

## RADIOCARBON DATING THE LAST GLACIAL-INTERGLACIAL TRANSITION (Ca. 14–9 <sup>14</sup>C ka BP) IN TERRESTRIAL AND MARINE RECORDS: THE NEED FOR NEW QUALITY ASSURANCE PROTOCOLS<sup>1</sup>

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**ABSTRACT.** The publication during the 1990s of Greenland ice-core records spanning the transition from the Last Cold Stage to the present interglacial (ca. 14–9 <sup>14</sup>C ka BP) presented new challenges to scientists working on marine and terrestrial sequences from this important time interval. In particular, there is now an overriding imperative to increase the levels of precision by which events during this period can be dated and correlated. We review some of the problems commonly encountered when using radiocarbon dating for these purposes, and consider some of the new approaches that will be required if this dating method is to provide a basis for a high precision chronology for the last glacial-interglacial transition.

### INTRODUCTION

Between approximately 14.0 and 9.0 <sup>14</sup>C ka BP the last glacial cycle drew to a close in a series of abrupt and apparently pronounced climatic oscillations, and was finally terminated by a sustained shift to warmer conditions at the start of the present (Holocene) interglacial. This transitional period has been referred to, *inter alia*, as the “last glacial-interglacial transition” (LGIT), the “last glacial-Holocene transition”, the “Late-glacial period” and the “Last Termination”. For simplification, we will use LGIT to denote this period of time. Because the LGIT is the most recent example in the geological record of a transition between a full glacial and an interglacial climatic régime, and because the geological evidence available for the reconstruction of events during this period is unsurpassed in terms of its level of preservation and diversity, it is one of the most intensively studied intervals in the entire Quaternary record. As a consequence, paleoenvironmental reconstructions for this period are some of the most highly resolved, in terms of their temporal and spatial detail, of any part of the Quaternary sequence (e.g. Lowe et al. 1994; Lundqvist et al. 1995; Lowe and Walker 1997a, Renssen and Isarin 1998; Walker et al. forthcoming).

The geochronological underpinning of these reconstructions, in terms of the development of site chronologies, regional syntheses and inter-regional correlations, has relied almost exclusively on <sup>14</sup>C dating. Indeed, it is difficult to over-estimate the importance of the role that <sup>14</sup>C has played in the development of the LGIT research agenda over the course of the last four decades. For example, the chronostratigraphic framework that has, hitherto, been most widely employed in NW Europe for the subdivision of the LGIT was defined exclusively in <sup>14</sup>C years (Mangerud et al. 1974). This scheme consisted of four principal elements: the “Bølling Chronozone” (including the “Oldest Dryas” episode), defined as the time interval between 13.0 and 12.0 <sup>14</sup>C ka BP, the “Older Dryas Chronozone” (12.0 to 11.8 <sup>14</sup>C ka BP), the “Allerød Chronozone” (11.8 to 11.0 <sup>14</sup>C ka BP) and the “Younger Dryas Chronozone” (11.0 to 10.0 <sup>14</sup>C ka BP). Each of these was considered to coincide with major climatic events or episodes.

The widespread application of the Mangerud et al. chronostratigraphic scheme and its associated terminology, even in regions or areas for which it was never originally intended (see Wohlfarth

<sup>1</sup>A contribution to the INTIMATE Programme of the INQUA Paleoclimate Commission

1996; Björck et al. 1998), reflected the widespread confidence of the scientific community in the  $^{14}\text{C}$  dating method. It was generally assumed that: a) the  $^{14}\text{C}$  technique provided a basis for dating and correlating climatostratigraphic boundaries with a reasonable degree of precision, and b) there was close correspondence between the  $^{14}\text{C}$  ages of the chronozone boundaries and the timing of climate change, at least in those regions where the scheme had been adopted. However, during the course of the last decade or so, questions have arisen about both of these assumptions. First, it has become increasingly apparent that, because of natural variations in the rate of  $^{14}\text{C}$  production,  $^{14}\text{C}$  years may have diverged from sidereal years by as much as 2.5 ka or more during parts of the LGIT (Bard et al. 1993). Superimposed on these long-term fluctuations have been shorter episodes of near-“constant age”, the so-called  $^{14}\text{C}$  “plateaux” (see e.g. Ammann and Lotter 1989; Bard and Broecker 1992; Lotter et al. 1992; Kromer and Becker 1992; Bard 1998; Stuiver et al. 1998). The fact that at least two of the chronozone boundaries in the Mangerud et al. (1974) chronostratigraphy fall within such  $^{14}\text{C}$  plateaux renders the scheme much less precise as a tool for dating and correlation than was appreciated only a quarter of a century ago (Walker et al. 1999a).

Secondly, the climatic changes themselves, which are frequently used to define the boundaries of the “Bølling”, “Allerød”, and “Younger Dryas” intervals in regional stratigraphic schemes, appear to have been diachronous across northwestern Europe (Coope et al. 1998; Witte et al. 1998). This, of course, calls into question the assumption that stratigraphic boundaries defined on the basis of climatic change are synchronous over large areas. Measuring the extent of diachroneity, however, is not easy when employing conventional approaches to  $^{14}\text{C}$  dating, again because of the limits on the levels of precision that are achievable. Thirdly, although it was subsequently acknowledged that the sequence of climatic changes during the LGIT may have been more complex than was initially embodied in the Mangerud et al. zonation scheme (see e.g. Walker 1995; Yu and Eicher 1998), neither the degree of climatic variability nor the abruptness of the climatic transitions was fully appreciated until the publication of the GRIP and GISP2 ice-core data (e.g. Alley et al. 1993; Dansgaard et al. 1993; Kapsner et al. 1995; Taylor et al. 1997; Alley 2000). These remarkable records are characterized by climatic events with durations as short as 150 ice-core years, and significant climate shifts measurable to within ice-core decades, or even ice-core years. Such temporal resolution appears to be well beyond the levels of precision presently attainable using  $^{14}\text{C}$  dating.

In the light of these new developments, and because of the demands that are increasingly being placed upon researchers to improve the levels of precision in the dating and correlation of LGIT events, new strategies for  $^{14}\text{C}$  dating need to be formulated. In this paper, we discuss the shortcomings of  $^{14}\text{C}$  dating as currently applied to LGIT records, and we suggest alternative approaches that might help resolve at least some of these difficulties.

#### **SOURCES OF ERROR IN $^{14}\text{C}$ DATES OBTAINED FROM LGIT SAMPLES**

Error sources in  $^{14}\text{C}$  measurements on samples of LGIT age fall into three categories: 1) site-specific geological problems that adversely affect sample integrity, 2) laboratory contamination and measurement precision, and 3) calibration of  $^{14}\text{C}$  dates to sidereal years. Each of these is considered in this section.

##### **Sample Selection and Integrity**

A wide range of materials is now employed in the dating of LGIT events, including fossil wood, terrestrial leaves and seeds, marine macro- (e.g. bivalves) and microfossils (e.g. foraminifera), fossil bone and bulk organic debris (e.g. “gyttja”, organic lake muds, and peats). Each has been affected to a greater or lesser extent by physical processes (including reburial or redeposition) and/or chemical

alteration, depending on the geological context through which the fossil material was transported and/or into which it was eventually deposited. These “site-specific” factors may influence the integrity of the samples and thereby adversely affect the resulting  $^{14}\text{C}$  ages. A considerable amount of recent research has been devoted to the identification and quantification of, and subsequent correction for, these site-specific factors. Indeed, it could be argued that these essentially “taphonomic problems” are the most important that need to be resolved, since attempts to correct for other potential influences (e.g. laboratory contamination and temporal variations in atmospheric  $^{14}\text{C}$  production) can only result in spurious levels of precision if the geological integrity of the samples remains in question.

The range of potential site-specific geological problems that can influence the suitability of samples for  $^{14}\text{C}$  dating is too diverse and complex to review in this short paper. Instead, we will focus on the problems that are particularly acute in the dating of LGIT sequences by examining those materials that are most commonly employed in the  $^{14}\text{C}$  dating of this time interval.

#### *Bulk Organic Sediment Samples*

A large number of published chronologies for the LGIT from sites in Europe and North America are based on  $^{14}\text{C}$  dating series obtained from bulk organic lake or pond muds (“gyttja”). It has long been recognized that a number of potential error sources can affect bulk samples of these sediments, including, for example, hard-water and mineral carbon errors, and biological and chemical fractionation processes (see e.g. Lowe 1991; Wohlfarth et al. 1993; Wohlfarth 1996; Lowe and Walker 1997). In some instances, it is possible to reduce the adverse effects of these site- or sample-specific factors through careful site and/or sample selection procedures and/or by the application of correction factors. However, identifying the sites and samples least affected by these problems, and establishing the scale of corrections to apply are far from straightforward. Lake or pond sediments are composed of a heterogeneous mix of clastic particles and organic debris, the latter derived from biota either inhabiting the lake or pond waters or from vegetation growing around the basin catchments (e.g. Colman et al. 1996; Batterbee 2000). The organic debris will, therefore, frequently include older organic materials eroded from the exposed lake edges (particularly during times of reduced water level) or from other organic deposits located in the catchment (e.g. exposed soils) which, often intermittently, may be influenced by fluvial, colluvial or other erosional processes. Thus bulk sediment samples consist of a variety of components which, if differentiated, would almost certainly generate a range of ages. The published  $^{14}\text{C}$  dates from the vast majority of bulk sediment samples are, therefore, almost certainly derived from averages of a range of activity values.

$^{14}\text{C}$  dates based on organic detrital limnic sediments obtained from LGIT sequences are often characterized by an “ageing effect” due to the dilution of the  $^{14}\text{C}:^{12}\text{C}$  ratio in organic residues. This problem is particularly acute where the local bedrock (or surficial material such as glacial till) is calcareous, or where mineral particles rich in carbon which is both refractory and geologically old have become incorporated into the lake deposits. Sediments accumulating in lakes in northern latitudes during the LGIT were particularly susceptible to “ageing” effects because of the glacier melt processes and widespread land instability (e.g. gelifluction or solifluction processes) prevalent at the time. Soils containing both older organic and inorganic materials would have been eroded from catchment slopes, while “glacial flour”, which often contains carbonaceous residues, would also have been washed into many lake basins (e.g. Björck and Moller 1987; Andrieu et al. 1993; Walker et al. 1993; Lowe et al. 1995). The problem is further exacerbated by the “hard water factor” where sub-aquatic photosynthesizing plants which take up carbonate from lake waters will further dilute

$^{14}\text{C}$  levels in organic lake muds, in exceptional circumstances adding up to 1200 yr to the apparent age of limnic material (Peglar et al. 1989).

That an “ageing” problem is commonly encountered in organic limnic sediments of LGIT age has been clearly demonstrated when  $^{14}\text{C}$  dates have been obtained on humic (acid washed, alkali soluble) and humin (acid washed, alkali insoluble) fractions of the same sediment sample. Older carbon residues, which would induce an ageing effect in a single bulk sediment date, tend to be reflected in the “humin” age determinations which may be older than the “humic” date by hundreds or, in some cases, thousands of years (Walker and Harkness 1990). Similar differences in ages have been noted between AMS  $^{14}\text{C}$  measurements on separate macromolecular compounds (e.g. lipids, amino acids and cellulose) extracted from the same bulk samples (e.g. Lowe et al. 1988). Equally, where parallel  $^{14}\text{C}$  measurements have been obtained on both the sediments and on plant macrofossils, the age estimates on the macrofossils are frequently younger than those obtained from their host sediment matrix (e.g. Coope and Brophy 1972; Coope and Joachim 1980; Cwynar and Watts 1989).

In recent years, therefore, there has been a tendency to assume that the  $^{14}\text{C}$  dating of plant macrofossils offers a more reliable means of establishing a chronology for the LGIT, since this avoids many of the difficulties encountered in the dating of bulk sediment samples (see e.g. Peteet et al. 1995; Lowe et al. 1995a; Björck et al. 1996; Gulliksen et al. 1998; Hughen et al. 1998; Kitagawa and Van der Plicht 1998). However, this is an assumption which has seldom been tested rigorously and, as is shown in the following section, it may not always hold true.

#### *Dating Plant Macrofossils*

With the rapid development of, and expansion in, AMS facilities over the last decade, plant macrofossils are now being used as a routine material for  $^{14}\text{C}$  dating of LGIT sequences since a reasonable analytical precision can be obtained from as little as 2 mg of organic carbon. This amount of organic C can typically be extracted from just a few fossil seeds, fruits or bracts, or even from a single fossil leaf. There has been a tendency to avoid aquatic taxa (e.g. *Potamogeton*) in such dating practices as many aquatic plants photosynthesize subaquatically and hence build into their cellular material the  $^{14}\text{C}:^{12}\text{C}$  ratios of the lake waters they inhabit, which may, in turn, be depleted by “hard-water” influences. However, a less critical approach has been taken towards the terrestrial plant macrofossils that have been dated, and it has generally been assumed that reliable AMS  $^{14}\text{C}$  ages can be obtained, irrespective of the species of plant remains that are being dated.

This assumption has recently been challenged, however. Turney et al. (2000) report AMS  $^{14}\text{C}$  dates from *Salix herbacea* leaves, *Carex* seeds and bulk organic detritus from a LGIT profile at Finglas River in southwest Ireland. These show systematic age differences between the dated series from the two types of macrofossils, with those obtained from *Salix herbacea* leaves being 900–1500  $^{14}\text{C}$  yr younger than those obtained from *Carex* seeds from the same horizons in the profile. The *Carex* results tend to be more in accord with a third series of dates obtained from samples of bulk organic detritus, although the latter invariably registered the oldest age in each dated horizon. Careful evaluation of the three dating series and of the litho- and biostratigraphic contexts suggested that the *Salix* leaves provided the most reliable age estimates, that the bulk organic detritus samples were probably contaminated by mineral carbon and/or older organic detritus reworked from the catchment soils which also incorporated the *Carex* seeds. Similarly, recent investigations at the site of St. Bees in northwest England show systematic differences in  $^{14}\text{C}$  age between terrestrial plant macrofossils of LGIT age and coleopteran remains (Walker et al. 1999b).

These data suggest that certain types of plant macrofossil (e.g. *Carex* seeds in the case from Finglas River site) may provide age estimates that are as aberrant as those obtained from contemporaneous lake sediments in which mineral carbon or hard-water-errors may occur. The conclusion must therefore be that, in certain sites and under certain conditions, AMS dates on terrestrial plant macrofossils are no more superior to bulk sediment samples in the dating of events during the LGIT. Indeed, it may well be that the most reliable chronology can be obtained not from the plant macrofossils, but rather from the “humic” sediment component, i.e. that part of the sediment fraction where there will effectively be no contamination by older carbon residues. In terms of dating strategy, therefore careful biostratigraphical investigations are required, coupled with the careful screening of plant macrofossils prior to dating, especially where high-precision geochronology is the objective.

#### *Marine Reservoir Effect*

Fossils obtained from marine sequences display an “apparent age”, or marine reservoir effect, caused by the slow mixing of ocean waters, and the upwelling of  $^{14}\text{C}$ -depleted waters near some coasts. Accordingly,  $^{14}\text{C}$  laboratories normally advise on an appropriate correction factor for those  $^{14}\text{C}$  measurements obtained from marine fossils, which is derived by measuring  $^{14}\text{C}$  activity in contemporary marine organisms. Examples of the scale of this marine reservoir correction are  $190 \pm 40$  for coastal Peru and Chile (Southon et al. 1995),  $355 \pm 20$   $^{14}\text{C}$  yr for coastal Iceland (Håkansson 1983), about 400  $^{14}\text{C}$  yr for coastal waters of the UK, parts of the North Atlantic and submerged corals around Barbados (Harkness 1983; Bard et al. 1987, 1991; Southon et al. 1992), about 580  $^{14}\text{C}$  yr for the Adriatic Sea and parts of the eastern Pacific (Shackleton et al. 1988; Langone et al. 1996) and  $788 \pm 33$   $^{14}\text{C}$  yr for the northeastern Pacific (Southon et al. 1992). It is important that the values of marine reservoir correction factors are firmly established, in view of the fact that marine circulation played a key role in driving climate perturbations during the LGIT (Björck et al. 1996). Uncertainty over the appropriate correction factor to apply reduces the level of precision in correlations between marine and terrestrial LGIT sequences (e.g. Asioli et al. 1999) and between marine and ice-core records (e.g. Voelker et al. 1998). It also limits the precision with which the timing of global melt-water discharge and sea-level rise can be reconstructed (e.g. Bard et al. 1996).

A more serious difficulty has recently emerged with respect to marine reservoir effects, namely that marine reservoir values may not only have varied spatially, but also temporally during the LGIT. Comparison of  $^{14}\text{C}$  ages obtained from marine planktonic foraminifera and from terrestrial deposits of the same age, age-equivalence having been established by tephrochronology (using the Vedde Ash Bed, Wastegård et al. 2000), suggest that the atmosphere-sea surface  $^{14}\text{C}$  difference in the North Atlantic was approximately 700–800  $^{14}\text{C}$  yr at the time of deposition of the Vedde Ash (during the “Younger Dryas/Greenland Stadial 1”) compared with the present difference of around 400–500  $^{14}\text{C}$  yr (Bard et al. 1994). By contrast, reservoir ages along the Norwegian coast during the preceding interstadial (“Bølling/Allerød/Greenland Interstadial 1”) were comparable with present-day values (Bondevik et al. 1999). In the Baltic Sea, however a marine reservoir age in excess of 1000 yr has been inferred for the early Holocene (Björck et al. 2000). Indeed over the course of the last 50 ka, the magnitude of the reservoir effect in the Iceland and Norwegian Seas may have varied by as much as 1600  $^{14}\text{C}$  yr, perhaps as a result of variations in geomagnetic field intensity, coupled with ocean circulation changes (Voelker et al. 1998).

Clearly, further work is needed in order to improve our understanding of temporal (and spatial) variations in the magnitude of marine reservoir effects in different ocean sectors. Comparison of  $^{14}\text{C}$  measurements obtained from marine and terrestrial fossils whose age-equivalence has been estab-

lished by some independent means (e.g. tephrochronology or paleomagnetic stratigraphy) would appear to be the best way forward.

### **Analytical Precision and Laboratory Contamination**

Two problems that are frequently encountered in the dating of LGIT sediment sequences, and which affect the levels of analytical precision that can be attained, are: 1) the fact that most organic sediments that accumulated during the LGIT, especially in northern latitudes, have a low organic C content, and 2) the majority of investigations of this time period have been based on coring which inevitably restricts the size of sediment samples that are available for dating. The latter applies equally to the AMS dating of plant macrofossils, as many LGIT sediments have relatively low fossil concentrations.

In the case of radiometric dating, a combination of low organic C content and limited sediment sample size impacts adversely on analytical precision. As a consequence, the standard errors on LGIT samples are typically around, or even in excess of, 100  $^{14}\text{C}$  yr, even in the case of measurements completed relatively recently (e.g. Preece and Bridgland 1999). This level of precision is not markedly different from that achieved in the early days of  $^{14}\text{C}$  dating. However, where open section sites have been sampled, and commensurately larger volumes of material extracted for dating purposes, counting statistics have improved dramatically with levels of precision of  $\pm 45$  to  $\pm 50$  being routinely achieved (Walker et al. 1993, 1999b). Nevertheless, further improvement in precision would seem unlikely, unless unusually long counting times are employed (Pilcher 1991).

In the case of AMS dating, the limits on precision are much more a function of the purity of the targets prepared and the performance capability of the AMS equipment (e.g. Van der Plicht 1995; Chen et al. 1995). It should be noted, however, that samples for AMS dating are often very small (less than 4 mg by weight and with a carbon yield measurable in  $\mu\text{g}$ ), and often with a low organic C content. Although significant improvements have been made in both target preparation and in equipment sensitivity, the levels of precision of  $^{14}\text{C}$  dates achieved routinely by AMS measurement of samples of LGIT age remains between  $\pm 50$  and  $\pm 150$  (e.g. Sirocko et al. 1993; Björck et al. 1996; Gulliksen et al. 1998; Voelker et al. 1998; Preece and Bridgland 1999; Asioli et al. 1999; Rühlemann et al. 1999), even where weighted means from multiple measurements have been employed (Burr et al. 1998). Indeed,  $\pm 100$  yr and above is frequently the norm, and it is one of the few disappointments of the AMS method that, despite initial aspirations to achieve higher levels of analytical precision, in many instances counting statistics have not improved significantly.

Clearly, in view of the temporal resolution that is now required in the reconstruction of events during the LGIT, further improvement in levels of  $^{14}\text{C}$  dating precision remains a research priority. Although increased precision may be best achieved, perhaps, through “wigggle-matching” (to calibration curves) of a series of  $^{14}\text{C}$  dates (see below), there is, nevertheless, an imperative to improve analytical precision still further through improved sample preparation protocols, use of additional counters to enable longer counting times, more rigorous analysis of variability in laboratory standards, and the development of even purer blanks and targets (e.g. McNichol et al. 1995; Schneider et al. 1995; Bird et al. 1999).

The general tendency towards the use of smaller samples, and/or samples of very low organic C content, in AMS  $^{14}\text{C}$  dating places even greater *onus* upon the  $^{14}\text{C}$  “user” community to take stringent precautions against contamination. The inadvertent introduction of minuscule amounts of modern organic C into samples of low  $^{14}\text{C}$  activity can have a major impact on the eventual date. Laboratory procedures should therefore be adopted which are designed to reduce the chances of even micro-lev-

els of contamination to an absolute minimum, including, for example, ensuring that the final selection and washing of samples takes place in ultra-clean laboratories or within laminar-flow (positive pressure) cabinets, and avoiding contact with any organic solvents during the treatment of samples prior to despatch to the dating laboratory. The point is elaborated by Wohlfarth et al. (1998), whose work suggests that even the long-term storage of wet macrofossil samples in the laboratory can lead to contamination through the growth of fungi or micro-organisms.

### Calibration

For over two decades, a precise  $^{14}\text{C}$ -calibration curve based on dendrochronologically-dated samples has been available for much of the Holocene, but this has only recently been extended back to approximately 12,000 dendro-years BP (Kromer and Spurk 1998; Spurk et al. 1998), i.e. towards the close of the LGIT (12,000 dendro- or calendar years are equivalent to ca. 10,600  $^{14}\text{C}$  yr). Attempts have also been made to extend calibration further back into the LGIT interval, and beyond using *inter alia*, paired U-series and  $^{14}\text{C}$  dates on coral samples (Bard et al. 1998; Burr et al. 1998),  $^{14}\text{C}$  dating of fossils contained in, or bulk sediment samples obtained from, annually-laminated lake and marine sediments (Hughen et al. 1998a, 1998b; Kitigawa and Van der Plicht 1998a, 1998b), and synchronization or “tuning” of  $^{14}\text{C}$ -dated marine or terrestrial sequences to ice-core records (Voelker et al. 1998). It has been suggested that some of these data sets should be amalgamated to produce an integrated calibration curve for the period 12,000 to >45,000 yr ago (Jöris and Weninger 1996; Van Andel 1998), but Van der Plicht (1999) has argued that the various data-sets deviate from each other to such a degree (by several millennia at approximately 30,000 yr ago, for example) that little confidence can be attached to calibrations derived using such an approach.

The calibration data-set most widely employed for samples dating beyond the dendro-based calibration curve is INTCAL98 (Stuiver and Van der Plicht 1998). The INTCAL98 data-set relies mainly on coral data (dated using both the U-series and  $^{14}\text{C}$  methods), but the paired age-points are few in number and many of the dates have relatively large statistical errors. Furthermore, a standard marine reservoir age of about 500 yr was adopted to link the measured  $^{14}\text{C}$  ages to the terrestrially defined  $^{14}\text{C}$  timescale, but this clearly fails to take account of the temporal variations in the magnitude of the marine reservoir effect (see above). As a consequence, using INTCAL98 to calibrate  $^{14}\text{C}$  dates from the LGIT often significantly increases the statistical uncertainty of the age estimates.

The  $^{14}\text{C}$  “plateaux” (episodes of near-constant  $^{14}\text{C}$  age) that characterize parts of the LGIT time interval (e.g. Ammann and Lotter 1989; Lowe 1991; Bard and Broecker 1992; Lotter et al. 1992; Kromer and Becker 1992; Austin et al. 1995; Wohlfarth et al. 1993) could, in theory, provide a basis for improving the precision of  $^{14}\text{C}$  dates using a procedure analogous to the “wobble-matching” approach. An elegant application of this method at a site in western Norway which employed 70 AMS  $^{14}\text{C}$  dates on both terrestrial plant macrofossils and NaOH-soluble fractions of lake sediment, “pin-pointed” the age of the Younger Dryas-Holocene transition to between 11,500 and 11,600 calendar yr BP (Gulliksen et al. 1998). Five clearly defined episodes of near-constant  $^{14}\text{C}$  age have been recognized during the LGIT centering on approximately 12.7–12.6, 11.4–11.3, 11.0–10.9, 10.4–10.3, and 10–9.9  $^{14}\text{C}$  ka BP (see e.g. Kromer and Becker 1993; Goslar et al. 1995; Björck et al. 1996; Wohlfarth 1996). However,  $^{14}\text{C}$  plateaux are poorly resolved in the INTCAL98 data (Stuiver and Van der Plicht 1998) while in other calibration data sets, such as that based on the Cariaco Basin sequence (Hughen et al. 1998a), plateaux-like features occur over different intervals. At present, therefore, the  $^{14}\text{C}$  data from the LGIT do not permit the kind of precise “wobble-matching” that the dendro-based calibration curve makes possible for the dating and correlation of Holocene events.

HIGH-PRECISION CORRELATION OF LGIT SUCCESSIONS: THE ROLE OF  $^{14}\text{C}$  DATING

In recent years, remarkably similar paleoclimatic reconstructions for the LGIT have been derived from terrestrial fossil records, from marine sediment sequences, and from Greenland ice-core records. Fossil beetle data from the UK (Atkinson et al. 1987), oxygen isotope variations in lake sediments in Switzerland and Germany (Siegenthaler et al. 1984; Von Grafenstein et al. 1999), oxygen isotope ratios, dust content and snow accumulation records in Greenland ice cores (e.g. Alley et al. 1993; Dansgaard et al. 1993; Taylor et al. 1993), and variations in biological productivity in ocean floor sediments in the tropical Atlantic Ocean (Hughen et al. 1996) all show the same general and highly distinctive pattern (Figure 1), which is now so recognizable that it serves as a kind of *leitmotif* for the LGIT (see Lowe and Walker 1997b).

Such a high degree of conformity between terrestrial, marine and ice-sheet records has prompted speculation that the major climatic changes during the LGIT were broadly synchronous, and that they were therefore orchestrated by a common forcing mechanism—possibly the North Atlantic ocean conveyor (Broecker et al. 1985). Many LGIT scenarios for the North Atlantic region are now based on this assumption, the marine or terrestrial records being “tuned” to the GRIP or GISP-2 ice-core records, as their highly resolved chronologies and wealth of paleoclimatic detail make them ideal templates for climatic reconstruction (e.g. Jöris and Weninger 1996; Hughen et al. 1998a; Voelker et al. 1998; Von Grafenstein et al. 1999). Indeed the INTIMATE group<sup>2</sup> (Björck et al. 1998; Walker et al. 1999a) has recommended that the GRIP ice-core record be regarded as the stratotype for the LGIT in the North Atlantic region, and advocate an “event stratigraphic” approach to inter-regional correlation, one which is based on comparing regional paleoclimatic signals (“events”) with those recorded in the Greenland stratotype. The events are the pronounced, high-amplitude cold (stadial) and warmer (interstadial) episodes that are so clearly manifest in the GRIP isotope trace, but which can also be recognized in other proxy climate records from around the North Atlantic region (Figure 1).

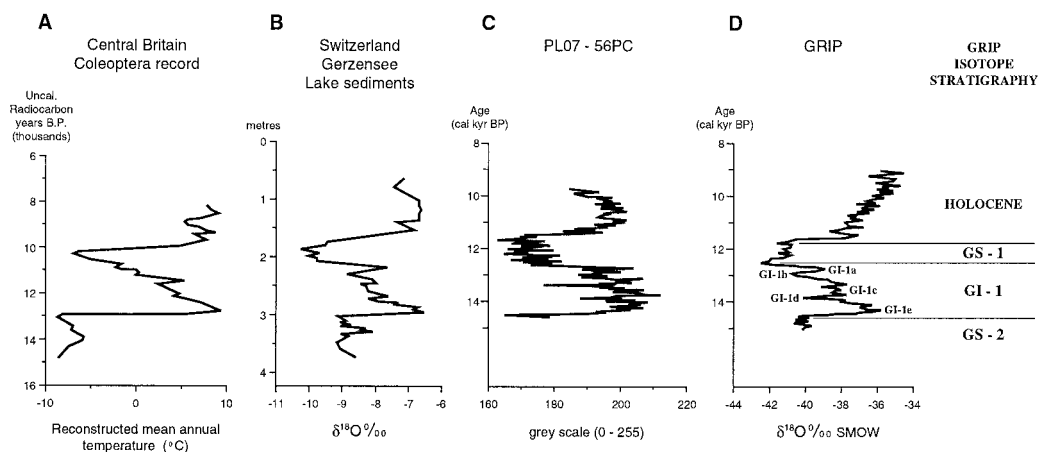


Figure 1 Mean annual temperature variations in Britain during the LGIT (A) (from Atkinson et al. 1987) compared with (B) stable oxygen isotope variations in sediments in Switzerland (Siegenthaler et al. 1984), (C) gray-scale variations reflected in laminated sediments in the Cariaco Basin, tropical Atlantic (Hughen et al. 1996), and (D) oxygen isotope variations in the GRIP ice-core record (Dansgaard et al. 1993). The GRIP isotopoe stratigraphy for the FGIT is described in Walker et al. (1999).

<sup>2</sup>INTIMATE (INTEgration of Ice-core, MARine and TERrestrial records) is a core program of the INQUA (International Quaternary Union) Palaeoclimate Commission.



Walker et al. (1999a) emphasize that this scheme is not intended as a basis for tuning the records to the GRIP stratotype: “tuning” does not *test* whether paleoenvironmental records are synchronous, but rather assumes it. Instead, it is suggested that site and/or regional investigations should comprise three stages: 1) the identification of local (climatic) events, based on independent (proxy) evidence, 2) comparison of the local records with the GRIP stratotype, using “marker” events in both, where feasible, and 3) the use of *independent dating evidence* to establish the degree of synchronicity (or otherwise) between the local and GRIP events. The third step is by far the most difficult to execute, because there is no direct means of establishing the relationship between ice-core and other chronologies.

Attempts have been made to effect time-stratigraphic correlations between terrestrial and/or marine LGIT and ice-core records, using calibrated  $^{14}\text{C}$  dates (e.g. Lowe et al. 1995; Sirocko et al. 1996; Yu and Eicher 1998; Asioli et al. 1999). The results have been generally encouraging, in that they show a degree of compatibility between the timing and duration of the principal LGIT climatic events. However, the often large errors associated with the  $^{14}\text{C}$  dates (generally  $\pm 300$  yr or even greater, at  $2\sigma$ , *excluding* the additional uncertainties introduced by the new INTCAL98 calibration data-set) impart a generally low level of precision to the correlations. Nevertheless the results suggest that  $^{14}\text{C}$  dating could provide a basis for time-stratigraphic correlation, at a relatively high level of precision, *if* materials of sufficient stratigraphical integrity for  $^{14}\text{C}$  dating could be found in those LGIT sequences capable of being analyzed at high temporal resolution and *if* the dendro-based calibration curve, or some alternative offering the same degree of precision and accuracy, extended through the LGIT.

An alternative means of establishing the precise ages and durations of events on land or in the oceans during the LGIT is by the use of varved sequences. A number of annually laminated limnic sequences spanning all or a substantial part of the LGIT have been described in recent years, including those of Lake Gościąg in Poland (Goslar et al. 1992, 1993), several former and extant lakes in Germany (e.g. Hajdas et al. 1993, 1995; Litt et al. forthcoming), the Cariaco Basin in the western Atlantic (Hughen et al. 1998a, 1998b) and Lake Suigetsu in Japan (Kitigawa and Van der Plicht 1998a, 1998b). The problem with these varved sequences, however, is that some may have been interrupted by hiatuses of unknown duration, while most do not extend to the present-day, which makes precise connections to the calendar timescale problematic. Errors associated with the age estimates of LGIT events derived from varve counting are therefore difficult to quantify. In any case, even if it could be established that the varve chronologies are reliable indicators of time, they would provide a chronology for local events only, and could not be used for dating of, and correlation between, more distant sites which do not contain varved sediments.

One approach that offers considerable potential as a basis for correlation between LGIT marine, terrestrial and ice-core records, is tephrochronology. In northwestern and central Europe, visible and microscopic layers of several distinctive tephtras have been found in LGIT sequences. These include the widely disseminated *Vedde Ash*, dated by  $^{14}\text{C}$  to the “plateau” at 10.4–10.3  $^{14}\text{C}$  ka BP (Björck et al. 1992; Birks et al. 1996; Wastegård et al. 1998; Wastegård et al. 2000); the *Laacher See Tephra*, which is found throughout north-central Europe and dated to around 11.2 ka  $^{14}\text{C}$  BP (e.g. Van den Bogaard and Schminke 1985; Hajdas et al. 1995b); the well-dispersed Icelandic *Saksunarvatn Ash*, dated to 10,210  $\pm$  30 cal BP (corrected dendrochronology—see Gulliksen et al. 1998); and the *Borrobol Tephra*, so far found only at sites in Scotland and Northern Ireland, and dated to around 12.26  $^{14}\text{C}$  ka BP (Turney et al. 1997; Lowe et al. 1999). Several tephra layers, including the Vedde Ash (Bard et al. 1994) and collectively termed *North Atlantic Ash Zone I*, occur within LGIT sediment sequences on the floor of the North Atlantic (e.g. Kvamme et al. 1989). At least two of these, the Vedde Ash and Saksunarvatn Ash, have also been found in the GRIP ice-core, the former dated to

10,240 ± 30 BP and the latter to around 12.0 ka GRIP ice-core years (corrected GRIP chronology from Grönvold et al. 1995; S Johnsen, personal communication).

Since each of these tephra layers forms a time-parallel horizon, they not only constitute markers for linking marine, terrestrial and ice-core sequences, but they also have the potential to underpin correlations based on  $^{14}\text{C}$  dating or varve chronology. However, the relatively limited number of tephtras that have so far been detected, coupled with their restricted geographical distribution, means that this potential has yet to be fully realized. Nevertheless rapid progress is being made in this field, and it is anticipated that tephrochronology will become an increasingly powerful tool in dating and correlating LGIT events (Lowe et al. 1999). That said, however, there still remains a need for a more universal dating method, i.e. one that is applicable to all sites in both the marine and terrestrial realms, and the only method that, at present, has such widespread application is  $^{14}\text{C}$  dating.

### **PROTOCOLS FOR IMPROVING THE PRECISION OF $^{14}\text{C}$ DATES OBTAINED FROM LGIT SEQUENCES**

It might reasonably be anticipated that the dendro-calibration curve will eventually be extended to the beginning of the LGIT, or that some other, equally detailed and reliable means will be found for the precise calibration of  $^{14}\text{C}$  dates beyond the present limit of the dendrochronologically derived curve (Van Andel 1998). Once this has been achieved, it will then be possible to establish, with more confidence, the calendar ages of events represented in marine and terrestrial LGIT sequences, by “wobble-matching” to the major inflexions. Indeed it appears that such “inflexions”, which reflect sudden changes in atmospheric  $^{14}\text{C}$  enrichment ( $\Delta^{14}\text{C}$ ), were even more pronounced during the LGIT than during the Holocene, because of abrupt changes in ocean ventilation during the LGIT. These would have affected the rate of exchange of  $^{14}\text{C}$  between the atmosphere and oceans and hence modulated the effects of the geomagnetic field strength, which seems to be the principal driver of the  $\Delta^{14}\text{C}$  variations (Voelker et al. 1998).

Until a more reliable basis for calibration is achieved, however, the question that arises is how best to ensure that the  $^{14}\text{C}$  data-sets are of adequate quality for use in calibration procedures. It is axiomatic that if high-precision dating is to be achieved, those chronologies should only be based on data that meet the highest quality assurance criteria. Yet published reports of  $^{14}\text{C}$  chronologies obtained from LGIT sequences rarely provide more than skeletal information about sampling strategies, about the nature of the materials supplied for dating, or about the laboratory protocols and pretreatments employed prior to  $^{14}\text{C}$  measurement. In too many cases where dates obtained from bulk sediment samples are reported, there appears to have been no, or very limited, advanced testing of the suitability of the materials for  $^{14}\text{C}$  dating (measures of the organic C content of the sediments; measures of percent carbonate; “humic” versus “humin” enrichment values, etc.). Often, too, there is little or no information provided on the laboratory procedures employed during the extraction of the material from cores or monoliths, or the length of time that has elapsed between the collection of the samples and delivery to the dating laboratory, of the conditions under which samples were stored, etc. In discussion of the dates, too, it is seldom acknowledged that the quoted error ranges represent only the compounded uncertainties associated with isotopic analyses (and note that these are quoted to  $1\sigma$  only), and by no means reflects the realistic extent of chronological confidence. Objective interpretation should include some quotient to represent the accumulated effects of geological, sample contamination, and pretreatment uncertainties, although these are rarely quantifiable. True confidence ranges will, therefore, in most instances be significantly greater than the analytical error as expressed in  $^{14}\text{C}$  years.

Table 1 Issues to be considered in the design of laboratory protocols and reporting of  $^{14}\text{C}$  age determinations that may be used in high-precision dating and correlation

- 
1. *Sample integrity (e.g. for bulk organic sediment samples)*
    - provider's sample number;
    - single measurement, or part of a series of measurements?  
if 'yes', specify provider and radiocarbon lab. numbers for all other samples in series;
    - information of stratigraphic (temporal) resolution of sequence from which samples have been obtained;
    - nature of material dated;
    - organic C content (LOI);
    - carbonate content;
    - nature of any clastic residue;
    - wet weight of sample submitted to laboratory;
    - total organic C (dry wt.) used in radiocarbon measurement;
    - tests for radiocarbon activity heterogeneity of bulk material (e.g. 'humic' versus 'humic'; other fractions tested);
  2. *Laboratory handling (provider)*
    - date of collection of samples;
    - date of submission of samples to radiocarbon laboratory;
    - storage conditions between date of collection and date of submission;
    - treatment of samples over storage period;
    - nature of solvents, dispersants (etc.) used;
    - conditions under which samples extracted and packaged for transfer to radiocarbon laboratory (e.g. whether under controlled air conditions);
  3. *Radiocarbon laboratory procedures*
    - date samples received;
    - date samples counted;
    - storage conditions between date of receipt and date of count start;
    - pretreatment procedures adopted;
    - formal laboratory sample number;
    - count statistics;
    - corrections applied (e.g. fractionation; reservoir effects);
    - comments on any unusual chemical effects during pretreatment;
  4. *Calibration procedures applied*
    - calibration data-set employed (specify version – e.g. CALIB4.0);
    - specify any smoothing or rounding procedures employed;
    - single calibration, or part of a series of calibrations?  
if 'yes', specify provider and radiocarbon lab. numbers for all other samples in series.
- 

A distinction needs to be made between dates that give only a general indication of age, and those intended to form a basis for precise geochronologies. Many  $^{14}\text{C}$  dates are obtained simply as "rangefinder" age estimates, in order to be able to allocate a stratigraphical unit or horizon to a specific time interval. Such dates were never intended for high-precision reconstruction. Nevertheless, we would contend that, even in such cases, some minimal information ought to be provided about the nature of the materials employed for dating, about the handling of the materials prior to, and during,  $^{14}\text{C}$  measurement, and about the counting procedures. Otherwise it is very difficult to assess the reliability of the resulting chronologies, and to recognize where reasonably narrow counting errors are excessively misleading. Where, on the other hand, the objective is to date events or horizons *precisely*, then there is a greater *onus* on the operator to provide contextual information about the mate-

rials dated, their stratigraphic integrity, and the laboratory procedures adopted. This becomes crucial in the assessment of  $^{14}\text{C}$  dates for inclusion in international databases, the purpose of which is to provide a geochronological underpinning for large-scale paleoenvironmental reconstructions.

There is a need, therefore, for the user community to recognize and to adopt more stringent laboratory protocols in order to meet the minimal quality assurance standards that will enable appropriate screening of  $^{14}\text{C}$  dates prior to inclusion in international databases. Table 1 (above) lists some of the factors that might need to be considered in the design of such protocols, and includes suggestions as to the sorts of questions that might need to be posed. Some of this information is included in the laboratory reports provided to the user by the  $^{14}\text{C}$  laboratory, and subsequently published in *Radiocarbon*. However, other equally important information does not appear in these reports, and there is no consistent codification of the contextual data. These and related matters have recently been considered by the INTIMATE international collaboration group (see e.g. Björck et al. 1998; Walker et al. 1999a), whose principal aims are to achieve more precise correlations between marine, terrestrial and ice-core records from the LGIT. A series of international workshops over the next four years is designed to further these aspirations and to offer appropriate advice on dating and correlation to those contributing to the work and aims of the INTIMATE program. We would, however, encourage the wider  $^{14}\text{C}$  user-community also to consider the need for improved protocols in both field and laboratory sampling, as well as in the reporting of  $^{14}\text{C}$  dates. In addition, we would invite reflection on how best to codify the detailed contextual information that might be required for the screening of  $^{14}\text{C}$  dates prior to inclusion in the major international databases that are now being assembled.

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