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ABSTRACTS OF THE 14TH INTERNATIONAL RADIOCARBON CONFERENCE

HIGH-PERFORMANCE ¹⁴C GAS-PROPORTIONAL COUNTING SYSTEM APPLYING PULSE-SHAPE DISCRIMINATION

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Experiments were started in 1984 at the Geological Survey of Finland (GSF) with a new ¹⁴C dating system that consists of a passive shield of ~ 90-year-old iron, a liquid-scintillation anticoincidence guard and three CO₂ proportional counters of conventional size. The counting room is an air-conditioned Faraday cage constructed of limestone concrete.

The application of pulse-shape discrimination greatly improves the precision of our radiocarbon analyses by distinguishing sample counts and background counts. The purity of the gas is monitored by recording the cosmic muon pulse-height spectrum. At present, the dating limit of a two-day measurement in the largest, 1.735 L counter, is 57,000 years.

Brief details are given of the construction and commissioning of the dating system.

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INTERVAL ESTIMATION OF THE FLORUIT OF AN ARCHAEOLOGICAL PHENOMENON: A GENERAL METHOD AND APPROXIMATION FOR SUMMARIZING A GROUP OF ¹⁴C DATES

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It became apparent at the last International ¹⁴C Conference that no clear definition of the aims of summarizing a set of ¹⁴C dates was available. The present paper provides the formation and solution of a specific aim through the definition and interval estimation of a floruit of ¹⁴C ages. This is achieved through a method of non-parametric density estimation carried out on all possible historical dates which may have generated each observed ¹⁴C date. The method takes into account not only the error on the ¹⁴C date itself, but also the error in the estimated calibration curve and the slope of the curve at such historical ages. As the method is computationally intensive, the authors also consider some simple approximations to the production of such interval estimates, which can be evaluated through the use of paper, pencil and ruler.

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RADIOCARBON DATING OF BONE OSTEOCALCIN: REFINEMENTS IN PROCEDURES FOR ISOLATING A PURE FORM OF NON-COLLAGEN PROTEIN

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Widely accepted methods used to chemically pretreat bone samples can, in some cases, yield unreliable ¹⁴C age determinations when applied to fossil bones, characterized by low to trace amounts of organic residues that do not exhibit a collagen-like amino acid pattern. The lack of effective pretreatment procedures for seriously biochemically degraded bone limits the critical application of ¹⁴C dating for this sample type – effectively eliminating the possibility of obtaining reliable ¹⁴C measurements on bone samples from most tropical environments older than a few thousand years. These environments include regions from which some of the most interesting late Pleistocene hominid fossils derive.

¹⁴C determinations have been obtained on osteocalcin, a non-collagen protein in bone. It has been proposed that osteocalcin retains isotopic integrity even in samples where collagen has been denatured by natural hydrolysis and other diagenetic processes, and where collagen and collagenderived products are retained in only low and trace amounts. The basis of the suggested isotopic integrity of osteocalcin is that it binds to hydroxyapatite, the major mineral component of bone, and, in this bound form, it is biogeochemically stable. In addition, it appears that the distribution of osteocalcin in nature is limited to vertebrates, and has not been detected in arthropod exoskeletons or in microorganisms.

Concerns have been expressed about the purity of the osteocalcin fraction isolated using published procedures and the degree to which organics other than osteocalcin might bind to hydroxyapatite. We will report on ¹⁴C data obtained on osteocalcin bone fractions by electrophoresis, immunological testing, amino acid residues and C/N ratios.

RADIOCARBON DATING OF PALEOSEISMICITY ALONG AN EARTHQUAKE FAULT IN SOUTHERN ITALY

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On November 23, 1980, a major earthquake ($M_5 = 6.9$) struck a large area of the southern Apennines (Campania and Lucania regions, southern Italy). Such a seismic event, the largest in Italy over the last 80 years, almost completely destroyed 15 villages and caused extensive damage to other villages and towns including Naples. The quake produced the first well-documented example in Italy of surface dislocation, represented by a fault scarp 38 km long. To assess the seismological hazard for the area, a study, including radiocarbon dating of organic materials from layers displaced by paleoseismic events, has been undertaken. Peat and charred wood samples were collected on the walls of two trenches excavated across the 1980 fault with a mechanical

¹Department of Physics, University of Rome, 00185 Rome, Italy ²Department of Earth Sciences, University of Rome 00185 Rome, Italy backhoe at Piano di Pecore di Colliano, Salerno, southern Italy (44°44'N, 15°22'E). The trenching site is in a little intermontane basin, where a pond recurrently formed because of partial damming of the seasonal stream by faulting activity. Here, the sedimentary suite is faulted and warped by five quakes (including that of 1980), which were comparable in terms of vertical throw and deformation pattern. Chronological data for pre-1980 events, coupled with detailed stratigraphic analysis, yielded a slip rate of 0.4 mm/yr and a recurrence rate of 1700 yr.

RADIOCARBON RESULTS FOR THE BRITISH BEAKERS

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The beginning of the Bronze Age in the British Isles has traditionally been marked by the appearance, in the archaeological record, of Beaker assemblages, mainly characterized by the Beaker pottery form, itself. Ceramic typologies based on this style, undoubtedly continental in origin, have been used both for relative dating and as evidence of the social and economic developments of the period.

Systematic radiocarbon dating has been attempted for the continental European Beaker material (Lanting, Mook & van der Waals 1973), but no such program has been carried out on British material. An examination of the existing radiocarbon results for the British Beakers showed many to be flawed in some way, particularly in the use of materials, such as mature wood, where there is no *a priori* reason for assuming a direct relationship between sample death and context. An attempt has been made at the British Museum to test the validity of archaeologically derived chronologies for the Beaker pottery of the British Isles. This involved analysis of a group of carefully selected human bone from Beaker burials, where there is a known direct association between ceramic usage and the cessation of carbon exchange. Twenty such samples have been identified and measured. The results presented here, combined with the 15 previously produced, supposedly reliable determinations, show no relationship between pottery style and calendar date of deposition.

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¹⁴C ACTIVITY AND ³He CONTENT IN INTERSTITIAL WATERS FROM CORAL REEF: EVIDENCE FOR THE ENDO-UPWELLING CONCEPT

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In the central desert region of the tropical ocean, atolls constitute oases. The classical model of atoll functioning, based on horizontal exchanges between lagoon and oligotrophic oceanic surface water, is unable to balance nutrient budgets to account for high organic production. The geothermal endo-upwelling concept (Rougerie & Wauthy 1986) is based on a vertical ascent of deep-and rich-nutrient oceanic water, driven by geothermal heat flow through the atoll internal

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structure, supplying the nutrients to the reef building. The intermediate deep water in the Pacific is ¹⁴C depleted, compared to the surface water, and also records the ³He anomaly resulting from hydrothermal activity. These properties of intermediate waters may be recorded in the interstitial waters in the coral reef. We use the agreement between these two parameters to check the validity of the concept of geothermal endo-upwelling.

REFERENCE

EVALUATING DISSOLVED INORGANIC CARBON CYCLING IN A FORESTED LAKE WATERSHED USING CARBON ISOTOPES

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Dissolved inorganic carbon (DIC) is an important component of the alkalinity balance on freshwater systems. A comprehensive evaluation of DIC cycling is essential to predict the impact of anthropogenic activities, such as acid rain, on natural systems. In this paper, we will discuss isotopic and chemical data of soil CO_2 and DIC (groundwater, streams and lake water) samples taken in a forested lake watershed on the Precambrian Shield, Canada. Some of the main results of this ongoing research can be summarized as follows:

Soil CO₂ profiles show higher CO₂ concentrations during the summer months. The average δ^{13} C for the soil CO₂ is -22.0%; groundwaters are characterized by a mean δ^{13} C of -23%. This indicates that weathering of silicates is the main process responsible for the generation of alkalinity in this watershed. Groundwater in the middle part of the basin is characterized by ¹⁴C activities in the range of 116 pMC to 120 pMC. These values are close to the atmospheric CO₂ during 1990. Lower ¹⁴C activity (112 pMC) is observed in groundwater in the discharge areas. This suggests (assuming piston flow) that the mean residence time of groundwater from recharge to discharge areas is in the order of 30 years in this basin. Stream DIC in the middle part of the basin presents similar ¹⁴C activities to those of groundwater; however, lower ¹⁴C activities are observed in streams discharging into the lake. These data suggest the contribution of older water to the stream along the stream course. Carbon isotope and chemical data on lake water particulate organic carbon (POC) and dissolved organic carbon (DOC) will also be discussed.

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INVESTIGATING CARBON SOURCES FOR METHANE AND DISSOLVED ORGANIC CARBON IN A REGIONAL CONFINED AQUIFER USING ¹⁴C

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Environmental isotopes (¹³C, ²H) have been widely used to investigate the origin of methane in subsurface systems. However, few investigations have used radiocarbon dating as a means of identifying carbon sources for methane (CH₄) and dissolved organic carbon (DOC) in groundwater. In this paper, we discuss ¹⁴C data for CH₄ and DOC from groundwater samples taken from a regional Quaternary-age, confined aquifer located in southern Ontario. The potential carbon

Rougerie, F and Wauthy, B 1986 Le concept d'endo-upwelling dans le fonctionnement des atolls-oasis. Oceanologica Acta 9: 133-148.

sources for methane in this aquifer are DOC, organic matter present in the aquifer materials and kerogen present in the bedrock. DOC in groundwater is mainly associated with the soil zone, in the recharge areas.

A comparison of the regional distribution of CH_4 and DOC in the aquifer with the nature of the underlying Ordovician formations indicate that bedrock lithology does not influence the distribution of CH_4 and DOC in the aquifer. Stable isotopic data (^{13}C , ^{2}H) indicate the predominant pathway of methane formation is microbial reduction of CO_2 . ^{14}C ages for CH_4 in the aquifer and underlying bedrock range between 30,000 and 40,000 BP, thereby eliminating the possibility of a significant Paleozoic bedrock carbon source for the CH_4 . The bedrocks are reservoirs for natural gas (thermocatalic methane) in other parts of southern Ontario.

DOC concentrations range between 1 to 17 mg C/L. Typical DOC concentration in groundwater is around 3 ppm. A strong linear correlation was observed between DOC and CH_4 concentration. These results suggest that DOC and CH_4 are associated with a common organic in the aquifer sediments. This carbon source is likely buried Wisconsinan-age peat deposits, confirmed in several boreholes. ¹⁴C ages of fulvic DOC that are similar to ¹⁴C ages of CH_4 support this hypothesis. In some wells, however, DOC ¹⁴C ages are significantly younger than CH_4 , suggesting this gas is migrating from production zones to other parts of the aquifer.

LATE PLEISTOCENE GEOCHRONOLOGY OF EUROPEAN USSR

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The proposed chronology of the Mikulino (Riss-Würm) interglacial and the Valdai (Würm) glacial epoch is based on ²³⁰Th/²³⁴U and ¹⁴C dates of many standard sections. The ²³⁰Th/²³⁴U dating of well-preserved shell samples indicates that Boreal (Eem) transgression sediments of the Barents and the White Seas show an age within the ranges of 86,000 to 114,000 years. The shells of the Kozangat transgression of the Black Sea yield an age about 95,000 to 115,000 years. Taking into account that ²³⁰Th/²³⁴U ages are reduced for shells, the dates are compatible enough with the chronology of deep-sea sediments (Substage 5e, 128,000-116,000 yrs ago). Mollusk shells of the Krotkov Cape liman clays (Taman Peninsula) date to about 95,000-100,000 years. The pollen spectrum of the clays correspond to tajga forest. This climatic event is concordant with an abrupt climatic deterioration that occurred 116,000-110,000 years ago (Würm I). The sediments of Early Valdai interstadials, corresponding to two Early Würm interstadials, give ${}^{14}C$ ages of \ge 45,000-56,000 years. The sediments of the Early Glacial stage (Shestikhino climatic deterioration) show ¹⁴C ages of \geq 47,000-49,000 years. The depositions of the Middle Valdai interstadial complex were studied from western Belorussia to the foothills of the Ural Mountains and from Abchazia to the coast of the Barents Sea (31 sections). ¹⁴C ranges of these sediments are from 52,000 ± 1690 to 25,440 ± 270. Many ¹⁴C dates of Late Valdai age were obtained for sediments overlain by glacial layers in marginal zones of the maximum glaciation stage from western Belorussia to the Pechora basin. These data indicate the Late Valdai age of the maximum glacial stage. The deglaciation period involves several climatic amelioration and deterioration phases.

RADIOCARBON DATING AND THE ANCIENT SETTLEMENTS IN THE SOUTHERN NEGEV AND SINAI DESERTS

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Until recently, the common scholarly concept was that the desert was generally vacant of human presence, and only seldom penetrated and settled by people from the neighboring fertile countries. Desert sites were usually regarded as temporary and short-lived. Throughout the last decade, this view is undergoing drastic change because of two factors. One was the withdrawal of the Israeli Army from the Sinai into the Negev, which resulted in intensive archaeological research. The second was the use of ¹⁴C for dating the sites. At present, there are 72 dates from these areas, 54 are from the 7th to 2nd millennia BC. For the first time, remains were dated to periods hitherto thought to be populated; and from several dwelling, cult and burial sites, we have evidence of constant use during a few millennia. Almost a complete sequence of settlement in the desert comes to light from the Neolithic period to the present. It is now evident that the desert was never abandoned, but only knew fluctuation in activity. The desert population maintained a great continuity in ethnicity, culture and religion, unlike the frequent cultural changes of the settled countries caused by military traumas and political events.

RADIOCARBON VARIATIONS FROM TASMANIAN CONIFERS: FIRST RESULTS FROM LATE PLEISTOCENE AND HOLOCENE LOGS

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Dendrochronological studies have been carried out on two Tasmanian conifer species in the Stanley River area of western Tasmania. The chronology extends back to AD 800 for Huon pine (*Lagarostrobos franklinii*) and AD 1400 for celery-top pine (*Phyllocladus aspleniifolius*). Apart from living or recently felled trees, sections have been taken from 58 logs preserved in floodplain sediments. Two of these logs have late Pleistocene ages, centered around 13,000 and 12,700 BP. There are four logs between 8000 and 9000 years old, and one centered at 7300 BP. The remaining logs have various ages between 6200 BP and the present day.

Radiocarbon measurements have been carried out on decadal samples from the two late Pleistocene logs, providing short (260-yr) segments of a calibration curve when plotted against individual ring numbers. The older log shows strong and rapid variations, whereas radiocarbon variations are quieter in the log centered at 12,700 BP.

Decadal measurements on the 7300-year-old log have been wiggle-matched with radiocarbon calibration curves from bristlecone pine and south German oak. Detailed intercomparisons among the different laboratories are planned, and should permit a preliminary estimate of the offset between the northern and southern hemispheres during early and mid-Holocene times.

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²³⁰Th/²³⁴U AND ¹⁴C AGES OBTAINED BY MASS SPECTROMETRY ON CORALS FROM MURUROA ATOLL, FRENCH POLYNESIA

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We have measured the ages of coral specimens collected from offshore the atoll of Mururoa, French Polynesia. The samples were collected by means of a deviated drill (30°), at about 170 m below the present sea level. Two geochronological techniques were used: ²³⁰Th/²³⁴U by thermal ionization mass spectrometry (TIMS; Lamont-Doherty) and ¹⁴C by accelerator mass spectrometry (AMS; Tandetron of Gif-sur-Yvette). The results (Table 1) show significant differences between the two geochronometers corresponding to Δ^{14} C values of 280 ± 30‰ and 340 ± 40‰ (2 σ) at about 15,600 and 17,600 Th-yr BP, respectively. These values are in reasonable agreement with those published by Bard, Hamelin and Fairbanks (1990) and Bard *et al* (1990a) and in excellent concordance with the redetermination of some of the Barbados ¹⁴C ages by AMS (Bard *et al* 1990b). This new comparative study of U-series and radiocarbon ages demonstrates that the large discrepancies first evidenced in Barbados are not the results of local alteration processes and that reef corals may be used successfully to calibrate the radiocarbon time scale.

Sample	¹⁴ C age (yr BP $\pm 2\sigma$)	Th/U age (yr BP $\pm 2\sigma$)
315	$13,160 \pm 280$ $13,160 \pm 300$	15,660 ± 90
315		$15,550 \pm 80$
315		$15,550 \pm 100$ $15,550 \pm 100$
313	$14,770 \pm 340$ $14,690 \pm 320$	17,650 ± 130
313		$17,630 \pm 100$ 17,630 ± 100
313	,	$17,500 \pm 100$ $17,500 \pm 120$

TABLE 1. Age results obtained by AMS and TIMS. The replicates were obtained on different aliquots of the same powdered samples. The ¹⁴C ages are conventional ages corrected for a reservoir age of 400 years.

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METHODS FOR DATING OF ORIENTAL TEXTILES BY ACCELERATOR MASS SPECTROMETRY

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In this paper, we will report on a series of measurements of ¹⁴C age made on small samples of Oriental textile using AMS. Many of these samples have been subjected to serious contamination over years of use and storage. Pretreatment of textiles is important to remove contaminants which can greatly affect the measured age.

Radiocarbon ages on textiles subjected to different sample pretreatments will be compared to the art-historical estimate of age. Treatments such as acid-base-acid cleaning, successive solvent extractions and extractions of amino acids will be discussed. The reliability of the various fractions dated will also be discussed. Some examples of Asian textiles, which have been verified using AMS ¹⁴C, will also be presented.

³⁶CI IN THE SNAKE RIVER PLAIN AQUIFER: ORIGIN AND IMPLICATIONS

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Between 1952 and 1984, low-level radioactive waste was injected into the Snake River Plain Aquifer at the Idaho National Engineering Laboratory at Idaho Falls, Idaho. These wastes were generated at the nuclear fuel reprocessing facility on the site. Recent measurements of ³⁶Cl in monitoring and production wells down-gradient from the injection well have detected high atom concentrations of this radionuclide from the point of implacement to the reservation boundary. The source of the ³⁶Cl is believed to originate, principally, by activation of stable chloride associated with fuel rods while in the reactors. Comparisons are made between tritium and ³⁶Cl concentrations in aquifer water, and the utility of using ³⁶Cl as a future tracer of subsurface water dynamics at the site is discussed.

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ABSOLUTE MINIMUM AGE OF THE LATE GLACIAL - HOLOCENE TRANSITION BY RADIOCARBON CALIBRATION AND STABLE ISOTOPE ANALYSES OF A 1477-YEAR GERMAN PINE DENDROCHRONOLOGY

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The German Preboreal pine dendrochronology has recently been extended to the Younger Dryas. This became obvious by stable isotope analyses (²H, ¹³C) of the early part of the tree-ring series. We consider the rapid and significant increase of deuterium and ¹³C within the sequence to be related with the climatic change at the end of the Late Glacial. High-precision ¹⁴C measurements of the corresponding part of the pine tree rings show a plateau of constant ¹⁴C ages of 10,000 BP

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THE USE OF NATURAL ¹⁴C AND ¹³C IN SOILS FOR STUDIES ON GLOBAL CLIMATIC CHANGE

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The amount of the "greenhouse" gases, CO_2 and CH_4 , produced by the decomposition of organic matter in terrestrial and paddy soils and emitted from the soil, can be estimated from depth profiles of the $\delta^{13}C$ value and ¹⁴C activity of soil organic matter, using simple mathematical models. By sampling the soil as thin layers, we can also determine whether the non-gaseous decomposition products are fixed to clay or transported downwards to the groundwater table. The influence of temperature and soil moisture regime on the carbon isotope depth profiles is shown by comparing a range of investigated soils from various climatic regions. A change of vegetation from C_3 to C_4 plants, which might take place during a predicted temperature rise in certain areas, thereby influencing the carbon balance, can be clearly detected by $\delta^{13}C$ depth profiles.

A HIGH RESOLUTION ¹⁰Be RECORD IN POLAR ICE

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A project has been started to establish a ¹⁰Be record in Greenland ice with a time resolution of one year. The upper part of a 300 m ice core from Dye 3, Greenland, has been used for this study. The time scale in this core has been determined using acidity peaks caused by volcanic eruptions and annual variations of the H_2O_2 concentration.

The main results obtained so far are:

- 1. The comparson of the ¹⁰Be record with solar activity as determined by sun spots, aurorae and Aa-index reveal a good correlation regarding the 11-year Schwabe cycle as well as the longer-term trends.
- 2. During the Maunder minimum period (AD 1645-1715), the ¹⁰Be concentration is higher by about 50%.
- 3. The 11-year Schwabe cycle is also present during the Maunder minimum, in spite of the missing sun spots.

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ANTHROPOGENIC RADIOCARBON IN THE IRISH SEA AND SCOTTISH COASTAL WATERS

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The anthropogenic component of environmental radiocarbon is important in two ways:

- 1. It may deliver a major radiation dose to both local and global populations
- 2. Excess ¹⁴C near discharge locations may provide tracer information on the physicochemical forms and the partitioning between ¹⁴C and stable carbon.

The Irish Sea and Scottish coastal waters receive significant inputs of ¹⁴C from discharges by Amersham International plc into Cardiff Bay and British Nuclear Fuels plc, who discharge directly into the Irish Sea from their Sellafield fuel reprocessing plant. We have studied the distribution of ¹⁴C activities in a range of sample types throughout this region. Initial work focused on the spatial distribution within a range of biological materials which derive their carbon from different sources. This has been further extended to include the various geochemical phases of seawater, *ie*, dissolved organic and inorganic carbon and particulate organic and inorganic carbon, together with the organic and inorganic carbon phases of surface sediments. The results are discussed in relation to radiological considerations and the application of ¹⁴C to existing mathematical models derived using ¹³⁷Cs.

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RADIOCARBON DATING OF MORTAR IN IRELAND

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Radiocarbon dates based on charcoal particles embedded in mortar and thus preserved over time were found to yield reliable radiocarbon dates when checked against buildings of known age. Presented here are a series of dates from early masonry structures in Ireland dating from the 9th century AD onwards. Generally, the architectural-historical and radiocarbon dates run in parallel. However, there are a few surprises in the case of structures whose ages were, until now, uncertain owing to a lack of diagnostic stylistic features. In general, the radiocarbon dates provide a firm base for chronological placement of many early medieval buildings in Ireland, encouraging their preservation.

RADIOCARBON DATING OF ANODONTA IN THE MOJAVE RIVER BASIN

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Radiocarbon dates were obtained from *Anodonta* shells from the Mojave River Basin and compared with tufa and AMS radiocarbon dates based on the organic fraction of rock varnish. In addition, modern *Anodonta* shells were assayed for their radiocarbon content and a calibration factor calculated for this geographic region. This study was carried out in context with chronological research into the natural history of ancient Lake Manix, California. *Anodonta* dates, when corrected, provide good correlation with other conventional radiocarbon dates from downstream locations in the Silver or Soda Lake basins. The numerical results of this study will be presented.

HIGH-SPEED ACQUISITION OF MULTI-PARAMETER DATA USING A MACINTOSH II CX

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Accelerator mass spectrometry systems based on > 3 MV tandem accelerators often use multianode ionization detectors and/or time-of-flight detectors to identify individual isotopes through multi-parameter analysis. A Macintosh IIcx has been programmed to collect AMS data from a CAMAC-implemented analyzer, and to display the histogrammed individual parameters and a double-parameter array. Data is transferred through a Nu-Bus to CAMAC dataway interface, which allows direct addressing to all functions and locations in the crate. Asynchronous data from the rare isotope is sorted into a CAMAC memory module by a list sequence controller. Isotope switching is controlled by a one-cycle timing generator. A rate-dependent amount of time is used to transfer the data from the memory module at the end of each timing cycle. The present configuration uses 10-75 ms for rates of 500-10,000 cps. Parameter analysis occurs during the rest of the 520 ms data collection cycle. Completed measurements of each sample are written to files that are compatible with standard Macintosh data bases or other processing programs.

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BACKGROUND AND CONTAMINATION STUDIES AT THE ISOTRACE AMS FACILITY

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For several years, background and contamination studies have been performed at the IsoTrace. In particular, the contamination of every stage of the sample processing was studied, and it was demonstrated that the practical limit of radiocarbon dating at IsoTrace is currently 60,000 BP. This limit was caused by a fixed contribution to the contamination in acetylene synthesis and graphitization processes. The combustion and hydrolysis stages in the sample processing were contamination-free. The consequences of these studies for the routine dating of old samples will be discussed.

RADIOCARBON DATING OF COPPER ARCHAEOLOGICAL ARTIFACTS

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The extraordinary preservation of small amounts of wood, bark wrap and cordage in copper harpoons and spears from Archaic period provenience of unspecified age provides a unique opportunity for radiocarbon dating with accelerator mass spectrometry (AMS). Results of the work carried out at IsoTrace, in conjunction with a copper research project addressing trace element, REE, PGE and Pb distributions in native and European copper, will be discussed.

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INTERSTADIAL MARINE DEPOSITS AT CAPE STORM, ELLESMERE ISLAND

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At Cape Storm, southern Ellesmere Island, a spectacular series of Holocene raised beaches is underlain by a till-like deposit and then by a second generation of marine deposits, dominantly sand. This lower marine unit is unusual, in terms of High Arctic sites, in that fragments of marine algae, *Laminaria* sp and *Sphacelaria plumosa* are preserved in the permafrost environment, together with several species of pelecypods.

Over 15 ¹⁴C age determinations are now available, including several from a 20-cm-thick debris layer at ~63 m asl, close to the top of the lower marine unit. Four age determinations on marine algae range from $35,800 \pm 1080$ years (GSC-2584-3) to $43,840 \pm 560$ years (TO-1298), whereas three dates on aragonite shells of *Mya truncata* range from $34,670 \pm 390$ years (TO-223) to 40,500

 \pm 1660 years (GSC-2786). In addition, a sample of siphon sheath/periostracum from *Mya truncata* gave an age of 43,370 \pm 330 years (TO-114).

The general correspondence between the dates on different materials, the presence of a cold water ostracod fauna, and the absence of such boreal-subarctic pelecypods as *Chlamys islandica* and *Mytilus edulis*, suggest that the mid-Wisconsinan interstadial age is correct. This conclusion is reinforced by the fact that amino acid ratios, on a variety of pelecypods, are the lowest in the region for pre-Holocene shells.

COSMOGENIC ISOTOPES AND THE GEOMAGNETIC FIELD IN THE PAST

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We propose a new method of paleomagnetic investigation, based on the analysis of cosmogenic isotope abundance in terrestrial samples. Based on this method, temporal variation of the geomagnetic field in the past, including the epoch of the Brunhes-Matuyama magnetic reversal, is reconstructed. We show that the minimum magnetic field occurs 3000-4000 years after 730,000 BP, and there are quasiperiodic variations with the 10,000-year period.

STOCHASTIC PROPERTIES OF GEOPHYSICAL SERIES

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Modern approaches to deterministic chaos investigations give us an opportunity to obtain such important characteristics of the series as the Ranyi dimensions, Kolmogorov entropy and Lyapunov exponents. These values characterize the level of chaos in a dynamic system, and allow us to estimate the minimal number of independent variables governing the system. Using this method, we have calculated the values mentioned above for the radiocarbon and beryllium series. We have found a high level of chaos in these series. Moreover, correlation dimension, D_2 , is non-uniform with time. For instance, during the Maunder minimum, D_2 turns out to be sufficiently higher than that for the neighboring time intervals.

RADIOCARBON DATING OF SOME OF THE DEAD SEA SCROLLS

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The name, "Dead Sea Scrolls," refers to some 1200 scrolls that were found in caves in the hills on the western shore of the Dead Sea during the last 45 years. They range in size from small fragments to complete books from the holy scriptures (Old Testament), uncanonized sectarian books, from letters and commercial documents. Papyrus and parchment are the writing materials. Only in a very few cases were direct information on the date of writing found in the scrolls. In

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all other cases, the dating is based on indirect archaeological and paleographical evidence. To check this evidence, the radiocarbon ages of 14 selected scrolls were determined, using the technique of accelerator mass spectrometry. Sample preparation, measuring procedure and results will be discussed and the calibrated ages compared with the expectations.

OCEANIC AND SOLAR FORCING OF NATURAL GEOGRAPHIC VARIATIONS IN ATMOSPHERIC $\Delta^{14}\mathrm{C}$

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A three-dimensional global tracer transport model (derived from a general circulation model) simulates geographic variations in atmospheric Δ^{14} C in response to oceanic and solar boundary conditions. Transient model results address the controversial existence of a strong higher-latitude 11-year cycle in atmospheric Δ^{14} C. The meridional atmospheric Δ^{14} C gradient is mainly forced by latitude-specific atmosphere/ocean CO₂ gas exchange rates, which are, in turn, controlled by regional wind strengths and sea-surface temperatures and by air/sea pCO₂ differences (Δ pCO₂) and oceanic ¹⁴C/¹²C deficiencies (Δ R). Assuming an observation-based Δ R latitudinal distribution, several oceanic Δ pCO₂ scenarios yield the measured preindustrial northern-southern hemispheric Δ^{14} C difference of 4‰. These alternative ¹⁴C-calibrated oceans have implications for the preindustrial latitudinal gradient of atmospheric pCO₂.

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GROWTH RATE INVESTIGATIONS OF TROPICAL TREES WITH THE NEW ERLANGEN AMS FACILITY¹

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At the Erlangen tandem accelerator, the setup of the new AMS components has been completed (Baumgärtner *et al* 1990); a high-current ion source, a 90° injection magnet, a purely electrostatic beam transport system, a 15° electric deflection, a 55° analyzing magnet and a gas-filled ΔE -E detector have all been installed. In the Cs sputter ion source, a spherical Ta ionizer is used to achieve high sputter rates and a small sputter focus. The separately pumped target chamber, which contains the sample magazine, is opened to the source chamber only during the automatic exchange of the sputter target.

¹Research was supported by the Deutsche Forschungsgemeinschaft.

Growth rate investigations of tropical trees are often difficult, because information on growth rhythm is rare. The interpretation of visible growth zones, which are caused by periodic floods and dry seasons, are not always unique. Therefore, we performed radiocarbon measurements along the cross-section of the tree and on predated individual growth zones. Since the variation of ¹⁴C in the atmosphere between 1950, the so-called bomb peak in the 1960s, and the present level is known (eg, Worbes 1989), the age profiles and, thus, growth rates of tropical trees may be deduced (Leavitt & Long 1989). Details of the apparatus, the sample preparation technique and the measurements will be presented.

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THE IMPACT OF CALCITE DISSOLUTION ON RADIOCARBON DATING OF DEEP-SEA SEDIMENTS

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The combination of bioturbation and dissolution in deep-sea sediments is expected to lead to a reduction in the radiocarbon age of core-top material (relative to that expected in the absence of dissolution). The reason is that dissolution should reduce in mass grains with large mixed-layer residence times. In an attempt to document this impact, we carried out by accelerator mass spectrometry (AMS), radiocarbon measurements on core-top material from cores of a series of depths in the equatorial Pacific. These cores range from little dissolution to extensive dissolution. We were surprised to find no evidence for age reductions. This suggests that dissolution occurs before grains have been mixed into the 5-8-cm-thick zone defined by nearly uniform ¹⁴C age *versus* depth.

EXPERIMENTAL DETERMINATION OF THE ¹⁴C INITIAL ACTIVITY OF CALCAREOUS DEPOSITS¹

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The initial ¹⁴C activity (A_o) of dissolved inorganic carbon (DIC) reflects geochemical processes of groundwater formation and influences the calculation of ¹⁴C age of calcareous deposits. ¹⁴C activity of carbonate precipitated from fresh water is practically identical to that of HCO₃⁻ dissolved in

water. Hence, the simplest method of determining A_o would be to measure ¹⁴C activity of either bicarbonate precipitated from water or of calcium carbonate collected in sediment traps or precipitated on artificial substrata in the case of tufa-depositing streams. However, the present ¹⁴C activity of dissolved biearbonates in water cannot be used to correct the age of old carbonate sediments for the so-called "hard water effect" because of the contamination of ground and surface waters with bomb-produced ¹⁴C. A reconstruction of the initial activity of DIC is still possible by ¹⁴C activity measurements of samples of pre-bomb calcareous deposits of known age in lake sediments or tufa and organic samples (eg, wood) from the same layer. We describe several methods of experimental determination of A_o , which we used in a case study of geochronology of carbonates in the Plitvice Lakes area.

The ¹⁴C activity of depth profiles can be correlated with sediment depth. The slope of the regression line gives the sedimentation rate, and the intercept gives the initial activity. Care must be taken of the uppermost part of the sediment, which is usually less thick than the lower part, and is affected by the global increase of natural ¹⁴C activity.

Assuming that wood fragments embedded in the lake sediment are contemporary with the surrounding sediment, initial ¹⁴C activity can be calculated as the ratio of the ¹⁴C activity of the sediment to that of the wood. In carbonate-rich waters, submerged material becomes coated with a calcareous crust having the ¹⁴C activity close to the A_o of water. Several prebomb test samples of wood coated with calcite crust were found, and the initial ¹⁴C activity was calculated. The A_o values obtained by different methods for Lake Prošće range from 71.6 to 72.2%, and those for Lake Kozjak from 74.8 to 75.9%. These values are also in accordance with the calculated values based on the downstream increase of ¹⁴C activity of bicarbonates in fresh waters open to the atmosphere (Srdoč *et al* 1986).

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AMS RADIOCARBON MEASUREMENTS AT THE UNIVERSITY OF WASHINGTON

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Preliminary results of recent radiocarbon studies on Galapagos Islands corals and on pollens extracted from lake sediment cores are presented. Recent technological improvements to our AMS system are also discussed.

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DATING OF CLIMATE AND HUMAN HISTORY: A BASIS FOR CLIMATE IMPACT ASSESSMENT

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Radiometric dating of interdisciplinary data sets is a vital prerequisite to link human and environmental history in time. However, a time-relationship between a certain climatic and archaeological change does not necessarily imply a cause-and-effect linkage. A sharp distinction should be drawn between time correlation and causal association. Experiments and models are necessary to assess whether a climatic change during a certain period affected a particular culturaleconomic activity. Development of agricultural systems in the Eastern Mediterranean from incipient Epi-Paleolithic strategies down to modern times might be studied experimentally, in relation to climatic and other environmental parameters. It is important to know at which thresholds, both climatic and social, system breakdown of a certain subsistence strategy or economic activity is likely to occur. System analysis in relation to climate impact assessment should follow the linkage in time between human and environmental history.

ESTABLISHING CALIBRATED ¹⁴C CHRONOLOGIES: PROBLEMATIC TIME ZONES AND HIGH-PRECISION DATING, WITH REFERENCE TO NEAR EASTERN ARCHAEOLOGY

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The pattern of past variations in the amount of atmospheric ¹⁴C affects the degree of accuracy that can be attained with radiocarbon calibration. Four principal time zones occur in the period since 3400 BC, in which calibration precision is highly limited:

3340-2920 BC 2890-2590 BC 770-405 BC AD 1660-1950

The reason is the rather horizontal trend of the calibration curve over these stretches of time. As a result, radiocarbon dates cannot be accurately calibrated in the following BP time zones (parallel with the above historical periods):

 4570 ± 20 BP to 4310 ± 20 BP 4250 ± 20 BP to 4050 ± 20 BP 2530 ± 20 BP to 2390 ± 20 BP 230 ± 20 to Present

Even high-precision ¹⁴C dates in these time zones may yield 1-sigma calibrated age ranges of several hundred years. However, for other time periods, in which the calibration curve has a more evenly descending trend, high-precision radiocarbon dating is essential for obtaining calibrated ¹⁴C dates with medium- to high-precision. Examples are presented, with particular reference to Near Eastern archaeology, using the computer calibration program of Van der Plicht and Mook (1988).

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DATING ARCHAEOLOGICAL ENTITIES IN THE DESERTIC LEVANT

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Assembling the radiocarbon dates for the Holocene period in the Southern Levant and especially from desertic sites, enables us to modify the chronological boundaries between archaeological periods and cultures. The calibration of many dates indicates the need to reconsider several accepted cutural explanations and the meaning of archaeological gaps in sub-regional sequences. Using the calibration curve as a partial indicator for climatic fluctuations, we will examine here a few archaeological cases dated to the mid-Holocene through the historical periods.

COMMON SPECTRAL FEATURES IN THE 5500-YEAR RECORD OF TOTAL CARBONATE IN SEA SEDIMENTS AND RADIOCARBON IN TREE RINGS

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We present the results of the analysis of the carbonate time series obtained from the Ionian Sea core GT90-3 and corresponding Δ^{14} C from trees. The 3.6-m core extends backward in time to 5520 BP, and extends the results obtained earlier (Castagnoli *et al* 1990a,b; in press) from core GT14 and core GT89-3.

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RADIOCARBON ABUNDANCE IN TREE RINGS: A TOOL FOR ECOLOGICAL INVESTIGATION

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We describe here the program of collaboration on ¹⁴C abundance measurements in tree rings with an aim of studying ecological phenomena. The first results of joint measurements of ¹⁴C abundance in tree rings are discussed.

THEORETICAL AND EXPERIMENTAL ASPECTS OF SOLAR FLARE MANIFESTATION IN RADIOCARBON ABUNDANCE IN TREE RINGS

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We suggest a method to determine solar cosmic-ray fluxes and spectra of the past based on a comparison of different cosmogenic isotope records. Experimental data on radiocarbon abundance in tree rings of 1989–1991 are analyzed with an aim of observing 1989 powerful solar flares. We have obtained seven intervals with a high probability for powerful solar flares for the period of the 13th to 20th centuries AD.

A SEARCH FOR ¹⁴C TREE-RING SOLAR FLARE EFFECTS

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Radiocarbon of early and late wood of pine tree rings from the northern and southern hemispheres has been measured to discover the effects of solar flares of the years 1942, 1946 and 1989. These solar flares produced considerable groundlevel enhancements of cosmic rays. Results obtained will be discussed on the basis of wood formation, the solar particle rigidity spectrum and the expected ¹⁴C yield.

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REACTION PATH PERSPECTIVE OF RADIOCARBON DATING OF GROUNDWATER

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Since the publication of the pioneer paper by K O Münnich (1957), ¹⁴C systematics has been used in a large number of hydrogeological studies. Because of the complexity of carbon geochemistry, numerous models have been proposed to correct the dilution effect of "dead" carbon in groundwater systems. However, the applicability of a fixed-model interpretation of ¹⁴C data remains limited.

As recharge water percolates through the CO_2 -rich vadose zone, the consumption of dissolved CO_2 during water-rock interactions is constantly replenished by a CO_2 reservoir in the vadose zone. The chemical and carbon-isotopic composition of the water is modulated by the CO_2 reservoir. As the water moves deeper into the groundwater system, the chemical and carbon-isotopic compositions of water change as a result of dissolution, precipitation and ion exchange reactions. Redox processes involving carbon further complicates the interpretation of chemical and isotopic compositions of groundwater. All proposed ¹⁴C dating models only consider dissolution of carbonate minerals under open- or closed-system conditions, with respect to a CO_2 reservoir. Consequently, fixed-model correction of ¹⁴C age may be erroneous.

A simple DIC alkalinity equation:

$$Q = \frac{2DIC - alk - H_2CO_3^\circ}{DIC}$$

is derived to account for simple dissolution of carbonate from open- to closed-system conditions. However, for systems involving precipitation and redox reactions, reaction path simulations are essential for reliable ¹⁴C dating of groundwater.

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A MODEL OF HUMUS FORMATION IN SOILS BASED ON RADIOCARBON DATA OF NATURAL ECOSYSTEMS

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Recent soil is an open system. In a soil profile, organic matter is deposited over a long period, and is continuously renewed during its lifetime. Until now, we have not had a clear understanding of the physical meaning of ¹⁴C data and their correlation with the true age of recent soils and other time-related parameters. We think that information on specific activity of ¹⁴C in humus makes it possible to state that soil is not younger than the obtained date. We also aim to define the rates and duration of the processes of humus accumulation and the coefficients of humus mineralization that can be used for assessing stability of different organic profile types. We have suggested the model of a monogenetic soil under stable conditions of pedogenesis. With the help of this model, we showed that the most stable fraction of humus is, humic acids with a ¹⁴C date of 1000 ± 40 BP deposited about 3000 years from zero time, and unchanged ¹⁴C activity. We can also define

coefficients of mineralization of humus and humic acids. Since the 1950s, nuclear tests in the atmosphere have increased significantly the ¹⁴C concentrations, and calculation of ¹⁴C age of soil organic matter is not possible because the specific activity of humus is higher than the specific activity of the NBS standard. However, knowing the coefficients of humus mineralization change of ¹⁴C activity in the atmosphere and the specific activity of humus, we can reconstruct the ages of different soils.

IDENTIFICATION OF LANDFILL GAS USING RADIOCARBON DATING

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The microbial decomposition of organic materials in landfills produces large quantities of methane. Landfill methane has been identified as a significant contributor to the Greenhouse Effect, and can result in hazardous situations if it migrates off-site. It is now also considered a potential energy resource. Thus, control of methane emissions from landfills is of environmental as well as of economic interest. Methane, the primary component of landfill gas, is also the primary component of natural gas, coal gas and marsh gas. Landfill gas generally contains high concentrations of carbon dioxide and may contain heavy organic compounds, such as vinyl chloride. Although gases from different sources can sometimes be distinguished by their chemical compositions, the chemical composition of gas can change as it migrates through the subsurface. Thus, chemical analysis alone does not always provide a method of identifying gas that has originated in a landfill.

Much of the organic material that decomposes in a landfill contains elevated ¹⁴C concentrations as a result of atmospheric testing of nuclear weapons in the 1950s and early 1960s. Thus, ¹⁴C analysis provides a definitive method for distinguishing landfill methane from methane derived from many other sources. The data presented show that ¹⁴C analysis of both methane and carbon dioxide, when combined with chemical and stable isotope analyses, can provide information about the environment through which the gas has passed as well as about the migration pathway.

A ¹⁴C DATING PROTOCOL FOR USE WITH PACKARD SCINTILLATION COUNTERS EMPLOYING BURST COUNTING CIRCUITRY

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Considerable research has been carried out in this laboratory into the use of Packard scintillation counters employing burst counting circuitry. The nature of the pulse-shape discrimination circuit has led to a redefinition of our sample vialing philosophy and scintillation cocktail optimization. Results are presented to demonstrate:

- 1. How efficiency may be enhanced with a two-component cocktail compared with the use of a primary fluor only
- 2. How a cocktail was developed in which efficiency and quenching were relatively unaffected by variations in the cocktail ratio
- 3. The rationale behind the use of screw-cap vials rather than sealable ampules
- 4. The advantage of using nickel/chromium-plated brass caps compared with standard plastic caps.

THE STATISTICS OF LOW-LEVEL COUNTING USING THE NEW GENERATION PACKARD SCINTILLATION COUNTERS

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The new generation of liquid scintillation counters with low-background count-rate capability raises questions concerning the application of standard Poisson statistics to the resulting counts. In addition, the stability of these systems must be verified over long time periods. In this paper, we investigate the suitability of Poisson statistics applied to count rates derived from the Packard 2260XL and 2000CA/LL counters. We consider their long-term stability and discuss the implications of this new technology for minimum age calculations.

RADIOCARBON DATING OF IRON ARTIFACTS

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During the late 1960s, N J van der Merwe (1969) obtained radiocarbon measurements on 11 iron pieces, ranging in carbon content from medium carbon wrought iron (1.2 kg used) to high carbon cast iron (30 g), thereby demonstrating the feasibility of the technique for iron. In the early 1980s, Sayre *et al* (1982) repeated two of van der Merwe's measurements and carried out two analyses on a recently re-recovered Elizabethan (?) iron bloom. Thirty grams were required of this medium carbon wrought iron to obtain an age using small proportional counters.

A number of iron artifacts were analyzed recently at IsoTrace using accelerator mass spectrometry (AMS). Samples ranged in size from 3.4 g of a medium carbon ($\sim 0.4\%$) wrought iron bloom to 274 mg for a high carbon (1.79%) wootz steel fragment. This reduction in sample size posed no problem for accelerator dating, which now permits analysis of samples that previously were too small or too valuable to be analyzed. For larger samples, multiple analyses can reveal variations that may aid the evaluation of a sample's history.

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THE SYNERGISM BETWEEN RADIOCARBON DATA AND MULTIVARIABLE CHEMICAL DATA FOR THE APPORTIONMENT OF ATMOSPHERIC PARTICLES

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Source apportionment of atmospheric aerosols is gaining increasing importance from the perspective of local and regional health and visibility impairment and from the perspective of global transport and global climate. Health effects arise from mutagens and carcinogens, such as certain of the polycyclic aromatic hydrocarbons; visibility and climatic effects relate to light scattering and absorption as well as cloud nucleation.

Carbon isotopes have become popular for apportioning anthropogenic and natural particle sources, and some work has been reported on univariate studies involving isotopes and chemical variables. In the present work, we shall demonstrate the important increase in understanding that comes from astute combination of ¹⁴C data with multivariate chemical data. Such methods of analysis yield information on the number of primary particulate sources, the source signatures and a quantitative apportionment of source intensities.

¹⁴C DATING OF MICRITIC CaCO₃ PEDOGENIC NODULES FROM VLEI DEPOSITS, HAASKRAAL PAN, SOUTH AFRICA

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Pedogenic calcareous nodules were ¹⁴C dated from various stratified soil horizons in a meander of the Zoetvlei River in South Africa. The nodules occur stratified throughout 2–6 m of alluvial sediments. The nodules probably formed during drying episodes, and as a result of periodic lowering of water-table cycles in the phreatic zone. The archaeological evidence suggests the area was inhabited by Middle Stone Age (Orangian industry) and Early Stone Age (Acheulian industry) groups, antedating the last 40,000 years and beyond the range of ¹⁴C. Twenty ¹⁴C dates were run on the stratified nodular sequences, all yielding finite ¹⁴C dates, suggesting contamination of older sediments with more recent carbonate.

Stained and impregnated nodules were thin-sectioned for petrographical analyses. Vugs and pores in the nodular micritic matrix were lined with secondary micrite and sparry calcite. XRD patterns of the nodules indicate the presence of the expandable chlorite montmorillonite-mica clay mineral group inherent in the soil matrix.

Uranium series dating of the nodular horizons from 14 sample sites reveals major drying episodes at about 44,000, 81,000 and 171,000 years. Older nodules retain a high porosity but are less permeable than the younger nodules. Stable ¹³C and ¹⁸O isotopes of older nodules record an isotopically lighter signal, suggesting cooler conditions affecting the groundwater reservoir.

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ANOMALOUS 11-YEAR ∆¹⁴C CYCLE AT HIGH LATITUDES

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At the Twelfth International Radiocarbon Conference in Trondheim, Norway, Fan *et al* (1986) reported a 25‰ peak-to-trough variation in Δ^{14} C during solar cycles 11 to 14 in tree rings from the MacKenzie Delta at 68°N. Our preliminary TAMS measurements on single-year tree rings from that area and those cycles do not confirm as large a variation, nor does there appear to be a significant correlation with the sunspot cycle. However, the $\Delta\%$ values appear to be significantly lower than for Stuiver and Quay's (1981) $\Delta\%$ measurements on tree rings from the Olympic Peninsula (47°46'N, 124°06'W). This is possibly an environmental effect, such as release of ¹⁴C-depleted CO₂ trapped under snow cover and released at the time of early growth in the Spring. Work is continuing to set tighter limits on the postulated high-latitude effect.

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SHARED PERIODICITIES IN SOLAR AND A14C RECORD VARIATIONS

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There are numerous outstanding periods in power spectra of the detrended, high-precision $\Delta^{14}C$ data. Among these are the 2120-year (Hallstattzeit), 212-year (Suess) and 88-year (Gleissberg) periods. A 53-year periodicity consistently appears above the background noise, although not as strongly as the Gleissberg. The amplitude peaks derived from several varied spectral methods support the proposition that many features in the $\Delta^{14}C$ data spectra are harmonics of the single 2120-year Hallstattzeit periodicity. Based upon this proposition, the frequency resolution of lower frequency harmonics can be significantly improved using the well-resolved higher frequency harmonics. Similarly, although the 11-year Schwabe cycle is highly attenuated by the low-pass nature of the carbon cycle, and is further obscured by solar-flare ¹⁴C production that tends to occur out of phase with the solar wind modulation of ¹⁴C, observed modulation of the Schwabe cycle by the Hallstattzeit harmonics in annual solar activity and $\Delta^{14}C$ records more precisely defines the period of the Schwabe cycle. Reconstruction of observed solar variability over the last three centuries can be effectively modeled using amplitude modulation of the 11-year cycle by the lowerfrequency solar harmonics. These low-frequency harmonics may result from Hallstattzeit-period variation acting as a ~500-year gate selectively suppressing solar activity and enhancing ¹⁴C production beginning every ~1620 years. For example, whereas the 212-year Suess cycle dominated from the 14th through the 17th centuries, the 88-year Gleissberg cycle appears enhanced. Hence, the ¹⁴C-R, coherence through the Hallstattzeit harmonics provides a means of predicting the trend of future solar activity.

RADIOCARBON ANOMALIES AND LATE HOLOCENE CLIMATIC CHANGE IN COASTAL SOUTHERN CALIFORNIA

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The connection between solar activity, climate and radiocarbon anomalies proposed by Eddy (1977) and Schmidt and Gruhle (1988) is supported by a paleoclimatic record from coastal California. Pollen analysis and five radiocarbon dates for a 687 cm core provide a detailed chronology of environmental change in a marsh at the head of Newport Bay, Orange County, California. From ca 7000 to 4500 BP, the site was a freshwater marsh, trees were more abundant than today, and grassland was the regional vegetation. Sediment deposition kept pace with sea-level rise during much of the record, but after 4500 BP, sea water regularly reached the coring site, and salt marsh was the local vegetation. Brief periods of dominance by freshwater vegetation at 3800, 2800, 2300 and after 560 BP correlate with episodes of global cooling during the late Holocene. The correspondence of these periods with major ¹⁴C anomalies support a causal connection with solar variability.

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AN EVALUATION OF CHEMICAL METHODS TO INHIBIT ISOTOPE FRACTION OF TRITON DURING HYDROLYSIS OF CALCIUM CARBIDE TO ACETYLENE IN BENZENE SYNTHESIS

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The hydrogen isotopefractionation effect in benzene synthesis during the hydrolysis of metal carbides has been previously reported. The formation of the hydroxide carries with it approximately 30% of the tritium activity from most water samples used in the synthesis of benzene for low background activity measurements. In low sample activity groundwater assessment studies, low background liquid scintillation counting (LSC) is an essential tool. At present, most water samples are distilled and prepared for LSC directly using an emulsifier type of scintillation cocktail. However, one significant limitation of this approach is that the tritium counting efficiency ranges between 15% to 20% for a typical sample at 50% to 60% water load. Another disadvantage is that the pulse-height energy spectrum of the severely quenched tritium water sample largely overlaps with that of the background spectrum. Another method to prepare low activity water samples is to electrolytically enrich the tritium as water and count the enriched water sample to improve the measurement sensitivity.

This paper presents and evaluates different chemical methods to inhibit the formation of triton as the hydroxyl hydrogen during benzene synthesis to reduce the isotope fractionation effect. A comparison is made between direct water counting, enriched water counting, and synthesis of benzene from water using the inhibitory methods to improve tritium counting efficiency and overall measurement sensitivity.

THE SEARCH FOR CARBON IN ROCK COATINGS TO CONSTRAIN THE AGES OF LAND FORMS AND ARCHAEOLOGICAL ARTIFACTS

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Accelerator mass spectrometry (AMS) has reduced the amount of carbon required for radiocarbon measurement to levels where it is feasible to extract organic carbon from rock coatings. Organic mats encapsulated by accretions of rock varnish, pedogenic carbonate and amorphous silica yield minimum ages for the exposure of the underlying rock to varnishing, the onset of soil formation and amorphous silica deposition. Radiocarbon dating of these organic deposits constrains the ages of natural and human features without any previous numerical age control. Glacial moraines, alluvial fans, paleoventifacts, slope colluvium, lava flow, landslides and lake shorelines are examples of land forms that now have some numerical age control. Artifacts, ground drawings and petroglyphs can also be dated for the first time by extracting organic carbon from within and underneath rock coatings.

HIGH-PRECISION ∆¹⁴C IN THE SURFACE OCEAN AND CLIMATIC VARIABILITY

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High-precision radiocarbon measurements of annual coral bands from the past 400 years will be presented. Data from several sites in the Pacific and Atlantic Oceans will be discussed in light of decadal timescale variability that has been identified in the prebomb Δ^{14} C records. Stable oxygen isotope (δ^{18} O) records, an indicator of sea-surface temperature changes, will be used to deconvolute the two processes that can cause changes in surface ocean radiocarbon, changes in mixing with the surrounding water mass(es) and changes in the atmospheric forcing function. Correlations with existing records of climate change will be presented.

ISOTOPIC COMPOSITION OF GROUNDWATER AND THE UPPERMOST PART OF LAMINATED SEDIMENTS OF GOŚCIĄŻ LAKE

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Measurement results of radiocarbon concentration in the carbonate and organic samples from the uppermost part of laminated sediments from Gościąż Lake (central Poland) are presented. The whole laminated sequence of Gośiąż Lake comprises more than 12,000 varves. Isotopic composition of carbon, hydrogen and oxygen of groundwater from the surrounding area also has been investigated. The results of our analysis indicate the different types of water present in an area close to the lake. The ¹⁴C concentration in the carbonate sediment is a bit higher than in carbonate and bicarbonate dissolved in water, and shows the presence of a short-term fluctuation, which is positively correlated with a change in the sedimentation rate.

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EVALUATION OF A PROTOTYPE LOW-LEVEL LIQUID SCINTILLATION MULTISAMPLE COUNTER

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At the 13th International Radiocarbon Conference in Dubrovnik, Yugoslavia, June 1988, the results of the first phase of a study on the pulse spectrum of a simple, single photomultiplier liquid scintillation counter were presented. The present study is a continuation of this work. Several improvements have since been made to the system, based on the knowledge obtained from the original work and from the current study. A new, improved photomultiplier has been incorporated and a more efficient active guard installed. A special quartz vial has been designed to reduce background and to improve the photoelectron factor of merit (keV/photoelectron). Four photomultipliers are installed in the system, allowing multiple samples to be counted simultaneously.

The measurements presented here cover background spectra and counting efficiency obtained with the system under various conditions in the laboratory. These measurements and derived parameters, such as the figure of merit (E^2/B) and the factor of merit (S/\sqrt{B}) , are used to evaluate the system performance. Results of measurements on the photoelectron factor of merit and further background spectra, obtained with the system at an underground site, are discussed.

CALCIUM-41 AS A LONG-TERM BIOLOGICAL TRACER FOR BONE RESORPTION

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Accelerator mass spectrometry (AMS) measurements of 41 Ca provide a unique possibility for measuring bone resorption over multiyear periods in humans. This possibility results from the long half-life of 41 Ca (100,000 yrs), the high sensitivity of AMS, the low specific activity of 41 Ca, the low energy released per decay and the low natural abundance of 41 Ca. These factors allow oral administration well below the annual limit of intake for the general public, and still provide an easily measured signal in blood or urine resulting from resorption of prelabeled bone over multiyear time periods.

We have tested this possibility by prelabeling 14 dogs simultaneously with ⁴¹Ca and ⁴⁵Ca (half-life 165 days) and then following both isotopes in blood for 28 weeks. We measured the ⁴¹Ca by AMS at the University of Pennsylvania. The two isotopes correlate well over this period. We will continue to monitor these isotopes for a year or more, long after the ⁴⁵Ca becomes unmeasurable.

This study paves the way for using ⁴¹Ca to characterize postmenopausal changes in bone resorption with time in individual women who receive a ⁴¹Ca oral dose several years before their menopause. Such a capability might allow an identification of women at risk of developing postmenopausal osteoporosis, with negligible exposure to radiation and before significant bone loss has occurred.

PRIME LAB: A DEDICATED AMS FACILITY AT PURDUE UNIVERSITY

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Purdue University has made a major commitment to establish a dedicated research facility for accelerator mass spectrometry (AMS) based on its existing FN tandem. The facility has been named the Purdue Rare Isotope Measurement Laboratory (PRIME Lab). The accelerator is in good operating condition and is well suited for measuring the full range of AMS isotopes. AMS will be the primary activity on the accelerator and could utilize as much as 90% of the beam time. A search is underway for a senior geoscientist, who will base a research program on AMS and will play a major role in establishing priorities for the new facility.

The start-up phase has been funded by Purdue and the National Science Foundation/Earth Science Division/Instrumentation and Facilities Program. We will first measure the radionuclides, ¹⁰Be, ²⁶Al and ³⁶Cl, at 3–5% precision and a relatively low capacity. With additional funding, we plan to provide rapid isotope cycling and fully automate the AMS measurements. We will add capability for the isotopes, ¹⁴C, ⁴¹Ca and ¹²⁹I, improve the precision to better than 1%, and increase the capacity to several thousand samples per year. The first results were obtained in early 1991 and are reported here.

Our first priority is a vigorous Purdue-based research program in the earth and planetary sciences. If properly funded, we plan to become a national AMS facility, providing a service to the earth and planetary science communities.

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EVIDENCE OF EARLY HOLOCENE SUMMER PRECIPITATION IN THE MIDDLE EAST

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Physical and botanical evidence for early Holocene summer precipitation in the Middle East is discussed. The well-established low-latitude increase in summer rainfall at this time apparently extended to the mid-latitudes of the Middle East, a region that today is characterized by long, rainless summers. Remnants of desert lakes radiocarbon dated to this period, pollen data, and plant remains in archaeological deposits provide evidence for summer-season precipitation. At present, Poaceae (grasses) are severely restricted by summer drought throughout the region. Pollen assemblages from early Holocene lakes in the Rub' Al-Khali (Empty Quarter) of Saudi Arabia, and from freshwater deposits buried beneath the Arabo-Persian Gulf, show high percentages of Poaceae pollen. In regional pollen diagrams, Chenopodiaceae and Artemisia pollen, dominant during the Pleistocene, were replaced by Poaceae pollen in the early Holocene. There was a concurrent increase in the pollen of summer-green trees, another indicator of summer rainfall. The earliest remains of those wheat varieties that prefer summer rainfall, the hexaploid varieties and onegrained Einkorn, Triticum boeticum spp thauder, are from regions that today experience a severe and extended summer drought. This elimination or reduction in summer drought was probably due to strengthened monsoon and other convective rainfall resulting from the increased summer insolation of the early Holocene. Local modifications may be attributed to orographic influences, proximity to moisture sources and interaction with westerly disturbances.

REMOTE DETECTION OF UNDERGROUND BIOREMEDIATION BY SOIL CARBON DIOXIDE ANALYSES

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In-situ bioremediation of contaminated soil and groundwater is a recently developed and promising technology. This paper describes a method of remotely detecting and monitoring underground biodegradation of gasoline hydrocarbons. Groundwater and soil vapor samples were taken from a field site containing a documented plume of unleaded gasoline in an underground aquifer. Carbon dioxide was isolated from both groundwater and soil vapor and analyzed. Mass spectrometry was used to determine stable carbon isotope ratios, and accelerator mass spectrometry (AMS) was used to determine radiocarbon activity. Bacteria counts and hydrocarbon analyses were also performed on the groundwater samples. Bacteria counts included those of total bacteria, viable bacteria and aerobic hydrocarbon-degrading bacteria. Hydrocarbon analyses revealed varying concentrations of benzene, toluene, ethyl benzene and other hydrocarbons, depending upon distance from the center of the original spill. Definite relationships among radiocarbon activity, stable carbon isotope ratios and levels of hydrocarbons in groundwater and soil vapor were found. These results indicate the potential use of remote sampling of soil vapor to identify and monitor natural or remediated underground biodegradation of gasoline hydrocarbons.

ACCELERATOR MASS SPECTROMETRY AT THE LUND PELLETRON ACCELERATOR

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Three years ago, funds were raised to equip the 3 MV Pelletron accelerator at the Lund University Institute of Physics for accelerator mass spectroscopy (AMS). During the initial period, we have been working exclusively with ¹⁴C.

The Pelletron accelerator has now been adjusted for mass spectroscopy. The generating volt meter has been modified and part of the high-energy side of the accelerator has been reconstructed to provide better location of the focusing devices. A Wien velocity filter and detectors for measuring the energy (E) and energy loss (ΔE) have been installed. About 30 samples of elementary carbon, using Fe or CO as catalyst, were produced at the Department of Radiation Physics in Gothenburg during the last two years. The ¹²C⁻ ion current from these samples is about 3–5 μ A, developed with an ANIS ion source.

We are now planning ¹⁴C analyses in the fields of archaeology, Quaternary geology and radio-ecology.

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CONFRONTATION OF GEOLOGIC STRATIGRAPHY AND RADIOMETRIC DATES FROM UPPER PLEISTOCENE SITES IN THE LEVANT

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It is difficult to reconcile the lithostratigraphy and paleoenvironmental interpretations of some major sites (Tabun, Skhul, Qafzeh) with recently determined radiometric (ESR, TL) ages. Additional comparative material is available from Yabrud rockshelter in Syria where Acheulio-Yabrudian levels have been dated ca 225,000 years (ESR on horse teeth), ca 150,000 years (ESR on burned flint) and ca 200,000 years (TL on burned flint). The stratigraphic positions of the Amudian (Pre-Aurignacian) components at Yabrud, Tabun and Adlun also can shed some light on the credibility of the radiometric dates. It is interesting , moreover, that the ESR/TL dates for those sites all cluster around the same value (100,000 years), in spite of quite different paleoenvironmental settings and different cultural associations and human physical types. This raises the question of the lack of sensitivity of those dating techniques, reminiscent of radiocarbon dating of many marine mollusks that formerly clustered around 30,000 years before methodological refinements and cross-dating with other methods showed them to be spurious.

ACCELERATOR MASS SPECTROMETRY OF ¹⁴C AT THE AUSTRALIAN NATIONAL UNIVERSITY

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The accelerator mass spectrometry program based on the 14UD Pelletron accelerator at the Australian National University has historically been committed to the measurement of ³⁶Cl in environmental samples. Recently, however, motivated by a need within the university for a small-sample capability to complement the existing decay-counting facility at the Radiocarbon Laboratory, a ¹⁴C capability has also been implemented. At the present time, this system is operated on a medium-precision, low-throughput basis with slow cycling between isotopes. The beam currents of the stable isotopes are measured in a Faraday cup, which is located immediately in front of the ¹⁴C detector. The special features of the system will be described, our experience with this mode of operation and with sample preparation will be reviewed, some early projects will be discussed, and envisaged developments will be outlined.

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A COMPARISON OF ¹⁴C AND U/Th AGES ON CONTINENTAL CARBONATES

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A basin of internal drainage is located in a discharge of a large confined aquifer from Northern Sahara. Lacustrine deposits of Holocene age were dated by both radiocarbon and U/Th methods on inorganic carbonates and on mollusk shells (*Cardium*). Small crystals of inorganic calcite were

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precipitated close to equilibrium with atmospheric CO₂. Radiocarbon ages are in sequence and are believed to be representative. Apparent ages of *Cardium* are about 2000 years older than expected from a supply of old carbon through the vertical seepage of the confined aquifer. No correction is possible from the ¹³C content of the carbonates, possibly because of biological effects in the system.

The age discrepancy between carbonate shallow water and interstitial waters of bottom mud cannot be reconciled by U/Th measurements. Uranium appears to be thoroughly leached, leading to excesses of ²³⁰Th and to ages that are too old. Assuming the age is representative of the inorganic calcite, we can evaluate the amount of leached uranium in the shallow deposits and its accumulation in the deeper reduced horizons of the profile.

We also discuss implications for age estimates of continental deposits.

SAPROPEL CHRONOLOGY AND NILE RIVER DISCHARGE DURING THE HOLOCENE

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Organic-rich layers (more than 2% organic carbon) occur in deep eastern Mediterranean Sea sediments. These sapropels were attributed to the stratification of the seawater column due to a large input of fresh water, mainly associated with a Nile River discharge which inhibited the thermohaline convection. ¹⁴C AMS determinations performed on the base of the sapropel show the synchroneity of the event at 8600 BP over the whole eastern Mediterranean Sea, recording the beginning of the major pluvial period during the Holocene. Oxygen isotopic values from core MD 84641 taken in the Nile River fan show that the large input of Nile water was maximum between 8600 and 8000 BP, and decreased drastically after that time.

THE HOLOCENE CLIMATIC RECORD IN THE SALT CAVES OF MOUNT SEDOM, ISRAEL

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The climatic history of the Dead Sea during the Holocene was reconstructed from data on outflow levels and geometry of caves in Mount Sedom, and from ages of 33 plant remains found in the caves. The salt diapir of Mount Sedom emerged from the Dead Sea early in the Holocene. A small watershed on the top of the mountain accumulated water, which dissolved shafts and, afterwards, caves in the rock salt, and also swept plant remains into the caves. The widths of the caves varied between less than 100% and 400% of the width of the presently active cave, due to variations in the amount and intensity of rains.

The upper and middle Holocene was divided into ten stages of alternating dry and humid climate, each characterized by the stand of the Dead Sea (high stands at 7000, 4500, 3000 and 1000 BP), and by the climate of the Dead Sea region and the conditions on the top of Mount Sedom. The changes in climate are within a period of about 1000 years. Good correlation was found with the

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sedimentological record of the southern basin of the Dead Sea, with archaeological information and, closer to the present, with the historical record. On a larger scale, good agreement was found with the very detailed data on water levels of the Black Sea, and fair agreement with lake beds in Africa.

AMS RADIOCARBON RESULTS OBTAINED FROM GRAPHITE TARGETS PRODUCED AT THE WOODS HOLE OCEANOGRAPHIC INSTITUTION BETWEEN 1986 AND 1990

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In July 1986, a radiocarbon target preparation laboratory was established at the Woods Hole Oceanographic Institution for producing graphite to be analyzed at the National Tandem Accelerator Mass Spectrometry (AMS) Facility at The University of Arizona, Tucson. By August 1990, 865 graphite targets had been prepared using the Fe-Zn reduction method described by Slota *et al* (1987). Our laboratory procedures during this time included the careful documentation of weights of all starting samples, catalysts and final graphite yields, as well as the volume of CO_2 gas evolved during hydrolysis or combustion. These numbers can be used to calculate a wide range of statistical parameters, thus allowing us to evaluate our laboratory, in particular, and this method, in general.

Of the 865 targets produced to date, 786 have been AMS dated. We have results on 498 $CaCO_3$ hydrolysis reactions (467 AMS dated), 128 closed-tube combustion reactions (113 AMS dated), and 38 DICs from seawater reactions (32 AMS dated). In addition, results have been obtained from 160 NBS oxalic acid standards I and II (142 AMS dated), 35 CaCO₃ hydrolysis blanks (26 AMS dated), and 5 closed tube combustion blanks (5 AMS dated).

Results of our data set, which includes values such as the mean and standard deviation of graphite yields, backgrounds and the ratio of the two oxalic acid standards, as well as the 1 σ error vs ¹⁴C age for all samples analyzed, will be presented.

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THE USE OF CONTINENTAL CARBONATES COMPARED WITH OTHER MATERIALS IN THE DATING OF A PALEOLAKE

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The possible usefulness of different kinds of components of geological materials for radiocarbon dating has scarcely been studied. Since carbonates and organic matter are very usual components of geological materials used in radiocarbon dating, we have undertaken a study on the application of these materials to the radiocarbon dating of a paleolake. The results of the study show a systematic shift of carbonates and organic matter with respect to contemporary wood. Moreover, organic matter is systematically younger than carbonates. The apparent age of carbonates has been explained as the result of hard-water effect and the apparent age of organic matter has been attributed to the existence of aquatic plants, which exert their chlorophyllic function at the expense

of the dissolved CO_2 , and from which organic matter is deposited. Disagreement between carbonates and organic matter is assumed to be caused by terrestrial plants that contribute also to the sedimentation of organic matter.

THE PALEOHYDROLOGY OF PALESTINE

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Based on numerous radiocarbon data obtained from groundwater and speleothem samples collected in Palestine and Arabic countries, a differentiated picture of the paleohydrology has been obtained. I will compare these data with available information on the climatic changes observed in North Africa and the Indian subcontinent.

CHARCOAL DATING - OXIDATION IS NECESSARY FOR COMPLETE HUMIC REMOVAL

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In the radiocarbon dating of sedimentary deposits, it has been common to consider that dates on suitably pretreated charcoal are consistently reliable. For deposits with a low carbon content, the traditional methods for the removal of 'fulvic and humic acids' from charcoal, using dilute acid and alkali solutions, are found to be inefficient. Dates obtained on fine sedimentary charcoal from sites with different climatic and depositional histories yield maximum ages only when an oxidation stage is included in the pretreatment sequence. The results described here demonstrate that after oxidation, newly solubilized humic-like material can be extracted with dilute alkali, and that this fraction has about the same age as the traditional 'humic acid' fraction. Inconsistent results with a nitric acid oxidation technique may indicate incomplete removal of humic materials rendered soluble by the oxidation.

Since the charcoal in these experiments has a similar particle size to the pollen in the same deposits, and the pollen and charcoal yield the same age when dated separately, it is likely that the charcoal arrives by the same mechanisms as the pollen (commonly wind or water deposition). This increases confidence that the dates obtained are giving a reliable chronology of sedimentation. Two of the factors that influence the final age for such charcoals are: 1) destruction of clay/organic complexes and 2) alkali extraction after oxidation. These findings imply that some revision in age structure of sedimentary deposits will be necessary for those sites where traditionally pretreated materials have been dated.

PALEOENVIRONMENTS AND PREHISTORIC SITES IN ISRAEL

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Prehistoric sites in Israel are situated in a variety of settings that range from the Mediterranean region in the north to the arid climates of the Negev Desert in the south. There is a large degree of variability of sedimentary environments through both time and space. In the desertic belt, for

example, Lower and Middle Pleistocene prehistoric sites are almost nonexistent, whereas Upper Pleistocene sites are relatively more common. Moreover, Middle and Upper Paleolithic, Epipaleolithic and Historic sites occur in a wide variety of sedimentary environments, including alluvial, lacustrine and aeolian – as exemplified by both dune sand and loess deposits. In the more humid coastal ranges, locally preserved lacustrine and fluviatile deposits contain Lower and Middle Pleistocene sites among which 'Ubeidiya is the oldest known outside of Africa. In contrast to the Negev, Middle and Upper Paleolithic sites are relatively poorly known from open-air sites, and are best preserved in cave settings. In addition, open-air environments of Neolithic and later sites seem to be tied to colluvial deposits, though sometimes exposures are relatively poor, owing both to Holocene sedimentation and modern agricultural practices.

Numerous paleosols occur in the region, and are characterized by calcic horizons in the southern and eastern areas, and grade to redder, clayier loams in the Mediterranean zone. Some of these fossil soils occur in close association with prehistoric sites throughout the area. Lithic artifact assemblages and organic materials excavated at a site can be dated quite well with radiocarbon and, thus, these dated sites provide a solid temporal framework for the sedimentary units associated with them. Depositional environments inferred from the sediments, on the other hand, not only provide valuable information about the changes of former environmental conditions – including climate – under which prehistoric humans and their cultures evolved, but also furnish criteria for evaluating changes in prehistoric settlement patterns over time.

THE IAEA ¹⁴C INTERCOMPARISON EXERCISE 1990

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As a follow-up of the meeting of experts convened at the International Atomic Energy Agency (IAEA) in February 1989, and the International ¹⁴C Workshop held in Glasgow in September 1989, the ¹⁴C Quality Assurance Program was formulated. As part of this program, a set of five new intercomparison materials has been prepared as a joint effort of several radiocarbon teams and IAEA staff. They represent natural materials frequently encountered in everyday practice of radiocarbon laboratories. We assume that the new intercomparison exercise will lead to precise evaluation of ¹⁴C concentration levels for these materials, which will be stored in the Agency as "IAEA Quality Assurance ¹⁴C Materials," and will be available upon request to all radiocarbon laboratories. The intercomparison materials were distributed among 137 laboratories in May 1990. In February 1991, a meeting of experts was convened in Vienna to evaluate the results and to make recommendations with regard to further use of these materials by the radiocarbon community. The paper presents the results of this new intercomparison exercise.

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TRENDS IN ¹⁸O IN LAND SNAIL SHELLS FROM THE NEGEV DESERT DURING THE HOLOCENE: IMPLICATIONS FOR CHANGES IN RAINFALL SOURCE AREAS

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The δ^{18} O value of the shell carbonate of land snails is related to the ¹⁸O content of precipitation, which in turn, relates to the source region and trajectory of the rain-bearing air masses. Analyses of ¹⁸O of shell carbonate of 76 radiocarbon-dated Holocene samples of the land snail, *Trochoidea* seetzeni, from the northern Negev Desert were carried out, and the results compared to modern snails from the same region. Early Holocene δ^{18} O values are similar to modern ones, and do not give any indication of the penetration of monsoonal rains into this region at this time. Centered around 6500-6000 BP, a large depletion in ¹⁸O is observed. A change in the atmospheric circulation pattern for this period is thus indicated, most likely an increase in the frequency of storm systems reaching the region from northeastern Africa. By 3500 BP, δ^{18} O values had reached modern levels, and indicate a stable pattern of atmospheric circulation since that time.

DATING OF POST-AD 1650 SAMPLES BY ASPARTIC ACID RACEMIZATION

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An evaluation of the age-predictive abilities of D/L ratios of various amino acids (alanine, alloisoleucine/isoleucine, proline, aspartic acid, methionine, glutamic acid and phenylalanine) was carried out based on 38 radiocarbon-dated Holocene samples of the land snail, *Trochoidea seetzeni*, from the Negev Desert. Results indicate that each of the amino acids has a similar correlation with age. The rates of racemization or epimerization vary by about $\pm 10-15\%$ between samples, and thus permit age estimates from D/L ratios with about this precision. Within most of the time range of applicability of radiocarbon dating, this precision is not as good as that of radiocarbon dating. However, for very late Holocene samples, amino acid racemization initially (*ie*, at low D/L values), and thus offers the best possibility of dating very young samples with good precision. This method should be particularly useful for samples younger than about AD 1650, since radiocarbon analysis provides no time resolution within this age range.

300-YEAR DECLINES IN ATMOSPHERIC ¹⁴C CONCENTRATION OF THE PAST

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Research in the non-random character of natural medium- and short-term ¹⁴C variations of the past has led to many conclusions on the occurrence of cyclicities, as well as the recurrence of characteristic patterns. One of a few independent cyclicities seems to be that of a ca 960-year period, confirmed by both Fourier and MEM analyses. The best known characteristic features are the maxima of Maunder and Spörer types, recurring rather irregularly. Another peculiarity appears to be 300 years long, a nearly monotonical fall of Δ^{14} C, reconstructed by radiocarbon measurements

in tree rings of oak from Lublinek (ca 9750-10,050 cal BP). Such a pattern repeats many times throughout the Holocene. Most of the periods of 300-year declines recur more or less regularly, with the mean time span of 966 years. The amplitude of 300-year declines varies, the highest occurring in the earliest periods. It seems also, that a similar drop occurs about the YD/PB boundary. For earlier times, the available data are too dubious, and searching for characteristic patterns seems to be fruitless.

GERARDIA: THE BRISTLECONE PINE OF THE DEEP-SEA?

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Layers from the proteinaceous skeleton of the colonial zoanthid sea anemone, Gerardia, were subjected to carbon isotope and chemical analyses. This specimen was collected from 600 meters in the Florida Current in the northwestern Atlantic Ocean using the Deep Submergence Research Vessel, ALVIN. Radiocarbon (Δ^{14} C) values in the layers decreased linearly with distance from the outer growing edge (-75‰) to the central core of the trunk (-250‰). If these Δ^{14} C values are reflective of time since accretion of the layers, then the ¹⁴C age of this specimen is 1700 ± 100 BP. The δ^{13} C values of the layers did not vary (-16.0 ± 0.2‰), suggesting an unchanged source of carbon over its lifetime. The C:N ratios (wt %) in the layers are 2.8 to 3.0, indicative of protein. The amino-acid assays reveal high concentrations of glycine, histadine and alanine.

The existence of a deep-sea animal that accretes a layered, proteinaceous skeleton is unique. A lifespan of greater than a millennium pinpoints this organism as a possible marine equivalent of long-lived trees, such as the bristlecone pine.

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INTERPRETING THE SMALL COSMOGENIC ISOTOPE SIGNAL

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The technical ability to detect very small isotope concentration changes and/or to use very small samples opens new fields of analysis, but also requires critical reevaluation of all sources of variability outside the direct measurement. Environmental noise, that is, spatial and temporal variability in the concentration of cosmogenic isotopes in the atmosphere and in reservoirs in contact with the atmosphere, lake plants, snow and sediments, may be caused by variable mixing within the troposphere, and between troposphere and stratosphere. Seasonal cycles in biospheric productivity and oceanic ventilation may contribute to ¹⁴C variability; ¹⁰Be concentrations can vary with long-range transport by zonal and meridional circulation.

The detection limit for cosmogenic isotopes is set by the equipment background, which is constant and follows Poisson statistics in counters, by laboratory contamination and by sample contamination, which is different for each sample. Appropriate sample treatment procedures can minimize or eliminate laboratory contamination and keep the residual constant. *In-situ* production as a cause of ¹⁴C contamination stays generally well below the detection limit. In rocks and minerals, it provides a dating tool. Interpretation of the results, however, is complicated by a strong dependence of the production rate on the mass shielding the material from cosmic radiation.

EXAMINATION OF BACKGROUND CONTAMINATION LEVELS FOR GAS COUNTING AND AMS TARGET PREPARATION IN TRONDHEIM

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The radiocarbon dating laboratory in Trondheim relatively often dates samples with ages > 30,000 BP. Contaminated background materials could easily affect the accuracy of very old dates (Beukens 1990). Measurements on different materials show that such effects are very small for our conventional gas counting.

Contamination levels in the target preparation line (for dating at the Uppsala accelerator) have been investigated. A significant lower background level is obtained for Icelandic double spar than for marbles, probably due to a crystal structure that is more insensitive to contaminating processes. The background level for combusted wood of interglacial age is acceptable (ca 0.35 pMC), whereas a lower background is obtained for combusted gelatin.

A comparison between dates obtained on old samples (> 40,000 BP) by both conventional gas counting and AMS demonstrates good agreement between the two dating techniques.

REFERENCE

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RADIOACTIVE LEVELS OF ¹⁴C AND ³H IN WHITE SPIRITS OF CHINA

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White (and yeast) spirits is a general name for strong alcoholic beverages in China. Spirits are made with grains that are leavened and distilled in water. We can learn about distribution and shifting of cosmogenic isotopes such as ¹⁴C and ³H from levels of ¹⁴C and ³H activities in white spirits.

This paper reports levels of ¹⁴C and ³H in 65 Chinese white spirits samples between 1986 and 1987. Experiments were made by measuring and analyzing each sample, which is prepared by mixing 40 cm³ pure spirits with 60 cm³ scintillator solution, using a low background liquid scintillation spectrometer.

Radioactive levels of 65 kinds of spirits samples are as follows:

- Variant range of ¹⁴C activity = 38.8-80.2 Bq.dm⁻³
- Average ¹⁴C activity = 57.4 ± 8.2 Bq.dm⁻³
- Variant range of ³H activity = 98.2-170.6 Bq.dm⁻³
- Mean of ³H activity = 149.2 ± 17.3 Bq.dm⁻³.

In ordinary strong alcoholic beverages that contain 57–60% alcohol, the mean ¹⁴C and ³H activities are 60.3 Bq.dm⁻³ and 153.8 Bq.dm⁻³, respectively, but in spirits of lower alcoholic content (38–40%), the mean ¹⁴C activity is 46.1 Bq.dm⁻³, that is, 23.5% less than ordinary spirits, and the mean ³H activity is 114.6 Bq.dm⁻³, that is, 25.5% less than ordinary spirits.

We compared the ¹⁴C and ³H contents of five kinds of staple grains from both Sichuan and Guangdong provinces. We learned that the level of ¹⁴C activity in spirits is equivalent to that in grains, and the level of ³H activity in spirits is ten times higher than in grains and water. White spirits has fully concentrated ³H and ¹⁴C from both grain and water, and activities increase with increasing alcoholic content. ³H in white spirits probably is averaged from both water and grain, and ¹⁴C is averaged mostly from grain.

ABSOLUTE RADIOCARBON CHRONOLOGY OF THE AUBREY CLOVIS SITE, TEXAS, BASED ON SOIL HUMATE STRATIGRAPHY

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The Aubrey Clovis site was discovered in the outlet channel of Ray Roberts Lake, Denton County, Texas. The Clovis occupation surface, including two camp areas and a bison-kill-processing locus, was buried 8–9 m below the Trinity River floodplain. A radiocarbon chronology was developed to establish the geochronology of late Pleistocene lacustrine and spring deposits and a thick section of Holocene alluvium. A 12 m stratigraphic column was sampled at 17 horizons, with ages ranging from 14,200 to 1730 BP without reversals. Dated materials include peat residue, peat soluble fractions, alluvium and soil A-horizons. Procedures for the pretreatment of sediment samples include removal of carbonates, extraction of total humates, separation from clay minerals and concentration of humates to solid form. Despite exposure to the atmosphere for several years and seepage of bicarbonate-rich waters, excellent results were achieved. Stable isotope data from these organic-rich sediments clearly demarcate the shift from lacustrine to fluvial environments at the locality.

RADIOCARBON DATING OF GROUNDWATER CONTAINING MICROBIAL METHANE

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Many aquifers in glacial deposits contain microbial methane formed from decomposition of organic materials contained in the sediments. Microbial decomposition can have a significant effect on both the δ^{13} C value and the ¹⁴C content of the dissolved inorganic carbon (DIC) and, thus, on the radiocarbon age of the water. This study addresses the problem of radiocarbon dating of groundwater containing microbial methane.

Groundwater samples were collected under pressure from several different depths in Illinoian glacial deposits and from the underlying bedrock at a site in east-central Illinois. The dissolved

gases were extracted from the water samples, measured volumetrically and analyzed by gas chromatography. The DIC was precipitated as barium carbonate and analyzed for both δ^{13} C and ¹⁴C. The DIC had δ^{13} C values of -3 to -1‰, similar to those observed for carbonates in the tills. If these heavy δ^{13} C values are used in readily available groundwater dating models without accounting for the effects of microbial methane formation, the resultant calculated age is unrealistically young.

Preliminary results show a positive correlation ($r^2 = 0.91$) between the $\delta^{13}C$ of the DIC and the concentration of methane in the water. With this correlation, a more realistic estimate of the water-rock interaction can be calculated and used for correcting the ¹⁴C age of the groundwater.

ISOTOPIC ANALYSIS OF CARBON IN A GEOTHERMAL SYSTEM

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The Surdulica geothermal system in the southeast part of Yugoslavia belongs to the granodiorite aquifer. Geothermal waters are in a range of $60^{\circ}-130^{\circ}$ C, and show bicarbonate sodium content, slightly alkali (pH = 7.0-7.5), mineralized (1.1-1.3 gr/L), with high fluoride and silica concentrations. The carbonate content of water increases with depth to the bottom of the system. Spring waters from altitudes higher than 800 m (top of the system, 1922 m asl) contain HCO₃ and CO₂ lower than 80 mg/L and 10 mg/L, respectively. During water flow, carbonate content fluctuates from 200 mg/L at the middle elevations (600 m asl) to 500 mg/L in geothermal waters reaching, in some parts of the aquifer, values of 3 g/L for HCO₃ and 500 mg/L for dissolved CO₂.

According to these data, the isotopic content of total dissolved inorganic carbon (TDIC) also changes. At the surface vegetation cover, $\delta^{13}C = -27\%$ and ¹⁴C content of 120 pMC were recorded. Data on ¹³C concentration vary from -15‰ in the springs at the middle elevations up to the interval of -5 to 0.5‰ for geothermal waters at the bottom.

In the surface water zone, we discovered infiltration of water at the top of the system (above 1300 m asl) with 30-50 pMC. The hot thermal spring zone in the foothills of the massif (400 m asl) is characterized by tritium-free water with very low ¹⁴C content (2-7 pMC). Because of the evident influence of dead carbon, ¹⁴C dating of these geothermal waters is difficult. For $A_o = 85$ pMC, the ages were estimated in the range of 10,000–28,000 years.

¹⁴C MEASUREMENTS ON LAMINATED LAKE SEDIMENTS

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With the development of the AMS technique, ¹⁴C measurements on laminated lake sediments became especially interesting because the measurement of milligram-size samples is now possible. The selection of well-defined terrestrial macrofossils (leaves, needles, seeds of trees) from the sediment, excludes "hard water" contamination.

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Soppensee, a small lake in central Switzerland, provides a continuous sequence of annual layers for the period, 6000-12,000 BP. Samples of macrofossils were collected from sections spanning 10-30 years and analyzed at the AMS facility at ETH-PSI.

The main goals of the project are: detailed information on climate changes by pollen stratigraphy, analysis of stable isotopes and extension of the existing ¹⁴C tree-ring chronology.

CATEGORIZATION OF ORGANIC SEDIMENTS IN ARID ENVIRONMENTS AND THE EFFECT OF SUNLIGHT ON THEIR COMPOSITION

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A series of sediment samples having a relatively low organic content, representing different microenvironments, have been subjected to a stepwise treatment involving carbonate removal, solvent extraction and separation into humic and non-humic fractions. Specific mobile organic extracts have been further separated according to polarity, then categorized using solid phase separation techniques and thin layer chromatography. The humic component has been categorized simply by H/C, O/C and C/N atom ratios, whereas the non-humic residues have been treated with HF/HClmixtures. The remaining organic component has also been categorized using the above techniques. There is strong evidence that many of the mobile organic compounds have been subjected to photochemical modification by the action of the UV component of sunlight. Implications of this effect for the pretreatment of organic sediments for ¹⁴C dating will be discussed.

A REVIEW OF ATTEMPTS TO DATE POTTERY AT OXFORD

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The main problem with dating pottery by radiocarbon is that many different carbon sources, of different radiocarbon age, may contribute to the potsherd carbon content. Also, the process of firing is liable to destroy information that might help separate possible sources. We describe several pottery dating projects in which we have dated separate fractions (such as humics, lipids and classes of residual carbon). Although in some cases there is sufficient consistency between results to accept that this approach can give a credible date, in other cases, no date has been possible, and general conclusions are difficult to make.

PROBLEMS AND ADVANCES IN THE RADIOCARBON DATING OF BONE: A SHORT REVIEW OF THE WORK OF AMS LABORATORIES

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Although the reliability of ¹⁴C dates of bone has increased greatly since AMS methods permitted better pretreatment on smaller samples, most old, badly contaminated or severely weathered bone still give serious problems. Several groups have recently proposed improvements to sample purification methods, often supported by a number of ¹⁴C measurements. We present here an overview of these improvements. The issue is complicated by the following:

- 1. Different problems are presented depending on age, preservation and degree of contamination of bone.
- 2. Methods may or may not be developed with routine application in mind.
- 3. Determining the conditions for which any method can be regarded as reliable is not at all straightforward.

DEVELOPMENTS IN SAMPLE COMBUSTION TO CARBON DIOXIDE, AND IN THE CARBON DIOXIDE ION SOURCE SYSTEM

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The operation of a commercial system as modified for preparation of CO_2 for the CO_2 source is described. AMS samples are automatically combusted in a CHN analyzer, and stable isotope measurements are made on line. The performance of this equipment is described, with reference to yield, sample contamination, memory effect, accuracy of isotope measurement, convenience and cost.

The current status of dating using the CO_2 source is described. This is the only source in operation at Oxford, and has been in routine dating for 18 months. An assessment of the practicalities of operation will be made, including the latest measurements on background, memory, sample size requirements and operating schedules. Modifications to the sputter beam optics and to the gas handling systems will be described.

GEOCHRONOLOGIC AND PALEOCLIMATIC CHARACTERIZATION OF QUATERNARY SEDIMENTS IN THE GREAT HUNGARIAN PLAIN

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We present here radiocarbon ages and δ^{18} O isotope ratios of mollusk shells from fluviatile and aeolian Quaternary deposits. We have identified ten malacofaunal levels and some species, serving as chronological indicators, from 7000 - 32,000 BP. These paleoclimatological investigations, according to the rules of sedimentology, are based on the oxygen isotope analysis of properly collected *Pupilla muscorum* shells. Oxygen isotope ratios of remote deposits from the same time period showed good agreement. Temperature values obtained from the results of the isotopic studies and of a malacothermometer constructed on the basis of the dispersion and climatic indicators of mollusk species are also closely correlated.

Comprehensive studies of paleoclimatic changes, through chronological, isotope chemical and biological analyses showed the same climatic periods as found in northern and western Europe. The climate of our study area was of a rather continental character at the end of the Pleistocene.

ASSESSMENT OF 0.3 ML MINIVIALS FOR LIQUID SCINTILLATION COUNTING OF BENZENE

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An evaluation is made of 0.3 mL minivials for radiocarbon dating undersized samples by liquid scintillation counting of benzene. A calibrated wood standard was diluted by varying amounts of 'ancient' CO_2 , with synthesized benzene counted in both conventional 3 mL vials and 0.3 mL minivials in a 1220 Quantulus. The accuracy and precision of results are compared for samples ranging in weight from 240 to 50 mg carbon.

Two problems associated with handling small samples are examined, namely memory effects within the vacuum system, and signal within the dilution gas. Careful measurement of these parameters allow corrections to be made permitting accurate age determination in both conventional vials and minivials. For samples containing less than 240 mg carbon, minivials produce more accurate results than conventional 3 mL vials.

EVALUATION OF HIGH-PURITY SYNTHETIC QUARTZ VIALS AND ACTIVE PLASTIC HOLDERS IN LIQUID SCINTILLATION COUNTING OF BENZENE

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An evaluation is made of high-purity synthetic silica vials in both delrin and active plastic holders (Pico AdaptersTM) for radiocarbon dating, utilizing liquid scintillation counting of benzene. Synthetic silica vials in the form of simple cylinder cells utilizing the standard 7 mL vial design are presented. A passive delrin holder for supporting 7 mL quartz or glass vials has also been designed.

The counting efficiency and background of the silica vials are compared with teflon and low-K glass vials for both delrin holders and Pico Adapters[™] in the 1220 Quantulus and Packard Tri-Carb 2066 XL fitted with a plastic detector guard. In the 1220 Quantulus, synthetic silica vials in Pico Adapters[™] have the highest Figure of Merit (FOM), closely followed by teflon and silica in delrin holders. In the Packard Tri-Carb 2660 XL, low-K borosilicate glass vials in Pico Adapters[™] give the highest FOM.

A RADIOCARBON CHRONOLOGY OF LATE NEOLITHIC SETTLEMENTS IN THE TISZA-MAROS REGION OF HUNGARY

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An archaeological research program has been ongoing for the past 15 years. We are investigating chronological questions and settlement patterns in the Hungarian Tisza region for the time of transition between the Late Neolithic and Early Copper Ages. In the course of this project, nearly 50 charcoal and bone samples have been dated from the Neolithic stratified tell settlements of Hódmezővásárhely-Gorzsa, Szeged-Tápé-Lebő, Hódmezővásárhely-Kőkénydomb, Szegvár-Tűzkőves and from the broadly distributed settlement of Deszk-Vénó. The new data outline the time sequence of the given settlements of the Hungarian Late Neolithic Age as follows:

Tápé-Lebő	6290 ± 60 - 5760 ± 60
Szegvár-Tűzkőves	6160 ± 60 - 5790 ± 60
Hódmezővásárhely-Kőkénydomb	$6150 \pm 60 - 5750 \pm 60$
Hódmezővásárhely-Gorzsa	$5970 \pm 100 - 5580 \pm 100$
Deszk-Vénó	5420 ± 60

DETERMINING THE ORIGIN OF GEOTHERMAL WATERS IN NORTHWESTERN YUGOSLAVIA BY ISOTOPIC METHODS¹

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The origin of geothermal water from several hot springs in northwest Croatia and east Slovenia, Yugoslavia, has been investigated by isotopic analyses. Two contradicting hypotheses on the origin of geothermal water were postulated (meteoric vs volcanic), both based on previous geological studies and physico-chemical measurements of geothermal waters. This work is aimed at solving the problem of the origin of geothermal water in the investigated region.

We measured the physico-chemical properties of geothermal waters (t, pH, conductivity, alkalinity, concentrations of Ca^{2+} , Mg^{2+} , Na^+ , Cl^- , SO_4^{2-} , dissolved CO_2 and O_2) as well as the concentration of radioactive (¹⁴C, ³H) and stable (¹³C, ²H and ¹⁸O) isotopes. We also analyzed calcareous deposits from hot springs at Varaždinske toplice spa, where the process of calcium carbonate precipitation is very pronounced. The results of measurements at Varaždinske toplice spa indicate a meteoric origin of this geothermal spring.

A comparison of our analyses with chemical and isotopic analyses of geothermal waters in the USA (Mammoth Hot Springs and Pagosa Springs) is presented.

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RECENT RADIOCARBON DATING OF UPPER PALEOLITHIC SITES IN THE EASTERN MEDITERRANEAN: A REVIEW OF THE WORK AT OXFORD

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The importance of the eastern Mediterranean region for the later Paleolithic has been demonstrated by the series of early thermoluminescence (TL) dates for anatomically modern hominids reported from cave sites in the area (Valladas *et al* 1987, 1988). Recent accelerator mass spectrometry (AMS) ¹⁴C dates from the 'Levantine Aurignacian' levels at Kebara Cave (Hedges *et al* 1990) have added to this story as have ¹⁴C dates from similar and later (*ie*, 'Levantine Aurignacian' and 'Proto-Kebaran') horizons at Ksar 'Aqil (Mellars & Tixier 1989).

The intention of this paper is to review some of the recent AMS dating undertaken by the Oxford Laboratory on Late Upper Paleolithic and Epipaleolithic sites in the Levant, in particular from sites that belong to the 'Kebaran' and 'Natufian' industrial traditions. The dates to be mentioned are on charred bone, seeds and charcoal, and come from the follwing sites: 1) the Natufian levels at Hayonim (Cave and Terrace), Mt Carmel; 2) a number of Kebaran sites in the Jordan Valley (Ohalo II, Urkan-e-Rubb IIa and Fazael; 3) several Terminal Pleistocene sites in the western Negev; 4) a further set of dates on the Wadi el-Jilat complex of sites in Jordan. Finally, some mention will be made to the difficulty in obtaining archaeologically acceptable dates with such material.

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MODELING THE Δ^{14} C RESPONSE TO A COMPLEX SOLAR FORCING

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Spectral analysis of the high-precision Δ^{14} C calibration record indicates the Δ^{14} C variations may not be responses to several separate sinusoidal forcings, but may, in fact, represent a system of harmonics from a single complex solar variation. Such a solar variation would likely express itself with both variations in the solar magnetic field and in solar energy output. The former variation would affect ¹⁴C production. The latter would undoubtedly have a climatic effect. After adapting the outcrop-diffusion model of Seigenthaler and Oeschger, we have used a computer software package (developed from "System Dynamics" as conceptualized by Jay W Forrester) to model the ¹⁴C system's time-variant behavior. The model permits the behavior of each parameter of the cycle to be studied separately. Production and climate-sensitive model parameters were individually varied at the harmonic periodicities to test model sensitivity. These suite forcings were then compared to the spectral and statistical characteristics of the Δ^{14} C, solar activity and climate time series. The hypothesis of a single complex solar variation not only offers a testable cosmogenic isotope production and climatic forcing but may provide a guide to the parameterization of more computationally complex coupled ocean-atmosphere general circulation models.

THE PHYSICS AND ASTROPHYSICS OF COSMIC-RAY VARIATIONS

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Cosmic rays striking the Earth are observed to vary on a wide variety of time scales. Variations over periods up to several tens of years are primarily caused by the Sun and its varying activity. Variations over larger time scales are, at least in part, due to the same causes, although geomagnetic variations also play a significant role. Extra-solar-system processes such as supernova explosions may play a role at the largest time scales.

The physical processes underlying the variations caused by the Sun and the interstellar medium, and their observational support, will be discussed.

CONSTRAINING THE INITIATION AND EVOLUTION OF ANOXIA IN THE BLACK SEA BY AMS RADIOCARBON DATING

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The Black Sea is the only extant, relatively large ocean basin containing substantial volumes of anaerobic deep waters. Bottom sediments collected from the deeper portions of this basin exhibit a lithological succession, from banded organic-poor lutites to finely laminated sapropelic muds to laminated organic-rich coccolith marls. This basin is an important natural laboratory for understanding the formation mechanisms of black shales, so widely distributed in the geological record. Despite numerous geological and chemical studies, there still remains considerable uncertainty about the temporal and chemical evolution of the deep anoxic waters and its imprint on the bottom sediments preserved in this basin. Central to this uncertainty has been the lack of a well-constrained chronology.

Early attempts at deriving a chronology for these sediments were based on a limited number of beta-decay radiocarbon analyses. Later chronological studies using varve counting showed that the radiocarbon ages appeared older by several thousand years. Some argue that the radiocarbon chronology is anomalously old due to input of detrital carbon and carbonates; others assert that each varve is an annual couplet, consisting of a lithogenic dark laminae deposited in late winter/spring, and a biogenic light laminae deposited in summer/fall. However, a varve is not produced every year due to a much reduced biogenic flux in some years, thus making age determinations by varve counting anomalously young.

In an attempt to further constrain this controversy, we have obtained 100 AMS radiocarbon analyses on both organic carbon and carbonates preserved in Black Sea sediments collected in 1988 from throughout this basin and along a 200-2200 m depth transect. Results from seven cores show that the age of the Unit II/I boundary, which defines the transition from sapropelic muds to varved sediments, is AMS radiocarbon dated at 3100 BP in both the organic carbon and carbonate fractions. This closely agrees with the older ¹⁴C ages and differs from the varve chronology by

some 2000 years. The age of the Unit III/II transition, which defines the transition from lacustrine to marine conditions, is dated in nine cores at 7350 BP in the organic carbon fraction, and 14,000 BP in the carbonate fraction. The organic carbon dates agree closely with the earlier ages and disagree with the varve chronology by some 2000 years. Most important, we see no change in the age of this boundary with water depth, as has been observed with varve counting, and as has been assumed in many models of the temporal development of anoxia in this basin. This result suggests the varve record is incomplete due to sedimentological processes, and that models of anoxia development need to be rethought.

MEASUREMENT OF COSMOGENIC ¹⁴C PRODUCED BY SPALLATION IN TERRESTRIAL ROCKS AND IN METEORITES

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The production of radioisotopes at the earth's surface by cosmic-ray effects has been discussed for many years. Only in the past few years, with more sensitivity for the radioisotopes by accelerator mass spectrometry (AMS), detection of ¹⁰Be, ²⁶Al and ³⁶Cl produced in this way have been measured by several groups. We report here on our efforts to measure cosmogenic ¹⁴C in terrestrial rocks at high altitude, and comparisons to other exposure-dating methods.

The production of ¹⁴C by cosmic-ray spallation effects in extraterrestrial samples, such as meteorites is well known. The amount of ¹⁴C left in a meteorite can be used to determine its terrestrial residence time at the earth's surface. In this paper, we will discuss measurements of spallogenic ¹⁴C in small meteorite samples from Antarctica, and the significance of the ages calculated. These ages allow us to place limits on infall rate of meteorites, and movement of the meteorites in ice.

A MINIVIAL FOR SMALL SAMPLE ¹⁴C DATING

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A 0.3 ml teflon minivial has been designed for ¹⁴C dating of small samples in a liquid scintillation counter. A special adaptor of standard vial size is used to optimize the position of the vial with respect to the phototubes and to intercept the light path between them, thus reducing optical cross-talk.

Better performance can be achieved by using customized vials than by diluting small samples for counting in large vials. Counting efficiencies up to 80% have been achieved in 0.3 ml vials typically with 0.05 cpm background.

We discuss the application of the minivial design criteria to other materials and demonstrate the power of electronic background reduction devices.

VARIATION IN TREE-RING WIDTHS GROWN IN MACKENZIE DELTA FROM AD 1528 TO 1972

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We measured the tree-ring widths of a white spruce (AD 1528-1972), grown in Mackenzie Delta $(68^{\circ}N, 130^{\circ}W)$, Canada. We compared the results with ring widths of a Qilian Mountain cypress and the corresponding climate records of China. The trends of these two sets of data were very much alike and closely correlated to climate variation. This means that the ring width of a tree, growing in arctic regions, is a sensitive indicator of global climate. The autocorrelation function of the ring widths was calculated for solar activity analysis. We also discuss the correlation between the tree-ring widths and ¹⁴C concentration in the rings.

BOMB-PRODUCED RADIOCARBON IN TREE RINGS

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Bomb-produced radiocarbon concentrations in 1961–1983 tree rings of a Dailing (47°N, 129°E) spruce were measured. The observed Δ^{14} C values rose to a peak of 909‰ in 1964, and then fell to 682‰ in 1967 and to 238‰ in 1983. By comparison with the Δ^{14} C values in tree rings grown at different latitudes and the atmospheric Δ^{14} C values in both northern and southern hemispheres, we found that the change of Δ^{14} C from 1961 to 1967 is mostly due to atmospheric mixing, thus showing a clear meridional variation with practically no longitudinal difference. Since the beginning of 1968, the decline is likely caused by the exchange of CO₂ between the atmosphere and the surface of the sea and by uptake by the terrestrial biosphere; the decay time is about 17 years. It is also evident that the Δ^{14} C values in the Dailing spruce from 1977 to 1982 are affected by a series of bomb tests in China.

HIGH-PRECISION RADIOCARBON MEASUREMENTS AT THE UNIVERSITY OF ARIZONA: LIQUID SCINTILLATION COUNTING TECHNIQUES

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The University of Arizona beta-counting laboratory purchased an LKB Quantulus liquid scintillation counter for dedicated high-precision radiocarbon measurement on single-year tree-ring samples. The settings and stability of the counter, selection of vials, standardization and background determination, and reproduction of previous high-precision samples and interlaboratory comparison of previously dated wood samples will be discussed.

EXPOSURE AGE DATING OF TERRESTRIAL MATERIALS WITH ²⁶Al AND ¹⁰Be

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Ages of surfaces must be known to understand the rates and the natures of geomorphologic processes that shape them. Most dating methods applicable to surfaces are at best relative: they rely on physical or chemical changes occurring within the surface with rates that depend on climate (temperature, rainfall and chemistry).

Six years ago at the 12th Radiocarbon Conference in Trondheim (Klein *et al* 1986), we presented the first results of exposure dating using ¹⁰Be and ²⁶Al. ¹⁰Be and ²⁶Al (as well as ³He, ¹⁴C, ²¹Ne, ³⁶Cl and ⁴¹Ca) are produced by cosmic rays in surface rocks, at a constant rate, independent of climate. '*In-situ*' production of ¹⁰Be and ²⁶Al makes it possible to determine surface ages ranging from a few hundred years to ~7 million years. Complicated exposure histories in which a surface has been affected by erosion, or by episodes of burial, can be identified. When two radionuclides are measured, the total exposure time can be determined, and a minimum age placed on the duration of burial. With three radionuclides, the time of burial can be determined and its duration more precisely defined. Three nuclides also make it possible to assess the impact of sporadic erosion (rock spalling, freeze fracturing...).

During the past six years, we have calibrated production rates and applied multi-nuclide techniques in the study of several geologic systems: to the rate and timing of glacial retreats, to the history of individual pieces of Libyan desert glass, to dating volcanic eruptions, and to determining the climatic history of the Dry Valleys and major ice sheets in Antarctica. A general overview of the current status of the technique with illustrations of applications will be presented.

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RADIOCARBON SOURCE APPORTIONMENT OF ATMOSPHERIC AEROSOLS FROM BOISE, IDAHO¹

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Atmospheric aerosols were collected during the winter of 1986–1987 in Boise, Idaho, to apportion this material between the two primary source emissions, residential wood combustion (RWC) and

¹This work is supported in part by the US EPA IAG #DW13931227-01-4.

motor vehicles. Filter samples were extracted with dichloromethane to isolate the non-volatile organic material for radiocarbon analysis, because this material potentially includes carcinogenic compounds that may pose a risk to human health. The extract was also subjected by the US Environmental Protection Agency (EPA) to mutagenicity testing to determine its bioactivity. The ¹⁴C results showed that RWC was the dominant contributor to this chemical fraction obtained at two receptors (sampling sites) and during both day and night. Ancillary ¹⁴C and composition measurements of total carbon, elemental carbon and the polycyclic aromatic hydrocarbon fraction were obtained from a few select samples. These results and details of the chemical separations will be presented.

COSMIC-RAY GENERATION DURING THE SUPERNOVA EXPLOSION

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A synchronous increase in the ¹⁰Be and ¹⁴C production rate in the Earth's atmosphere 30,000-40,000 years ago has been discovered. The time profile and amplitude increase show that a proper source of increased intensity of cosmic rays was located not far from the solar system ~50 pc. The most probable source is a supernova explosion.

SCHWABE CYCLE MANIFESTATION IN RADIOCARBON ABUNDANCE IN ANNUAL TREE RINGS

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Annual data on ¹⁴C abundance in tree rings over the period, AD 1700-1940, are considered. We show that the obtained hyperfine structure consists of 10-12-year components. Fourier spectral analysis shows the existence of the following principal periods, 10.0, 11.0, 12.5 and 15.5 years. Their amplitudes in ¹⁴C variation are 0.29%, 0.36%, 0.23% and 0.20%, respectively.

TEMPORAL-SPECTRAL ANALYSIS OF THE CHARACTERISTICS OF SOLAR ACTIVITY OVER THE PAST 400 YEARS

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The results of comparative temporal-spectral analysis of data on solar activity over the past 400 years are considered. The data used include the time series of the annual Wolf numbers, Aaindices, borealis auroras and cosmogenic ¹⁴C, and ¹⁰Be abundance in tree rings and polar ice cores, respectively. We show that the Hale cycle, occurring during the Maunder minimum, was dominant.

VARIATION OF RADIOCARBON CONTENT IN TREE RINGS DURING THE MAUNDER MINIMUM OF SOLAR ACTIVITY

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We discuss the results of high-precision measurements of radiocarbon abundance in annual tree rings during the time interval including the Maunder minimum. The following conclusions are made:

- 1. During the Maunder minimum, radiocarbon abundance in annual tree rings changed in time, which means that the Sun modulates intensity of cosmic rays even during conditions of extra-low sunspot number.
- 2. We show that during the Maunder minimum, the periods in the time interval, 17-26 years, are more pronounced.
- 3. When the Sun enters the deep minimum and emerges from it, a change of heliomagnetic modulation of the cosmic-ray flux occurs, namely, periods of more than 11 years become more important.

A TANDEM TIME-OF-FLIGHT HIGH-SENSITIVITY MASS SPECTROMETER FOR COSMOGENIC ISOTOPE MEASUREMENTS

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We describe the tandem mass spectrometric setup, TRACE, which includes two steps. The first is a multichangeable ion laser source combined with a high-sensitivity time-of-flight mass spectrometer. The second is a charge-exchange chamber for interaction of ions with energy of about 1 KeV/nucleon passing through thin foils (100 A), and a mass spectrometer for negative single-charged ion analysis. In the first step, molecular ions are eliminated by selection ions with a charge of $Q \ge + 3$. In the next step, isobars are separated by the negative ion instability of interfering elements ¹⁴N, ²⁶Mg and ¹²⁹Xe. We discuss the results of experiments and calculations. The advantages of our TRACE setup are compared with other AMS units.

THE INTERNATIONAL RADIOCARBON DATA BASE AND THE SOUTHEAST MEDITERRANEAN PILOT PROJECT

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One of several pilot projects that emerged from the First American Workshop on the International Radiocarbon Data Base (IRDB) (Kra 1988) is entitled "Paleoenvironment and Human History in the Southeast Mediterranean." Work has begun on data entry, using the software program,

PARADOX 3.5 and an IBM PC. Initial entries are based on the comprehensive data compiled by James Weinstein (1984) on the Southern Levant, and expanding the data to include reference material from *RADIOCARBON* as well as original sources. A computer demonstration of the project will be given, showing the possibilities for retrieving relevant paleoenvironmental and archaeological data. The potential for evaluating technological assessments and exercising quality control will also be presented.

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ATMOSPHERIC RADIOCARBON ACTIVITY VARIATION IN JAPAN

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Radiocarbon activity was measured periodically at an altitude of about 10 km at ground level between 1964 and 1985. The high-altitude sampling was performed by a jet fighter plane, and samples were obtained by adsorption on molecular sieves. The ground-level samples were obtained by absorption in sodium hydroxide solutions. Radiocarbon activity was measured by CO_2 gas counting, and expressed as percent excess above the normal level, which is defined by 95% NBS standard oxalic acid.

In the beginning of our measurements, the ¹⁴C activity was widely scattered, but this seems to be due to the different activity in three large air masses that cover the Japan island randomly and alternatively. As the differences of ¹⁴C activity in the air masses decreased with time, the spread of ¹⁴C activity scatter also decreased.

RADIOCARBON DATING OF FOSSIL WOOD FROM SOUTHERN POLAND

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In the last decade, a great number of trunks of subfossil oaks have been found during exploitation of gravel peats in the Vistula valley, up and downstream from Krakow, southern Poland. Preliminary radiocarbon measurements carried out in our laboratory point to a range of radiocarbon ages of individual oaks from 700 to 8400 BP. On the basis of about 100 trunks that were investigated, three floating chronologies were established, covering 700, 225 and 400 years, with ¹⁴C dated younger ends of the chronology estimated at 1690 ± 90 , 3050 ± 80 and 7800 ± 100 BP, respectively. Owing to the large quantity of the material, we hope that further measurements will enable us to link the chronologies and extend them to the present. Characteristic ¹⁴C variations were also observed in the calibration curve.

CHRONOLOGY OF LAKE SEDIMENTS FROM THE KASHMIR HIMALAYAS AND THEIR ENVIRONMENTAL IMPLICATIONS

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We present results on several sediment cores of ~ 6 m depth raised from Lake Manasbal in the foothills of the Himalayas in Kashmir (34°9'N, 74°52'E), at altitude 1770 m. We determined the chronology of the sediments by ¹³⁷Cs, ²¹⁰Pb and ¹⁴C in four cores. We examined depth variation of a variety of environmental monitors, such as magnetic susceptibility, low-frequency and frequency-dependent components (χ_L and χ_{fd}) to understand influx of soil-rich sediments from the catchment areas into the lake. Pigments such as chlorophyll, carotene, oscillaxanthin, myxoxanthophyll and C/N ratios were measured to assess variations in autochthonous and allochthonous organic inputs into the lake.

¹³⁷Cs shows a distinct peak at a depth of 6-10 cm in short cores, indicating sedimentation rates of 2-3 mm/yr for the top part of the cores. In the long cores, the top strata were invariably disturbed. ¹⁴C dates show sedimentation rates in the range of 0.5 to 1.5 mm/yr. A peak in χ_{fd} indicates the influx of highly weathered material into the lake during periods of high precipitation. We will also discuss environmental implications of the organics.

HALF-LIFE OF ⁴¹Ca

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There is renewed interest in an accurate value of the half-life of ⁴¹Ca because, with AMS, it has become possible to measure ⁴¹Ca at natural concentrations in both extraterrestrial and terrestrial materials. In 1974, a rather precise half-life of $(1.03 \pm 0.04) \times 10^5$ yr was reported by Mabuchi *et al* (1974) as the result of a specific activity measurement. However, the dependence of this result on neutron capture cross-sections, a substantial disagreement with older specific activity measurements and a precision which seemed to be smaller than the inherent uncertainties of the method, prompted us (Kutschera *et al* 1989) to perform a new specific activity measurement. In the present measurement, we used a sample of enriched calcium material whose ⁴¹Ca abundance (1.237%) was determined directly by mass spectrometry. The activity was measured with an Si(Li) detector via the 3.3 keV X rays emitted in the electron capture decay of ⁴¹Ca to ⁴¹K. The result, $t_{44} = (1.01 \pm 0.10) \times 10^5$ yr, is in excellent agreement with the very recent value of Klein *et al* (1991), $t_{44} = (1.03 \pm 0.07) \times 10^5$ yr, who determined the half-life from the decay of ⁴¹Ca relative to ³⁶Cl in Antarctic meteorites of different terrestrial ages. The original half-life result of Mabuchi *et al* (1974) has been corrected to (1.13 ± 0.12) $\times 10^5$ yr using recent information on neutron capture cross-sections and X-ray fluorescence yields, and a more realistic estimate of the

uncertainties. From these three independent half-life measurements, a weighted mean of

$$t_{\frac{1}{2}}$$
 (⁴¹Ca) = (1.04 ± 0.05) × 10⁵ yr

is calculated, which is recommended as the most accurate value of the ⁴¹Ca half-life.

A brief discussion of "unsettled" half-lives for radioisotopes which are of potential interest for future AMS studies (eg, ³²Si, ⁵⁹Ni) will also be presented.

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PROPOSED STUDIES OF ¹⁴CO AND ¹⁰Be IN POLAR ICE TO DELINEATE COSMIC-RAY FLUX CHANGES IN THE PAST 40,000 YEARS

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Lal *et al* (1990) demonstrated that cosmic-ray *in situ* interactions with oxygen in ice crystals produced appreciable amounts of ¹⁴CO, which can be used as a tracer for measuring both ice ablation and accumulation rates. We propose to extend the studies of ¹⁴CO and ¹⁰Be to determine the cause of the large variations in the ¹⁰Be concentrations documented by Raisbeck and his colleagues. The bases of the proposal are: 1) any changes in the geomagnetic field intensity will affect the concentration of ¹⁰Be in ice but not that of ¹⁴CO; 2) any changes in the flux of cosmic rays within the heliosphere would lead to an increase in both the fallout of ¹⁰Be and *in-situ* production of ¹⁴CO in ice. The fractional increase would be larger for ¹⁰Be for the second case. Climatic changes would modulate the ¹⁰Be concentrations but not those of ¹⁴CO. These factors should enable us to uniquely decide on the cause of changes in the ¹⁰Be concentrations observed.

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IN-SITU COSMOGENIC ¹⁴C IN TERRESTRIAL ROCKS: ANALYTICAL METHOD AND RESULTS

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We have developed a wet analytical method for the quantitative extraction of *in-situ* cosmogenic ¹⁴C from rocks. The experiments are confined to extraction of ¹⁴C from quartz digesting it with HF *in vacuo* using CO and CO₂ as carrier gases. Kel-F is used for the digestion vessel. Results of determination of system blanks for CO₂ and ¹⁴C are presented along with results for terrestrial rock and samples in which cosmogenic ¹⁰Be and ²⁶Al have been determined (Nishiizumi *et al* 1991). We find that about half of the *in-situ* ¹⁴C is present in quartz in the form of ¹⁴CO, similar to the case observed for polar ice (Lal *et al* 1990). The technique developed complements that of high-temperature fusion developed by Jull *et al* (1991) for studies of *in-situ* ¹⁴C in terrestrial and extraterrestrial samples. The technique has the advantage of low blanks and separate assays of ¹⁴C activities in the CO and CO₂ phases. The measurement of ¹⁴C activity in CO phase allows practically contamination-free determination of the ¹⁴C activity.

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COSMOGENIC IN-SITU ¹⁴C IN POLAR FIRN AND ICE SAMPLES

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A recent paper (Lal *et al* 1990) showed conclusively that Antarctic ablation ice from Allan Hills contained appreciable amounts of cosmogenic *in-situ* ¹⁴C, and that about 60% of this was in the chemical form of CO, the rest in the form of ¹⁴CO₂. For dating ice using atmospheric trapped CO₂, it is important to know the fraction of *in-situ*-produced ¹⁴C present in it. We are carrying out experiments to determine the amounts of ¹⁴CO and ¹⁴CO₂ in accumulation firn and ice from Greenland and Antarctica. We hope to present these results which should unambiguously estimate the contributions of *in-situ* ¹⁴C to the total ¹⁴C activity present in accumulation ice.

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IMPLICATIONS OF 813C VARIATIONS IN C3 PLANTS OVER THE PAST 55,000 YEARS

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The transition from most-recent glacial to postglacial (ca 10,000 BP) marks a pronounced change in δ^{13} C of atmospheric CO₂ and CO₂ concentration, and perhaps in light levels, soil moisture and other environmental conditions. Such environmental factors may influence plant carbon-isotope fractionation and, therefore, may have induced changes in plant δ^{13} C through time. Radiocarbondated organic matter with reported δ^{13} C values have been examined for trends or changes in δ^{13} C in response to these major environmental dislocations. A significant δ^{13} C decline from pre- to post-10,000 BP in both wood and, with a smaller magnitude, in all plant matter is consistent with an increase in the ratio of intercellular to atmospheric CO₂ partial pressures from glacial to postglacial.

A NEW TREE-RING WIDTH, $\delta^{13}C$ AND ^{14}C INVESTIGATION OF THE TWO CREEKS TYPE LOCALITY

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We have recently made an extensive collection of wood from the original Two Creeks site in order to study it with respect to radiocarbon activity and stable-carbon isotope composition. The wood includes isolated logs as well as a trunk in growth position rooted in the Two Creeks soil zone. Five pieces of wood were initially selected for detailed analysis. Radiocarbon analysis was performed on ABA-treated wood and on base-soluble fractions, both on complete specimens and on the outermost rings alone. Ring widths were measured to attempt standard cross-dating, and δ^{13} C chronologies were developed using five-year ring groups as another means of cross-dating. We discuss our results from these analyses in the context of the reported age and duration of the Two Creeks interval.

FOSSIL RADIOACTIVITY FROM NEARBY SUPERNOVAE

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We consider the present levels of various fossil radioactivities that might be expected from Galactic supernovae in the past, and compare them with currently available data. In particular, we estimate the expected activities produced directly from supernovae nucleosynthesis and indirectly by

supernova neutrinos, gamma rays and new accelerated cosmic rays interacting in interstellar, solarsystem and terrestrial material. Comparing these estimates with the various observations, we set constraints both on the nearby supernova occurrences in the past and on the total Galactic rate of supernova occurrence.

RELIABILITY OF RADIOCARBON DATING IN TEPHROCHRONOLOGICAL STUDIES OF KAMCHATKA AND KURILES

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Tephrochronological studies of the Kurile-Kamchatka region are being conducted to reconstruct the history of formation and eruptive regime of volcanoes, and to identify the marker tephra beds which describe the largest explosive eruptions of the Holocene. The dating of volcanic events is based on the ¹⁴C age of organic materials (buried soil, peat, wood and charcoal). Buried soils are the principal material for layered dating of tephra beds. Up to 10-20 thin soil intercalations alternating with tephra beds occur in each section. When there are sufficient quantities of organic material in the buried soil, successive alkaline extracts (from the same sample) are dated. Extracts of minimum and maximum age can be considered as close to the onset and termination of a soil bed formation process which is completed by pyroclastic conservation. Thin intercalations of organic material can be found in Holocene peat bogs underlying or overlying the ash beds, their age being practically synchronous with the time of tephra fallout. Carbon samples of sufficient purity and quantity can be obtained from peat both for cellular tissue and alkaline extract dating. Maximum and minimum ages of alkaline extracts can be considered as most reliable for dating. Wood and charcoal are found underlying or distributed in tephra beds in considerably smaller amounts compared with peat, but they also present good dating material because of their small individual age and distinct positioning in the section.

RADIOCARBON DATING IN THE 50,000- TO 65,000-YEAR RANGE WITHOUT ISOTOPIC ENRICHMENT

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In principle, it is possible to detect ¹⁴C levels in natural materials in the 10-11 half-life range $({}^{14}C/{}^{12}C \sim 10^{-15})$ using commercially available liquid scintillation (LS) detectors. In practice, defensible finite age determinations involve careful consideration of several critical elements in the analysis in addition to the final radioactive measurement stage. These critical elements are: 1) the integrity of the sample itself, in terms of contaminating younger material and of *in situ*-produced ¹⁴C; 2) the availability of "dead" background material; 3) chemical blank in laboratory preparation of, in this case, benzene; and 4) stability of background and efficiency of the LS counting system.

Our results so far, determined by LS on LKB-Wallac "Quantulus" measurement systems in an underground counting chamber, show comparability among benzene produced in the lab from anthracite and marble, benzene from cellulose extracted from suspected 125,000-year-old wood, and benzene produced commercially from petroleum. We conclude that the technical barriers to LS dating of samples in the 50,000- to 65,000-year range are less restrictive than the availability of suitable sample material.

ISOTOPIC COMPOSITION OF ATMOSPHERIC METHANE IN THE SOUTHERN HEMISPHERE

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Measurements in both the northern and southern hemispheres show that the concentation of atmospheric methane is increasing at about 1% per year. This increase is of concern because methane plays a significant role in the radiative properties of the atmosphere and in tropospheric and stratospheric chemistry. Isotopic determinations of atmospheric methane and its sources can provide important constraints on source strengths and sinks of the gas. We present methane isotopic data from the Antarctic and Baring Head, New Zealand. The measurements show small but significant gradients in ${}^{13}CH_4$ and ${}^{14}CH_4$ when compared to similar data collected in the northern hemisphere. The data are interpreted in terms of current models of tropospheric atmospheric chemistry, and ${}^{14}CH_4$ determinations are used to evaluate levels of fossil methane in the atmosphere.

TRAVERTINE RADIOCARBON DATING: AN APPROACH FROM THE STUDY OF RECENT TRAVERTINE ISOTOPIC COMPOSITION

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The travertine complex of Llorà (Girona, Spain) shows continuous development during the Holocene, related to spring-fed streams along a hill slope. To date the travertine, we use recently precipitated travertine isotopic composition to evaluate the initial ¹⁴C activity, instead of considering values obtained by indirect methods. The purpose of this method is to understand every process that determines the ¹⁴C activity of travertine, and to justify the measured values as well as their variations. We undertook the following steps:

- Hydrochemical and isotopic analyses of water from springs and from several points along streams. Data show chemical and isotopic reequilibrium of spring waters under atmospheric conditions, *ie*, under open-system conditions.
- Isotopic analyses of recently precipitated travertines. Their δ^{13} C values agree with the δ^{13} C of dissolved inorganic carbon. As a result of the aforementioned reequilibrium, the ¹⁴C activity increases notably as water flows far from springs.
- Isotopic analyses of ancient travertines. Samples rigorously selected yield δ¹³C and δ¹⁸O values similar to those of recently precipitated carbonates. This similitude allows us to apply the measured ¹⁴C activity in recent travertines as the initial ¹⁴C activity of ancient travertines after correcting for present atmospheric ¹⁴C enrichment.

Finally, the concordance of ages among several samples of the youngest layer of the ancient travertine formation (~ 4000 BP) leads us to conclude that we can offer a highly reliable method of travertine ¹⁴C dating.

THE OPTIMIZATION OF TARGET PRODUCTION FOR ACCELERATOR MASS SPECTROMETRY

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We have been investigating a number of methods for the production of graphite targets to run in the accelerator mass spectrometer at Woods Hole Oceanographic Institution (WHOI). The goal of the accelerator facility at WHOI is to achieve a high throughput of samples so that methods should be rapid, reproducible, and lend themselves to automation. The techniques that appear most promising are the reduction of CO_2 in an H₂ atmosphere over a Co or Fe catalyst (Vogel *et al* 1987) or a closed-tube reduction using a Co or Fe catalyst and Zn as a reducing agent (Slota *et al* 1987). The former method has the advantage that it is fast (2–6 hours) and can be monitored continuously during the reaction; the latter method may be appropriate for producing large batches of graphite from samples that are not C-limited. We are using a residual gas analyzer (RGA) to study the process of graphitization in order to find ways to improve the efficiency of the process. Using a capillary inlet system, we are able to continuously monitor the composition of the gases in the graphite reactors. Results from our studies using the RGA will be presented as well as results from the performance in our accelerator of targets produced using different methods.

Additionally, the status of automation in the preparation laboratory will be discussed. We are automating as many aspects of the target preparation process as possible using an HP3852 process controller linked with an HP9000 work station. We are able to continuously monitor, both graphically and digitally, the graphitization process in all our reactors. All ovens and valves are operated remotely using software commands.

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THE INFLUENCE OF OPTIMIZATION CRITERIA ON THE QUALITY OF RADIOCARBON DATING

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Counting is one of the most important steps in reaching accurate results in radiocarbon dating by the liquid scintillation technique. Among the factors that influence the quality of counting, the most important is the choice of the most suitable counting window. Two criteria are generally used for the selection of this instrumental condition: the maximum figure of merit and the balance point. The maximum figure of merit can be described as the greatest counting efficiency for the best background; the balance point relates to the best stability of counting efficiency among samples and through time.

In order to obtain the best counting performance, we have systematically studied the effect of the concentration in the counting solution of Bu-PBD and bis-MSB as primary and secondary

scintillators, respectively. We have used the maximum figure of merit and the figure of merit corresponding to the balance point as parameters for the evaluation of the quality of counting for each composition studied.

THE IMPACT OF ACCELERATOR DATING AT THE EARLY AGRICULTURAL SITE OF ABU HUREYRA ON THE EUPHRATES

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The early village of Abu Hureyra is significant because of its great size (ca 11.5 ha) and long sequence of occupation (ca 11,500–7000 BP) that spans the transition from late Pleistocene hunting and gathering to early Holocene farming, and the cultural change from Epipaleolithic to Neolithic. The 40 accelerator dates obtained for Abu Hureyra have produced three important results.

- 1. They have demonstrated that the site was inhabited for much longer than the few conventional radiocarbon dates for the site had suggested. The gap between the Epipaleolithic and Neolithic villages seems to have been brief.
- 2. They have dated a change in climate and vegetation during the life of the Epipaleolithic village. That change precipitated a shift in the foraging way of life of the early inhabitants just before the inception of agriculture.
- 3. Dating of individual bones and seeds has documented that the wild progenitors of sheep and several cereals were present near Abu Hureyra in the late Pleistocene, outside their present areas of distribution. This has implications for where those species may have been domesticated.

THE EFFECT OF MICROBIAL RESPIRATION ON REACTION-PATH MODELING OF GROUNDWATER AGES¹

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The respiration of heterotrophic microorganisms provides a source of dissolved inorganic carbon (DIC) that complicates groundwater age dating by ¹⁴C. Although fractionation of carbon isotopes during dissolution/precipitation reactions is well understood and can be modeled, the carbon fractionation during microbial respiration is largely undefined. Reaction-path modeling of DIC sources associated with microbial respiration of lignite in the Middendorf aquifer in South Carolina is demonstrated. Tritium, ¹⁴C and groundwater chemistry were analyzed along three flow paths. The ¹⁴C ranged from 89 pMC in the recharge zone to 9.9 pMC in the distal borehole; the δ^{13} C remained relatively constant at ~ -22‰. To account for increasing inorganic carbon and to calculate the groundwater ages, two geochemical reaction paths were modeled: lignite oxidation with calcite dissolution and lignite oxidation with carbon isotope fractionation. Carbon isotope analysis of particulate organic carbon from the core sediments was used to model the carbon isotope fractionation, and the groundwater ages obtained ranged from modern to ~ 13,000 BP.

¹Research supported by the US DOE/OHER Subsurface Science Program

MEASUREMENT OF ¹⁴C CONCENTRATIONS OF STRATOSPHERIC CO₂ BY ACCELERATOR MASS SPECTROMETRY

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Air samples of about 16 liters at normal state (1 atm, 0°C) were collected from the stratosphere on September 1, 1989, off Sanriku-Shore, Miyagi Prefecture, North Japan (39°N, 142°E). The samples were collected by a cryogenic sampling device with a liquid helium cooling system, at altitudes of 19, 20, 21, 22, 24, 26, 27, 29 and 30 km. The sampling apparatus flew to the stratosphere on an observation balloon of the Institute of Space and Astronautical Science. Carbon dioxide of a few milligrams of carbon was separated from air samples using a cryogenic method at Tohoku University, and ¹⁴C concentration of the CO₂ was measured by a Tandetron accelerator mass spectrometer at Nagoya University. An Fe-graphite target was prepared by reducing CO₂ on Fe-powder with hydrogen in a Vycol tube at 500°C. The concentrations, Δ^{14} C, of CO₂ were 270–310‰ at altitudes of 21–30 km, and 130‰ at 19–20 km. The Δ^{14} C values at 21–30 km were higher than the Δ^{14} C of the current tropospheric CO₂ of around 100–150‰.

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CALIBETH – AN INTERACTIVE COMPUTER PROGRAM FOR CALIBRATION OF RADIOCARBON DATES

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A computer program for convenient calibration of radiocarbon data has been developed. It has a simple user interface, which includes pull-down menus, windows and mouse support. All the important information, such as calibration curves, probability distribution and the results in text form, are displayed on the screen and can be rearranged by the user.

Two versions of CalibETH, one for an IBM-PC and one for the Macintosh, are available. On an IBM-PC, CalibETH runs under the graphics interface, GEM, from Digital Research.

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EVALUATION OF ACTIVE/PASSIVE GUARD MATERIALS WITH AFTER-PULSE ELECTRONICS FOR LOW-BACKGROUND LIQUID SCINTILLATION COUNTING

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Background reduction of a liquid scintillation counter has been evaluated using a spectrum of active/passive guard materials and after-pulse electronics. Guard materials possessing both active and passive properties fall under two basic categories. Passive properties center around near 100%

photon transmission at the 330-420 nm wave length. Active properties require 380-420 nm photon production with a prompt primary decay fluorescence approaching 2 nsec and a secondary delayed emission from 100-600 nsec.

Four materials meeting the above criteria have been evaluated. Their active/passive guard properties are discussed as well as their capabilities for background reduction.

¹⁴C PROFILES IN THE NORWEGIAN AND GREENLAND SEAS BY CONVENTIONAL AND AMS MEASUREMENTS

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 CO_2 in the atmosphere is the most important climate gas, and more knowledge about CO_2 uptake in the ocean is of prime importance in predicting future development. Radioactive carbon from nuclear tests, during a period of about 30 years, has been a very useful tracer in CO_2 exchange studies. Up to now, the measurements have been based mainly on conventional counting techniques with large CO_2 samples (ca 5 L). The AMS technique on small CO_2 samples (ca 5 ml) has made it much easier to perform sampling, and this development has especially stimulated the use of ¹⁴C as a tracer in the ocean.

In higher latitudes, the ocean acts as a sink for CO₂. The present paper is concerned with CO₂ (DIC), ¹⁴C and ¹³C measurements in the Norwegian and Greenland Seas. During cruises in 1989 and 1990, it was possible to obtain several ¹⁴C profiles, and also to repeat a few GEOSECS profiles from 1972. The shape of these profiles changes with time, and gives information about the mixing rate and the age of the deep water. From changes in the profiles, we see that the deep water in this area has only reached about 10–20% of the ¹⁴C concentration in the ocean surface during a period of 25 years.

SUBMERGED PLANTS AND THE SLOW RESPONSE TO CHANGES IN RADIOCARBON ACTIVITY OF ATMOSPHERIC CARBON DIOXIDE

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Various submerged plants from some lakes close to Uppsala, Sweden, have been studied for a decade. The ¹⁴C activity was lower than that of atmospheric CO_2 when the investigation started, but now it is higher for some of the plants, indicating a slow response to the present decrease of the excess due to nuclear-weapon tests. Plant species react differently to different processes of CO_2 uptake. Thus, the activity values of plants taking their CO_2 from the sediment are dependent on different conditions in the same lake. The NaOH-soluble fraction of lake sediments is the preferred fraction after normal pretreatment unless terrestrial debris can be used, or a more sophisticated pretreatment is developed and applied. Also, since emergent lake plants have different ¹⁴C activities than submerged plants, the reservoir age of lake sediments may vary throughout the history of the lake, but this also may result from the position in the lake.

¹⁴C ACTIVITY IN DIFFERENT SECTIONS AND CHEMICAL FRACTIONS OF OAK TREE RINGS, AD 1938–1981

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The ¹⁴C activity in tree rings from an oak growing in the suburbs of Uppsala, Sweden, has been studied for the period, AD 1938–1981. The results are compared with the atmospheric carbon dioxide records from Abisko, northern Sweden, where local or regional contamination from fossil-fuel combustion can be neglected. We have studied the influence from different chemical pretreatment procedures in use. HCI-NaOH-HCI treatment is compared with cellulose extraction. We have also analyzed the extract. Each ring has been split into two samples corresponding to early (spring) and late wood. A more refined partitioning has been applied to the years 1963 and 1964. The ¹⁴C measurements were made at the Uppsala AMS facility whereas δ^{13} C was determined with a conventional mass spectrometer. A cellulose yield of ca 25% and 35% was obtained for the early and late wood, respectively. We observed a delay in the ¹⁴C activity response in the tree rings compared with the activity in the atmosphere.

RADIOCARBON GEOCHRONOLOGY OF THE LARSEN COVE, MARAMBIO ISLAND (SEYMOUR ISLAND), EAST OF THE ANTARCTIC PENINSULA

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On the northern coast of the Larsen Cove, Marambio Island (Seymour Island), periglacial landforms, such as frost domes, ice wedges, ice-wedge casts, gelifluction terraces and lobes have developed, along with raised beach topography at several levels along the coast. In order to estimate formative ages of frost domes and lower raised beaches, five algae samples were collected from the coastal sediments and radiocarbon dated. The results revealed that the frost domes were formed about 2200 BP, whereas the lower marine terrace was formed about 3100 BP. The figure of reservoir correction for the surveyed area was about 1000 years.

RADIOCARBON DATING OF OYLUM HÜYÜK, TURKEY

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Radiocarbon dates were determined from charcoal and bones from an excavation at Oylum Hüyük, Turkey just north of Aleppo, Syria. This site is a large tell located strategically on trade routes linking Turkey, Mesopotamia and the general Levant. These radiocarbon dates provide, for the first time, a chronological correlation of this brand-new site with the surrounding Anatolian, Mesopotamian and Egyptian chronologies, using calibrated radiocarbon measurements.

LOW-LEVEL LIQUID SCINTILLATION COUNTING FOR RADIOCARBON DATING

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Recently, liquid scintillation counting (LSC) has become the method of choice for radiocarbon dating. In order to perform radiocarbon dating, it is essential to optimize the instrument performance (E^2/B). This can be accomplished by maximizing the counting efficiency and minimizing the instrument background. This maximum instrument performance can be achieved using the patented Time-Resolved LSC (TR-LSC) technique on a modern Packard liquid scintillation counter. The TR-LSC technique rejects most nonquenchable background pulses and accepts valid scintillation pulses which result from the ¹⁴C in the sample. The optimization of this technique, in regard to sample preparation, cocktail composition and counting regions will be discussed in detail, as well as the use of a new slow fluor plastic guard detector. This detector helps to further reduce background without substantially changing the counting efficiency of the sample. Data will be presented to support the use of the TR-LSC technique for radiocarbon dating.

DISTRIBUTION OF BOMB RADIOCARBON IN THE ANTARCTIC OCEAN: IMPLICATIONS REGARDING IRON FERTILIZATION

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The stimulation of plant production in the nutrient-rich surface waters of the Antarctic Ocean through the addition of dissolved iron was proposed as an effective way to partially compensate the rise in atmospheric CO₂ content generated by fossil-fuel burning and deforestation. A successful fertilization would drop the CO₂ partial pressure (pCO₂) in Antarctic surface waters, and CO₂ would flow from the atmosphere into the surface waters of the Antarctic. However, the atmospheric pCO₂ drop would be buffered by the CO₂ outflow from the non-Antarctic portion of the surface ocean. To draw down the atmospheric pCO₂ to that for the iron-fertilized Antarctic, it would be necessary also to draw down the ΣCO_2 content of the surface layer and part of the underlying thermocline for the entire non-Antarctic portion of the ocean. This CO₂ would all have to be crammed into the Antarctic water column. Inventories of bomb-produced ¹⁴C in the Antarctic Ocean, as measured during the GEOSECS surveys of the world ocean, show that the Antarctic waters contain far less bomb ¹⁴C than invaded this region from the atmosphere. By contrast, the temperate regions of the southern hemisphere ocean have inventories exceeding the amounts received by invasion. The explanation for these deficiencies in the Antarctic Ocean is upwelling coupled with lateral transport to adjacent temperate regions. If this is true, only a limited volume on top of the upwelling plume would be available for CO_2 storage. The CO_2 delivered to the Antarctic from the atmosphere and non-Antarctic surface ocean would drive the pCO2 of Antarctic

surface water back up toward the value dictated by the warm surface ocean. Results of calculations based on the distribution of bomb ¹⁴C reveal that, after 100 years of totally successful fertilization, the atmosphere's CO₂ content would be lowered by 30 ± 15 ppm. Thus, if a century from now, the atmosphere's CO₂ content were 500 ppm without fertilization, with full fertilization over the entire century, it would instead be between 455 and 485 ppm.

A NEW RADIOCARBON DATING LABORATORY IN BRAZIL

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A new radiocarbon dating laboratory was established in 1989 at the Centre for Nuclear Energy in Agriculture (CENA), University of São Paulo, Campus of Piracicaba. The purpose of the laboratory is to cover at least partially the rapidly growing demand for radiocarbon analyses in Brazil, mainly connected with ongoing large environmental programs, eg, IAEA/010, isotope-aided studies of the Brazilian Amazon.

The laboratory utilizes the benzene synthesis method and liquid scintillation counting. The yield of benzene synthesis is in the range of 82%-91%. The counting efficiency of the liquid scintillation spectrometer is about 56%, and the background for 3 ml sample size about 1.10 cpm. The available instrumentation allows the analysis of up to 150 samples per year, with an average precision of approximately 1.0 pmc. The intercalibration cross-check with several radiocarbon laboratories revealed agreement of the reported dates within the quoted analytical error.

At present, our efforts are directed towards increasing the analytical capacity of the laboratory through the acquisition of new equipment, improving the precision and accuracy of the analyses and initiating paleoenvironmental research programs in Brazil.

	Inter	comparis	son results (yr	s BP)		
Sample	CENA*	δ ¹³ C ‰	GIF**	GEOC[†]	WAT [‡]	δ ¹³ C ‰
Shell (Carat.1)	4020 ± 90	-1.7	4040 ± 90	-		-
Shell (Carat.2) ^s	4780 ± 90	-1.4	4630 ± 100	-		-
Charcoal (CSP-2) [§]	1100 ± 100	-26.9	1180 ± 60	-		-
Wood (Ba-284)	3710 ± 80	-26.9	-	3700 ± 90		-
Wood (GA)	7630 ± 90	-24.8	-	7735 ± 70		-
Coral (11-3)	3760 ± 70	-1.5	-	-	3630 ± 90	-1.7
Coral (C-21)	6260 ± 80	-0.8	-	-	6240 ± 90	-0.9
Wood (10793)	$10,900 \pm 100$	-25.8	-	-	10,900 ± 100	-25.1
Wood (16035)	$10,100 \pm 90$	-25.0	-	-	10,300 ± 80	-24.6
Wood (16034) ^{\$}	$10,200 \pm 120$	-23.7	-	-	10,400 ± 90	-25.4

Intercomparison results (yrs BP)

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⁴Samples diluted with spectrographic benzene

SOLAR SIGNALS FROM ¹⁴C IN TREE RINGS

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The ¹⁴C record in wines has shown many interesting features. The strongest signal which is imprinted in it comes from the 11-year solar cycle. Another possible feature could be a frequency modulation of solar origin, also detectable, despite the limited length of the record (carrier period 11 years, modulating period 22 years).

The solar signature is also present in annual tree rings, though weaker; the length of the record is short, in any case. Long tree-ring records are now available in decadal and bi-decadal sequences. However, as it is well known, conventional spectral analysis of such temporal sequences, is affected by aliasing, which is always present in equispaced sequences where a signal lies beyond the Nyquist frequency. Thus, it is difficult to clearly define the real presence of solar cycles on the scale of centuries. Aliasing can be avoided in the analysis of unequispaced time sequences. On the other hand, unequispaced sequences ask for other more sophisticated treatments, such as Bayesian analysis.

A comparison is carried out between the detectability of signals of solar origin in unequispaced and equispaced sequences, when the latter contain aliased frequencies.

A frequency modulation model for wines is compared with an annual sequence in tree rings, for the last 400 years.

DETERMINING RECURRENCE INTERVALS OF GREAT SUBDUCTION ZONE EARTHQUAKES IN SOUTHERN ALASKA BY RADIOCARBON DATING

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Rupture on the eastern segment of the Aleutian arc subduction zone produced the great 1964 Alaska earthquake and caused vertical and horizontal tectonic displacements over more than 140,000 km². ¹⁴C dates from subaerial peat and forest layers submerged below the pre-1964 hightide level indicate the 1964 earthquake was preceded by gradual regional interseismic subsidence of 2.5-9.5 mm/yr for at least 800 years. Older ¹⁴C dates from buried layers of peat and rooted tree stumps yield recurrence intervals for 1964-type tectonic events of about 600-950 years and interseismic subsidence rates of 3-5 mm/yr over the past 3000 years in the area of Copper River delta on the mainland. ¹⁴C dates from peat and driftwood on emergent marine terraces on Middleton Island at the outer edge of the continental shelf in the Gulf of Alaska yield recurrence intervals for major tectonic events of 400-1300 years over the past 5000 years and an average uplift rate of 10-11 mm/yr.

GEOCHEMICAL MODELING AND RADIOCARBON DATING OF GROUNDWATER: RECENT SOFTWARE DEVELOPMENTS AND FIELD EXAMPLES

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Geochemical reaction models are usually required for radiocarbon dating of groundwater. The adjustment of ¹⁴C data for geochemical reactions by the inverse modeling approach has been generalized and put into the interactive FORTRAN 77 code, NETPATH. NETPATH solves net chemical and isotope mass balance and isotope evolution in a groundwater system using observed chemical and isotopic data. The processes of dissolution, precipitation, oxidation/reduction, incongruent reaction, gas exchange, mixing, evaporation, dilution, isotope fractionation and isotope exchange can be considered. Given a set of chemical and isotopic constraints, and a set of plausible reactant and/or product phases for the system, NETPATH computes every possible geochemical mass balance reaction for an observed initial and final water composition along a flow path. Rayleigh distillation calculations are applied to each geochemical reaction model to predict carbon, sulfur and strontium isotopic compositions at the endpoint, including the reaction-adjusted ¹⁴C content. Reaction identification and assignment of adjusted ¹⁴C ages is usually non-unique because of a lack of sufficient mineralogic and kinetic data for the system, and a lack of definition of the initial ¹⁴C content. Examples of the application of NETPATH to ¹⁴C dating of groundwater in the Floridian aquifer of Georgia and Florida, USA are presented.

¹⁴C GAS COUNTING: IS THERE STILL A FUTURE?

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Reviewed here are developments in low background proportional counters and chambers used for low-level internal gas counting of ¹⁴C and other soft beta emitters. I have studied the influence of counter geometry and the nature of the gas filling on counter characteristics and describe in detail operational characteristics of multi-element proportional chambers. Further detector developments for ultra-low-level gas counting based on cryogenic liquids and time projection chambers are discussed. My observations show that proportional and drift chambers together with multichannel electronics operating on-line with a computer open up new possibilities in ultra-low-level gas counting.

¹⁴C MEASUREMENTS IN OAK TREE RINGS AROUND 8000 BP

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During excavations for a hydroelectric power plant on the Danube River south of Bratislava, oak trees were found about 20 m below the surface. We present here results of ¹⁴C measurements, made in the Bratislava and Tbilisi ¹⁴C laboratories, on single tree rings of one of these oak trees.

We used proportional counting and liquid scintillation counting for ¹⁴C analysis of cellulose prepared from wood samples. The obtained ¹⁴C results are compared with similar measurements of ¹⁴C in tree-ring samples from central Europe.

RADIOCARBON DATING IN PALEOECOLOGICAL STUDIES

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One of the most acute problems in environmental studies is assessing human and natural influences on the development of geoecosystems. To obtain reliable results, we must first construct an accurate time scale. For this, radiocarbon dating is the most widely used. In this paper, I analyze the problems and remedies in high-precision paleoecological investigations. Because of different geneses and structures, the main sources of paleoecological information, bog and lake deposits, should be observed separately.

Bog deposits present the following problems:

- 1. Gathering material for dating from a definite layer
- 2. Separating admixtures of different ages migrating in water from the material dated
- 3. Separating roots and rhizomes penetrating from upper layers into the layer being dated, and estimating their influence
- 4. Extrapolating ages in the sections between the dated layers.

The situation is more complicated in dating lake deposits. "Hard water effect" and allochthonous material cause deviations of ¹⁴C ages of 2000–2500 years. Elaborating geochemical or limnological criteria that enables us to estimate the reliability of results has not been successful up to now. For this reason, I have used palynological methods along with geochemical and limnological data. The pollen diagram for a bog situated in the vicinity of a lake is divided mathematically into zones, the borders of which are ¹⁴C dated. The ages are then extrapolated to the zone boundaries of the lacustrine pollen diagram. Naturally, a time scale obtained in such a manner is not absolutely reliable.

DATING GROUNDWATER IN COASTAL PLAIN AQUIFERS IN SOUTHERN MARYLAND USING DOC AND DIC ¹⁴C AGES: AN EVALUATION BY COMPARISON WITH OTHER DATING METHODS

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The Aquia (Paleocene) and Magothy (Late Cretaceous) Formations of the Atlantic Coastal Plain represent two of the most productive aquifers in southern Maryland. We have made ¹⁴C measurements using accelerator mass spectrometry (AMS) on dissolved organic (DOC) and inorganic carbon (DIC) of Magothy and Aquia groundwaters. The DOC ¹⁴C ages are consistently younger than the DIC ¹⁴C ages at each sampling site, as expected, based on the bicarbonate (2.4–4.1 mM) concentrations in these waters. Once the DIC ¹⁴C ages have been corrected for addition of dead carbon using a mass balance approach, both DIC and DOC ¹⁴C ages are similar. However, a DOC ¹⁴C age of 7270 years in the recharge region of the Aquia is unexpectedly high. Both bomb ³⁶Cl and ³H have been measured at the same site (Purdy 1991). Once downgradient DOC ¹⁴C ages have been corrected for recharge DOC ¹⁴C values, an approximate flow rate of 3.8 m/yr has been extrapolated over the entire aquifer based on these ages.

Independent age estimates were obtained from Cl⁻, ¹⁸O and ²H distributions. All three species vary in a systematic manner and are expected to reflect the temporal, meteoric changes in precipitation influenced by sea-level shifts over the past 50,000 years. Groundwater with minimum Cl⁻, ¹⁸O and ²H values has been correlated with rainfall of 18,000 BP, when the recharge region was at a maximum distance from the coast during the most recent low sea-level stand. A flow rate based on this age is approximately 1.8 m/yr. A third dating approach involving computer modeling using hydrodynamic parameters (Fleck & Vroblesky, in press) yields a flow rate of 4.8 m/yr.

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A PRECISION HIGH-THROUGHPUT ACCELERATOR MASS SPECTROMETER FOR ¹⁴C

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We will describe the performance of a newly installed ¹⁴C accelerator mass spectrometer which simultaneously measures all three carbon isotopes (masses 12, 13 and 14), at an energy of 10 MeV. Using a graphite sample prepared from recent oxalic acid, ¹⁴C counting rates in excess of 150 counts/second are routinely recorded in the final detector. The pulse-height spectrum of these events indicates that no scattered ¹²C and ¹³C ratios indicate constancy at the level of 2‰. We will

describe automatic setup and tuning routines, which are designed to allow the total AMS system to be set up under computer supervision. Critical elements are subsequently auto-tuned so that each section of the instrument operates in a flat-topped region.

THE GEOCHRONOLOGY OF SEDIMENTS IN LAKES OF WEST CHINA

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The Xinjiong and Tibet plateau are located in the west of China where there are a number of inland lakes that are the highest above sea level and the largest of their kind in the world. Inexhaustible supplies of minerals, such as halite, glauber salt, strintium minerals, borates and sylvine have accumulated in these salt lakes.

We report here the ¹⁴C dating of several saline lakes on the Xinjiong and Tibet plateau and their cycles of sedimentation within an estimated period of about 50,000 years. From the data obtained, we consider it appropriate to draw the boundary in the studied area between the Pleistocene and the Holocene at about 10,000 BP. In this paper, we discuss the changes of paleoclimate, which will be useful background information for studying and forecasting future climate change.

¹⁰Be PROFILES AS A STRATIGRAPHIC TOOL

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Using accelerator mass spectrometry (AMS), it is now possible to measure detailed profiles of ¹⁰Be in ice cores and marine sediments. In addition to providing information on the parameters (primary cosmic-ray intensity, solar modulation, geomagnetic modulation) which control cosmogenic production rates, such data may be useful as a stratigraphic tool for establishing relative and/or absolute chronologies for the reservoirs in which they are measured.

We have now measured detailed ¹⁰Be profiles in several ice and sediment cores covering all or part of the last climatic cycle (~100,000 years). We will discuss these results in terms of cosmogenic production rate variations, and their potential application to establishing chronologies, as mentioned above.

RADIOCARBON AGES OF CARBONATE MATERIALS FROM GANGETIC ALLUVIUM

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The subsurface stratigraphy of the central alluvial plain of the Indo-Gangetic Plain contains lenticular beds of nodular concretions made up of calcareous sediments (Kankar). These deposits were formed by diagenetic processes in response to fluvial action and climate of the region. In the absence of organic materials, the soil carbonates were radiocarbon dated to derive the rate of sedimentation in the Gangetic plain.

¹⁴C ages of calcareous nodules recovered from deep borings at Ranipur, Kanpur and Fatehpur are reported here. The average rate of sedimentation derived for the southern part of the central

alluvial plain is 2.4 cm/100 years. The ¹⁴C ages of thick deposits of Kankar also coincide with the glacial periods at 18,000 and 30,000 years. Alluvial drowning and meandering of the rivers in the Gangetic Plain have left in their wake many abandoned channels, ponds and lakes. Radiocarbon ages of marl deposit (shell layer) from an abandoned channel in Unnao district suggest that this process may be in response to sea-level rise during the early Holocene. The sedimentation rate for this channel has been calculated as 0.7 cm/100 years for the 40-cm-thick shell layer.

ON THE POSSIBILITIES OF USING THE ¹⁴C METHOD IN THE STUDY OF LATE PLEISTOCENE GEOCHRONOLOGY

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The use of radiocarbon dates in the study of Late Pleistocene geochronology is accompanied by two permanent problems: reliability of "old" finite ages and real precision of dates of Middle Weichselian age. The latter is much discussed in the study of the development of the last Scandinavian glaciation in the Russian Plain in East Europe. Skepticism about ¹⁴C dates is caused by objective and subjective reasons. One of the objective reasons – the presence of allochtonous material, leads to controversial results within a geological section, but in some cases, allows us to obtain additional information on the genesis of the studied deposits. From among the large number of subjective reasons for more serious mistakes, the leading dangers are the inability to avoid contamination and to measure very low radioactivity of samples.

RADIOCARBON DATING: A DIDACTIC VIDEOTAPE

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In order to familiarize archaeologists with the basics of radiocarbon dating and to acquaint users with the experimental procedure, we prepared a videotape in which radiocarbon dating by liquid scintillation counting is explained. The exposition is divided into the following sections:

- Geochemical basis of radiocarbon dating considering the carbon isotopes, their distribution, formation and disappearance of radiocarbon
- Measurement of ¹⁴C activity by liquid scintillation, explaining its basis
- Treatment of samples
- Synthesis of benzene
- Necessity of using a contemporary standard
- Age calculation and its range; justification of the uncertainty
- Calibration of radiocarbon ages and its justification

RADIOCARBON DATING IN THE SAN PEDRO VALLEY, SOUTHEASTERN ARIZONA

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In the San Pedro Valley in eastern Arizona, ¹⁴C measurements and major element chemistry were used to construct a geochemical framework for the interpretation of basin hydrology. Residence

times in the upper and lower San Pedro basins, after correcting the measurements for the groundwater chemistry, were greater than 12,000 years. The ¹⁴C data show the presence of a regional confined aquifer, a closed hydrological system and a locally extensive unconfined aquifer, an open system. ¹⁴C, ³H, δD and $\delta^{18}O$ depict areas of recharge, discharge, mixing and direction of flow. The ¹⁴C groundwater ages do not increase in a downvalley direction, the assumed direction of groundwater movement in most of the intermontane basins in the region. Recharge to the confined aquifer in the lower San Pedro basin is from precipitation falling on the Galiuro Mountains along the eastern part of the valley. Recharge occurs along the entire mountain front near the alluvium-mountain contact and, subsequently, as groundwater flows normally to the axis of the mountains and discharges along the center of the basin. Where the aquifer is unconfined, recharge to the groundwater occurs over a much larger area of the basin with increasing mixing in the direction of flow. The hydrogeological concept of the isotope model is fully supprted by the water chemistry and by the reactions and associated mass transfer defined by the chemical model. Weathering of primary silicate minerals also does not increase in a downvalley direction but along the direction of flow.

PULSE-HEIGHT ANALYSIS FOR GAS PROPORTIONAL COUNTERS

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Pulse-height analysis can be combined with conventional gas proportional counting of radiocarbon to provide: 1) reduced background levels; 2) more sensitive monitoring of gas purity and plateau slope effects; 3) detection of radon contamination; 4) improved evaluation of guard counter performance, etc. I have investigated the performance of a prototype PHA system in the underground Menlo Park laboratory (cosmic-ray flux 0.28 cpm/cm²) using a low-cost microcomputer (80286-16) to collect pulse-height spectra from four counters simultaneously. I found that 15-20% of the background pulses lie above the end-point of the ¹⁴C spectrum. Variations in gas multiplication were monitored via the cosmic-ray spectrum, which exhibits a pronounced peak corresponding to the energy deposited by a meson crossing the diameter of the counter tube. The position of this peak is used to adjust the ¹⁴C window in the anticoincidence spectrum. This procedure was found to reduce the plateau slope of a counter with $S_0 = 44$ cpm from 0.75%/100 volts to 0.2%/100 volts. About half of the excess pulses in a gas sample containing radon were found to lie above the ¹⁴C window, allowing the detection of radon. The measurements also revealed a possible defect in the guard counter array, as two counters showed an imprint of the cosmic-ray spectrum in their background ¹⁴C spectra. It will be of interest to compare these results with measurements done in a ground-surface laboratory.

CARBONATE CONTAMINATION REVISITED

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Bard *et al* (1990) have recently presented a set of uranium series disequilibrium dates on a stratigraphic sequence of previously radiocarbon dated coral samples (Fairbanks 1989). The authors present the hypothesis that the discrepancies between the two sets of dates contain information on the history of radiocarbon secular variations. These data can also be interpreted in light of a simple model of carbonate contamination (Robinson 1980, 1981). The model suggests

incorporation of radiocarbon from a source close to modern by a first-order rate process. The possible values of the rate constant were evaluated from the population of carbonate dates obtained by the Menlo Park laboratory during the first few years of its operation. Although the dating limit imposed by the apparatus was about 52,000 years, carbonate materials from a variety of environments yielded a maximum age of about 40,000 years. In certain less favorable situations, the maximum ages obtained were only in the 25,000–30,000-year range. Unfortunately, coral samples were not available for inclusion in the data set used to calibrate the model. A contamination rate constant that would give a limiting age of about 30,000 years well reproduces the radiocarbon *versus* absolute (U/Th) age variations in the Bard/Fairbanks data. Unfortunately, these workers did not attempt to measure the contamination in their corals (by radiocarbon dating the last interglacial samples). Perhaps the background levels available at their laboratory were not sufficiently low to make this feasible.

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RADIOCARBON DATING OF PREHISTORIC ROCK PAINTINGS

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Prehistoric pictographs occur worldwide, but until recently, they could not be dated directly. We have developed a new method that allows direct ¹⁴C dates to be obtained on these artifacts. The technique is applicable even to paintings that occur on limestone (CaCO₃) walls. Preliminary work has been done on a pictograph from the Lower Pecos region of southwest Texas. A sample of the painting, which had spalled from the shelter wall by natural processes, was subjected to a low temperature (<150°C) oxygen plasma to selectively remove the organic carbon-containing material used in the paint without contamination from the limestone substrate. The primary product of the plasma reaction is CO₂, which was isolated and converted to graphite. The Zürich ETH accelerator mass spectrometer was then used to determine the ¹⁴C content of the sample. An age of 3865 ± 100 BP was obtained, and is in good accord with the archaeological context, which has set the range between 2000 and 6000 BP. This technique is applicable to rock art samples which incorporated organic matter into the paint binder or vehicle. This preliminary result indicates that the method is feasible and can be used on rock art worldwide.

25 YEARS OF RADIOCARBON DATING SOILS: A PARADIGM OF ERRING AND LEARNING

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Soil organic matter sequesters about three times the carbon existing totally in the living biomass and nearly the same regarding the total carbon in the atmosphere. Using models, such as Jenkinson's and, presently more widely used, Parton's Century Model, soil organic matter fractions of different functions are defined, based on their ¹⁴C ages. Rejuvenation of soil carbon was believed the principal impediment for absolute soil dating. Recent studies, eg, Becker-Heidmann (1989) indicate that a soil ¹⁴C age of > 1000 years cannot have more than 0.1% carbon rejuvenation. Earlier observations were problematic, with age *versus* depth increase in ¹⁴C profile curves showing an inflection of reduced age in the deepest samples, *ie*, the rim of the organic carbon-containing epipedon. Our ¹⁴C dating of earthworms and study of their feeding habits could explain this phenomenon in mollic horizons. Becker-Heidmann (1989) showed, in a thin-layer soil profile dating, a highly significant correlation between the highest ¹⁴C age and the highest clay content in the profiles. Thus, optimization of soil dating is, to a lesser degree, related to the used extraction solvent then to soil texture fractions. Examples will be presented. Our observations tend to mitigate error ranges inherent in dating dynamic systems such as soils, with their eluviation, illuviation and turbation processes.

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THE ORIGIN AND TURNOVER OF DISSOLVED ORGANIC CARBON IN FORESTED WATERSHEDS DETERMINED BY CARBON ISOTOPIC (¹⁴C AND ¹³C) MEASUREMENTS

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Dissolved organic carbon (DOC) is important in the acid-base chemistry of fresh waters and in determining the mobility, persistence and toxicity of metals and pollutants in natural waters. Yet, very little is known about the origins and cycling of DOC in natural watersheds. In this study, carbon isotopes (¹³C and ¹⁴C) are used to study the origins, transport and fate of DOC in a small, acid-stressed watershed near Haliburton, Ontario. Carbon isotopic measurements were made on DOC from precipitation, streams, soil A and B horizon percolates, shallow and deep groundwaters, bogs and lake water. In addition, ¹³C and ¹⁴C measurements of soil organic matter, phytoplankton and sediments were made to determine the relative importance of these potential DOC sources. These data were used to construct whole watershed mass balances for DOC. Analyses of DO¹⁴C, only recently possible with the aid of accelerator mass spectrometry (AMS), indicate that a large fraction of the DOC turns over rapidly (50% in about 40 years). ¹⁴C measurements of humic and fulvic acid fractions of the DOC indicate differences in the origin and turnover of these components.

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PRELIMINARY MEASUREMENT OF RADIOCARBON IN BLACK CARBON FROM MARINE SEDIMENTS

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Black carbon (or soot) is found throughout the troposphere and becomes incorporated into marine sediments. Natural fires were the primary preindustrial source of this material. Soot forms a distinctive component of marine sediments, characterized by insolubility in hydrochloric and hydrofluoric acids and low density ($<2g/cm^{-3}$). Although they represent a very minor fraction of total sedimentation, black carbon fluxes as great as 3 μ g cm⁻² yr⁻¹ have been recorded in the bottom of the sea, and adequate quantities for AMS dating can readily be extracted from many places on the sea floor.

If this carbon were principally derived from contemporary ¹⁴C, and were delivered into the sediment relatively promptly, this would provide datable material in many marine cores, even those free of carbonates; further, comparison of black carbon with benthic and planktonic foraminifera in calcareous cores would provide a paleoindicator of atmospheric, mixed-layer and deep-sea carbon reservoirs. Black carbon has been separated by flotation from North Atlantic marine sediments, treated by various means to remove adsorbed organics, and converted to AMS targets by the Vogel method. Initial results gave somewhat greater ages than associated calcareous materials, and slightly older than, or equal to, associated organics. Further investigations are underway.

DETERMINATION OF RADIOCARBON IN AUSTRIAN WINE AND VINEGAR

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A very simple, yet sensitive method of determining ¹⁴C (and tritium) in wine, using a commercially available ultra-low-level liquid scintillation counter has previously been described (Schönhofer 1989). The analytical parameters have been investigated and optimized. Now, a complete set of wine samples from 1955 to 1987 has become available from the HBLVA Klosterneuburg (a wine-making school), near Vienna. The samples are all from the same well-known area and are guaranteed to be from the respective year, and not mixed with wine from another year's harvest. The counting efficiency was determined by addition of an internal standard; thus, precision was greatly enhanced. Results showed that the type of grape had no influence on the ¹⁴C concentration. Using the obtained time-dependent concentration curve, the age of samples could be determined successfully.

Acetic acid was extracted from vinegar by solvent extraction. It was possible to determine ¹⁴C in acetic acid as it is done in alcohol, after optimizing parameters, such as the scintillation cocktail, mixing ratio and volume. With one exception, all the samples of analytical-grade acetic acid for laboratory use showed a ¹⁴C concentration typical for the environmental concentration of about

1976. However, it can be assumed that the reagents are not produced from biological sources. Some samples of vinegar that were supposedly produced from wine showed the same (too high) ¹⁴C concentration; some contained the same concentration as wine of recent harvest. Also, samples of acetic acid produced from wood, technical-grade acetic acid and one from "ancient times" were analyzed. The results are discussed and a possible explanation is given.

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ESR AND U-SERIES DATING OF PREHISTORIC SITES IN THE EASTERN MEDITERRANEAN

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The Eastern Mediterranean has been a thoroughfare for hominids since the time of the Acheulian culture. Sites that were occupied by the earlier populations (Middle and Lower Paleolithic) are beyond the range of ¹⁴C, but can now be dated effectively by trapped charge techniques (TL, OSL, ESR), and in some cases, by U-series. A consistent chronology based on TL dates on burned flint and ESR on tooth enamel have been obtained at the Early Modern human site of Qafzeh, the Neanderthal site of Kebara and the Acheulian site of Yabrud. ESR dates for Skhul and Tabun have been obtained using museum specimens of teeth. The addition of U-series analyses of tooth enamel will further refine these dates. Concordance can be tested with high-precision mass spectrometric U-series on stalagmites at a few sites (eg, Hayonim and Nahr Ibrahim). ESR is uniquely applicable to the oldest sites (Ubeidiya, Gesher Benot Yaacov), whereas OSL may be useful for some coastal sites (eg, Evron).

FURTHER ANALYSIS OF THE INTERNATIONAL INTERCOMPARISON STUDY (ICS)

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The major findings of the ICS have already been published (Scott, Long & Kra 1990), but a number of questions remain unresolved. In this paper, we address a number of key issues broadly defined into two categories of user and technical relevance. These include:

- 1. The analysis of the known-age wood samples provided in Stages 2 and 3 of the ICS
- 2. Further investigation of the quoted errors and their relationship with the perceived precision and accuracy, which are of interest to users of ¹⁴C dates
- 3. The influence of method of pretreatment and investigation of the corresponding $\delta^{13}C$ data base, of more technical relevance to laboratories.

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ANNOUNCEMENT OF A FURTHER INTERCOMPARISON EXERCISE

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Following recommendations of the Glasgow International Workshop on Intercomparison of Radiocarbon Laboratories (Scott, Long & Kra 1990), a further international intercomparison is planned. This new intercomparison is complementary to the existing IAEA intercalibration and will make use of natural samples whose ages will be unknown to the participants. The study has been funded by the UK Research Councils (SERC and NERC), and samples will be free to all participants.

We anticipate that this intercomparison will be ongoing, with distribution of samples in late 1991 and presentation of the results at a later meeting.

The paper will present full details of the samples available and the time scale of the study. Briefly, we envisage that the new study will be more focused than the ICS (Scott, Long & Kra 1990), and will include natural samples in both pretreated and unpretreated forms.

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PAST VARIATIONS OF δ^{13} C IN ATMOSPHERIC CO₂ RECONSTRUCTED FROM ANALYSES OF POLAR ICE CORES

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 δ^{13} C of atmospheric CO₂ has changed in the past 200 years due to combustion of fossil fuels and deforestation, which both yield CO₂ with lower δ^{13} C than that of atmospheric CO₂. Knowledge of the time history of this isotopic signature helps us to understand the carbon cycle and its anthropogenic perturbation. We have developed, in our laboratory, a technique to extract CO₂ from air trapped in polar ice and to analyze its stable isotopic composition. We have measured δ^{13} C and δ^{18} O on CO₂ samples extracted from a shallow ice core from Dye 3, Greenland, covering the time since about 1800. Gravitational separation can enrich the isotopically heavier gases in the firm and thus lead to an enrichment of 13 CO₂. Therefore, we have also measured by mass spectrometry the isotopic composition of oxygen and nitrogen in the trapped air and corrected the measured δ^{13} C results accordingly for gravitational enrichment. The results agree relatively well with those obtained earlier by Friedli *et al* (1987) on an ice core from Siple Station, Antarctica. The data will be compared with the anthropogenic δ^{13} C decrease as expected from carbon cycle model simulations.

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RADIOCARBON AND ARCHAEOLOGICAL CONTEMPORANEITY

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The identification of contemporaneity within sets of archaeological assemblages is crucial for the archaeological examination of cultural processes over time and space. Conventional radiocarbon dating has rarely led to satisfactory resolution of the contemporaneity problem. An AMS age determination program using maize kernels from single-component Mohawk Iroquois sites of brief duration is expected to solve the problem for the 16th and 17th centuries. Variations in the calibration curve and clues from the archaeological record combine to permit dating to within limits narrow enough to resolve contemporaneity within the sequence. This, in turn, allows analysis of demographic change during the period, especially epidemic-induced depopulation that followed 1634.

THE RESERVOIR EFFECT IN THE COASTAL WATERS OF PORTUGAL AND ITS VARIABILITY DURING THE HOLOCENE

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Radiocarbon dating on marine shells collected alive before 1950 from western and southern coasts of Portugal has indicated that the differences, ΔR , in reservoir age of these regional areas of the Atlantic Ocean and the reservoir age of the model of Stuiver, Pearson and Braziunas (1986) are 280 ± 35 years and 235 ± 25 years, respectively. The corresponding values of the apparent age of marine shells are 700 ± 35 years (western coast) and 660 ± 25 years (southern coast). These values suggest the occurrence, in those areas, of an active upwelling similar to that existing in coastal waters of California (USA).

On the other hand, radiocarbon dating on marine shells and on charred wood or bones collected from the same level (and closely associated) at various archaeological sites of Portugal, representing different periods of time in the Holocene, has shown that before 1200 BP, the mean apparent age was 360 ± 35 years, which is representative for all the Portuguese coastline, and corresponds to a coastal environment weakly influenced by the upwelling of deep water. After 1100 BP, it would have increased to the values referred to above. These results suggest that between 1200 and 1100 BP, a change in some climatological parameters along the Portuguese coast might have occurred, causing a significative intensification of coastal upwelling off Portugal.

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THE VOSTOK ¹⁰Be SPIKES AND AN INTERSTELLAR SHOCK WAVE?

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¹⁰Be in the Vostok ice core discloses two prominent increases not accompanied by corresponding ¹⁸O enhancements. Raisbeck *et al* (1987) attribute these to cosmic-ray (CR) increases from geomagnetic field reversals or excursions. Sonett *et al* (1987) alternatively propose CR increases from propagating supernova shells. A possible source is the North Polar Spur (NPS) (Davelaar *et al* 1980). The recent uranium/thorium dating of Barbados corals implies radiocarbon ages that are too young (Bard *et al* 1990). This is in accord qualitatively with a ¹⁴C burst. We review whether the atmosphere inventory memory is consistent with reasonable ocean-atmosphere transfer rates.

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ACCURACY, PRECISION AND THROUGHPUT OF ¹⁴C MEASUREMENTS AT THE CENTER FOR ACCELERATOR MASS SPECTROMETRY (AMS)

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The AMS facility at Lawrence Livermore National Laboratory (LLNL) has been making ¹⁴C measurements for over one year. During that year, measurement precision, specified as the deviation among multiple measures on single samples, varied between 1% and 5%. Accuracy has remained within the stated precision during most tests, indicating that the errors are seldom systematic. The sources of the imprecisions are being discovered and successively eliminated. The stability of the accelerator has been a limiting factor, which improves as the accelerator is conditioned to higher voltages and better control is implemented. The output of the multisample, high-intensity ion source and its emittance have been improved through replacement of the ionizer and modification of the source geometry. Further modifications are planned to better couple the ion source to the isotope injection optics. ¹⁴C count rates have been in excess of 250 cps for modern material. Less than a minute is required to make individual measurements to 1% counting precision for contemporary samples. We introduced an efficient program for collecting multiparameter data on a Macintosh II CX, which stores summarized data directly into a spreadsheet format for rapid summation and analysis. Over 500 unknown samples for archaeology, atmospheric science, biomedicine, geology and oceanography have been measured in the first year of operation.

RADIOCARBON MEASUREMENTS IN SOUTH PACIFIC OCEAN WATERS IN THE VICINITY OF THE SUBTROPICAL CONVERGENCE ZONE

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Radiocarbon distribution profiles have been measured along three transects in the southern Pacific, two east of New Zealand and one east of Tasmania. Use of accelerator mass spectrometry (AMS), with its small-sample-size capability, made it possible to sample near-surface waters with a depth resolution of a few tens of meters. Sampling of deeper water was guided by salinity and temperature data transmitted by a CTD probe. In the case of measurements taken over the Chatham Rise, east of New Zealand, the radiocarbon profiles are highly structured, and can be correlated with known circulation patterns in this region. The other two sets of data are less influenced by the local bathymetry, and show profiles more typical of the deep ocean. The results will be discussed in terms of the rate of penetration of bomb-radiocarbon they imply, and their significance for ocean-atmosphere exchange of carbon dioxide.

THE INFLUENCE OF RADON IN BENZENE SYNTHESIS FOR RADIOCARBON AGE DATING

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Previous research presented at the 1988 Dubrovnik Conference documented the interference caused by the incorporation of ²²⁶Ra and ²²²Rn as natural contaminants in radiocarbon age dating using very low background liquid scintillation counting techniques (Hood *et al* 1989). Although it was demonstrated that the use of vacuum and dry-ice alcohol temperatures during the catalyst extraction of sample benzene appeared to eliminate the radon, it raised concern in our laboratory. We decided to investigate further the problem of radon in benzene synthesis. Since radon is quite soluble in organic solvents commonly used in liquid scintillation counting, such as benzene, we primarily studied the uptake of radon by benzene open to the atmosphere. The different chemical reagents were studied as sources of radon in benzene, such as oxygen stored in steel cylinders, the sample itself, water for carbide hydrolysis, and the catalyst used to trimerize acetylene to benzene. Also presented will be the spectra of beta-emitting daughter products of the ²²⁶Ra decay series and their interference in the ¹⁴C counting region of interest.

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ANTHROPOGENIC INFLUENCE ON THE ¹⁴C ACTIVITY OF RECENT LAKE SEDIMENTS: A CASE STUDY¹

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Anthropogenic activities that introduce excess of nutrients and other pollutants into rivers and lakes are causing significant changes in aquatic environments. The excess of nutrients greatly accelerate the process of eutrophication. Lake marl formed in the period of eutrophication differs in several aspects from that formed in oligotrophic water.

Recent sediment cores, 30 cm long, from Prošće and Kozjak lakes located in Plitvice National Park, central Croatia, Yugoslavia, were analyzed for ¹⁴C activity of lake marl and organic carbon in the sediment, the ratio of stable isotopes (¹³C, ¹⁸O) and the distribution of diatoms. The abrupt increase of a population of diatom species, *Cyclotella operculata unipuctata* and *Achnanthes clevei rostrata*, in the uppermost 5 cm layer was attributed to eutrophication of the lake water. ¹⁴C activity measurements helped to determine the time period of an increased input of nutrients into lakes. The increase of the ¹⁴C activity in lake sediment due to the bomb effect was used for age determination of the depth profiles.

A molecular characterization of the hydrocarbons isolated from the sediment was performed. Computer-assisted high-resolution gas chromatography/mass spectrometry was applied to estimate contributions of biogenic, fossil and pyrolytic hydrocarbons.

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ENVIRONMENTS OF THE PAST 20,000 YEARS BASED ON AMS ^{14}C , $\delta^{13}C$ AND $\delta^{15}N$ VALUES ON PROTEINS AND INDIVIDUAL AMINO ACIDS

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Climate change is described by comparing modern to ancient environmental conditions. Analysis of past climatic conditions and predictions for future change are improved by 1) better time control for climatic changes, and 2) developing new proxies of past climates. Fossil mammals are suited for paleoecology because they are datable by ¹⁴C AMS – bone contains isotopic signatures of animal diets, species associations reflect paleoenvironments and extinctions, and biome changes represent environmental control.

AMS ¹⁴C research includes dating of megafaunal species, such as Holocene "remnants" and species never dated. Extinction of the Late Pleistocene megafauna is estimated as $10,900 \pm 100$ yr BP. Fossil associations of micromammals (rodents and insectivores) no longer living together (disharmonious faunas) imply equable climates from 18,000-10,000 BP. Direct dating of disharmonious fossil rodent species confirmed the equability model because the fossil micromammals were contemporaneous in Great Plains faunas. Information on paleovegetation is

from ¹⁴C, δ^{13} C and δ^{15} N analysis of individual amino acids from collagen. Carbon isotopes monitor C₃ and C₄ plant abundances, N isotopes record soil moisture and ¹⁴C provides chronological control. Using individual amino acids controls for diagenesis of proteins.

IMPLICATIONS OF DIPOLE-MOMENT SECULAR VARIATION FROM 50,000 TO 10,000 YEARS FOR THE RADIOCARBON RECORD

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Sparse paleointensity data from 10,000 to 50,000 years suggest that the average dipole moment (DM) was 50–75% of the average of 8.67 (× 10^{22}) Am² for the past 5 million years and 8.75 for the past 11,000 years. A linear ramp function, increasing the DM from 4 to 8.75 Am² between 50,000 and 10,000 BP, generates a total ¹⁴C inventory of 122 dpm/cm², yielding better agreement with recent inventory assays, which include ¹⁴C in sedimentary sinks. Using the Lingenfelter and Ramaty (1970) production function and a model DC gain of about 110, this DM function would give a Δ^{14} C > 200‰ from 20,000 to 30,000 BP, similar to the Barbados coral record. This particular production function leaves a decaying tail showing a decrease of 25% during the Holocene. Another problem in this time period is the existence and nature of the Laschamp event ca 45,000 BP. Such an event, with a DM equal to 25% of its average value and lasting 5000 years, would only increase the present-day inventory by 0.3–1.2 dpm/cm², but could produce a ¹⁴C spike of 500‰. We will present a family of DM histories that are consistent with Holocene and Pleistocene ¹⁴C records.

A REVISED SWEDISH CLAY VARVE CHRONOLOGY: PRESENT STATE OF THE ART

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A comprehensive revision of the late- and postglacial Swedish varve chronology (the Swedish time scale) is in progress. So far, a new chronology covering a 1000-year period of Late Weichselian deglaciation from the youngest Younger Dryas moraines has been worked out, and the postglacial time scale has been connected to the present. Older, pre-Younger Dryas parts of the time scale are covered by floating chronologies, which have not yet been definitely correlated.

The clay varve chronology now dates the end of the Younger Dryas to about 10,700 calendar years BP (10,738 +50/-225 BP). The end of the Younger Dryas is then morphologically and clay-stratigraphically defined as the end of the stagnation period at the youngest Fennoscandian moraines in Sweden. This stagnation is followed by rapid and uncomplicated ice recession. The date is younger than the value obtained by mass spectrometry of U-Th in Barbados corals (ca 11,500 BP) for the Pleistocene/Holocene boundary, but very close to the number of annual layers counted down to this boundary in the Dye 3 deep ice core from Greenland (10,720 \pm 150 BP).

CARBON ISOTOPE LABELS AS MIRRORS OF GLOBAL CHANGE

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The imprint of global change on the physical and chemical properties of carbonaceous compounds is diverse and often controversial in interpretation. Anthropogenic effects have to be separated from changes induced by internal variability in the dynamic reservoirs, and regional (or subregional) influences may dominate the signal. Global change information can be derived from ${}^{13}C/{}^{12}C$ and ${}^{14}C/{}^{12}C$ isotope records only when the global signal dominates.

Biological fractionation complicates the interpretation of tree-ring records. The ¹⁴C/¹²C fractionation component can be eliminated through δ^{13} C normalization, but this approach is not applicable to ¹³C/¹²C research. Coral ¹³C/¹²C also is liable to biological fractionation changes, and regional differences may exist. Despite these drawbacks, the paleo-information derived from the carbon isotope records in ice cores, corals and tree rings is internally consistent, and a well-defined time history of atmospheric CO₂ content change and terrestrial carbon reservoir size can be given.

The ${}^{14}C/{}^{12}C$ record reflects changes in modulation by the solar wind and Earth magnetic dipole intensity. Although climate also may play a role in the century-type oscillations, the solar influence is most likely dominant, and a 10,000-year history of the Sun's surface conditions can be given.

AMS RADIOCARBON DATING OF MEDIEVAL DOCUMENTS ON PARCHMENT

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The collection of medieval charters and rolls on parchment was radiocarbon dated by the accelerator mass spectrometry (AMS) method. We describe our method of sample pretreatment. We also present a chronology of the charters based on radiocarbon dates and a comparison to calendar dates obtained by historical study of the documents.

RADIOCARBON ANOMALIES OBSERVED FOR PLANTS GROWING IN ICELANDIC GEOTHERMAL WATERS

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In the upper part of the Markarfljót drainage area in southern Iceland, plant remains have been observed within thick beds of lacustrine sediments. Small samples of different plant species from the same horizon were collected and radiocarbon dated at the Aarhus AMS facility. Terrestrial plants yielded an age of ~9000 BP, whereas aquatic moss (*Fontinalis antipyretica* Hedw) yielded

¹Science Institute, University of Iceland, IS-107 Reykjavík, Iceland ²Institute of Physics, University of Aarhus, DK-8000 Aarhus C, Denmark the surprisingly high ¹⁴C age of ~16,000 BP. The age of the terrestrial plants is believed to reflect the true age of the sediment. It is proposed that the anomalously high ¹⁴C age of the aquatic moss may be an effect of the moss having grown in geothermal water, as the area is known to be geothermally active today. In a test of this hypothesis, modern aquatic moss growing in geothermal water showed a similar ¹⁴C anomaly, with measured ages ranging from 6000 to 8000 BP, which may be explained by the equally high ages measured for the corresponding water samples. The ¹⁴C content of geothermal springs and neighboring rivers in the area ranges from 9 to 50 pMC, equivalent to 20,000–5500 BP.

A FULLY RELATIONAL DATA BASE FOR RADIOCARBON PROJECTS

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This paper describes the progression in technique and sophistication from a simple punched-card system for a radiocarbon data base, through computer-based 'flat' systems, to the powerful, menudriven fully relational data base capable of handling most aspects of the research laboratory projects.

COMPARISONS OF URANIUM SERIES AND RADIOCARBON DATES ON LACUSTRINE DEPOSITS OF THE EASTERN SAHARA

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Glacial conditions in the northern hemisphere are known to correspond with hyperarid conditions in the Sahara where past pluvial conditions correspond with interglacial conditions in northern Europe. Radiocarbon dating of lacustrine deposits in the eastern Sahara have indicated pluvial conditions between around 25,000 to 50,000 BP (oxygen isotope stage 3), whereas absence of archaeological evidence has failed to support this conclusion. Uranium-series dating of lacustrine carbonates in the same region, and, in some cases, on the same deposits as those dated by radiocarbon, also fail to support pluvial conditions between pluvials of the early Holocene (oxygen isotope stage 1) and those of isotope stage 5.

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THE CONTRIBUTION OF SECONDARY RADIATION TO THE BACKGROUND OF GAS PROPORTIONAL COUNTERS AND THE ANALYSIS OF BACKGROUND COMPONENTS

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We present here the results of background measurements in a low-level gas counting system taken at different shielding conditions, from the top of a mountain to deep underground. The results demonstrate clearly the importance of secondary radiation, formed by the interaction of charged cosmic-ray particles with the passive shield, in the background of a low-level counting system. It is further shown how the measurement of the background, under varying secondary radiation

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conditions and at varying gas pressures, make it possible to determine quantitatively the components of the background of gas proportional counters. The results of such measurements are presented and their consequences discussed.

GAMMA RADIATION FLUX IN RADIOCAROBN DATING LABORATORIES USING GAS PROPORTIONAL COUNTERS MEASURED WITH AN NaI CRYSTAL INSIDE AND OUTSIDE THE PASSIVE SHIELD

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A comparison of a large number of gas proportional counting systems used for radiocarbon dating has shown that there is large scattering in the background of similar systems. In order to find the cause of this scattering, the gamma background spectrum has been measured with a $2" \times 2"$ NaI low-level crystal outside and inside the passive shield of a few low-level laboratories. A discussion of the spectra and the cause of the difference in the background of the gas proportional counters is presented.

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REDUCTION OF CO2-TO-GRAPHITE CONVERSION TIME FOR ¹⁴C AMS SAMPLES

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Today, graphite is the most common type of carbon target for accelerator mass spectrometry (AMS). Graphite is produced by reduction of CO_2 by various methods, and it is a well-known phenomenon that the presence of a small amount of impurities (eg, sulphur compounds) may retard the reaction.

We present here some procedures that have considerably decreased the average reduction time for CO_2 from various types of sample material. In particular, CO_2 obtained by combustion of organic samples, such as plant macrofossils, wood and gelatin, showed previously large variations in reduction time (up to about 7 hours). Several methods of CO_2 purification have been tested. The most efficient is the presence of water vapor during combustion. Including the effect of a modified version of the sample preparation system, the CO_2 -to-graphite conversion time, in general, was reduced by more than a factor of three for samples in the range of 300 μ g to 2 mg carbon. Measurements on background samples have shown that ¹⁴C contamination during sample preparation did not increase by the implementation of these procedures, which are now used routinely during preparation of targets that are ¹⁴C AMS dated in Uppsala, Sweden.

RADIOCARBON AND STABLE ISOTOPE STUDIES OF C4 GRASSES

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We report on studies to evaluate the possibility of using C4 grasses as probes of the isotopic composition of atmospheric CO₂. Marino and McElroy (1991) have reported recently on the use of Zea mays to track secular variations of δ^{13} C over the last 40 years. Our experiments focus on both historically and ¹⁴C-dated samples of C4 grasses found in the southwestern United States. Isotopic studies on variations of δ^{13} C within single plants, and as a function of time, will be discussed.

REFERENCE

Marino, BD and McElroy, MB 1991 Isotopic composition of atmospheric CO₂ inferred from carbon in C4 plant cellulose. Nature 349: 127-131.

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NEW PERSPECTIVES FOR RADIOCARBON DATING ORGANIC DEPOSITS BY ACCELERATOR MASS SPECTROMETRY

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Radiocarbon dating of short-lived, *in-situ* specimens found in organic deposits, is, in principle, preferable to dating bulk samples, because the latter may contain a number of sources of error. We instituted a program of comparative AMS ¹⁴C dating of bulk samples and macrofossils occurring in the same stratigraphic level in minerotrophic peats and gyttjas. Our results show that coexisting macrofossils yield consistent ages. Although bulk and macrofossil ages often do not show significant differences, disturbing effects (notably a hard-water effect in gyttjas and ageing of clayey peats or gyttjas due to fluvial input of reworked, older organic debris) are present in a number of bulk samples. Further, contamination by recent roots may be a serious problem in surficial bulk samples. In these cases, dating results can be improved by ¹⁴C dating well-selected macrofossils by AMS.

UTILITY OF AMS ¹⁴C MEASUREMENTS IN SEDIMENT BIOGEOCHEMISTRY

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Variation of the radiocarbon content of sedimentary organic matter with chemical or physical pretreatment of the sample is well documented. The ability to separate sedimentary organic carbon into components with different turnover times (as evidenced by the radiocarbon content) can provide valuable information addressing fundamental questions about the origins, transport and ultimate fate of sedimentary organic materials. The small-sample capabilities of AMS are particularly well suited to this task.

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AMS ¹⁴C measurements of physically and chemically fractionated organic matter from sediments collected from the North Atlantic continental margin will be used to illustrate this approach to the study of carbon cycling in marine sediments. ¹⁴C data prove useful in distinguishing marine from terrestrial sources of sedimentary organic carbon, and in identification of the organic substrates from which pore-water DOC, DIC and CH₄ are derived. A byproduct of this research is the identification of organic fractions most suitable for use in radiocarbon dating of sedimentary organic matter.

AUTOMATIC CALIBRATION OF RADIOCARBON DATES

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A computer program for automatic calibration of radiocarbon dates has been developed at the Centre for Isotope Research (CIO) in Groningen. The program calculates the probability distribution along the calendar axis, based on the radiocarbon measurement and its standard deviation. The program can use any of the dendrochronological calibration sets as published in the literature, and has been distributed widely in the ¹⁴C community.

The program has been rebuilt extensively. From the main menu, one can select interactive calibrating, automatic calibrating, calibration averaging, spline calculations, smoothing, wiggle matching and special options. It is upgraded to Turbo-Pascal (version 5.0/5.5), and is working on XT and AT personal computers or true compatibles. Graphics supported are CGA, Hercules, EGA and VGA. For VGA, there is a color option. The calibration graphs can be dumped to a printer (Epson compatible) or color plotter (HP compatible).

The new Groningen program will be demonstrated during the conference.

A NEW DATA ACQUISITION SYSTEM FOR THE GRONINGEN COUNTERS

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The Groningen proportional counter system consists of 11 counter systems: 3 for tritium and 8 for radiocarbon. Three of the ¹⁴C combined form the large high-precision counter; the small counter setup was decommissioned after the Chernobyl accident.

Until 1989, the data-acquisition system was based on an Apple microcomputer/PIA interface. This locally designed system is replaced by an XT-compatible PC with an IEEE-based interface system. The amplifiers and discriminator units were completely rebuilt. The same XT/IEEE processes additional data, such a temperature and pressure of the counter and the barometer reading.

The software is completely written in Turbo-Pascal. The program allows data entry from all the counters or from a subset; an on-line display shows – of all counters – the status, the GrN-number of the sample being measured and the most important channel counts. All data are stored on files which are coded by the date of the measurement and the counter number. This allows automatic processing/reporting and – in the near future – connections to outside programs such as, for example, the new Groningen data base.

THE NEW GRONINGEN ¹⁴C DATA BASE

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Since 1982, we have set up a database system that contains information on all ¹⁴C samples measured in Groningen since 1953. The system consisted of a homemade program (partly in Machine Code) running on an Apple PC. This data base had an important disadvantage: the program itself was inaccessible, and exchange of output with other laboratories was, in practice, very difficult. Therefore, the system was transferred to a modern standard computer and programming language.

The data were successfully transferred (via the RS-232 interface) from the Apple to an Olivetti M290 (AT compatible) computer, equipped with a 40 Mb hard disk, a 3.5" high-density floppy disk drive and a 40 Mb tape streamer.

The new database program was completely written in Turbo-Pascal, using the Turbo-Pascal Database Toolbox. The program features versatile search routines. The output can be directed to either screen, printer or file. At present, four types of date lists can be selected: 1) the *RADIOCARBON* date list; 2) the HLF data transfer format; 3) a summary listing; 4) the IRDB format. Adding additional date lists to the program is straightforward, facilitating exchange with other laboratories or users.

The Groningen data base contains 17,000 records at present (September 1990) and is growing at a pace of about 1000 per year.

SEPARATION AND CHARACTERIZATION OF COLLAGEN-DERIVED PEPTIDES AND AMINO ACIDS

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The dating of low collagen and/or heavily contaminated bone is still problematic. One approach to a solution is the isolation and purification of collagen-derived specific peptides and amino acids. The enzymatic cleavage of collagen by means of collagenase produces a mixture of many small peptides, some of which have a very high collagen specificity. Subsequent reversed-phase and ion-exchange chromatography enables separation of hydrophilic and hydrophobic peptides, and isolation of specific tripeptides like GlyProHyp. The described HPLC techniques can be useful in two ways: 1) analytical, to assess the chemical intactness of collagen samples and 2) preparative, to isolate specific fractions of the mixture. The developed preparative approach will be described in the context of the need to balance increased specificity against sacrifice of yield.

DATING OF PRECOLUMBIAN MUSEUM OBJECTS

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Through accelerator mass spectrometry (AMS) dating, it has become possible to apply radiocarbon dating to precious museum objects. We are finding AMS to be very helpful in dating Precolumbian art. The origin of most of these objects is vague. They have been in museums and in private collections for many years. Most Precolumbian objects can only be placed in a general cultural context because no archaeological contextual information nor written documents exist. In this paper, AMS dates of different types of material will be discussed.

RADIOCARBON DATING OF LIME FRACTIONS AND ORGANIC MATERIAL FROM BUILDINGS

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AMS dating and proportional gas counting were used to analyze lime mortar. If separation was possible, lime nodules and mortar powder were dated. From each carbonate sample, the total amount of CO_2 and fractions were measured. In all but one case, the results from the isotopic fractionation provided information about the absorption of atmospheric CO_2 . In some cases, the nodules produced better radiocarbon dates than the mortar powder; in other cases, it was the opposite. In some samples, organic material was found and dated separately. Working with AMS and looking for small samples, we found more organic material (charcoal, hair, wax) in the mortar and construction materials than we were expecting to find.

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DESIGN AND PERFORMANCE OF A THERMAL DIFFUSION MICRO-COLUMN FOR ¹⁴C ENHANCEMENT OF CARBON MONOXIDE

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Isotopic enrichment of carbon monoxide by thermal diffusion is well known and has been routinely used in radiocarbon laboratories to extend the range of ¹⁴C dating (Grootes & Stuiver 1979). In these systems, at least 150 g of CO is required to obtain 9 g of CO with a theoretical enrichment factor of about 16 for ¹⁴C. This 9 g quantity of CO, converted to CO_2 , is required for precise low-level proportional gas counting (llc). At the time these systems were designed, llc was the only method available for precise radiocarbon analysis. To take advantage of the new direct atom counting techniques, a design was proposed for a small sample thermal diffusion system capable of concentrating the ¹⁴C contained in 1 g of CO into a 23 mg fraction (theoretical enrichments

greater than 40) (Grootes et al 1980). Our research group has been interested in this proposed system, and has recently commenced construction of a modified design.

Our system requires 0.8-3.2 g quantities of CO to operate at pressures between 1-4 atm. The diffusion column is comprised of a 2.5 m quart tube (4 mm ID) that is connected to a 1.5 L reservoir volume above, and a 1.2 mL enrichment volume below. The quartz tube is jacketed by a glass condenser tube (4 cm ID) in which chilled water is circulated. Inside the quartz tube is a Pt/Ir wire (0.12 mm diam) that can be heated resistively; it is held taut by a stainless steel spring in the reservoir volume. Concentricity of the wire with the walls of the quartz tube is maintained by a series of probes down the length of the condenser, which can be used to adjust the position of the quartz tube. The enrichment volume can be microsampled (in 30 μ L aliquots) by a glass 3-way valve, which is attached to a quadrupole mass spectrometer and a system to convert the enriched CO into an Fe/C accelerator target (Verkouteren *et al* 1987). The mass spectrometer is used to determine mass 30 ($^{12}C^{18}O + ^{14}C^{16}O$) enrichment yields and to test for chemical contamination.

We will report details of our design and preliminary performance data of this microchemical ¹⁴C enrichment system.

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RELATIONSHIPS AMONG CO₂, CO, CH₄ AND H₂ DURING REDUCTION OF CO₂ TO GRAPHITIC CARBON ON IRON WOOL

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For three years, our group has made high quality targets for ¹⁴C accelerator mass spectrometry by reduction of sample CO₂ to graphitic carbon on iron wool and *in situ* melting of this wool into a solid bead (Verkouteren *et al* 1987). The chemical yield $(85 \pm 8\%)$ and the length of time required for this reduction (1-2) days have been somewhat variable (Klouda *et al* 1990). These variabilities are dependent, in part, on the source of the sample and presence of trace chemical cofactors. We have noted, however, that samples from the same source can also differ in reduction behavior. This variability in behavior can be attributed to any number of factors that could potentially affect the kinetics of the reduction. Thermodynamically, the reduction should proceed quickly (Table 1). Here, we have used manometry and mass spectrometry to investigate the dependencies of our reduction process on variations in sample size, reduction volume, amounts of hydrogen, iron wool and zinc, positioning of the zinc heating furnace and its cycling time, level of cryogenic fluid in the cold trap, and other factors that might affect the relationships among carbon dioxide, carbon monoxide, methane, hydrogen, iron, zinc and water vapor.

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Pertinent equilibria and nee energies for reduction process		
Equilibria	ΔG ₈₇₃ (kJ/mol)	
$CO_2 + Zn \longleftrightarrow CO + ZnO$	-55	
$CO + CO \xleftarrow{Fe} C + CO_2$	-18	
$CO + H_2 \xleftarrow{Fe} C + H_2O$	-14	
$CO_2 + H_2 \xleftarrow{Fe} CO + H_2O$	+4	
$CO + 3H_2 \xleftarrow{Fe} CH_4 + H_2O$	-18	

 TABLE 1

 Pertinent equilibria and free energies for reduction process

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RAPID PRODUCTION OF GRAPHITE WITHOUT CONTAMINATION FOR BIOMEDICAL AMS¹

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The application of AMS to the detection of ¹⁴C makes possible a new class of sensitive experiments in molecular biology. We presently apply this technology to pharmacokinetics of xenobiotics, radio-immune assays and genetic damage and repair. Such experiments inherently produce large numbers of samples for the determination of biological variability in molecular interactions. The samples vary in ¹⁴C concentration over many orders of magnitude, sometimes unpredictably. In our initial experiments, we reduced CO₂ to filamentous graphite by hydrogen. Our throughput was limited by the number of reactors (4), and cross-contamination resulted in unacceptable uncertainties. Instead, we modified the known reduction of CO₂ by zinc in a sealed tube in order to rapidly and reproducibly make graphite without contaminations. Titanium hydride is added to the zinc in the bottom of the reaction tube. A smaller inserted tube contains the cobalt catalyst. The CO₂ is transferred from a break-seal combustion tube to the reaction tube through a completely disposable plastic manifold. Samples are transferred at 15-20 per hour. The sealed tubes are heated to 435°C in a muffle furnace. The number of samples to be prepared simultaneously is limited only by the size of the furnace. The process is complete to <1%fractionation within 5 hours. Cross-contamination is much less than 1 part in 50,000. The hydrogen from the decomposing hydride minimizes the production of volatile carbonyls and the transfer of volatile zinc compounds back to the graphite. The ion beams from these graphites are similar in intensity to those from hydrogen-reduced carbon, and no 'burn-in' is required. The isotope ratio among identical samples is reproducibly measured to better than 1%, and backgrounds of 0.7pMC are measured without any precleaning of the metals involved.

¹Work performed under the auspices of the US DOE by Lawrence Livermore National Laboratory under contract W-7405-ENG-48

THE NEW NATIONAL OCEAN SCIENCES ACCELERATOR MASS SPECTROMETER FACILITY AT WOODS HOLE OCEANOGRAPHIC INSTITUTION: PROGRESS AND FIRST RESULTS

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The installation of the new AMS machine at Woods Hole Oceanographic Institution (WHOI) is nearing completion. During the summer of 1990, extensive factory tests of the fully assembled single-injector machine produced very promising results. The new injector design implementing the recombinator principle, has proven to be successful. We have learned that the simultaneous injection of all three carbon isotopes (with a 100:1 chopped C-12 beam) is possible, without notable effects on the performance of the accelerator. Because of the elaborate background suppression measures in the high-energy leg of the system, removing the ME/q**2 ambiguity, an exceptionally strong and clean ¹⁴C signal (ca 40/s) was observed in first tests with NBS-OX II graphite samples at beam currents up to 50 microamperes. On-site tests at WHOI started in November 1990, and first routine operation results are anticipated by early spring of 1991.

³⁶CI IN THE STRATOSPHERE

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Cl and Cl compounds are important trace constituents for stratospheric chemistry, in particular with respect to O3 destruction. Stratospheric Cl chemistry has recently received increased attention with the observation of strong O₃ depletion in the Antarctic winter vortex and in the weaker and more complex Arctic winter vortices.

Cosmogenic ³⁶Cl is produced by spallation reactions from Ar mainly in the stratosphere, and the large amounts of ³⁶Cl introduced by nuclear weapon testing have been removed from the stratosphere by now. We are investigating the use of cosmogenic ³⁶Cl as a tracer for stratospheric Cl chemistry and for stratospheric/tropospheric exchange processes. In a first attempt, we try to determine stratospheric and tropospheric production rates, the partitioning of ³⁶Cl among particulates and gaseous Cl compounds, and the respective inventories and removal rates.

We have obtained three pairs of samples of stratospheric ³⁶Cl in particulates and HCl collected aboard the NASA WB-57B high-altitude aircraft, following techniques by Lazarus et al (1976). The preliminary results for two flights (#1: 13.7 km, 30-33°N, 97-107°W, 1.8-2.4 km above tropopause; #2: 17.6 km, 36-45°N, 92-94°W, 7.6 km above tropopause) are as follows:

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Samples	36 Cl/Cl × 10 ⁻¹⁵	³⁶ Cl m ⁻³ air STP	HCl (ppbV)
#1 particulates	63 ± 7	$1.45 \pm 0.16 \times 10^{3}$	0.26 ± 0.05
HCl	655 ± 52	$1.99 \pm 0.18 \times 10^{4}$	
#2 particulates	350 ± 20	$3.0 \pm 0.2 \times 10^4$	2.0 ± 0.2
HCL	3260 ± 130	2.9 \pm 0.2 \times 10^5	

These results show that about 90% of ³⁶Cl atoms are associated with HCl, the main gaseous Cl compound. More data will be available at the time of the conference for comparison to the calculated production rates and to the measured deposition rates (Elmore *et al* 1987; Conard 1986). ¹⁰Be has not been measured in these samples but ¹⁰Be/³⁶Cl ratios can be inferred from numerous data in the stratosphere at many latitudes and altitudes (Wahlen *et al*, unpublished data), and can be compared to ¹⁰Be/³⁶Cl in polar deposition (Conard 1986). This comparison suggests that Cl might at times be strongly removed from the polar stratosphere.

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THE COUNCIL FOR BRITISH ARCHAEOLOGY/RCD UNITED KINGDOM ARCHAEOLOGICAL DATA BASE

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The idea for a data base maintained specifically for archaeological ¹⁴C dates originating from sites within the United Kingdom was first proposed some 10 years ago. With support from the Council for British Archaeology, this data base is now a working reality available for researchers and submitters in UK archaeology. It contains dates from three sources: 1) published dates (*RADIO-CARBON* and elsewhere) included in the Council for British Archaeology Index of Archaeological Sites; 2) further published dates collated ready for publication in a Supplement to the Index; 3) some data from the Oxford and Groningen Laboratories transferred directly from their laboratory data bases.

In the course of the last 12 months, the original data have, after detailed checking, been completely re-entered in a revised and more practical format from that published previously (Walker *et al* 1990; Walker *et al*, in press). The new format makes greater use of free text entry and has fewer keyed fields, making the data base easier to read for the user and providing greater protection from wholesale copying. The database management system used remains STATUSE (Harwell Computer Power), this being particularly powerful in its ability to retrieve data from free text. Data can be retrieved on screen, hard copy or computer file and, by the use of purpose written macros within the data base, in any format required by the user. Although the internal format is designed to facil-

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itate 'on the spot' screen or paper presentation for research, text entry is still orientated towards the HLF fields style previously described (Wilcock *et al* 1988), as it is ideally suited to wider data exchange, in particular to the IRDB minimum entry fields (Kra 1989; Walker & Kra 1988).

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¹⁴C DATING AND MEASUREMENTS OF CLIMATIC PROXY INDICES OF LOESS SEQUENCE TO RECORD PALEOMONSOON VARIATION ON THE LOESS PLATEAU OF CHINA DURING THE LAST 16,000 YEARS

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A loess profile, 15 m thick, which has developed in the Baxie River Valley, west of the Loess Plateau, can be considered to be representative of loess-paleosol sequences formed over the last 16,000 years. The ¹⁴C determinations of various organic fractions and thermoluminescence (TL) dates of quartz from the profile indicate that the paleosol complex, 8-4.9 m deep below the top of the profile, which can be interpreted as recording the Holocene Optimum, and reflects the prevalence of Asian summer monsoon circulation, developed between 10,000 and 6000 BP. A loess layer, 1.25 m thick, at a depth of 9.25-8 m, is characterized by low organic content, low magnetic susceptibility and more positive organic δ^{13} C values. These data indicate that the loess layer records an abrupt change in Asian monsoon climate spanning 10,870-10,230 BP. A weakly pedogenic paleosol at 9.25-9.75 m depth marks the termination of the Last Glacial period and the reactivation of the summer monsoons. This paleosol spans the range from 12,500 to 11,000 BP. Two ¹⁴C determinations on the humin fraction of the organic component of the base of the higher paleosol complex and the top of the lower paleosol gave ages of $10,230 \pm 230$ cal BP and 10,870± 270 cal BP, respectively. These ages mark the beginning and termination of an abrupt event involving increased dust influx under the domination of winter-monsoon conditions. This abrupt change seems to have lagged behind the equivalent Younger Dryas event found in the North Atlantic region. Further work is needed to provide an explanation for this time lag.

The organic δ^{13} C values from the profile range from -21% to -24%. The more positive δ^{13} C value suggested that the proportion of C3-type plants in the river valley of the loess plateau increased when summer monsoon influence strengthened, and C4-type vegetation increased when the summer monsoon influence weakened. Magnetic susceptibility and organic content were low during loess deposition, also reflecting domination of winter monsoons over summer monsoons.

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A COMPARISON OF SOME AGE-ESTIMATE METHODS FROM THE EGYPTIAN SAHARA

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A variety of techniques was used to estimate the age of a sequence of five fossil lakes, with associated Middle Paleolithic sites, in two adjacent depressions in the Eastern Sahara of Egypt. Tight stratigraphic controls and multiple samples from the same sedimentary units permitted direct comparison of the results. The methods used were uranium (U)-series, electron spin resonance (ESR), three varieties of thermoluminescence (TL) (quartz sand, burned sediment and optical), ostrich eggshell epimerization and radiocarbon dating. A large series of age estimates was obtained, and two of the techniques yielded fairly consistent results: optical TL and ostrich eggshell epimerization. The results from the other methods were often erratic and in conflict with their stratigraphic positions. Nevertheless, the age estimates place the earliest of the Middle Paleolithic wet episodes as contemperoaneous with some part of oxygen isotope stage 7, and the four younger wet events with stage 5. The faunal evidence indicates a significant northward movement of biotic zones, with some micromammalian species occurring today 1500 km farther south.

A NEW TECHNIQUE FOR CONVERTING CARBON DIOXIDE TO AMS TARGET GRAPHITE

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With the advent of the new generation of high current, 60 sample, sources for AMS ¹⁴C dating, there is a need for a simpler way of converting CO_2 samples into graphite for making AMS targets. It would also be an advantage if the technique were suited to being automated.

Currently, ¹⁴C dating using beta counting provides much higher precision in the 45,000–60,000 year age range than does AMS ¹⁴C dating. This is exactly the opposite to what would be expected from theory. An accelerator mass spectrometer can count 0.1% of the ¹⁴C atoms in a sample in a few minutes, whereas a beta counter takes eight years. One would therefore expect AMS to be the method of choice for samples of low ¹⁴C content. The reason for this surprising result is that whereas most beta counting labs know their background/chemical blank to better than 1%, AMS labs have a relative high chemical blank (relative to the size of their sample) and rarely can reproduce it to better than 25%.

The relatively large size, relative to the sample, and the lack of reproducibility of the blank in AMS ¹⁴C dating severely limits the smallest sample that can be run at a reasonable precision. Clearly, it is difficult to date a moderately old 10 μ g sample if the uncertainty in ones blank is 1 μ g (modern).

This paper describes work towards developing a simpler graphitization system, with a lower/more reproducible blank, capable of mass production and compatible with automation.

TARGET ERROR - A POSSIBLE PROBLEM IN HIGH PRECISION AMS ¹⁴C DATING

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A number of AMS labs have attempted to carry out high-precision AMS ¹⁴C dating and appear to have run up against a barrier in the 2‰ to 4‰ region. This can be attributed to uncertainties generated in the apparatus itself. This paper proposes that at least part of the problem may instead lie in the properties of the graphite target. In particular, the assumption is that every part of the graphite in the target has the same propensity to produce beam current.

It is well known that the various graphitization reactions used to convert CO_2 to graphite can have very large isotopic fractionation. In the data presented in this paper, the first 20% of the graphite produced from NBS OX-II carbon dioxide ($\delta^{13}C = -17.8\%$) showed $\delta^{13}C = -33.1\%$ and the last 20%, $\delta^{13}C = +12.9\%$. If, for example, the propensity of the graphite produced in the last part of the reaction to produce ion beam is not the same as the rest of the sample, we cannot make the necessary isotope corrections required for high precision ¹⁴C dating.

The assumption presently being made is that the isotope fractionation *versus* yield curve and the propensity to produce ion beam current *versus* yield curve are the same shape for both the samples and the NBS standards. The fact that real samples tend to give lower beam currents than graphite prepared from the NBS oxalic standards suggest that this assumption may not be valid.

Ways will be suggested as to how this problem may be circumvented.

AMS RADIOCARBON DATING OF ICE: PROGRESS AND NEW RESULTS

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New developments in the "Sublimation Technique" for the ¹⁴C dating of ice will be described, together with the application of the technique to a number of problems.

IMPURITIES ARISING DURING BENZENE SYNTHESIS FROM ACETYLENE ON VANADIUM AND CHROMIUM CATALYSTS

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Impurities generated during benzene sythesis can affect liquid scintillation counting in radiocarbon dating. Acetylene from a single (tank) source was purified cryogenically and introduced onto vanadium and chromium catalysts in varying proportions of gas and catalyst. We then analyzed benzene produced from this acetylene source by gas chromatography (GC) and gas chromatography-mass spectrometry (GC-MS). These analyses demonstrate that the synthesized benzene contains, at most, traces (<0.1% total) of substituted benzenes. These products are principally toluene and ethylbenzene together with smaller amounts of xylenes, cumene and phenol. In addition, we have analyzed by GC the tank acetylene source and acetylene generated in the laboratory as well as their benzene products. The results of these analyses show the presence of C_1 - C_6 alkenes in the laboratory-produced acetylene. Preliminary results indicate that the amount

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and nature of the contaminants in the benzene are related to these alkene precursors in the acetylene and to the temperature at which the benzene is removed from the catalyst. The presence of impurities in the scintillation cocktail may affect counting reliability, and therefore we urge special care in accounting for contaminants.

A COMPARISON BETWEEN THE ORSAY AND NIST ¹⁰BE STANDARDS

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Ever since our first publication on ¹⁰Be measurements by accelerator mass spectrometry (AMS) (Raisbeck *et al* 1978), we have used ¹⁰Be/⁹Be standards produced by mixing an almost pure solution of ¹⁰Be (whose concentration was established by isotope dilution mass spectrometry in our laboratory) with pure ⁹Be. Although we always recognized the uncertainty in this standard (associated mainly with the uncertainty in the mass fractionation parameter of our spectrometer), we have continued to use it because: 1) it allowed us to make internally consistent measurements; 2) this ¹⁰Be/⁹Be ratio is independent of the ¹⁰Be half-life; 3) there appeared to be no widely available and generally accepted alternative.

The recent commercialization of a standardized reference solution (SRM 4325) of ¹⁰Be/⁹Be by the National Institute of Standards and Technology (NIST) has changed this latter situation. It appeared to us to be useful to compare our standard with this material, thus making it possible to compare the absolute values of our ¹⁰Be measurements with other laboratories using the NIST standard. We report here a series of AMS comparison measurements between the two standards, which were carried out at the Gif sur Yvette Tandetron. Implications regarding the ¹⁰Be half-life are also mentioned.

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INSTRUMENTS AND DATA SOFTWARE OF LOW-LEVEL LIQUID SCINTILLATION COUNTING USED FOR ¹⁴C DATING

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Our laboratory is engaged in low-background liquid scintillation research. We have three kinds of instruments. We discuss here our instrumentation and data process software of low-level liquid scintillation counting for radiocarbon dating. We also conduct research in gas proportional counting of radiocarbon. More than 90% of Chinese laboratories use liquid scintillation counting methods. Our associates include the Institute of Archaeology and Beijing University ¹⁴C laboratories, and the Geology Institute.

THE UPPER PALEOLITHIC AND EARLY HOLOCENE TIME HORIZONS OF ARABIA: RESULTS AND PROSPECTS

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With the recent reevaluation of the appearance of modern man in Africa, a myriad of other problems has arisen, concerning the archaeology of the Middle East. Of particular interest here is the definition and identification of the Upper Paleolithic in Arabia within the context of the earlier Middle Paleolithic. Archaeological and geological data may be used to help resolve the debate over industry and species within the Arabian peninsula. Radiocarbon dating, climatological and sea-core data will be used to focus particularly on southwest Arabia.

The early Holocene record for the same region can also be examined, especially in relationship to the Paleolithic and the later climatological and geological record. A wealth of diverse archaeological data can also be brought to bear on examining population adaptation in the peninsula since 10,000 BP.