	Black (10YR 2/1), yellowish brown (10YR 5/6) mottles, frequent very fine roots
10–22	Ap2g	Black, few yellowish brown and red mottles, many very fine roots
22–30	A2g	Brown to dark grayish brown, very few very fine roots
30–42	B1g	Brown to dark brown, brown and red mottles, few very fine roots
42–58	B21g	Brown to dark brown, many red and “straw” yellow mottles, very few very fine roots
58+	B22g	Brown, “straw” yellow mottle, very few very fine roots

TABLE 23. Klong Luang

Lab code	Depth (cm)	pH	C	δ <sup>13</sup> C	<sup>14</sup> C activity	<sup>14</sup> C age
HAM-2468	0–2	4.3	1.39	-22.6	106.01 ± 0.67	Modern
HAM-2469	2–4	4.4	1.31	-22.2	105.12 ± 0.66	Modern
HAM-2470	4–6	4.6	1.32	-22.2	99.99 ± 0.74	50 ± 60
HAM-2471	6–8	4.6	1.32	-21.9	104.83 ± 0.66	Modern
HAM-2472	8–10	4.3	1.41	-22.2	98.87 ± 0.79	140 ± 60
HAM-2473	10–12	4.4	1.38	-22.0	100.27 ± 0.75	Modern
HAM-2474	12–14	4.5	1.31	-21.2	104.03 ± 0.70	Modern
HAM-2475	14–16	4.5	1.21	-21.7	102.39 ± 0.65	Modern
HAM-2476	16–18	4.3	1.25	-20.8	89.67 ± 0.70	940 ± 60
HAM-2477	18–20	4.3	1.27	-20.2	95.60 ± 0.63	440 ± 50
HAM-2478	20–22	4.1	1.25	-20.4	85.71 ± 0.68	1310 ± 60
HAM-2479	22–24	4.0	1.30	-20.4	85.39 ± 0.59	1340 ± 60
HAM-2480	24–26	4.0	1.21	-21.0	80.22 ± 0.68	1840 ± 70
HAM-2481	26–28	4.2	1.15	-21.2	83.23 ± 0.60	1540 ± 60
HAM-2482	28–30	3.8	1.10	-20.9	81.20 ± 0.67	1740 ± 70
HAM-2483	30–32	4.3	0.98	-21.3	77.94 ± 0.69	2060 ± 70
HAM-2484	32–34	4.2	0.84	-21.2	80.39 ± 0.57	1820 ± 60
HAM-2485	34–36	3.7	0.81	-21.3	75.51 ± 0.65	2320 ± 70
HAM-2486	36–38	4.2	0.86	-21.1	79.21 ± 0.56	1930 ± 60
HAM-2487	38–40	3.9	0.79	-21.3	73.10 ± 0.63	2580 ± 70
HAM-2488	40–42	4.0	0.78	-21.0	75.84 ± 0.55	2290 ± 60
HAM-2489	42–44	4.0	0.72	-21.0	71.58 ± 0.63	2750 ± 70
HAM-2490	44–46	4.1	0.75	-21.0	74.94 ± 0.55	2380 ± 60
HAM-2491	46–48	4.0	0.74	-21.2	68.70 ± 0.61	3080 ± 70
HAM-2492	48–50	4.2	0.80	-21.1	74.04 ± 0.55	2480 ± 60
HAM-2493	50–52	4.0	0.79	-21.1	69.77 ± 0.65	2950 ± 70
HAM-2494	52–54	3.8	0.84	-21.3	73.02 ± 0.54	2590 ± 60
HAM-2495	54–56	3.9	0.77	-21.4	68.82 ± 0.69	3060 ± 80
HAM-2496	56–58	4.0	0.84	-21.7	67.15 ± 0.92	3250 ± 110
HAM-2497	58–60	4.2	0.88	-21.8	70.74 ± 0.54	2830 ± 60
HAM-2498	60–62	4.4	0.85	-21.9	65.47 ± 0.69	3450 ± 90
HAM-2499	62–64	4.3	0.84	-22.2	66.36 ± 0.60	3340 ± 70



**TACHIAT, PROFILE 1****Profile-Related Data**

Location (longitude, latitude):	7°22'N, 100°10'E
Location (country, next city or village):	Thailand, Tachiat, <i>ca.</i> 60 km northwest of Songkhla
Soil order and type (FAO classification):	Gleyic Acrisol
Soil order and type (USDA classification):	Typic Paleaquult, clayey-kaolinitic isohyperthermic
Soil order and type (local classification):	Bang Nara series
Parent material:	Marine sediment
Mean annual temperature:	27.6°C
Annual rainfall:	2500 mm
Vegetation and land use:	Farmers have grown rice at this site since land clearing > 40 yr ago. For the last 20 yr, rice has been planted nearly every rainy season, while during the dry season the soil was allowed to lie fallow.

Site description: 20 m asl

The field was located *ca.* 10 km from the lagoon of Phattalung on the experimental site of the Soils Department of Prince of Songkhla University. Puddling was usually done to a depth of ~13 cm. The hardpan was at *ca.* 40–50 cm depth. The surface had previously been undulating, but was leveled only a short time before sampling. Profile 1 was nearly unaffected by this, whereas Profile 2 was prepared within the former lower and now filled part of the field. The groundwater table at time of sampling was at 56 cm depth.

Date of sampling:	1990
Date of measurement:	1991
Collector:	Becker-Heidmann, Apinan Kamnalrut
Submitter:	Becker-Heidmann, Apinan Kamnalrut
Submitter's comment:	

The pH of the Tachiat soils is very low. Both soils have a pH of only 4, with nearly no variation throughout the profile. The soil contains no carbonate, and the carbon content is also rather low. The low  $\delta^{13}\text{C}$  value indicates little decomposition and humus maintenance. The only slight enrichment of  $\delta^{13}\text{C}$  between the surface soil and the bottom of the puddling layer supports this interpretation. In particular, no additional anaerobic decomposition with methane production can be assumed based on this depth distribution. In the subsoil, the  $\delta^{13}\text{C}$  value decreases, which means that there is no considerable transport of freshly decomposed organic matter into the deeper part of the soil. The  $^{14}\text{C}$  activity in the topsoil reflects the actual  $^{14}\text{C}$  activity of the atmosphere, supporting the thesis of reduced decomposition. Below the puddling zone, the  $^{14}\text{C}$  activity sharply decreases, again indicating impeded percolation, down to a value of 84 pMC, which corresponds to a  $^{14}\text{C}$  age of *ca.* 1400 BP. Obviously, the soil development within this alluvial sediment is relatively young. The comparable rice soil at Klong Luang shows  $^{14}\text{C}$  ages up to 3400 BP.

Literature references: Becker-Heidmann (1992); Drachenberg (1992)

**Description of Profile at Tachiat 1**

Depth / cm	Horizon	Description
0–18	Apg	Brown (10 YR 5/3), with reddish mottles
18–30	B1g	Very pale brown (10YR 7/4), spotted, with microcracks coated with iron (5YR 5/8)
30–40	B21g	Light gray (10YR 7/2), microcracks coated (5YR 5/8)
40–52	B22g	Brown with red mottles (10YR 4/8), high coarse sand content
52+	B23g	Very pale brown with red mottles, water table at 56 cm

TABLE 24. Tachiat, Profile 1

Lab code	Depth (cm)	pH	C	δ <sup>13</sup> C	<sup>14</sup> C activity	<sup>14</sup> C age
HAM-3070	0–2	4.1	1.07	–25.9	105.07 ± 0.75	Modern
HAM-3071	2–4	4.0	1.05	–24.6	105.76 ± 0.76	Modern
HAM-3072	4–6	3.8	0.97	–24.7	106.06 ± 0.76	Modern
HAM-3073	6–8	3.9	0.94	–24.6	106.52 ± 0.75	Modern
HAM-3074	8–10	3.9	0.99	–24.9	105.40 ± 0.75	Modern
HAM-3075	10–12	4.0	0.98	–25.3	105.89 ± 0.75	Modern
HAM-3076	12–14	3.9	0.99	–24.5	106.96 ± 0.74	Modern
HAM-3077	14–16	4.0	0.98	–24.9	105.87 ± 0.74	Modern
HAM-3078	16–18	4.0	0.99	–25.1	104.13 ± 0.74	Modern
HAM-3079	18–20	4.0	0.92	–24.4	105.30 ± 0.74	Modern
HAM-3080	20–22	3.9	0.91	–24.1	105.70 ± 0.75	Modern
HAM-3081	22–24	4.0	0.74	–23.9	104.07 ± 0.74	Modern
HAM-3082	24–26	4.1	0.51	–24.0	99.62 ± 0.72	50 ± 70
HAM-3083	26–28	4.1	0.40	–24.2	96.57 ± 0.71	290 ± 70
HAM-3084	28–30	4.0	0.38	–24.5	92.80 ± 0.71	610 ± 70
HAM-3085	30–32	4.0	0.34	–25.0	91.91 ± 0.69	680 ± 70
HAM-3086	32–34	3.9	0.38	–24.9	90.62 ± 0.69	790 ± 70
HAM-3087	34–36	4.0	0.33	–25.2	88.72 ± 0.70	960 ± 70
HAM-3088	36–38	4.0	0.33	–25.1	90.56 ± 0.70	790 ± 70
HAM-3089	38–40	3.9	0.32	–25.1	88.23 ± 0.68	1000 ± 70
HAM-3090	40–42	3.9	0.32	–25.5	89.61 ± 0.69	870 ± 70
HAM-3091	42–44	3.9	0.33	–25.4	88.65 ± 0.69	960 ± 70
HAM-3092	44–46	3.9	0.32	–25.3	86.63 ± 0.68	1150 ± 70
HAM-3093	46–48	3.9	0.32	–25.6	86.40 ± 0.70	1160 ± 70
HAM-3094	48–50	4.0	0.31	–25.3	86.09 ± 0.69	1200 ± 70
HAM-3095	50–52	3.9	0.31	–25.4	86.00 ± 0.67	1200 ± 70
HAM-3096	52–54	3.9	0.30	–25.6	85.52 ± 0.68	1250 ± 70
HAM-3097	54–56	4.0	0.31	–25.6	83.50 ± 0.67	1440 ± 70
HAM-3098	56–58	4.0	0.31	–25.6	84.08 ± 0.67	1380 ± 70
HAM-3099	58–60	4.0	0.31	–25.9	85.73 ± 0.68	1220 ± 70
HAM-3100	60–62	4.0	0.30	–25.4	85.91 ± 0.71	1210 ± 70

**TACHIAT, PROFILE 2****Profile-Related Data**

Same as Profile 1 except:

Date of measurement: 1991/1992

Submitter's comment:

The second profile does not differ from the first one regarding pH and carbon content in general. Only the humus layer is higher, as explained above. The δ<sup>13</sup>C value is nearly the same within the topsoil. There is one exceptionally high value at *ca.* 17 cm depth, but the δ<sup>13</sup>C value decreases in the subsoil and is generally lower than in Profile 1. Thus, we cannot detect a loss of organic substances by percolation. <sup>14</sup>C activity is slightly higher than in Profile 1, which means that the decomposition is even less intensive. The corresponding <sup>14</sup>C age within the subsoil reaches only *ca.* 1100 BP.

Literature references: Becker-Heidmann 1992; Drachenberg 1992

**Description of Profile at Tachiat 2**

Depth / cm	Horizon	Description
0–14	Ap1g	Dark gray with mottles, cracks, probably deposited Ap material

14–32	Ap2g	Dark gray with more mottles (Fe?), probably original Ap
32–52	B1g	Light gray with orange-brown mottles (5YR 5/8)
52+	B2g	Orange-brown–light gray mottled, red mottles (less than at Tachiat 1), water table at 56 cm

TABLE 25. Tachiat, Profile 2

Lab code	Depth (cm)	pH	C	$\delta^{13}\text{C}$	$^{14}\text{C}$ activity	$^{14}\text{C}$ age
HAM-3101	0–2	4.1	1.13	-25.0	105.65 ± 0.73	Modern
HAM-3102	2–4	4.1	1.15	-24.9	105.54 ± 1.07	Modern
HAM-3103	4–6	4.1	1.10	-25.1	105.40 ± 0.78	Modern
HAM-3104	6–8	4.0	1.13	-24.9	105.02 ± 0.76	Modern
HAM-3105	8–10	4.1	1.20	-24.8	105.94 ± 0.74	Modern
HAM-3106	10–12	4.1	1.12	-24.8	109.99 ± 0.76	Modern
HAM-3107	12–14	4.1	1.12	-25.3	107.53 ± 0.75	Modern
HAM-3108	14–16	4.1	1.01	-25.0	106.62 ± 0.74	Modern
HAM-3109	16–18	4.1	0.96	-24.9	109.81 ± 0.75	Modern
HAM-3110	18–20	4.1	1.00	-25.3	110.34 ± 0.75	Modern
HAM-3111	20–22	4.1	0.85	-25.2	110.01 ± 0.82	Modern
HAM-3112	22–24	4.0	0.96	-25.3	105.71 ± 0.76	Modern
HAM-3113	24–26	4.2	0.86	-25.6	109.06 ± 0.74	Modern
HAM-3114	26–28	4.2	0.64	-26.0	104.14 ± 0.74	Modern
HAM-3115	28–30	4.2	0.45	-26.1	101.31 ± 0.73	Modern
HAM-3116	30–32	4.2	0.38	-26.2	96.49 ± 0.72	270 ± 70
HAM-3117	32–34	4.2	0.30	-26.9	94.58 ± 0.71	420 ± 70
HAM-3118	34–36	4.0	0.29	-26.5	93.88 ± 0.72	480 ± 70
HAM-3119	36–38	4.0	0.30	-26.4	92.36 ± 0.69	620 ± 70
HAM-3120	38–40	4.1	0.30	-26.5	91.90 ± 0.69	650 ± 70
HAM-3121	40–42	4.0	0.31	-27.0	93.27 ± 0.71	530 ± 70
HAM-3122	42–44	4.1	0.29	-26.8	89.20 ± 0.69	890 ± 70
HAM-3123	44–46	4.1	0.31	-26.6	92.77 ± 0.71	580 ± 70
HAM-3124	46–48	4.1	0.31	-26.7	92.69 ± 0.71	580 ± 70
HAM-3125	48–50	4.0	0.31	-26.1	90.16 ± 0.72	810 ± 70
HAM-3126	50–52	4.0	0.29	-26.9	89.36 ± 0.72	870 ± 70
HAM-3127	52–54	4.1	0.29	-27.3	87.82 ± 0.69	1010 ± 70
HAM-3128	54–56	4.1	0.28	-27.6	87.19 ± 0.68	1060 ± 70
HAM-3129	56–58	4.1	0.28	-27.7	87.94 ± 0.70	990 ± 70

## TONSANG

## Profile-Related Data

Location (longitude, latitude):	18°52'N, 99°02'E
Location (country, next city or village):	Thailand, Tonsang near San Sai, ca. 10 km northeast of Chiang Mai
Soil order and type (USDA classification):	Typic Tropaqualf
Soil order and type (local classification):	Low humic gley soil, Hang Dong series
Mean annual temperature:	25°C
Annual rainfall:	1220 mm
Vegetation and land use:	The experimental site has been cultivated for rice and soybean rotation for ca. 10 yr.

## Site description:

The soil was very homogeneous. The groundwater level was at ca. 52 cm depth the first sampling day and rose to ca. 40 cm the second day.

Date of sampling: 1991  
 Date of measurement: 1992  
 Collector: Becker-Heidmann, Drachenberg, Arayangkoon  
 Submitter: Becker-Heidmann, Arayangkoon  
 Submitter's comment:

The pH value is generally high, *ca.* 6.3 throughout the B1 and B2g horizons and the surface layer. Amazingly, the pH depth distribution curve shows a minimum of 5.6 between 6 and 10 cm depth, which is the main rooting zone. The carbon content is very low because of either rapid turnover or low input of organic crop residues. The decrease of carbon with depth within the topsoil, which is unusual for a plowed horizon, supports the rapid turnover hypothesis. The  $\delta^{13}\text{C}$  value is generally high at the same level as the other studied tropical soils. If no C<sub>4</sub> plant has been grown in the past, the high value of 22‰ may be, in principle, a result of either anaerobic decomposition with methane production, or decomposing C<sub>4</sub> weeds, or exudation of carbohydrates from the roots during the past when the plot was used for rice. The increase from top to bottom of the Ap horizon by *ca.* 3‰ stands for high microbiological activity. The decrease within the B2g horizon is due to its anaerobic conditions (Becker-Heidmann 1990; Stout, Goh and Rafter 1981). The varying value near the top of the B2g horizon is probably a result of changes in the groundwater table. The depth distributions of carbon content,  $\delta^{13}\text{C}$  value and <sup>14</sup>C activity indicate a high decomposition rate rather than low input of organic matter.

Literature references: Becker-Heidmann (1992)

#### Description of Profile at Tonsang

Depth / cm	Horizon	Description
0–16	Apg	Loamy clay, dark grayish brown (2.5Y 4/2), many fine roots, iron at root channels
16–25	B1g	Silty clay, yellowish brown (2.5Y 5/4), many fine roots, red mottles (Fe)
25–52+	B2g	Sandy clayey loam, pale red (2.5YR 6/2), few roots

TABLE 26. Tonsang

Lab code	Depth (cm)	pH	C	$\delta^{13}\text{C}$	<sup>14</sup> C activity	<sup>14</sup> C age
HAM-2837	0–2	6.8	1.35	-22.0	117.92 ± 0.84	Modern
HAM-2838	2–4	6.3	1.29	-21.6	119.37 ± 0.66	Modern
HAM-2839	4–6	5.8	1.17	-21.1	120.17 ± 0.84	Modern
HAM-2840	6–8	5.6	1.00	-21.1	120.63 ± 0.86	Modern
HAM-2841	8–10	5.6	0.92	-21.0	122.13 ± 0.85	Modern
HAM-2842	10–12	5.7	0.77	-21.4	121.46 ± 0.86	Modern
HAM-2843	12–14	6.1	0.44	-20.6	115.39 ± 0.65	Modern
HAM-2844	14–16	6.3	0.26	-19.3	105.37 ± 0.79	Modern
HAM-2845	16–18	6.4	0.17	-19.5	101.76 ± 0.76	Modern
HAM-2846	18–20	6.3	0.22	-19.4	101.77 ± 0.63	Modern
HAM-2847	20–22	6.3	0.17	-20.1	100.87 ± 0.60	Modern
HAM-2848	22–24	6.3	0.21	-19.9	100.82 ± 0.74	Modern
HAM-2849	24–26	6.3	0.17	-20.5	101.77 ± 0.60	Modern
HAM-2850	26–28	6.3	0.16	-20.3	100.41 ± 0.60	Modern
HAM-2851	28–30	6.3	0.16	-20.1	96.98 ± 0.77	330 ± 70
HAM-2910	30–32	6.3	0.16	-21.0	98.38 ± 0.73	200 ± 60
HAM-2911	32–34	6.3	0.13	-20.0	96.80 ± 0.62	340 ± 50
HAM-2912	34–36	6.2	0.12	-21.1	95.78 ± 0.59	410 ± 50
HAM-2913	36–38	6.3	0.11	-20.6	94.87 ± 0.75	490 ± 60
HAM-2914	38–40	6.3	0.10	-20.3	95.04 ± 0.73	480 ± 60
HAM-2915	40–42	6.3	0.10	-20.6	95.80 ± 0.61	420 ± 50

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## OTHER ARTICLES

### GENOTOXICITY STUDY ON NICOTINE AND NICOTINE-DERIVED NITROSAMINE BY ACCELERATOR MASS SPECTROMETRY

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**ABSTRACT.** We have studied DNA adduction with  $^{14}\text{C}$ -labeled nicotine and nicotine-derived nitrosamine, 4-(methylnitrosamino)-1-(3-pyridyl)-1-butanone (NNK), by accelerator mass spectrometry (AMS) in mouse liver at doses equivalent to low-level exposure of humans. The dose ranges of nicotine and NNK administered were from  $0.4\ \mu\text{g}$  to  $4.0 \times 10^2\ \mu\text{g kg b.w.}^{-1}$ , and from  $0.1\ \mu\text{g}$  to  $2.0 \times 10^4\ \mu\text{g kg b.w.}^{-1}$ , respectively. In the exposure of mice to either nicotine or NNK, the number of DNA adducts increased linearly with increasing dose. The detection limit of DNA adducts was 1 adduct per  $10^{11}$  nucleotide molecules. This limit is 1-4 orders of magnitude lower than that of other techniques used for quantification of DNA adducts. The results of our animal experiments enabled us to speculate that nicotine is a potential carcinogen. According to the procedure for  $^{14}\text{C}$ -labeled-NNK synthesis, we discuss the ultimate chemical speciation of NNK bound to DNA. From the animal tests we derived a directly perceivable relation between tobacco consumption and DNA adduction as the carcinogenic risk assessment.

## INTRODUCTION

Carcinogens are generally metabolically activated electrophilic species covalently bound to nucleophilic sites in DNA, forming adducts (Margison and O'Connor 1979). DNA adduction may be an early detectable and critical step in chemical carcinogenesis. However, the relation between adduct formation and exposure has primarily been established at carcinogen doses much higher than actual human exposure owing to the limitations of detection sensitivity. To date, the most sensitive technique for adduct detection was  $^{32}\text{P}$ -postlabeling, which permitted a quantitative measurement at levels  $>1$  adduct per  $10^8$ - $10^{10}$  nucleotides (Gupta 1985). Phillips *et al.* (1988) measured DNA adduction with unidentified mixed polycyclic aromatic compounds in cigarettes in human lung tumors using the  $^{32}\text{P}$ -postlabeling method.

Felton *et al.* (1990) at Lawrence Livermore National Laboratory (LLNL) were the first to use AMS with a  $^{14}\text{C}$ -labeled carcinogen, 2-amino-3,8-dimethyl-imidazo (4,5-f) quinoxaline (MeIQx), for the quantification of food carcinogen-DNA adducts, with a detection limit of 1 adduct per  $10^{11}$ - $10^{12}$  nucleotides. The introduction of this technique enabled dose-response studies to be carried out over several orders of magnitude, including environmental, low-level doses for many carcinogens. In recent years, Vogel and his colleagues at LLNL have presented more studies on DNA adduction, biodistribution and pharmacokinetics of xenobiotics with AMS (Vogel and Turteltaub 1992; Turteltaub *et al.* 1992; Creek *et al.* 1994; Frantz *et al.* 1995).

Nicotine, 3-(1-methyl-2-pyrrolidinyl)-pyridine, is a major alkaloid in tobacco products, typically comprising 1-2 weight % of tobacco. The pharmacological effect of nicotine is a dominant factor for tobacco addiction. So far it is not clear whether nicotine itself is carcinogenic to humans. Nicotine was not mutagenic according to the Ames test, nor did nicotine administered by intraperitoneal injection at a maximum tolerable dose of  $0.8\ \text{mg kg}^{-1}$  increase urinary mutagenicity in Sprague-Dawley rats (Doolittle *et al.* 1991). However, it has been proposed, but not clearly demonstrated,

that nicotine could convert to carcinogenic nitrosamines in the human body (Hoffmann and Hecht 1985). 4-(methylnitrosamino)-1-(3-pyridyl)-1-butanone (NNK) is a potent and predominant genotoxic carcinogen produced by the nitrosation of nicotine and is widely present in both mainstream and sidestream cigarette smoke as well as unburned tobacco. The content of NNK in cigarettes is roughly 1/10,000 of the content of nicotine. Chronic treatment of rodents with NNK resulted in the induction of tumors in the lung, liver, nasal cavity and pancreas (Hoffmann and Hecht 1985; Hecht, Young and Chen 1980). Figure 1 shows the molecular structure of  $^{14}\text{C}$ -labeled NNK.

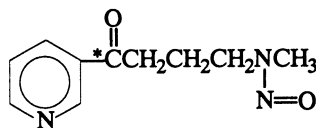


Fig. 1. Molecular structure of NNK with a single  $^{14}\text{C}$  atom at 1-position (\*)

Unfortunately, the genotoxicity of NNK in humans is difficult to determine accurately. The largest uncertainty results from the fact that studies with laboratory animals are generally carried out at doses well in excess of actual human exposure levels. Radioimmunoassay has been used to detect the methylated DNA adducts in rat lungs following a treatment with NNK at the lowest dose of 0.1 mg kg b.w.<sup>-1</sup> (Belinsky *et al.* 1990). However, this dose is *ca.*  $3 \times 10^3$ -fold higher than the actual daily exposure to NNK of smokers (Hoffmann and Hecht 1985).

In this study, we report sensitive measurements of DNA adduction with NNK in mice in a wide dose range beginning at a very low level just above the detection limit of our AMS. We affirm the genotoxicity of nicotine in mice at low dose levels with the aid of the high sensitivity of the AMS method.

## METHODS

$^{14}\text{C}$ -nicotine [pyrrolidine-2- $^{14}\text{C}$ ] is a Dupont-NEN product with very high specific activity of 1.9 GBq mmol<sup>-1</sup> (51.35 mCi mmol<sup>-1</sup>), equivalent to  $^{14}\text{C}$  in 82.14% of the molecules. We synthesized  $^{14}\text{C}$ -NNK (6.59 MBq mmol<sup>-1</sup>) by the procedure of Decker and Sammeck (1964). Nitrosation of  $^{14}\text{C}$ -4-(methylamino)-1-(3-pyridyl)-1-butanone·2HCl forming  $^{14}\text{C}$ -NNK was induced by sodium nitrite in the dark. We fed  $^{14}\text{C}$ -nicotine and  $^{14}\text{C}$ -NNK in 2% ethanol (10  $\mu\text{l}$  g b.w.<sup>-1</sup>) to mice (Kunming, 25–30 g) by stomach intubation (gavage). Doses of nicotine administered were from 0.4  $\mu\text{g}$  to  $4.0 \times 10^2$   $\mu\text{g}$  per kg b.w., and doses of NNK from 0.1  $\mu\text{g}$  to  $2.0 \times 10^4$   $\mu\text{g}$  per kg b.w. Mice were sacrificed at 24 h post administration and DNA adducts in the liver were isolated and purified as described by Gupta (1984). DNA purity was assayed by UV spectrometry,  $A_{260\text{nm}}/A_{280\text{nm}} = 1.84 \pm 0.02$ , with a standard ratio =  $1.82 \pm 0.05$ . Then we converted the purified DNA adducts to graphite samples to be used as ion source in the AMS. The conversion of DNA adducts to graphite was made after Vogel's protocol, consisting of oxidation of DNA to CO<sub>2</sub> by CuO and further reduction of CO<sub>2</sub> to graphite by Zn and TiH<sub>2</sub> in sealed tubes in a vacuum system (Vogel and Turteltaub 1992). The  $^{14}\text{C}$  contents were measured by a protocol developed for the 2×6 MV EN Tandem AMS facility at the Institute of Heavy Ion Physics, Peking University. Details on this procedure can be found elsewhere (Chen *et al.* 1990). Our AMS allows measurements of the isotope ratio  $^{14}\text{C}/^{12}\text{C}$  with a sensitivity of  $7 \times 10^{-15}$  and an instrument precision of 1%–4%. The isotope ratio was converted to the number of adducts based on some fundamental data (1 pMC =  $5.9 \times 10^{10}$  C gC<sup>-1</sup>; 1  $\mu\text{g}$  of DNA = 3240 pmol of nucleotide; carbon content in DNA = 30.0%  $\pm$  0.5%).

## RESULTS AND DISCUSSION

By careful handling of the samples (usually 1–3 mg) in disposable labware, the blank pMC of DNA from control mice was 1.19–1.29, proving that DNA isolation and graphite preparation were carried out without detectable  $^{14}\text{C}$  contamination. The  $^{14}\text{C}$  content in a core of a lump of Chinese anthracite was 0.07 pMC, used as a monitor of any contamination from the biological sample preparation system. We also used graphite from Chinese glucose prepared through the same sample preparation system as an internal standard.

We measured graphite samples from the DNA adducts in mice exposed to nicotine at different doses by AMS and calculated the numbers of DNA adducts. Each datum (o in the figures) was calculated from a mean value of 5–7 runs of AMS measurements. We have achieved a very low detection limit of 1 adduct per  $10^{11}$  nucleotides for  $^{14}\text{C}$ -labeled DNA adducts. This detection limit is 1–4 orders of magnitude lower than that of other techniques used for a quantitative assay of DNA adducts, such as  $^{32}\text{P}$ -postlabeling, radioimmunoassay or fluorescence spectrometry (Gupta 1985).

Figure 2 shows the dose response of nicotine adducts in mouse liver. DNA adducts increase with increasing dose levels in this log / log presentation. The DNA adduction with toxic chemicals or carcinogens causes genetic changes in cells, and some fixed changes can cause DNA replication errors, resulting in mutations. In some cases, the mutations occur in genes controlling cell proliferation and replication, leading to cancers. It can be concluded that DNA adduction may be an early but critical step in chemical carcinogenesis. Therefore, DNA adducts have been widely accepted as a valid internal dosimeter for monitoring exposure to toxic chemicals and carcinogens (Beland and Poirier 1993; Farmer 1994).

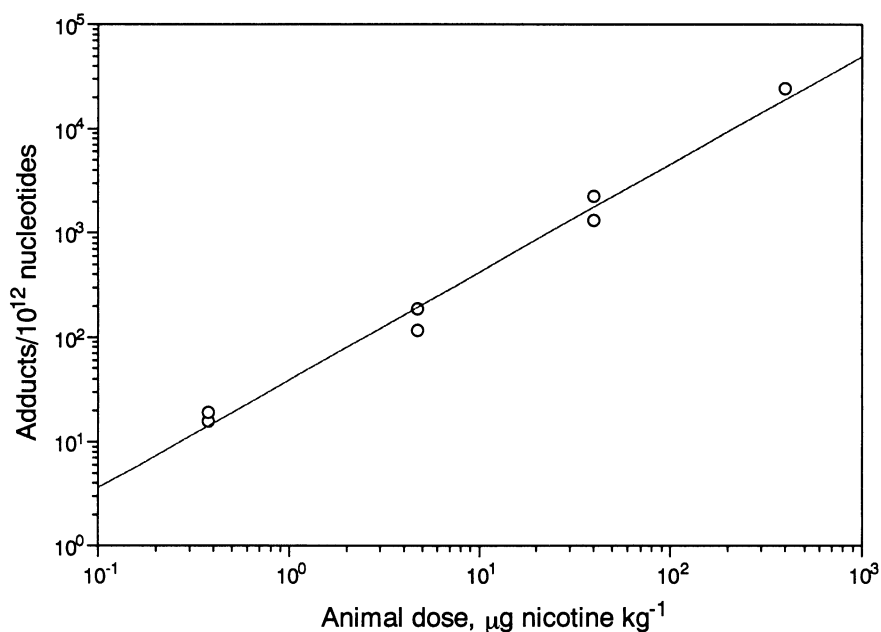


Fig. 2. Influence of nicotine exposure dose on DNA adduct levels. Regression results:  $Y = a + bX$ ,  $a = 1.59 \pm 0.07$ ,  $b = 1.03 \pm 0.05$ ,  $r = 0.993$



Our findings concerning nicotine-DNA adduction and its good linear dependence on the exposure dose levels raises doubts about previous claims that nicotine is noncarcinogenic. Based on our observations in the preceding paragraph, we speculate that nicotine, besides being a source of conversion to carcinogenic nitrosamines, is likely a potential carcinogen *per se*.

In the case of NNK, we covered a much wider range of doses, namely from  $0.1 \mu\text{g}$  to  $2.0 \times 10^4 \mu\text{g}$  per kg b.w. in measuring NNK-DNA adduction. Figure 3 shows the dependence of DNA adduction on the NNK exposure dose in mice. The lowest dose of NNK,  $0.1 \mu\text{g kg b.w.}^{-1}$ , is 3 orders of magnitude lower than that accessible by other techniques (Belinsky *et al.* 1990).

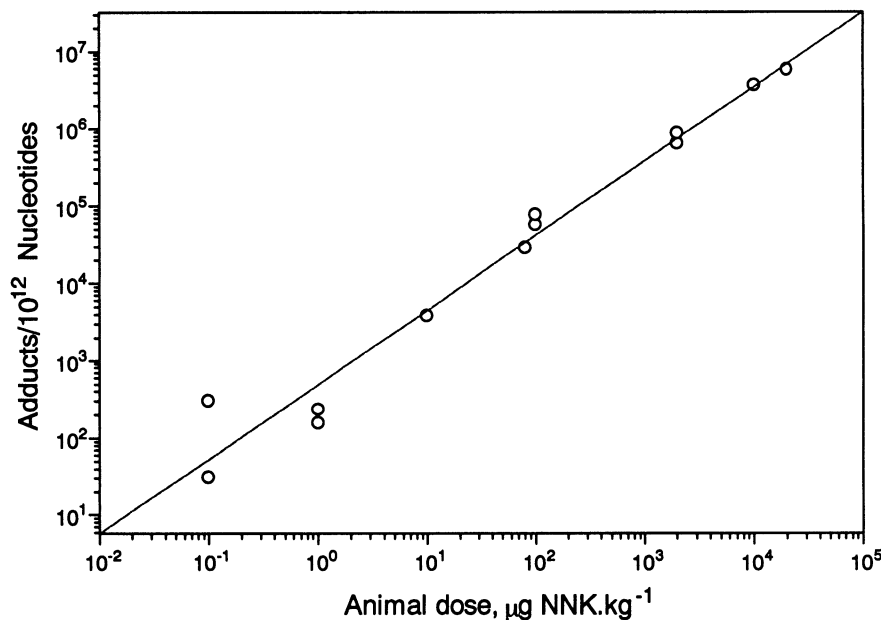


Fig. 3. Influence of NNK exposure dose on DNA adduct levels. Regression results:  $Y = a + bX$ ,  $a = 2.69 \pm 0.13$ ,  $b = 0.96 \pm 0.05$ ,  $r = 0.985$ .

Although AMS provides no direct structural information on DNA adduction, in our study it yields some evidence on the mechanism of the adduction reaction. Hoffmann and Hecht (1985) proposed that two possible routes may lead NNK to damage DNA: (1) methylation of DNA by metabolite  $\text{CH}_2\text{N}=\text{NOH}$  at the terminal of NNK; (2) reaction of DNA with the active metabolite 4-(3-pyridyl)-4-oxo-butyldiazohydroxide (Fig. 4). In our study only the  $^{14}\text{C}$ -containing portion of NNK bound to the separated hepatic DNA could be detected by AMS, and the labeled NNK was synthesized with  $^{14}\text{C}$  in the 1-position attached to the pyridine ring (Fig. 1). Hence, we have obtained an experimental finding to support route (2) in which the final active metabolite of NNK,  $^{14}\text{C}$ -labeled-4-(3-pyridyl)-4-oxo-butyldiazohydroxide, combines with DNA. However, our result does not provide any positive or negative evidence for the existence of route (1).

In the animal tests, we created an exposure to nicotine and NNK *via* oral gavage that was similar to the ingestion exposure route in humans by snuff-dipping. Actually, only very few people dip snuff nowadays. Hoffmann and Hecht (1985) estimated that one snuff dipper ingested 10–20 mg of nicotine by consuming 1 g of fine-cut tobacco. We will assume the lower value of 10 mg of nicotine in

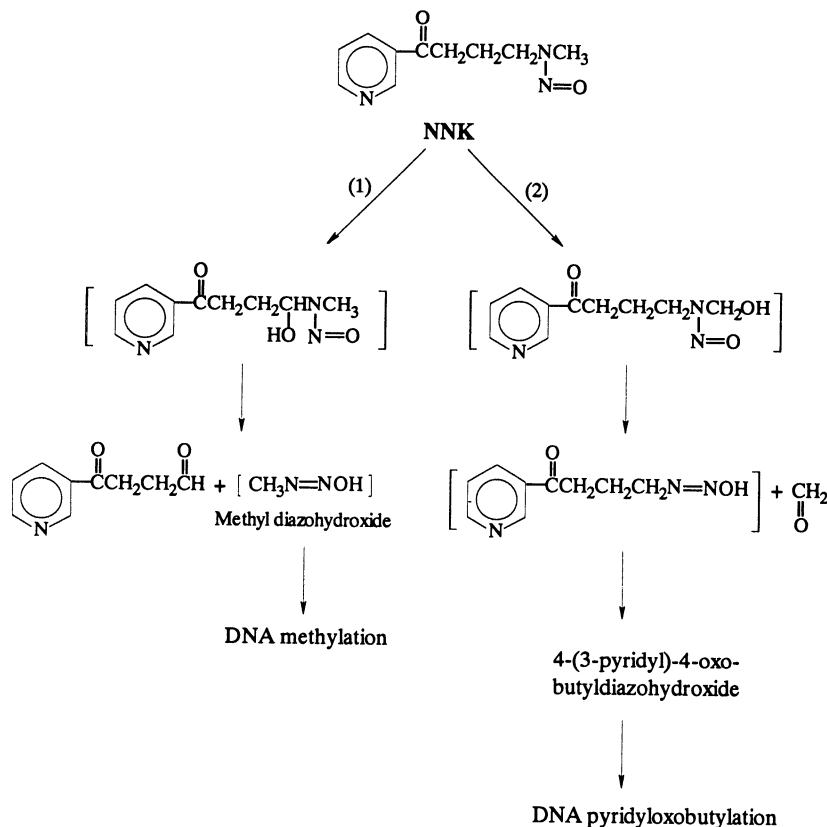


Fig. 4. Metabolic activation of NNK. Structures in brackets are likely intermediates that have not been isolated.

the following discussion. The exposure to NNK of a snuff-dipper consuming 10 g of tobacco was 16.1  $\mu\text{g}$ , averaged from the leading five U.S. fine-cut tobaccos (Brunnemann, Scott and Hoffman 1982). Based on these estimated values, basic human physiological data ( $3 \times 10^7$  DNA/cell,  $1.5 \times 10^{13}$  cells/liver), and the data from the curves in Figure 2 and Figure 3 we can make a carcinogenic risk assessment in terms of a directly perceivable relation between the tobacco consumption and DNA adduction (Table 1).

Our findings show that AMS provides a very sensitive tool for the quantification of DNA adducts using  $^{14}\text{C}$  tagged xenobiotics. Furthermore, it is evident that AMS would be a potential tool in biomedical research (Liu *et al.* 1994), particularly screening for genotoxicity and making risk assessment of drugs and chemicals at environmental exposure levels (Weisman 1996).

#### ACKNOWLEDGMENTS

We thank the National Natural Science Foundation of China for financial support. We are also very thankful to Prof. Hans von Gunten for his valuable comments, and to Profs. C. E. Chen and S. X. Yuan for their assistance.

TABLE 1. Exposure Dose Response of DNA Adduction in a Snuff-Dipping Person (70 kg b.w.)

Chemicals	Tobacco consumed (g)	Exposure dose ( $\mu\text{g}/\text{kg}$ b.w.)	Adducts / $10^{12}$ DNA	Adducts / liver cell	Adducts / liver	Equivalent consumption of cigarettes*
Nicotine	1	$1.4 \times 10^2 \dagger$	$7.2 \times 10^3$	$2.2 \times 10^{-1}$	$3.3 \times 10^{12}$	7‡
NNK	10	$2.3 \times 10^{-1} \S$	$6.6 \times 10^1$	$2.0 \times 10^{-3}$	$3.0 \times 10^{10}$	111#

\*By the exposure route of mainstream smoking inhalation.

†10 mg / 70 kg = 143  $\mu\text{g}/\text{kg}$

‡In the mainstream smoke of one brand of U.S. commercial filter cigarettes, the nicotine level is 1.5 mg per cigarette (Adams, O'Mara-Adams and Hoffman 1987), and the exposure of nicotine to a snuff-dipper consuming 1 g of fine-cut tobacco is 10 mg. Therefore, 10 mg / 1.5 mg = 6.66.

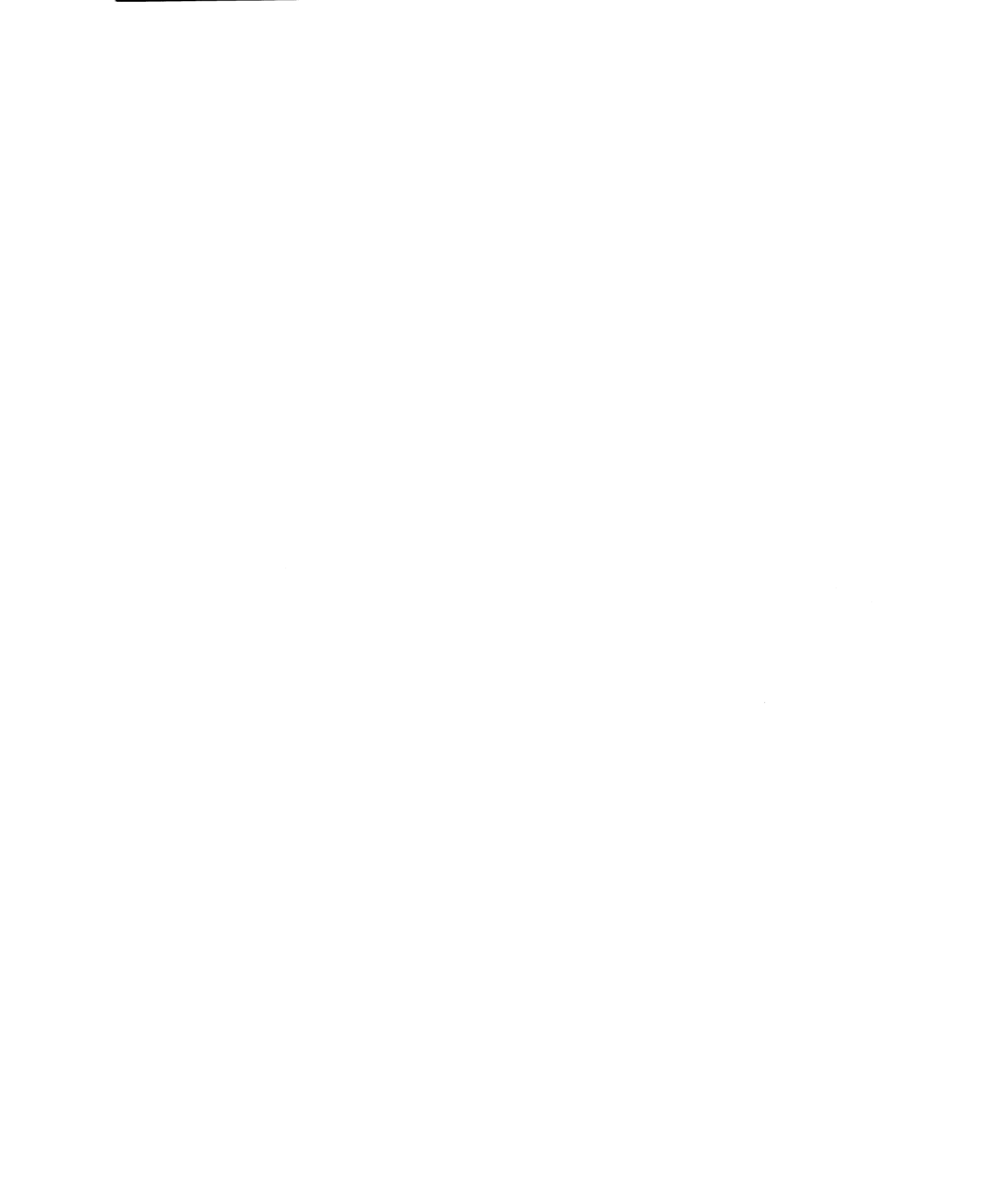
§16.1  $\mu\text{g}$  / 70 kg = 0.23  $\mu\text{g}/\text{kg}$ .

#The estimated exposure of a person to NNK of 20 cigarettes by the smoking inhalation route is 2.9  $\mu\text{g}$ , equivalent to 0.145  $\mu\text{g}$  per cigarette, and the exposure to NNK of 10 g tobacco by the snuff-dipping ingestion route is 16.1  $\mu\text{g}$  (Hoffmann and Hecht 1985). Therefore, the equivalent consumption from smoking cigarettes is 16.1  $\mu\text{g}$  / 0.145  $\mu\text{g}$  = 111.

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## AN ARCHAEOLOGICAL AND PALEONTOLOGICAL CHRONOLOGY FOR DAISY CAVE (CA-SMI-261), SAN MIGUEL ISLAND, CALIFORNIA

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**ABSTRACT.** We provide detailed contextual information on 25 <sup>14</sup>C dates for unusually well-preserved archaeological and paleontological remains from Daisy Cave. Paleontological materials, including faunal and floral remains, have been recovered from deposits spanning roughly the past 16,000 yr, while archaeological materials date back to *ca.* 10,500 BP. Multidisciplinary investigations at the site provide a detailed record of environmental and cultural changes on San Miguel Island during this time period. This record includes evidence for the local or regional extinction of a number of animal species, as well as some of the earliest evidence for the human use of boats and other maritime activities in the Americas. Data from Daisy Cave contribute to a growing body of evidence that Paleoindians had adapted to a wide variety of New World environments prior to 10,000 BP. Analysis of shell-charcoal pairs, along with isotopic analysis of associated marine shells, supports the general validity of marine shell dating, but also provides evidence for temporal fluctuations in the reservoir effect within the Santa Barbara Channel region.

### INTRODUCTION

Separated from the mainland for hundreds of thousands of years, California's Channel Islands provide some of the earliest evidence for fully developed maritime cultures in the New World and contain a fascinating record of biological evolution and extinctions along the California coast. Among the most important archaeological and paleontological localities on the Channel Islands is Daisy Cave (archaeological site CA-SMI-261), a cave and rockshelter complex that contains stratified artifact- and fossil-bearing deposits dating from the terminal Pleistocene to the present. It is located on the northeast shore of San Miguel Island *ca.* 40 km off the Santa Barbara mainland coast (Fig. 1). At present, Daisy Cave is the earliest documented coastal shell midden in North America, occupied sequentially by Paleoindian, Paleo-coastal, and more recent maritime peoples. Basketry and cordage remnants from the site date between *ca.* 8500 and 10,000 cal BP, doubling the documented antiquity of perishable woven technologies along the Pacific coast (Connolly, Erlandson and Norris 1995).

Five major archaeological components have been identified in the cave, with human occupations extending from *ca.* 12,000 to 700 cal BP. The earliest component, in which small quantities of chipped stone artifacts and marine shells indicate a relatively ephemeral occupation, shows that early maritime peoples used boats to visit or colonize the islands during the terminal Pleistocene. Much more extensive Early Holocene deposits, dated between *ca.* 8500 and 10,000 cal BP, have produced a diverse archaeological assemblage that includes basketry and cordage, a variety of chipped stone tools, and artifacts made from bone and marine shell. During the Middle Holocene, human occupation of Daisy Cave appears to be marked only by a thin shell midden stratum dated to *ca.* 6700 cal BP, but this component has produced a rich assemblage of culturally selected faunal remains. The Late Holocene was again marked by relatively intensive occupation; a diverse assemblage of woven artifacts, stone, bone, shell, and other artifacts has been recovered, including a unique "bead-maker's kit" (Rozaire 1978). Near the end of the occupational sequence, it appears that Chumash Indian people used the site primarily as a place to bury their dead.

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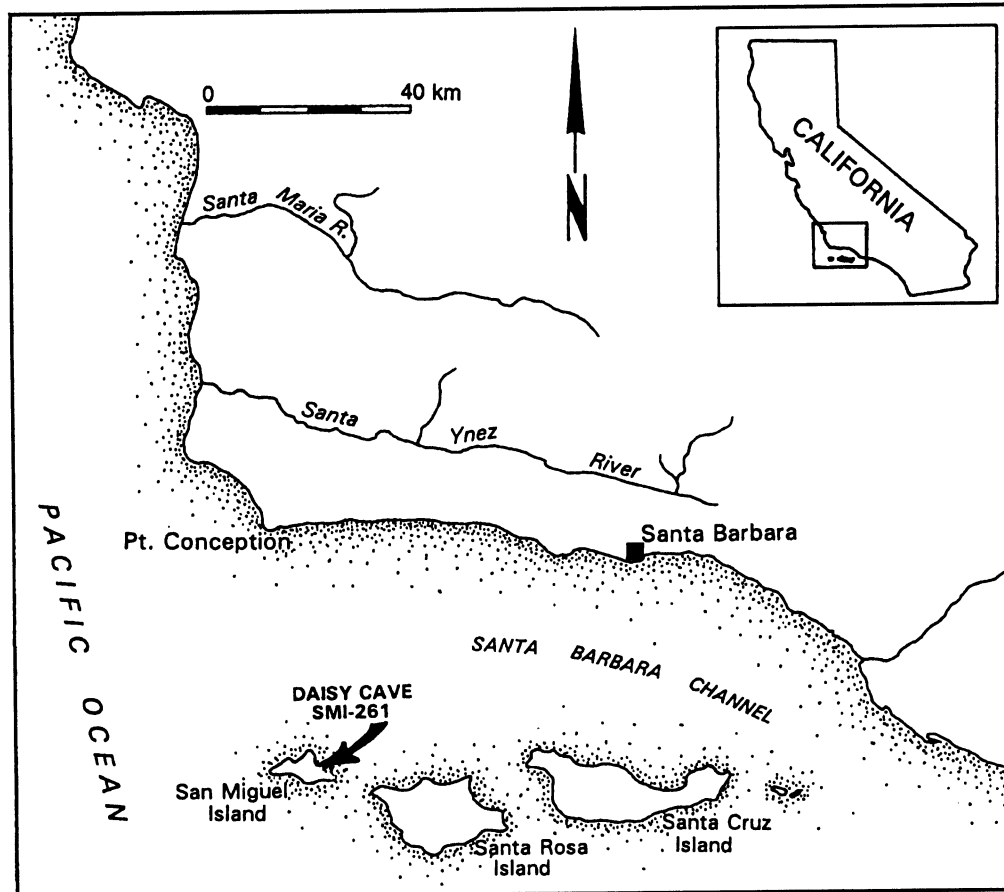


Fig. 1. Daisy Cave and the larger Santa Barbara Channel region

Faunal analyses of archaeological and paleontological specimens from the site have identified the remains of over 150 vertebrate and invertebrate species (Forgeng 1992), including several now extinct (Guthrie 1980; Walker 1980). This faunal assemblage results from the complex interaction of both cultural and biological (non-human) depositional processes. Recent research also shows that Daisy Cave contains a pollen record spanning much of the past 13,000 yr (West and Erlandson 1994), complementing a regional pollen record contained in varved sediments of the nearby Santa Barbara Basin (Heusser 1978). In short, Daisy Cave has produced a wealth of information the archaeology and paleoecology of the northern Channel Islands and the California coast (see also Connolly, Erlandson and Norris 1995; Erlandson 1991, 1993, 1994; Erlandson *et al.* 1996a, 1996b; Guthrie 1980, 1993; Kennett *et al.* 1996; Rozaire 1978, 1993; Walker 1980; Walker *et al.* 1978).

Until 1986, human occupation of Daisy Cave was thought to have begun only *ca.* 3000 yr ago (Guthrie 1980; Rozaire 1978; Walker *et al.* 1978). Since 1986, when  $^{14}\text{C}$  dating first demonstrated a much greater antiquity for the cave sequence, dating and other studies of the site or its contents have proceeded almost continuously. A comprehensive chronology for the cave deposits has not been published until now, however, and few details have been available on the context of individual  $^{14}\text{C}$  samples. In this paper, we provide detailed contextual information on 25  $^{14}\text{C}$  dates for the archaeo-

logical and paleontological deposits at Daisy Cave. These include dates on several matched pairs of marine shell and charcoal samples, which allow us to discuss methodological issues related to the comparability of marine and charcoal dates along the California coast and possible temporal fluctuations in the regional marine reservoir effect. In the sections that follow, we also discuss the general environmental and cultural patterns evident in the cave deposits, including vegetation changes in the site vicinity, evidence for the local or regional extinction of several animal species and general patterns of resource use by the human occupants of the cave. First, however, we provide background data on the structure of Daisy Cave, the history of research at the site and the methods employed in our study.

## DESCRIPTIVE BACKGROUND

### Physical Setting

San Miguel is the westernmost of the Northern Channel Islands, which contain an impoverished terrestrial flora and fauna compared to the adjacent California mainland. In contrast, the waters around the islands are nourished by upwelling and nutrient-rich currents that support a diverse and highly productive marine ecosystem. During the Last Glacial, the Northern Channel Islands were connected in a single island land mass known as Santarosae (Orr 1968). The ocean floor off Daisy Cave drops off rapidly, so the cave appears to have remained relatively close to the sea throughout the last 12,000 yr, even when sea levels were 25–50 m lower than at present. This sustained proximity to the sea partially explains the antiquity and extent of the archaeological deposits at the cave, since many Channel Island sites located along the current shoreline would have been several kilometers from the coast during the terminal Pleistocene.

Daisy Cave is located just above an isolated, rough and rocky stretch of shoreline on the northeast coast of San Miguel Island. The cave appears to have been formed during the Pleistocene by marine erosion associated with a high interglacial sea stand. Today, the cave is perched *ca.* 10 m above sea level immediately above the shoreline near the base of an ancient sea cliff of relatively erosion-resistant andesitic conglomerate. The site faces northward and is exposed to the full force of the northwesterly winds that buffet San Miguel Island much of the year (Johnson 1972: 63). The interior of Daisy Cave (Fig. 2), however, is one of the few places on the island where one can escape such winds. This shelter helps to explain the repeated and extensive use of the cave by humans, despite the lack of fresh water in the immediate vicinity.

Daisy Cave consists of a narrow fissure *ca.* 11 m deep and 1.5 to 3 m wide; an outer rockshelter *ca.* 4 m × 5 m wide and a stratified shell midden deposit on the slope in front of the cave and shelter. Archaeological and paleontological deposits have been found in all three site areas. The cave itself is divided into three narrow chambers separated by two low crawl spaces. Today, a person can stand comfortably in the front and back chambers, but the middle chamber provides sitting room only. The archaeological deposits at Daisy Cave seem to have covered over 200 m<sup>2</sup> at one time, but this extent has been reduced somewhat by coastal erosion.

### History of Excavation and Research

Daisy Cave is situated within the historical territory of the Chumash Indians, among the most socio-politically, economically and technologically complex hunter-gatherer cultures anywhere on earth (Arnold 1992; Moratto 1984: 118). The elaborate archaeological manifestations of Chumash society have attracted the attention of antiquarians, archaeologists, anthropologists and amateurs for over 125 yr. The earliest excavations of Daisy Cave probably occurred in the late 1800s and early 1900s.



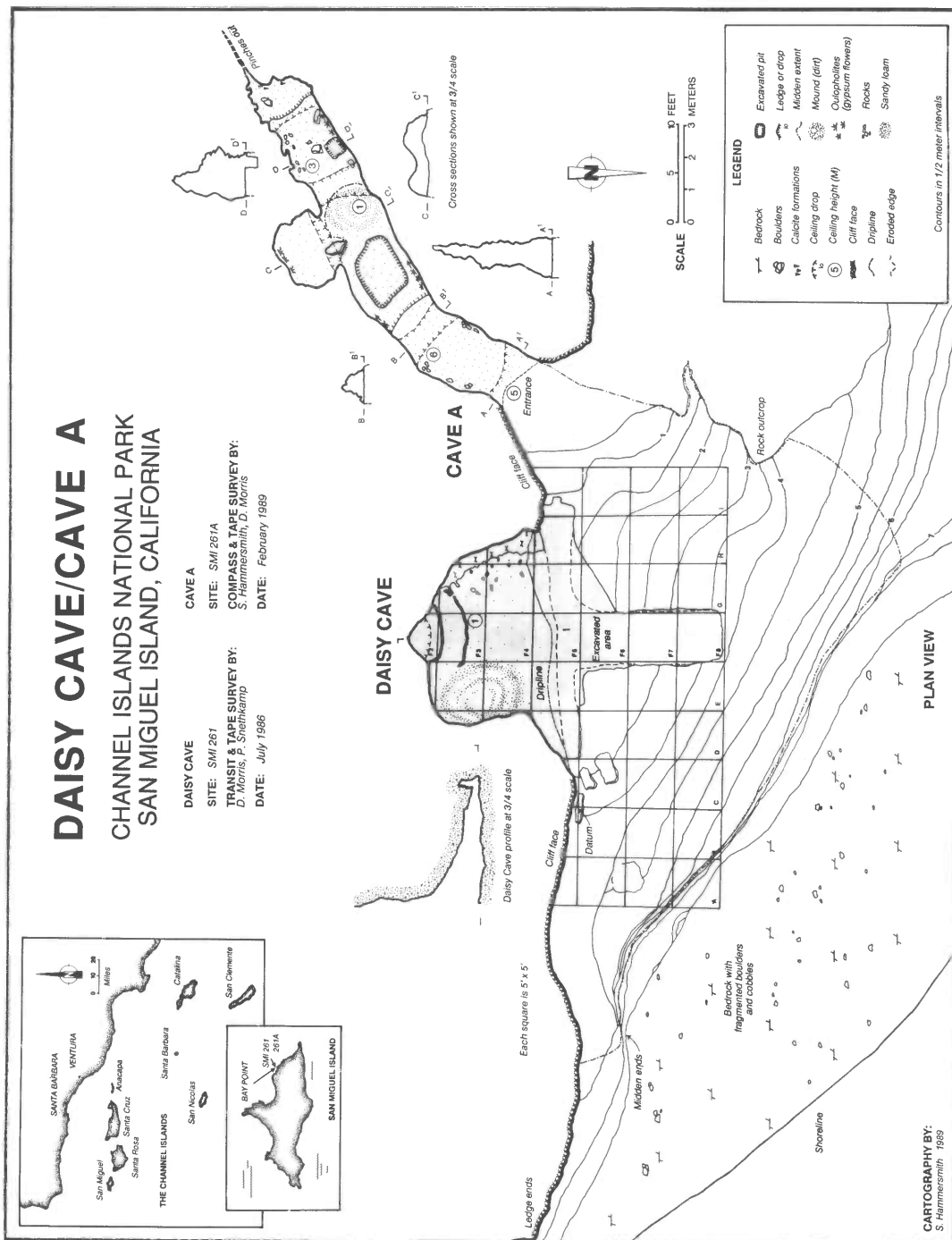


Fig. 2. Map of Daisy Cave and site CA-SMI-261, showing Rozaire's grid and excavation areas

The diary of a rancher's wife describes an 1888 outing in which a human skull was dug from a cave at "Eagle's Cliff" (Daly 1990), a historical place name for the Daisy Cave area. Another probable early excavator was Ralph Glidden, an energetic antiquarian who ransacked numerous San Miguel Island archaeological sites in 1919, with financial support from the Heye Foundation. Heye's (1921) report of Glidden's San Miguel Island expedition depicts a cemetery excavated near Bay Point. Neither Heye nor Glidden (1919) described the physical setting of this locality, but circumstantial evidence strongly suggests that the site was Daisy Cave since later investigations found the remnants of a badly disturbed cemetery inside the cave (Rozaire 1978; Walker *et al.* 1978). Phil Orr (1950: 14) of the Santa Barbara Museum of Natural History probably also describes a visit to the site (which he called Hoffmann Cave), a place where "ancient Indians had been buried."

In 1967–1968, the first well-documented archaeological investigation at Daisy Cave was directed by Charles Rozaire of the Los Angeles County Museum of Natural History. Using methods typical of California archaeology at the time, Rozaire's team excavated *ca.* 20% of the archaeological deposits at Daisy Cave, including the whole interior of the rockshelter, a trench through the midden in front of the shelter and a test pit in the middle chamber of the cave. Rozaire (1978) recovered a diverse array of artifacts and faunal remains and collected the badly disturbed skeletal remains of at least 26 people who had once been buried in the cave. Each 5 × 5 foot (1.52 m<sup>2</sup>) test pit was excavated in arbitrary 6-inch (15.2 cm) levels oriented horizontally. Many of these appear to have cross-cut the finely stratified cave sediments, especially in the deeper deposits in front of the cave and rockshelter, where the strata slope downward to the east and north. As a result, it is difficult to confidently associate much of this assemblage with anything other than very general time periods. Because 1/4-inch (~ 6 mm) screen was used to sift most of the excavated sediments, small artifacts and faunal remains are underrepresented in the recovered assemblage. At the time, the chronology of the cave occupation was estimated strictly from temporally sensitive artifacts—rare in early Channel Islands assemblages—and Rozaire (1978) and Walker *et al.* (1978) concluded that the excavated strata spanned only the last 3000 yr.

The true antiquity of the cave deposits was not recognized until 1985–1986, when Guthrie, Morris and Snethkamp collected materials from the site surface and stratigraphic profiles, cleared and redocumented the strata exposed in Rozaire's trench walls, excavated two small test pits inside the cave and collected two column samples from the trench walls in front of the rockshelter. The test pits in the cave recovered little, but the columns—carefully excavated in natural stratigraphic levels—recovered invaluable faunal and artifactual remains. Dating of shells from these columns first suggested a terminal Pleistocene age for the earliest human occupations at Daisy Cave and established the Pleistocene age of the underlying paleontological strata.

In 1989, Morris, S. Hammersmith and Erlandson finished mapping Daisy Cave and laid plans for additional site investigations. In 1991, the 1985–1986 collections were transferred to the University of Oregon for detailed analysis. In the summers of 1992, 1993, 1994 and 1996, Erlandson directed further investigations at Daisy Cave, designed to collect additional archaeological and paleontological samples, recover a sample of unique basketry and cordage identified in the Early Holocene levels at the site, explore the evidence for a terminal Pleistocene human occupation of the cave and extend the paleontological record further back into the Pleistocene. The 1992–1996 efforts completed the stratigraphically controlled excavation of three 50 cm × 100 cm wide test units in the deposits outside the rockshelter and an exploratory sounding extending Rozaire's test pit inside the cave deeper into stratified sediments beneath the cave floor.

## DATING METHODS

The 25  $^{14}\text{C}$  samples described below were analyzed at two laboratories. Twelve marine shell samples and one charcoal sample were analyzed at Beta Analytic, Inc. (Beta) of Coral Gables, Florida, using conventional liquid scintillation techniques. Eleven small charcoal samples and one marine shell sample were analyzed at the Center for Accelerator Mass Spectrometry (CAMS) at the University of California's Lawrence Livermore National Laboratory.

At Beta Analytic, the external surfaces of the shell samples were etched in dilute HCl acid to remove portions of the shell most susceptible to contamination *via* weathering and exchange with the surrounding matrix. The shell samples submitted were unusually well preserved, however, with little or no evidence for weathering or contamination. After pretreatment, the remaining shell from each sample was dissolved in a second acid wash to produce carbon dioxide. The charcoal sample was crushed and dispersed in deionized water and submitted to successive HCl-NaOH-HCl washes to eliminate carbonates, remove mechanical contaminants and secondary organic acids, and neutralize the solution. According to correspondence from Beta Analytic, benzene synthesis and counting proceeded normally for all samples submitted.  $^{13}\text{C}/^{12}\text{C}$  ratios were measured for each shell sample and  $^{14}\text{C}$  ages were adjusted accordingly.

At the CAMS facility, small charcoal samples weighing 2 to 4 mg were selected for dating from samples consisting mostly of carbonized twigs culled from flotation samples housed at the University of Oregon. Small twigs were selected to avoid the "old wood" effect (Schiffer 1986), a potentially significant problem on the Channel Islands where driftwood logs or wood from relict terminal Pleistocene forests may have been burned by the human occupants of the cave. Prior to dating, the charcoal samples were pretreated with alternating acid-base-acid rinses using HCl (1.0N) and NaOH (1.0N), with final rinses in acid to remove any absorbed  $\text{CO}_2$  and in deionized water to remove adhering organic acids or carbonate. The samples were dried and combusted at  $850^\circ\text{C}$  for 5 h in sealed quartz tubes to convert organic carbon to carbon dioxide. The evolved  $\text{CO}_2$  from each sample was converted to graphite using standard techniques (Vogel, Nelson and Southon 1987).  $^{14}\text{C}/^{12}\text{C}$  ratios were measured by AMS, as described elsewhere in detail (Davis *et al.* 1990). We assumed a  $^{13}\text{C}$  value of  $-25$  in calculating  $^{14}\text{C}/^{13}\text{C}$  ratios and correcting for mass-dependent fractionation.

To effectively compare dates for marine shell and charcoal samples, all  $^{14}\text{C}$  dates were calibrated via Stuiver and Reimer's (1993) CALIB 3.0.3 computer program (Table 1). These calibrated calendar dates were then used to construct a relatively high-resolution chronology for the finely stratified archaeological and paleontological deposits at Daisy Cave. Paired samples of marine shell and charcoal from the same stratigraphic levels were also used to examine the potential for temporal fluctuations in the intensity of marine upwelling and the regional reservoir effect ( $\Delta R$ ) in the Santa Barbara Channel area (Kennett *et al.* 1996).

## RADIOCARBON DATES FOR ARCHAEOLOGICAL AND PALEONTOLOGICAL SAMPLES

### Late Holocene Series

These samples are from two site areas: the upper strata excavated by Rozaire in the relatively shallow midden deposits inside the rockshelter; and from Stratum A, the uppermost of the deep midden deposits in front of the rockshelter (Fig. 3). Most of the sediments inside the rockshelter appear to have been stratigraphically superior to, or contemporary with, Stratum A. Artifacts found within the rockshelter sediments include a "bead-maker's kit" containing Purple Olive (*Olivella biplicata*) shell bead blanks, shell fishhook blanks, small chert bladelets and bladelet drills used in bead-mak-

TABLE 1: A Radiocarbon Chronology for Daisy Cave (CA-SMI-261), San Miguel Island, California\*

Provenience (Unit/Stratum)	Lab number	Material dated	Uncorrected <sup>14</sup> C age	<sup>13</sup> C/ <sup>12</sup> C adjusted	Calendar age range (cal BP)
Unit G3, 15–30 cm	Beta-56911	Giant chiton shell	1050 ± 80	1400 ± 80	780 (700) 640
Col. E-6, Str. A1	CAMS-8864	Charred twig	--	3220 ± 70	3360 (3430) 3480
Col. E-6, Str. A1	Beta-49997	Black abalone shell	3070 ± 80	3510 ± 80	2970 (3110) 3230
Col. E-6, Str. A3	CAMS-9095	Charred twig	--	3110 ± 60	3220 (3350) 3380
Col. E-6, Str. A3	Beta-15619	Red abalone shell	2990 ± 90	3430 ± 90	2860 (2980) 3140
Col. E-6, Str. C	CAMS-8862	Charred twig	--	6000 ± 70	6750 (6830) 6900
Col. E-6, Str. C	Beta-15620	Black abalone shell	5940 ± 110	6380 ± 110	6440 (6600) 6720
Col. E-6, Str. C	Beta-52359	CA mussel shell	6090 ± 80	6500 ± 80	6630 (6720) 6820
Col. E-6, Str. E1	CAMS-8866	Charred twig	--	7810 ± 60	8440 (8550) 8580
Col. E-6, Str. E1	Beta-15621	Black abalone shell	8030 ± 100	8460 ± 100	8530 (8680) 8930
Col. I-5, Str. J	Beta-19871	CA mussel shell	8290 ± 200	8700 ± 200	8730 (9010) 9340
Col. E-6, Str. E4	CAMS-8865	Charred twig	--	8040 ± 60	8730 (8960) 8990
Col. E-6, Str. E4	Beta-15622	Black abalone shell	8270 ± 120	8730 ± 120	8950 (9080) 9260
Col. E-6, Str. F1	CAMS-8867	Charred twig	--	8600 ± 60	9490 (9520) 9550
Col. E-6, Str. F1	Beta-15623	CA mussel shell	8470 ± 120	8900 ± 120	9170 (9330) 9430
Col. E-6, Str. F3	CAMS-8863	Charred twig	--	8810 ± 80	9650 (9770) 9910
Cave A, 26–30 cm	CAMS-12456	CA mussel shell	--	9110 ± 90	9420 (9480) 9580
Cave A, 26–30 cm	CAMS-14366	Charred twig	--	9180 ± 60	10,040 (10,060) 10,280
Col. E-6, Str. F3	Beta-49948	CA mussel shell	8940 ± 90	9360 ± 90	9650 (9830) 9900
Col. E-6, Str. G	Beta-52360	Black turban shell	10,180 ± 70	10,600 ± 70	11,250 (11,460) 11,670
Col. E-6, Str. G	Beta-14660	Red abalone shell	10,260 ± 90	10,700 ± 90	11,440 (11,670) 11,940
Col. E-6, Str. G	CAMS-9094	Wood charcoal	--	10,390 ± 130	12,060 (12,290) 12,460
Col. E-6, Str. I	CAMS-9096	Wood charcoal	--	11,180 ± 130	12,950 (13,090) 13,230
Col. E-6, Str. J	CAMS-14369	Charred twig	--	11,700 ± 70	13,520 (13,640) 13,770
Cave A, 150–60 cm	Beta-77070	Wood charcoal	15,780 ± 120	15,780 ± 120	18,530 (18,670) 18,810

\*Beta Analytic dates are based on conventional (LSC) radiocarbon dating; CAMS dates are based on accelerator mass spectrometry. CA mussel = California mussel (*Mytilus californianus*). Rounded calendar ages include midpoint (in parentheses) and age range at one sigma, calibration via Stuiver and Reimer's (1993) CALIB 3.0.3 program.

ing, the remnants of a woven basketry bag and other artifacts (Rozaire 1993). These rockshelter sediments also produced numerous other woven sea grass (*Phyllospadix* sp.) items, including cordage, rope and a textile fragment (Rozaire 1978). Tarring pebbles, heated and swirled inside baskets with bitumen to make them watertight, were also found primarily in the upper levels. The Late Holocene levels were rich in faunal remains, including sea mammals (cetaceans, pinnipeds and sea otters), a diverse array of birds, marine fish and shellfish (mussels, abalones, turbans, sea urchins, etc.) and limited amounts of terrestrial mammal (mice, shrew, bat, island fox and skunk). Stratum A, once one of the major occupational strata on the slope in front of the rockshelter, has been highly eroded due to sheet wash and other processes. Recent small-scale excavations in the remnants of this deposit have produced numerous faunal remains and expedient chipped stone artifacts, but relatively few formal tools or other artifacts.

#### Beta-56911. Unit G-3, 15–30 cm

1400 ± 80

Giant chiton (*Cryptochiton stelleri*) shell fragment, 15.6 g, from Rozaire's test pit excavated inside the rockshelter at Daisy Cave. Collected 1967–1968 by LACMNH archaeologists, submitted 1992 by Erlandson to estimate the age of the most recent human occupation of Daisy Cave.

*Comment* (J.E.): this shell sample came from the same unit and general level that produced the "bead-maker's kit." Similar caches have been found in Northern Channel Islands sites dating to the last 1000 to 2000 yr (e.g., Orr 1968). Beta-56911, with a calibrated midpoint equal to ca. 700 cal BP,

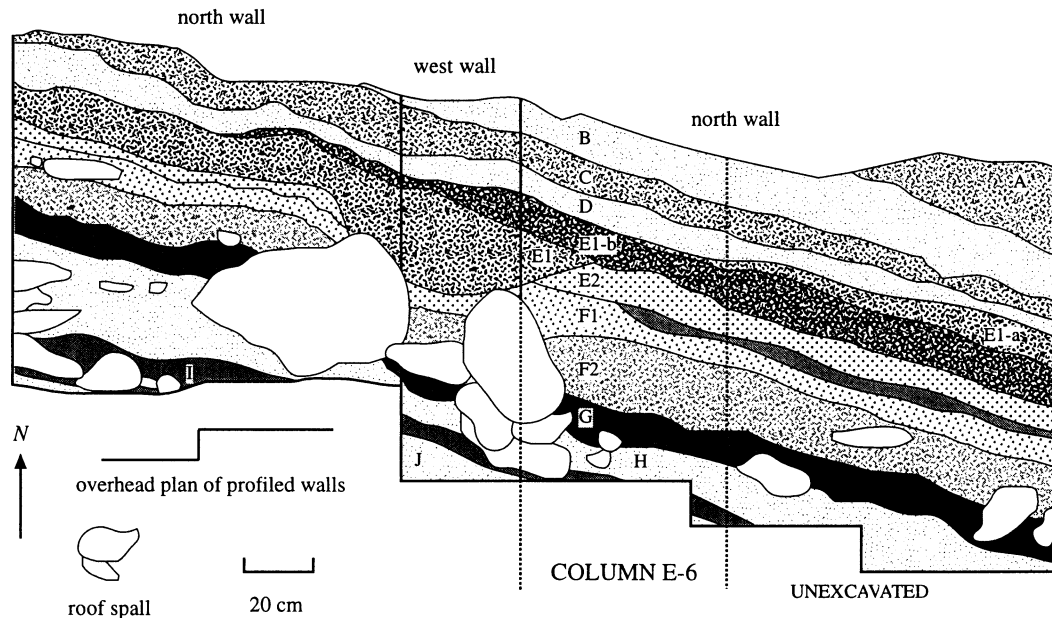


Fig. 3. Stratigraphy of archaeological and paleontological deposits along the dripline of the Daisy Cave rockshelter

is consistent with other data on the development of craft specialization in bladelet and bead production (Arnold 1987, 1992).

**CAMS-9095. Column E-6, Stratum A3**

**3110 ± 60**

A small burned twig fragment from near the base of Stratum A, this sample comes from the same 10–12 cm thick stratum as marine shell sample Beta-15619. Collected 1985 by Guthrie and P. Snethkamp, submitted 1993 by Erlandson and Kennett to test comparability of marine shell and charcoal dates from cave strata.

*Comment* (J.E./D.K./L.I.): calibration of this date suggests that Stratum A may have begun to accumulate *ca.* 3400 cal BP. Generally, the shell and charcoal dates are quite similar given the lack of precise spatial association, but the calibrated midpoint for the charcoal date is unexpectedly older (+370 yr) than the associated shell date (Beta-15619). Since an “old wood” problem should not affect this small twig, the difference may be due to other factors (see CAMS-8864).

**CAMS-8864. Column E-6, Stratum A1**

**3220 ± 70**

A small burned twig from the upper part of Stratum A, this sample came from the same 4–6 cm thick stratum as marine shell sample Beta-49997. Collected 1985 by Guthrie and Snethkamp, submitted 1993 by Erlandson and Kennett to test the comparability of shell and charcoal dates and help refine the chronology for the human use of Daisy Cave.

*Comment* (J.E./D.K./L.I.): once again, shell and charcoal dates generally correspond, but the calibrated midpoint for this date is considerably older (+320 yr) than expected. Consistent variation between the two shell/charcoal “pairs” from Stratum A suggests that differences between the shell and charcoal dates may be due to temporal fluctuations in the intensity of marine upwelling and the reservoir effect (see Kennett *et al.* 1996).

**Beta-15619. Column E-6, Stratum A3****3430 ± 90**

Red abalone (*Haliotis rufescens*) shell, 82 g, from 25 cm below surface and near the base of Stratum A. Collected 1985 by Guthrie and Snethkamp, submitted 1986 by Guthrie to establish the age of archaeological and paleontological strata in the upper part of the stratigraphic profile outside the rockshelter.

*Comment* (J.E./D.G.): this date first showed that the dense 30-cm-thick shell midden stratum capping the sequence in front of the rockshelter began accumulating *ca.* 3000 cal BP, roughly the age once estimated for the entire archaeological sequence at Daisy Cave.

**Beta-49997. Column E-6, Stratum A1****3510 ± 80**

Black abalone (*Haliotis cracherodii*) shell, 24.1 g, from upper 4–6 cm of Stratum A, the uppermost stratum identified outside the rockshelter in 1985–1986. Collected 1985 by Guthrie and Snethkamp, submitted 1991 by Erlandson and Morris.

*Comment* (J.E.): By 1992, the loose Stratum A sediments had been removed almost completely by sheet wash erosion. This date, slightly younger than those from the overlying samples, shows a mild stratigraphic reversal. The two shell dates from Stratum A overlap at only one sigma, however, so this variation may be insignificant.

*General Comments:* The four dates that bracket Stratum A outside the rockshelter suggest that this component may have accumulated relatively rapidly, possibly during a single occupation of the site beginning *ca.* 3200 ± 200 cal BP. The complex and finely stratified shell midden deposits once present inside the rockshelter suggest that Daisy Cave was occupied repeatedly during the Late Holocene, however, with additional refuse deposited around 700 cal BP. At some point, probably during the past 1000 yr, the cave itself seems to have been used primarily as a Chumash burial place.

**Middle Holocene Series**

Currently, a Middle Holocene human occupation of Daisy Cave is represented only in Stratum C, a 5–10-cm-thick shell midden identified in the deep midden deposits outside the rockshelter. However, the Stratum C cultural component may also have formed the base of the cultural deposits that Rozaire (1978) identified inside the rockshelter. After calibration, three dates for the occupation of Stratum C are highly consistent, averaging *ca.* 6720 ± 150 cal BP. Despite the relatively high density of archaeological materials in Stratum C, the thin nature of the deposits suggests that occupation of the site at this time was relatively brief. Due to the small volume of stratigraphically excavated sediments, relatively few artifacts can be confidently associated with this component. These include a few expedient flake tools, chipped stone debitage, a battered cobble hammerstone, a spire-ground Olivella bead, a fragment of worked bone and an abalone dish containing red ochre. The faunal assemblage is dominated by California mussel (*Mytilus californianus*) shells, but sea mammal, fish, bird and other vertebrate remains are also present.

The stratigraphy outside the rockshelter suggests that there was no substantial human occupation of Daisy Cave for long periods before and after the deposition of Stratum C. Above and below this occupational stratum are thin yellowish cave soils (Strata B and D) that appear to be culturally sterile. The few archaeological materials found in Strata B and D probably come from the accumulation of sediments over the uneven surfaces of the underlying midden strata, the downward trampling of cultural materials during later site occupations, or the limited redeposition of archaeological materials from upslope. Stratum B appears to have gradually accumulated during the Middle Holocene, between *ca.* 6700 and 3200 cal BP. The upper portions of Stratum D also seem to have accumulated early in the Middle Holocene. Faunal remains are relatively scarce in these noncultural strata, and

pollen analysis suggests that the vegetation in the site vicinity was dominated by modern taxa (West and Erlandson 1994).

**CAMS-8862. Column E-6, Stratum C** **6000 ± 70**

Small charred twig from flotation sample excavated by Guthrie and Snethkamp in 1985. Submitted 1993 by Erlandson and Kennett to refine the chronology of shell midden deposits in Stratum C and check the correlation between shell and charcoal samples from this short-term occupation level.

*Comment* (J.E.): after calibration, the midpoint of this date is 110 and 230 yr older than the midpoints for shell samples from the same stratum. All three dates overlap at two sigma, however, suggesting that this variation is not statistically significant.

**Beta-15620. Column E-6, Stratum C** **6380 ± 110**

Black abalone shell, 52 g, from 44 cm below the surface of midden. Collected 1985 by Guthrie and Snethkamp, submitted 1986 by Guthrie to establish the basic chronology of archaeological and paleontological deposits at Daisy Cave.

*Comment* (J.E.): this date, one of the initial  $^{14}\text{C}$  series for Daisy Cave, first established the antiquity of Stratum C and roughly doubled the known antiquity of human occupation of the site.

**Beta-52359. Column E-6, Stratum C** **6500 ± 80**

California mussel shell valve, 13.2 g, collected 1985 by Guthrie and Snethkamp, submitted 1992 by Erlandson to confirm the veracity of earlier date for Stratum C.

*Comment* (J.E.): analysis of second shell sample from Stratum C confirmed the antiquity of the layer and the Middle Holocene occupation of the cave.

### Early Holocene Series

Early Holocene archaeological and paleontological deposits have been identified inside Cave A, just inside the dripline of the rockshelter, and on the slope outside the cave and rockshelter. Of these, the deposits outside the cave/rockshelter are the most extensive, where Strata E and F are up to 50 cm thick and cover an area of roughly 120 m<sup>2</sup>. Dated between *ca.* 8500 and 9900 cal BP, the Early Holocene archaeological deposits appear to have accumulated during repeated short-term occupations of the site. Nonetheless, the extent and density of the refuse deposits suggest that the most intensive use of the site dates to this time period. Inside the driplines of the cave and rockshelter, these early shell midden deposits are loose and unconsolidated, almost completely undisturbed, remarkably well preserved, and interspersed with deposits of seabird guano. On the slope outside the driplines, most midden constituents in the same strata are still relatively well preserved, but seabird guano has been dispersed by rain and slopewash, and the strata are strongly indurated.

Artifacts recovered from the Early Holocene strata include three fragments of basketry and hundreds of strands of cordage made from sea grass, *Olivella* beads made by removing the spire or apex from whole shells, expedient chipped stone tools and toolmaking debris, small bipointed bone fish gorges, occasional leaf-shaped bifacial points or knives, and rarer stone, bone or shell artifact types. The most remarkable of these are the woven artifacts, which include what may be remnants of the toeflaps from two children's sandals (Connolly, Erlandson and Norris 1995). The shells of California mussels, black turbans (*Tegula funebris*) and black abalones dominate the faunal assemblage, but fish, sea mammal and bird remains are also present.

**CAMS-8866. Column E-6, Stratum E1****7810 ± 60**

Small charred twig fragment extracted from flotation sample recovered from the uppermost of the Early Holocene strata. Collected 1985 by Guthrie and Snethkamp, submitted 1993 by Erlandson and Kennett. After calibration, this is the youngest calendar date (8550 cal BP) available from the Early Holocene strata at Daisy Cave.

*Comment* (J.E.): this sample was submitted to test comparability of <sup>14</sup>C dates from marine shell and charcoal. Stratum E1 produced a fragment of twined basketry—one of the possible sandal fragments—and hundreds of fragments of sea grass cordage (Connolly, Erlandson and Norris 1995).

**CAMS-8865. Column E-6, Stratum E4****8040 ± 60**

Small charred twig fragment from a flotation sample from the lowest level of Stratum E. Collected 1985 by Guthrie and Snethkamp, submitted 1993 by Erlandson and Kennett.

*Comment* (J.E.): sample was submitted to test the comparability of marine shell and charcoal dates and to further refine the site chronology. After calibration, this date overlaps at one sigma with a shell date (Beta-15622) from the same stratigraphic level.

**Beta-15621. Column E-6, Stratum E1****8460 ± 100**

A single black abalone shell (52.5 g) from 55 cm below surface and the uppermost level of Stratum E. Collected 1985 by Guthrie and Snethkamp, submitted 1986 by Guthrie.

*Comment* (J.E.): this date, from the initial <sup>14</sup>C series for the site, almost tripled the known antiquity of human occupation at Daisy Cave. After calibration, this date overlaps at one sigma with the date (CAMS-8866) for a charred twig from the same level.

**CAMS-8867. Column E-6, Stratum F1****8600 ± 60**

Charred twig from a flotation sample collected 1985 by Guthrie and Snethkamp, submitted 1993 by Erlandson and Kennett to help refine the cave chronology and test comparability of marine shell and charcoal samples in close stratigraphic association.

*Comment* (J.E./D.K.): after calibration the two dates from Stratum F1 overlap at two sigma, but the younger shell sample may indicate that coastal upwelling was less intense at this time and that the regional reservoir effect was less pronounced than during historic times (Kennett *et al.* 1996).

**Beta-19871. Column I-5, Stratum J****8700 ± 200**

California mussel shell (19.3 g) from 108–111 cm below surface near the base of dense shell midden level in column sample located *ca.* 6 m west and upslope of Column E-6. Collected 1985 by Morris, submitted 1987 by Guthrie and Snethkamp to establish the antiquity of shell midden deposit closer to the mouth of Cave A.

*Comment* (J.E.): Stratum J from this column bears no relationship to Stratum J in Column E-6. The strata in Column I-5 were partly disturbed, probably by runoff along the base of the cliff. This sample appears to correlate with midden deposits near the base of Stratum E (see Beta-15622) located downslope in Column E-6.

**Beta-15622. Column E-6, Stratum E4****8730 ± 120**

Black abalone shell (63.4 g) from 75 cm below surface in the lowest level of Stratum E. Collected 1985 by Guthrie and Snethkamp, submitted 1986 by Guthrie to establish the antiquity of the early shell midden levels.



*Comment* (J.E.): this date, from the initial  $^{14}\text{C}$  series for the site, is consistent with stratigraphically overlying and underlying samples and established that much of the shell midden deposits outside Daisy Cave dated to the Early Holocene.

**CAMS-8863. Column E-6, Stratum F3** **8810 ± 80**

Small charred twig from lowest 7–8 cm of Stratum F, near the base of the dense shell midden deposits at Daisy Cave. Collected 1985 by Guthrie and Snethkamp, submitted 1993 by Erlandson and Kennett.

*Comment* (J.E.): this sample, from the same general midden level as Beta-49948, was submitted to confirm the cave chronology and compare paired marine shell and carbon samples to explore possible diachronic changes in coastal upwelling and its effects on the local reservoir effect. After calibration, the two samples are very similar, overlapping at one sigma.

**Beta-15623. Column E-6, Stratum F1** **8900 ± 120**

California mussel shell (91.0 g) from 80 cm below surface in the uppermost level of Stratum F, a relatively dense shell midden stratum containing numerous chipped stone artifacts. Collected 1985 by Guthrie and Snethkamp, submitted 1986 by Guthrie.

*Comment* (J.E.): this date, among the initial series for the site, clearly established the presence of maritime peoples on the Northern Channel Islands at *ca.* 9300 cal BP.

**CAMS-12456. Cave A, Profile A, Stratum VIII/IX** **9110 ± 90**

California mussel shell fragment from near base of shell midden exposed in sounding within the middle chamber of Cave A (Fig. 4). Shell, from a small bulk midden sample, collected 1993 from 26–30 cm below surface by Kennett and L. Haslouer, submitted 1994 by Erlandson and Kennett.

*Comment* (J.E.): this date suggests that the base of the shell midden inside Cave A also dates to the Early Holocene and is roughly contemporary with strata F2 and F3 outside the rockshelter. This is consistent with the presence of numerous fragments of sea grass cordage and abundant black turban shells, both common in synchronic levels outside the cave.

**CAMS-14366. Cave A, Profile A, Stratum VIII/IX** **9180 ± 60**

Small charred twig from shell midden lens exposed in sounding within the middle chamber of Cave A. Collected 1993 as part of shell/charcoal pair by Kennett and Haslouer, submitted 1994 by Erlandson and Kennett.

*Comment* (J.E.): after calibration, the midpoint of the date for this burned twig is almost 600 yr older than the associated shell, suggesting that marine upwelling was less intense at this time, that the cave occupants burned some older debris inside the cave, or some combination of the two. Calibration of this date suggests that the base of the midden inside Cave A may have been deposited as much as 10,060 yr ago.

**Beta-49948. Column E-6, Stratum F3** **9360 ± 90**

Fragments of California mussel shell (30 g) from the lowest level of the dense shell midden strata. Collected 1985 by Guthrie and Snethkamp, submitted 1991 by Erlandson and Morris to establish the age of the first unequivocal evidence for human occupation at Daisy Cave.

*Comment* (J.E.): calibration of this date suggests that the base of shell midden Stratum F outside the rockshelter was deposited *ca.* 9900 cal BP.

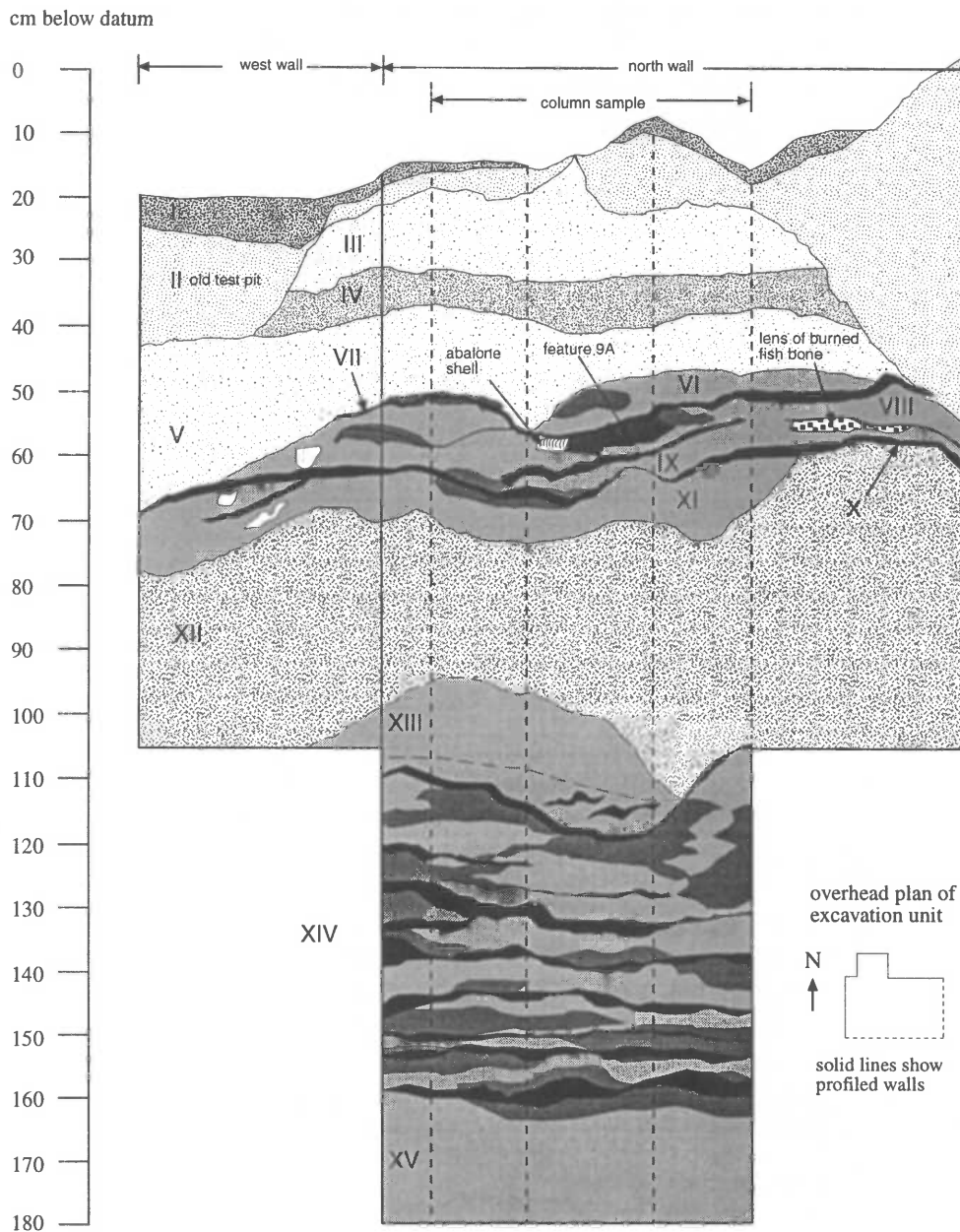


Fig. 4. Stratigraphy of test sounding in the middle chamber of Daisy Cave (CA-SMI-261A)

### Terminal Pleistocene Series

#### CAMS-9094. Column E-6, Stratum G

10,390 ± 130

Small twig fragment from same general provenience as Beta-14660 and Beta-52360, although shell is found primarily in the upper portions of the 4–6 cm thick Stratum G and materials from the

lower part of G were not differentiated during excavation. Collected 1985 by Guthrie and Snethkamp, submitted 1993 by Erlandson to test the accuracy of marine shell dates from same stratum.

*Comment* (J.E.): overall, this date fits well within the depositional chronology for Daisy Cave. After calibration this date, with a midpoint of *ca.* 12,290 cal BP, appears to be slightly older than shells from the same stratum. While the twig may have come from the lower portions of Stratum G, other evidence suggests that marine upwelling (and the regional reservoir effect) may have been less intense at this time, increasing the apparent disparity between the three dates from this stratum (Kennett et al. 1996).

**Beta-52360. Column E-6, Stratum G** **10,600 ± 70**

Black turban (*Tegula funebris*) shell fragments (13.2 g), broken and carefully cleaned of all adhering sediment, from the lowest shell midden stratum in Column E-6. Collected 1986 by Guthrie and Snethkamp, submitted 1992 by Erlandson and Snethkamp to check validity of Beta-14660.

*Comment* (J.E.): this date independently confirmed, based on the analysis of shell from a second gastropod species, that shell in Stratum G was deposited during the terminal Pleistocene. A >1000 yr gap between the deposition of shell at the base of Stratum F and the shells in Stratum G also suggests that displacement of shell or artifacts from above has not occurred.

**Beta-14660. Column E-6, Stratum G** **10,700 ± 90**

Red abalone shell (>100 g) from 110 cm below surface, at the base of shell-bearing deposits exposed in the north wall of Rozaire's test unit E-5, located along the dripline of the rockshelter. Collected 1985 by Guthrie and Snethkamp, submitted 1985 by Guthrie to establish the age of earliest apparent human occupation of the cave.

*Comment* (J.E.): this date first documented a very early human occupation of Daisy Cave and the presence of humans on the Northern Channel Islands at *ca.* 11,700 cal BP. The cultural origin of this abalone shell has been questioned (Erlandson 1991, 1994: 194), but subsequent work suggests that an ephemeral human occupation of Daisy Cave did take place during the terminal Pleistocene (Erlandson 1993; Erlandson et al. 1996b).

**CAMS-9096. Column E-6, Stratum I** **11,180 ± 130**

Small carbonized twig fragment from a dark brown cave soil below shell midden. Retrieved from flotation of sediments from column sample. Collected 1985 by Guthrie and Snethkamp, submitted 1993 by Erlandson to extend chronology of paleontological sediments underlying the archaeological strata.

*Comment* (J.E.): preliminary analysis of pollen from Stratum I suggests that pine trees were abundant in the cave vicinity at this time (West and Erlandson 1994).

**CAMS-14369. Column E-6, Stratum J** **11,700 ± 70**

Small carbonized twig fragment from thin cave soil below shell midden, retrieved from flotation of sediments from Column E-6. Collected 1985 by Guthrie and Snethkamp, submitted 1993 by Erlandson to extend chronology of paleontological strata at Daisy Cave.

*Comment* (J.E.): this date indicates that the base of the 1985 excavations terminated in levels equal to *ca.* 13,640 ± 120 cal BP. Pollen from this stratum also suggests that a pine forest existed in the cave vicinity at this time (West and Erlandson 1994).

**Beta-77070. Column CA, Stratum XIV****15,780 ± 120**

Chunks of wood charcoal (16.0 g), apparently from sizeable branches or logs, from the lowest of several dark charcoal-rich lenses located between 110 and 160 cm below the floor of the middle chamber of Cave A. Collected 1994 by Tveskov and Erlandson, submitted 1994 by Erlandson and Morris.

*Comment* (J.E.): the potential for an "old wood" problem was anticipated on submission of this sample, but the antiquity of this date was still unexpected. In the field these dark lenses, overlain by 30–50 cm of light brown sterile silt capped by an Early Holocene shell midden, were considered to be possible hearth features. They contain burned rock, welded sediments, burned bone, occasional wood fragments, and rare cultural artifacts. A large Monterey chert flake of unquestionable cultural origin was found *in situ* within the uppermost (undated) dark lens in Stratum XIV. A small chert flake was found in screen residuals from Stratum XIV, but its provenience is much less certain.

**DISCUSSION AND CONCLUSIONS**

Our chronological studies at Daisy Cave provide a detailed temporal framework for understanding environmental changes on the Northern Channel Islands and the southern California coast over the past 13,000 to 19,000 calendar yr. Pollen samples from the lower levels of Daisy Cave suggest, for instance, that prior to *ca.* 12,000 cal BP a pine forest grew outside the cave, probably covering a more extensive coastal plain exposed by lower sea levels (West and Erlandson 1994). The bathymetry off Daisy Cave is relatively steep, however, and even 12,000 yr ago this coastal plain was probably not more than several hundred meters wide. The presence of small relict stands of Bishop pine (*Pinus muricata*) and Torrey pine (*P. torreyana*) on the other Northern Channel Islands, and the recovery of a pine needle from Stratum G tentatively identified as *P. muricata*, suggest that this forest may have consisted of one or both of these species. The pollen spectra take on a more modern character after *ca.* 12,000 yr ago, when the percentage of pine pollen declines dramatically and Compositae, Rosaceae and *Quercus* (oak) pollen all increase significantly.

A comprehensive analysis of recently collected faunal remains from Daisy Cave has yet to be completed and contextual problems make it difficult to determine the age of many specimens excavated prior to the 1980s. Nonetheless, the remains of several species locally or regionally extinct have been found at Daisy Cave. Walker (1980) identified the remains of a relatively large deer mouse (*Peromyscus nesodytes*) in the lower levels of the cave, a species later replaced by a diminutive mainland cousin (*P. maniculatus*). Walker also identified the remains of the western spotted skunk (*Spilogale gracilis*) and an ornate shrew (*Sorex ornatus*), neither of which is present on San Miguel Island today. Guthrie (1980) identified the remains of an extinct vampire bat (*Desmodus stocki*), and an extinct flightless scoter (*Chendytes lawi*). Guthrie (1980: 695) also identified relatively large numbers of the bones of two sea birds that no longer breed on the island, the manx shearwater (*Puffinus puffinus*) and Leach's storm petrel (*Oceanodroma leucorhoa*), suggesting that they may have bred on the island in the past. Faunal remains from the shell midden strata also suggest that rocky intertidal habitats were productive throughout much of the past 10,000 yr. This contradicts some general claims (*e.g.*, Yesner 1987) that coastal habitats only became productive for human foragers to exploit when sea level stabilized during the Middle Holocene. Fish bones are relatively common in all the Holocene midden levels, and sea mammal and sea bird remains also are present.

Since at least 11,600 cal BP, the environmental record at Daisy Cave is accompanied by well-preserved shell midden deposits that document the adaptations of maritime peoples to the Santa Barbara Channel environment. Those shell midden deposits contain the remains of hundreds of animal

and plant species interspersed with other animal and plant remains deposited naturally as paleontological deposits. The available data suggest that Daisy Cave was occupied by humans primarily during five periods: an ephemeral episode of site use *ca.* 11,600 ± 150 cal BP; repeated occupations between *ca.* 8500 and 10,000 cal BP; a relatively brief episode *ca.* 6700 ± 200 cal BP; more intensive occupation(s) dating to *ca.* 3200 ± 200 cal BP; and one or more episodes *ca.* 700 ± 200 cal BP. The most poorly dated portion of this archaeological chronology is the younger end of the spectrum, because most of these deposits were excavated during the 1960s (Rozaire 1978) or have been eroded away. Additional occupational components may be discovered, however, with further dating of LACMNH collections.

Artifacts, ecofacts and features at Daisy Cave provide a wealth of information about human adaptations to the island and marine habitats and fluctuations in Channel Island environments through time. With the chronological and other analyses completed to date, several preliminary conclusions can be made about the environmental and archaeological records at Daisy Cave.

First, pine forest habitats appear to have been present on San Miguel Island—and probably were much more widespread in the Santa Barbara Channel area—until between 12,000 and 13,000 cal BP, when the frequency and percentages of pine pollen deposited at Daisy Cave decline dramatically. This is consistent with Axelrod's (1967: 110) suggestion that "an essentially continuous closed-cone pine forest blanketed the outer coast and islands during the late Pleistocene, and as recently as 14,500 to 12,000 years ago". It is also consistent with Heusser's (1978) pollen spectrum for the Santa Barbara Basin, in which the contribution of pine pollen decreases from *ca.* 40% to less than 20% in sediments dated to *ca.* 12,000 ± 500 cal BP. These vegetation changes are closely linked to postglacial warming, which also led to dramatic sea level rise and the breakup of Santarosae into its modern constituent islands. This marine transgression also shifted the coastline progressively closer to Daisy Cave during the terminal Pleistocene and the Holocene. By the Late Holocene, it appears that the environment of the Daisy Cave area had taken on an essentially modern character.

Data from Daisy Cave suggest that the Northern Channel Islands were settled by humans by at least 11,600 cal BP, a colonization that required relatively seaworthy watercraft and a substantial economic reliance on marine resources. Archaeological remains from the terminal Pleistocene cave occupation are limited because only small samples of these basal deposits have been excavated to date. Based on the available data, however, we attribute this occupation to maritime Paleoindian or "Paleocoastal" peoples (see Moratto 1984). This occupation appears to be as much as 1000 to 1500 yr earlier than several mainland Paleocoastal sites from the southern California coast, which are dated between *ca.* 9500 and 10,000 cal BP (Erlandson 1994). Thus, the terminal Pleistocene component at Daisy Cave currently represents the earliest known Paleocoastal occupation on the California coast. Currently, it seems most likely that these early maritime peoples were descended from even earlier Paleoindian peoples who appear to have left Clovis-like fluted points on the southern California coast (see Erlandson, Cooley and Carrico 1987) a millennium or more prior to the initial occupation of Daisy Cave. Nonetheless, the data from Daisy Cave provide additional evidence for the relatively early diversification of Paleoindian economies in western North America.

During the Early Holocene, Daisy Cave was occupied repeatedly by maritime peoples who relied heavily on marine resources like shellfish, fish and sea mammals. Preliminary dietary reconstructions suggest that fish made a significant contribution to the diet, at least while these people occupied Daisy Cave. Technological data support this assertion, as one of the most common types of finished artifact found in the Early Holocene levels is the small bone gorge, a straight bipoined hook. This contrasts with Early Holocene sites from the adjacent southern California mainland coast,

where fish bone and fishing tackle are relatively rare, and fishing seems to have played a much more supplemental subsistence role (Erlandson 1994: 263). By 8500 to 9000 cal BP, grinding stones (manos and metates) also make up a prominent part of many mainland artifact assemblages, suggesting that small seeds and other plant foods were important staples. At Daisy Cave, no unequivocal milling tools have been found in the lower archaeological levels. This is consistent with findings from other Early Holocene island sites and seems to be related to the relatively low diversity and productivity of edible seed-producing plants on the Channel Islands. Presently, Daisy Cave provides the only direct evidence for the types of basketry and other woven artifacts used by Early Holocene peoples along the Pacific Coast of North America (Connolly, Erlandson and Norris 1995).

Due to the limited amounts of cultural occupation and natural sedimentation that took place at Daisy Cave during the Middle Holocene, we know much less about the nature of cultural and environmental changes during this period. By the Late Holocene, however, Daisy Cave is once again the scene of relatively intensive and repeated human occupation. A detailed picture of Late Holocene cultural changes has yet to emerge from the site, but we suspect it is during this period that much of the cultural complexity and artistic elaboration typical of the ethnographic Chumash developed. At Daisy Cave, the evidence for these developments is seen in the presence of a fully diversified material culture that includes circular shell fish hooks, a wide range of beads and ornaments, a bird bone pan-pipe, a fragment of a redwood plank boat (*tomol*), asphaltum coated baskets and tarring pebbles, the bead-maker's kit containing standardized microblade drills and bead refuse typical of Chumash craft production, and a symmetrical and highly finished stone pestle. Although the archaeological record at Daisy Cave provides only a series of snapshots in time, the appearance of this elaborate and sophisticated Chumash material culture seems to be the culmination of over 11,000 yr in the evolution of local maritime societies.

Finally, on a more methodological note, the Daisy Cave chronology is marked by the relatively close correlation of  $^{14}\text{C}$  dates on marine shell and charcoal samples. Scientists working on the California coast have relied heavily on the dating of marine shells for decades, but there remains a pervasive oral history about the unsuitability of marine shell for  $^{14}\text{C}$  dating. Considering the numerous uncertainties and errors associated with comparing dates run by different laboratories on samples derived from different carbon reservoirs, our results provide a remarkably consistent chronology for the paleontological and archaeological deposits at Daisy Cave. In part, this is due to the finely stratified nature of the Daisy Cave deposits, the extremely good preservation of faunal and botanical remains at the site, the meticulous procedures used in recent excavations of the site, and the careful selection and pretreatment of  $^{14}\text{C}$  samples to avoid problems associated with contamination, the old wood effect, etc. Nonetheless, our study clearly demonstrates that marine shell samples can provide relatively high-resolution  $^{14}\text{C}$  dates for the construction of archaeological and paleontological chronologies. This is true despite the tentative identification of temporal fluctuations in upwelling and the regional reservoir effect for the Santa Barbara Channel region, based largely on the analysis of shell and charcoal samples from Daisy Cave (Kennett *et al.* 1996).

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## PARAMETERS OF A RADIOCARBON INSTALLATION

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**ABSTRACT.** I aim to define instrumental parameters of a radiocarbon laboratory installation whereby one can estimate its precision and a maximum age up to which its measuring results are reliable. The commonly accepted factor of merit (FM) relates the precision of measurement to Poisson statistics. Unlike the FM, the proposed parameters show the extent to which a  $^{14}\text{C}$  laboratory is affected by destabilizing factors that could cause additional measurement errors. Assuming that all destabilizing factors produce either a change in counting efficiency or additional fluctuations of the background counting rate, I have derived two parameters for consideration.

Several authors (Moljk, Drever and Curran 1957; Vinogradov *et al.* 1961) have suggested estimating the precision of a radiocarbon age with the help of the FM  $M = S/\sqrt{B}$  (where S and B stand for counting rates of standard and background, respectively). Using the FM, it is then possible to compare other  $^{14}\text{C}$  laboratories with respect to their precision and maximum age. Such a comparison is not appropriate in all cases. In some cases, investigators do not use the FM to estimate the precision of the measuring results (*e.g.*, Ryabinin 1978; Sementsov 1970). Sementsov (1970) evaluated the precision of measurement results by estimating them based on the experimental deviation of the counting rate of the standard  $\sigma_{\Sigma S}$ :

$$\sigma_{\Sigma S}^2 = [1/(n-1)] \cdot \sum_{i=1}^n (S_i - \bar{S})^2 \quad (1)$$

where

$S_i$  = a counting rate of a standard in  $i$ th test

$\bar{S} = \sum_{i=1}^n S_i/n$  = an average value of counting rates in  $n$  tests.

The FM was obtained under the assumption that measurement is subject to Poisson statistics. Undoubtedly, the process of a radioactivity and the formation of background signals obey Poisson statistics. Thus, according to the properties of the Poisson distribution, one can determine a counting rate deviation called the statistical deviation<sup>1</sup>  $\sigma_{st}$ .

$$\sigma_{st}^2 = S/t \quad (2)$$

where

$S$  = counting rate of standard

$t$  = counting time .

The statistical deviation is not an independent value because it is determined from the already known measured values  $S$  and  $t$ . If, under experimental conditions for the same sample, Equations (1) and (2) are in good agreement then one may claim that the  $^{14}\text{C}$  lab is optimal. Its instrumental errors are negligibly small and may be neglected. The agreement of the errors calculated according to Equations (1) and (2) is not always observed. Some investigators (Crevecoeur, Stricht and Capron 1959) have preferred to calculate errors according to Equation (1) in spite of the fact that it is nec-

<sup>1</sup>We use the word “deviation” hereafter implying “standard deviation” (1  $\sigma$ ). We do not use the term “standard deviation” so as not to confuse it with the term “standard”, which has another meaning.

essary to spend additional time making repeated tests before error calculation. Many investigators find that, according to their measurements, summary (experimental) errors exceed statistical errors considerably (Crevecoeur, Stricht and Capron 1959; Currie 1972; Zavel'sky 1972; Khait 1982). Since it is necessary to decrease the statistical deviation to an acceptable value, one must increase the counting time. For long counting times, it is very likely that the measurement process will be affected by external factors (*e.g.*, temperature changes, supply voltage fluctuations), the changes of which will also result in additional fluctuations in the counting rate and as a result, instrumental errors occur. Summary fluctuations contain both instrumental and statistical fluctuations. For future work, let us assume that all factors that influence the measurement process cause either a change in the count efficiency  $E$  or an additional counting rate deviation of the background. Accepting this assumption, the counting rate deviation for any  $^{14}\text{C}$  sample will be a function of the counting rate deviation of the standard and that of the background (Khait 1982), given as

$$\sigma_r^2 = [(R-B)^2/(S-B)^2] \cdot \sigma_s^2 + [1-(R-B)^2/(S-B)^2] \cdot \sigma_b^2 \quad (3)$$

where

- $R$  = counting rate of any sample
- $S$  = counting rate of a standard
- $B$  = counting rate of a background
- $\sigma_r$  = counting rate deviation of any sample
- $\sigma_s$  = counting rate deviation of a standard
- $\sigma_b$  = counting rate deviation of a background .

Comparison of the results calculated by Equation (1) and those calculated by Equation (3) shows good agreement (Khait 1982). The counting rate deviations in Equation (3) are instrumental deviations. A summary (experimental) deviation consists of the instrumental deviation  $\sigma$  and the statistical deviation  $\sigma_{st}$ . To the extent that these values are independent, then, in accordance with the laws of statistics and with previous studies (Crevecoeur, Stricht and Capron 1959; Currie 1972; Zavel'sky 1972), the following equations express the summary deviations  $\sigma_{\Sigma S}$ ,  $\sigma_{\Sigma r}$  and  $\sigma_{\Sigma b}$  of the three counting rates  $S$ ,  $R$  and  $B$  incorporating instrumental and statistical deviations

$$\sigma_{\Sigma s}^2 = \sigma_s^2 + S/t \quad (4.1)$$

$$\sigma_{\Sigma r}^2 = \sigma_r^2 + R/t \quad (4.2)$$

$$\sigma_{\Sigma b}^2 = \sigma_b^2 + B/t . \quad (4.3)$$

Equations (4.1)–(4.3) allow us to determine instrumental deviations (the first expression in the right side of the equations). However, for  $\sigma_r$ , the result given in Equation (3) is to be preferred to Equation (4.2). Equation (3) does not require repeated tests. As follows from Equations (4.1)–(4.3), the statistical deviations (the second expression in the right side of the equations) can be reduced by increasing counting time. Experience shows that with an increase of counting time it is impossible to reduce the summary (experimental) deviations  $\sigma_{\Sigma s}$ ,  $\sigma_{\Sigma r}$  and  $\sigma_{\Sigma b}$  (Currie 1972; Zavel'sky 1972). At present we cannot answer how instrumental deviation depends on counting time. To overcome this problem, we assume the same counting time for standard, sample and background is used and that they appear in the same order, *i.e.*, standard-background-sample. By following these rules, a possible dependence of instrumental deviation on counting time can be avoided. It is then possible to calculate  $\sigma_s$  and  $\sigma_b$  according to Equations (4.1) and (4.3) if we determine the preliminary summary deviations  $\sigma_{\Sigma s}$  and  $\sigma_{\Sigma b}$  according to Equation (1). It may happen that a statistical deviation is much less than an instrumental one. In that event, we can neglect the statistical deviation. In such a case, all further reasoning will also be true for the summary (experimental) deviation as well.

Equation (3) makes it possible to draw the following important conclusions:

1. It is possible to save counting time because there is no need to use the repeated tests to calculate the counting rate deviation for every sample.
2. It is necessary to repeat counting of the standard and background. These repeated measurements can be used in Equation (1) to calculate summary deviations.
3. It will suffice to measure the counting rate of any sample once and then calculate its deviation according to Equation (3). There is no need to use additional time for the measuring process.

The following expression allows us to calculate the deviation of a  $^{14}\text{C}$  age.

$$\sigma_T = \tau \cdot \sqrt{\sigma_s^2/(S-B)^2 + \sigma_r^2/(R-B) + \sigma_b^2 \cdot (S-R)^2/(S-B)^2 \cdot (R-B)^2} \quad (5)$$

where

$\tau$  = average time of life of  $^{14}\text{C}$ .

The investigators (Crevecoeur, Stricht and Capron 1959) used this equation for calculating the errors of a  $^{14}\text{C}$  age. As is apparent from Equation (3),  $\sigma_r$  is a value dependent on  $\sigma_s$  and  $\sigma_b$ . Substituting  $\sigma_r$  into Equation (5), according to Equation (3), we get the following transformed expression

$$\sigma_T = \sqrt{2} \cdot \tau \cdot \sqrt{\sigma_s^2/(S-B)^2 + \sigma_b^2(S-R)/(S-B) \cdot (R-B)^2} \quad (6)$$

Also

$$(R-B) = (S-B) \cdot \exp(-T/\tau) \quad (7.1)$$

$$R = (S-B) \cdot \exp(-T/\tau) + B \quad (7.2)$$

After substituting  $(R-B)$  and  $R$  of (7) into (6), the expression  $\sigma_T$  becomes

$$\sigma_T = \sqrt{2} \cdot \tau \cdot \sigma_s/(S-B) \cdot \sqrt{1 + \exp(T/\tau) \cdot [\exp(T/\tau) - 1] \cdot \sigma_b^2/\sigma_s^2} \quad (8)$$

Equation (8) is a function of the instrumental deviation of the sample's age. The behavior of Equation (8) is determined by its coefficients

$$P = \sigma_s/(S-B) \quad (9.1)$$

$$Q = \sigma_b/\sigma_s \quad (9.2)$$

Replacing  $\sigma_s/(S-B)$  and  $\sigma_b/\sigma_s$  with their symbols, Equation (8) becomes

$$\sigma_T = \sqrt{2} \tau \cdot P \sqrt{1 + \exp(T/\tau) \cdot [\exp(T/\tau) - 1] \cdot Q^2} \quad (10)$$

We may assume that the nature of the instrumental deviation of a  $^{14}\text{C}$  age for any  $^{14}\text{C}$  laboratory is given by Equation (10).

Two graphs in Figure 1 show the deviation as a function of age: graph 1 was carried over from a paper by Crevecoeur, Stricht and Capron (1959); graph 2 was plotted according to Equation (10) for the instrumental deviations of the liquid scintillation counter that was used in Moscow State University's Laboratory of Recent Sediments and Pleistocene Paleogeography in 1976.

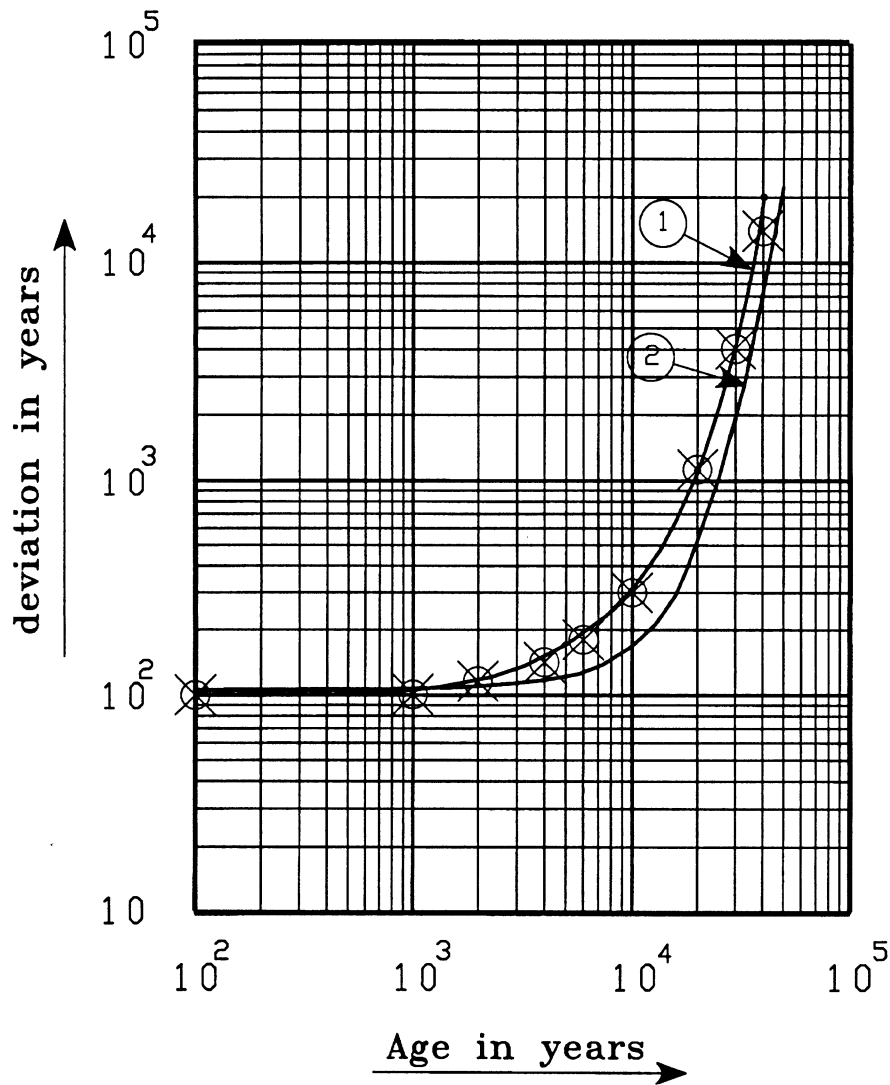


Fig. 1. Standard deviation as a function of age

From the similarity of the two graphs in Figure 1, it is very likely that our assumption mentioned above is correct. To verify this, the coefficients of the first graph were determined by solving two equations to give

$$1) T = 0; \delta_T = 100 \qquad 2) T = 30,000; \delta_T = 400.$$

Despite the fact that Figure 1 (Crevecoeur, Stricht and Capron 1959) does not contain the interval of ages from  $T=0$  to  $T=500$ , for the purpose of facilitating the calculation of its coefficients, we found it worthwhile to extend Figure 1 to intersect the error scale. In accordance with these chosen points, we have the following system of the two equations:

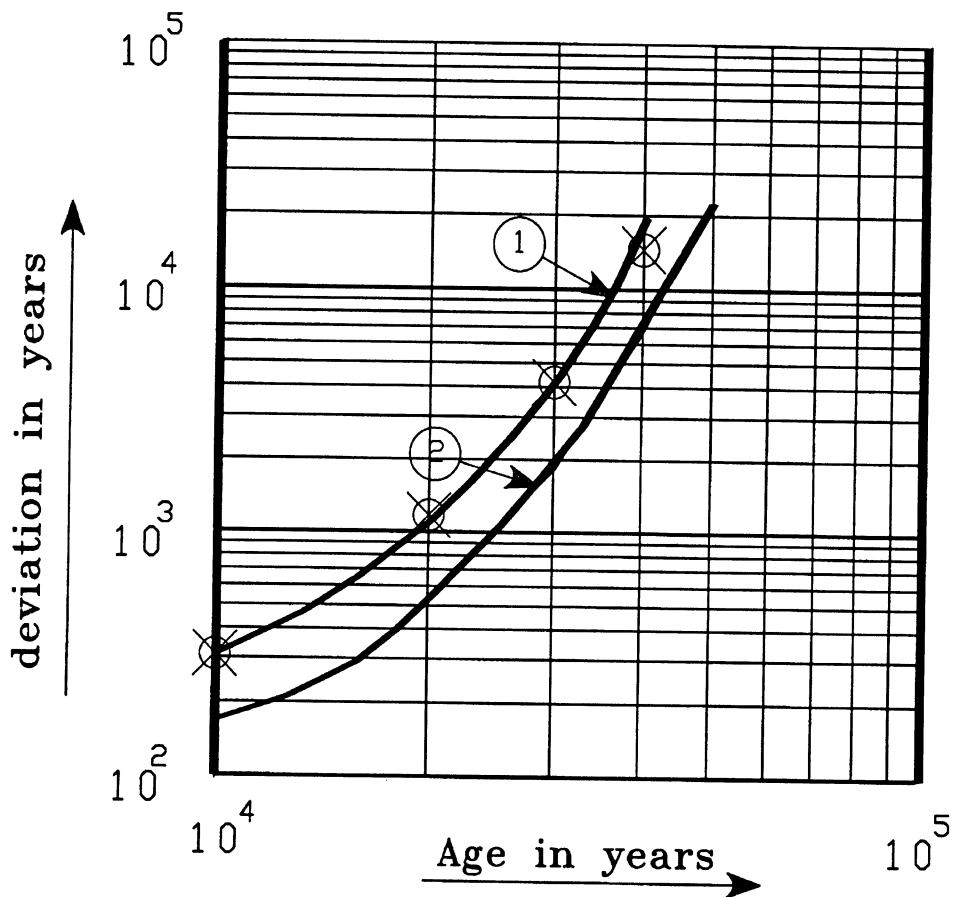


Fig. 2. Fragment of Fig. 1

$$\begin{aligned}
 100 &= \sqrt{2} \cdot \tau \cdot P \cdot \sqrt{1 + \exp(0/\tau) \cdot [\exp(0/\tau) - 1] \cdot Q^2} \\
 4000 &= \sqrt{2} \cdot \tau \cdot P \cdot \sqrt{1 + \exp(30,000/\tau) \cdot [\exp(30,000/\tau) - 1] \cdot Q^2} .
 \end{aligned}
 \tag{11}$$

The solution of the system gives the results  $P = 8.8 \cdot 10^{-3}$  and  $Q = 0.95$ .

Using these estimated coefficients, the expression for measuring the error on a  $^{14}\text{C}$  age for the gas counter presented by the authors (Crevecoeur, Stricht and Capron 1959) over the whole range of ages is

$$\sigma_T = 100 \cdot \sqrt{1 + \exp(T/\tau) \cdot [\exp(T/\tau) - 1] \cdot 0.9} .
 \tag{12}$$

From Equation (12), the locations of nine points were determined (Table 1) and marked by crosses in Figure 1. As we see, the locations of these points do not deviate considerably from graph 1. The two functions with three deviations  $\sigma_s$ ,  $\sigma_r$  and  $\sigma_b$  according to Equation (5) and also the paper (Crevecoeur, Stricht and Capron 1959) and with two deviations  $\sigma_s$  and  $\sigma_b$  according to Equation (12), as shown in Figure 1, coincide. This confirms the above supposition and also the fact that

instrumental errors prevailed in the results of the measurements presented in Crevecoeur, Stricht and Capron (1959). It is possible that there are instrumental errors in most  $^{14}\text{C}$  measurements.

Coefficients  $P$  and  $Q$  are the parameters of Equation (10) and are the proposed parameters for measuring counting performance of a  $^{14}\text{C}$  lab.

Assuming that  $T \ll \tau$ , we obtain an approximate result in accordance with (10), by neglecting the second expression under the root.

$$\sigma_T = \sqrt{2} \cdot P \cdot \tau . \quad (13)$$

Assuming that  $T \gg \tau$ , we get an approximate result for which the first expression under the root can be neglected

$$\sigma_T = \sqrt{2} \cdot P \cdot Q \cdot \tau \cdot \exp(T/\tau) . \quad (14)$$

Equation (13) does not contain the variable  $T$ . Consequently, instrumental deviation depends very little on the age for  $T \ll \tau$ . This is also seen in Figure 1 (for the first graph approximately up to 1000 yr, for the second graph up to 3500 yr). There is only one possibility to decrease instrumental errors over this range, namely lowering  $P$ .

Equation (14) for  $T \gg \tau$  shows an exponential dependence on the instrumental deviation of a  $^{14}\text{C}$  age. As shown in Equation (14), the product of coefficients  $P$  and  $Q$  characterizes the precision of age determination in this interval. According to Equation (9), this product is equal to

$$P \cdot Q = \sigma_b / (S - B) . \quad (15)$$

We can consider this product  $P \cdot Q$  as an individual parameter  $A$  (this is,  $A = P \cdot Q$ ) on which the precision of measurements in the range of  $T \gg \tau$  depends. In any case, as follows from Eqs. (9.1) and (15), to obtain a higher precision of measurement over the whole range of  $^{14}\text{C}$  ages that can be measured in a lab, the counting rate of the standard must be as high as possible, or, in other words, the *counting efficiency* must be as high as possible. To obtain a higher precision of measurements in the interval of young ages according to Equation (9.1), it is important to reduce the counting rate deviation of the standard to a minimum.

TABLE 1. Dependence of Standard Deviation of Age on Age

No.	1	2	3	4	5	6	7	8	9
Age ( $T$ ) in years	0	1000	2000	400	6000	10,000	20,000	30,000	40,000
Standard deviation ( $T$ ) in years	100	107	115	140	176	296	1100	3930	13,750

For a higher precision of measurements in the range of  $T \gg \tau$ , we must reduce the counting rate deviation of the background to a minimum according to Equation (15). The precision of measurement results depends on two instrumental parameters  $P$  and  $Q$  over the whole range of a  $^{14}\text{C}$  age. These parameters show the capability of a  $^{14}\text{C}$  laboratory to resist the effect of destabilizing factors on a measurement process. Comparison of these parameters of various  $^{14}\text{C}$  laboratories enables one to ascertain which of them can yield a higher precision of measurement in a particular part of the age scale.

The availability of three equations for the calculation of instrumental errors (Eqs. (10), (13) and (14)) allows us to divide the whole range of  $^{14}\text{C}$  measurements into three intervals: young, middle and ancient ages. Equation (10) is generalized. The calculations of instrumental errors by Equation (10) are always correct for the whole range of a  $^{14}\text{C}$  laboratory. We can consider Equation (10) as a function of the parameters  $P$  and  $Q$ . The influence of each of these parameters on the precision of measurements varies in different places on the age scale. Therefore, it is worth dividing the whole range of  $^{14}\text{C}$  ages into intervals. This permits us to estimate the precision of measurements with the help of approximating Equations (13) and (14) showing either a prevailing influence of one of these parameters ( $P$  or  $A$ ) their combined influence on the precision of measurement. So, the precision of measurement in the range of "young ages" depends solely on parameter  $P$ , but in the range of "ancient ages" it depends solely upon parameter  $A$ .

In other cases, the measurement error must be calculated according to Equation (10). To determine when Equations (10), (13) and (14) apply, we solve the following equations

$$\sqrt{1 + \exp(T/\tau) \cdot [\exp(T/\tau) - 1] \cdot Q^2} - 1 = \delta \quad (16)$$

$$\frac{\sqrt{1 + \exp(T/\tau) \cdot [\exp(T/\tau) - 1] \cdot Q^2} - Q \cdot \exp(T/\tau)}{Q \cdot \exp(T/\tau)} = \delta \quad (17)$$

where

$\delta$  = relative permissible difference.

The root of Equation (16) is a bound between the intervals of young and middle ages. We can find its value according to the expression

$$T_{ym} = \tau \cdot \ln(0.5 + \sqrt{0.25 + 2\delta/Q^2}). \quad (18)$$

The root of Equation (17) is a bond between the intervals of middle and ancient ages. We can find its value according to the following expression

$$T_{ma} = \tau \cdot \ln\left(-\frac{1}{4 \cdot \delta} + \sqrt{\frac{1}{16 \cdot \delta^2} + \frac{1}{2 \cdot \delta \cdot Q^2}}\right). \quad (19)$$

$T_{ym}$  is a bound up to which we can use Equation (13) for the calculation of instrumental errors (deviations)  $\delta_T$ .  $T_{ma}$  is a bound over which we can use Equation (14) for the calculation of instrumental errors. Then the difference in instrumental errors between each of the approximation equations and the generalized Equation (10) will not exceed a specified relative permissible difference  $\delta$ . Assuming the relative permissible difference to be 10% ( $\delta = 0.1$ ),  $T_{ym}$  for each of the above-mentioned counters will be equal:

$$\begin{aligned} T_{ym}(\text{gas counter}) &= 1397 \text{ yr} \\ T_{ym}(\text{scintillation counter}) &= 4042 \text{ yr} . \end{aligned}$$

Assuming the relative permissible difference to be 10% ( $\delta = 0.1$ ), the bound  $T_{ma}$  was determined only for the scintillation counter that was used in the  $^{14}\text{C}$  laboratory of Moscow University in 1976. It is equal to 9520 yr. It was not possible to determine  $T_{ma}$  for the gas counter used by Crevecoeur, Stricht and Capron (1959) because the relative permissible difference between the generalized



Equation (10) and the approximation Equation (14) could not be achieved at any of the points of the range of this gas counter.

As follows from Equation (13), the precision of measurements in the range of young ages depends almost entirely on the parameter  $P = \sigma_s/(S-B)$ . The lower the value for  $P$ , the smaller the error of a measurement result in this interval. The bound  $T_{ym}$  between the interval of young and middle ages determines the width of the interval of young ages ( $0 < T < T_{ym}$ ). Errors of measurement in this interval are minimum and almost constant. The higher the value for  $T_{ym}$ , the better the lab. The locations of the bounds  $T_{ym}$  in the graphs for two counters of Figure 1 are also in agreement with the fact that instrumental errors hardly ever change over the range of young ages. As follows from Equation (18), the parameter  $Q = \sigma_b/\sigma_s$  influences the location of  $T_{ym}$  on the age scale. Thus we can evaluate all the capabilities of a  $^{14}\text{C}$  laboratory using its two parameters  $P$  and  $Q$ . The growth of measurement errors becomes appreciable for  $T > T_{ym}$ . This fact can also be observed on the graphs of Figure 1. The precision of measurements in the interval of middle ages ( $T_{ym} < T < T_{ma}$ ), as follows from the generalized Equation (10), depends on the two parameters  $P$  and  $Q$ . As the age  $T$  grows, the influence of the parameter  $Q$  on the precision of dating grows too. As Equation (19) shows, the location of the bound between middle and ancient ages also depends on the parameter  $Q$ .

As follows from Equation (14), the instrumental error  $\sigma_T$  is an exponential function of the age  $T$  for  $T > T_{ma}$ . The growth of measurement errors in this interval is quicker than over the central range of ages. Therefore, the higher the bound between middle and ancient ages, the better the lab. The product of the parameters  $P$  and  $Q$  determines the precision of dating in the interval of ancient ages according to Equation (14).

Still missing from our list of parameters is the maximum age  $T_{max}$ . It is possible to deduce this important additional parameter as a function of the parameters  $P$  and  $Q$ . However, the maximum age is the subject of separate investigation and will be dealt with in future research.

Table 2 presents the parameters and the equations for their calculation. The results of the calculations of some of these parameters are also presented in this table for two counters under discussion. We have used equations by Moljk, Drever and Curran (1957) for the calculation of the maximum age. Crevecoeur, Stricht and Capron (1959) proposed that the maximum age should be determined if there are summary (experimental) errors of measurements. Moljk, Drever and Curran (1957) suggest determining the maximum age as a function of the FM. Table 2 lists the results of the calculations of  $T_{max}$  performed in both of these ways for the scintillation counter used in 1976 at Moscow University's  $^{14}\text{C}$  laboratory. The counting efficiency  $E$  is also included in Table 2.

It is possible to express the parameters  $P$  and  $A$  in terms of counting efficiency

$$P = \sigma_s/E \cdot d \cdot W \quad (20)$$

$$A = \sigma_b/E \cdot d \cdot W \quad (21)$$

where

$d$  = specific radioactivity

$W$  = weight of a sample .

It is possible to determine the weight of a sample to obtain the necessary values for  $P$  and  $A$ , ensuring the required precision of measurements.

TABLE 2. Parameters of a Radiocarbon Installation

No.	Parameter symbol	Parameter name	Calculation formula	Type of counter	
				Gas	Scintillation
				University of Louvain	Moscow University
1	$E$ (%)	Counting efficiency	$\frac{S-B}{d \cdot w} \cdot 100$	--	27
2	$M$	FM	$S/\sqrt{B}$	--	28
3	$P$		$\sigma_s/(S-B)$	$8.8 \times 10^{-3}$	$9.33 \times 10^{-3}$
4	$Q$		$\sigma_s/\sigma_B$	0.95	0.42
5	$A$		$P \cdot Q = \sigma_B/(S-B)$	$8.36 \times 10^{-3}$	$3.92 \times 10^{-3}$
6	$T_{ym}$ (yr)	Bound between intervals of young and middle ages	$\tau \cdot \ln\left(0.5 + \sqrt{0.25 + \frac{2 \cdot \delta}{Q^2}}\right)$	1397	4042
7	$T_{ma}$ (yr)	Bound between intervals of middle and ancient ages	$\tau \cdot \ln\left(-\frac{1}{4 \cdot \delta} + \sqrt{\frac{1}{16 \cdot \delta^2} + \frac{1}{2 \cdot \delta \cdot Q^2}}\right)$	--	9520
8	$T_{max}$ (yr)	Maximum age	$\tau \cdot \ln \frac{S-B}{\sqrt{2} \cdot \delta_B}$	--	43,360
			$\tau \cdot \ln\left(\frac{1}{6} M \sqrt{t}\right)$	--	40,500

## CONCLUSION

We have obtained a system of instrumental parameters for a  $^{14}\text{C}$  lab as a result of an investigation of instrumental errors of measurements. Two of these parameters,  $P$  and  $A$ , are fundamental. They are fundamental because they will suffice to determine completely the precision of measurements over the whole age range capability of a  $^{14}\text{C}$  laboratory. We can replace the parameter  $Q$  with the parameter  $A$  according to the formula  $A=PQ$ . Then the two parameters  $P$  and  $A$  will be fundamental. Additional parameters, such as the bounds of age ranges and the maximum age, enable us to evaluate quickly the performance of a  $^{14}\text{C}$  lab. The FM is also a fundamental parameter as the precision of measurements is directly dependent upon it. This parameter is suitable for an ideal case only, that is, for measurements without fluctuations in counting efficiency and without additional fluctuations of background, and therefore I propose that  $P$  and  $A$  should also be considered fundamental.

The rest of the parameters are additional, being related to the fundamental ones in a certain way. The additional parameters enable us to quickly evaluate the performance of a  $^{14}\text{C}$  lab, for example, such parameters as the bounds of intervals and the maximum age. From the above reasoning, the FM is a fundamental parameter as the precision of measurements is directly dependent upon it. It is neces-

sary to allow for an essential precondition of obtaining this FM. This parameter is suitable for an ideal case only, that is, for measurements without fluctuations of count efficiency and without additional fluctuations of the background.

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**NOTE**

**<sup>14</sup>C DATING OF AN ISRAELITE BIBLICAL SITE AT KUNTILLET AJRUD (HORVAT TEMAN): CORRECTION, EXTENSION AND IMPROVED AGE ESTIMATE**

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We have found three printing errors in Table 2 of our paper <sup>14</sup>C Dating of an Israelite Biblical Site at Kuntillet Ajrud (Horvat Teman) (Meshel, Carmi and Segal 1995). The errors are in the <sup>14</sup>C ages of samples 1830, 1831 and 1832. The corrected table with three additional dates is given below. The three additional samples are as follows:

RT 2095: A bunch of human hairs from Locus 41, Basket 352

RT 2096: Fine woven fabric from Locus 104, Baskets 30–31

RT 2097: Straw from Locus 256, Basket 1385.

The hair sample is evidently out of context. On the <sup>14</sup>C ages of all the other samples, we applied the bootstrap technique (Aczel 1995) to improve the age estimate of the site. The estimate after 1000 bootstraps is 2549–2607 yr BP at a confidence level of 68%, and the corresponding calibration is 801–770 BC at 100% confidence (Stuiver and Reimer 1993). By applying the bootstrap technique we succeeded in reducing the calibrated age range from 70 to 31 yr.

TABLE 1. <sup>14</sup>C Dates from Kuntillet Ajrud

Sample (RT-no.)	Δ <sup>14</sup> C (‰)	δ <sup>13</sup> C (‰)	<sup>14</sup> C age (yr BP)
1826	-250.6 ± 3.7	-23.2	2275 ± 40
1827	-283.4 ± 4.2	-20.9	2635 ± 45
1827	-290.0 ± 5.0	-20.9	2710 ± 60
1828	-263.3 ± 4.0	-21*	2410 ± 45
1829	-284.4 ± 3.9	-21.4	2645 ± 45
1829	-285.5 ± 5.0	-21.4	2660 ± 50
1830	-264.5 ± 6.4	-21.02	2425 ± 70
1831	-276.2 ± 4.0	-21.42	2555 ± 45
1832	-289.0 ± 4.3	-21.52	2695 ± 50
1833	-280.3 ± 4.2	-21.8	2600 ± 45
1834	-271.9 ± 5.3	-21.0	2505 ± 60
1835	-283.8 ± 4.3	-22.4	2640 ± 50
1836	-269.5 ± 4.4	-17.3	2480 ± 50
2094	-285.8 ± 4.2	-22.8	2660 ± 45
2095	-24.6 ± 7.9	-15.23	155 ± 65
2096	-271.0 ± 3.6	-21.4	2495 ± 40
2097	-297.0 ± 4.9	-21	2785 ± 55

\*Estimated

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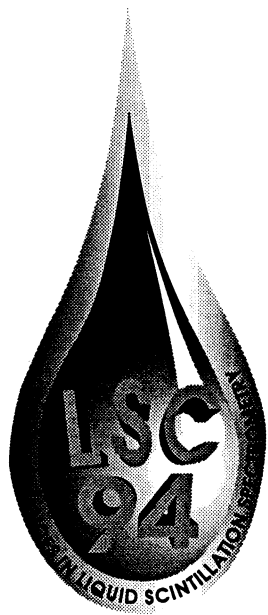
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*Half-life of  $^{14}\text{C}$ .* In accordance with the decision of the Fifth Radiocarbon Dating Conference, Cambridge, England, 1962, all dates published in this volume (as in previous volumes) are based on the Libby value, 5568 yr, for the half-life. This decision was reaffirmed at the 11th International Radiocarbon Conference in Seattle, Washington, 1982. Because of various uncertainties, when  $^{14}\text{C}$  measurements are expressed as dates in years BP, the accuracy of the dates is limited, and refinements that take some but not all uncertainties into account may be misleading. The mean of three recent determinations of the half-life,  $5730 \pm 40$  yr, (*Nature*, 1962, Vol. 195, No. 4845, p. 984), is regarded as the best value presently available. Published dates in years BP can be converted to this basis by multiplying them by 1.03.

*AD/BC Dates.* In accordance with the decision of the Ninth International Radiocarbon Conference, Los Angeles and San Diego, California, 1976, the designation of AD/BC, obtained by subtracting AD 1950 from conventional BP determinations is discontinued in *RADIOCARBON*. Authors or submitters may include calendar estimates as a comment, and report these estimates as cal AD/BC, citing the specific calibration curve used to obtain the estimate. Calibrated dates should be reported as "cal BP" or "cal AD/BC" according to the consensus of the Twelfth International Radiocarbon Conference, Trondheim, Norway, 1985.

*Measuring  $^{14}\text{C}$ .* In Volume 3, 1961, we endorsed the notation  $\Delta$ , (Lamont VIII, 1961), for geochemical measurements of  $^{14}\text{C}$  activity, corrected for isotopic fractionation in samples and in the NBS oxalic-acid standard. The value of  $\delta^{14}\text{C}$  that entered the calculation of  $\Delta$  was defined by reference to Lamont VI, 1959, and was corrected for age. This fact has been lost sight of, by editors as well as by authors, and recent papers have used  $\delta^{14}\text{C}$  as the observed deviation from the standard. At the New Zealand Radiocarbon Dating Conference it was recommended to use  $\delta^{14}\text{C}$  only for age-corrected samples. Without an age correction, the value should then be reported as percent of modern relative to 0.95 NBS oxalic acid (Proceedings of the 8th Conference on Radiocarbon Dating, Wellington, New Zealand, 1972). The Ninth International Radiocarbon Conference, Los Angeles and San Diego, California, 1976, recommended that the reference standard, 0.95 NBS oxalic acid activity, be normalized to  $\delta^{13}\text{C} = -19\text{‰}$ .

In several fields, however, age corrections are not possible.  $\delta^{14}\text{C}$  and  $\Delta$ , uncorrected for age, have been used extensively in oceanography, and are an integral part of models and theories. Thus, for the present, we continue the editorial policy of using  $\Delta$  notations for samples not corrected for age.

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