

RADIOCARBON "WIGGLES" IN GREAT LAKES WOOD AT ABOUT 10,000 TO 12,000 BP

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ABSTRACT. High-resolution radiocarbon calibration for the last 14,000 cal yr has been developed in large part using European oaks and pines. Recent subfossil wood collections from the Great Lakes region provide an opportunity to measure ^{14}C activity in decadal series of rings in North America prior to the White Mountains bristlecone record. We developed decadal ^{14}C series from wood at the classic Two Creeks site (~11,850 BP) in east-central Wisconsin, the Liverpool East site (~10,250 BP) in northwestern Indiana, and the Gribben Basin site (~10,000 BP) in the Upper Peninsula of Michigan. Initial AMS dates on holocellulose produced younger-than-expected ages for most Two Creeks subsamples and for a few samples from the other sites, prompting a systematic comparison of chemical pretreatment using 2 samples from each site, and employing holocellulose, AAA-treated holocellulose, alpha-cellulose, and AAA-treated whole wood. The testing could not definitively reveal the source of error in the original analyses, but the "best" original ages together with new AAA-treated holocellulose and α-cellulose ages were visually fitted to the IntCal04 calibration curve at ages of 13,760–13,530 cal BP for the Two Creeks wood, 12,100–12,020 cal BP for Liverpool East, and 11,300–11,170 cal BP for Gribben Basin. The Liverpool East age falls squarely within the Younger Dryas (YD) period, whereas the Gribben Basin age appears to postdate the YD by ~300 yr, although high scatter in the decadal Gribben Basin results could accommodate an older age nearer the end of the YD.

INTRODUCTION

The calibration of the radiocarbon timescale is now well established, in large part through ^{14}C measurement on tree-ring chronologies of European oak and pine for the last 13,000 calendar yr (Kromer et al. 1998; Friedrich et al. 1999; Reimer et al. 2004). In North America, tree rings of bristlecone pine from the White Mountains in California going back about 8600 yr had been used in early efforts to calibrate ^{14}C dates (Klein et al. 1982). Such chronologies from tree rings provide an opportunity for high-resolution calibration, but differences among these high-resolution records may also be useful in detecting effects of past atmospheric circulation and the global carbon cycle.

In an effort to determine the record of ^{14}C production in North America prior to the bristlecone record, we have used wood from numerous sites containing subfossil wood of the Great Lakes region. The long-range goal of the project is to use tree rings to characterize environmental variation between about 14,000 and 4000 cal BP, but in this study we report on ^{14}C analysis of ring sequences from 3 of the older sites of special interest and precisely estimate their absolute ages by matching to the IntCal04 calibration curve. Additionally, some apparent age errors prompted further tests of sample preparation methods.

SITES

The classic Two Creeks site (about 44°20′N, 87°32′W) (Goldthwait 1907; Thwaites and Bertrand 1957; Broecker and Farrand 1963; Leavitt and Kalin 1992; Kaiser 1994) was key to determining the date of the last major glacial advance in the continental USA. A mature spruce forest at that site in east-central Wisconsin (about 40 km east-southeast of Green Bay) was drowned by rising lake levels

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and buried by lake sediment as a re-advance of the Laurentide ice sheet blocked northern drainage outlets, and then was overridden ~11,500 BP by the Lake Michigan lobe during the Two Rivers advance, which deposited a characteristic red till above the older sediments.

At the Liverpool East site (Schneider and Hansel 1990) near the southern end of Lake Michigan (about 41°33′N / 87°17′W) near Gary, Indiana, spruce trees became established following the retreat of glacier ice from northern Indiana and a subsequent rise in lake level during the Calumet phase. The trees were then buried by the deposition of dune sand and lacustrine deposits until ~10,300 BP, late in the Younger Dryas event (Panyushkina et al. 2005).

Finally, the Gribben Basin site (about 46°21′N/87°24′W) (Hughes and Merry 1978; Lowell et al. 1999; Pregitzer et al. 2000) represents an extensive spruce forest in the Upper Peninsula of Michigan near Marquette. The site was buried by outwash ~10,000 BP during a re-advance of the Laurentide ice sheet in the Lake Superior basin.

METHODS

One cross-section of a long-lived tree from each of the sites was initially selected for developing the ^{14}C chronologies. Sequences of 10-yr ring groups were separated along single radii from each of the wood cross-sections. For Gribben Basin, the sequence was composed of thirteen 10-yr subsamples from specimen UPJH-144A; Liverpool East had nine 10-yr subsamples from specimen LSAS-49 plus, for comparison, 3 additional 10-yr subsamples from LSIP-72 that overlapped (by tree-ring crossdating) 3 subsamples from LSAS-49; and Two Creeks initially produced seventeen 10-yr subsamples from TCSL-71A. Later, an even longer-lived specimen (TSIP-12A) was discovered at the Two Creeks site, and based on crossdating with TCSL-71A, provided an opportunity to extend the Two Creeks record. TCIP-12A contributed 8 more 10-yr subsamples, 4 of which immediately predated the TCSL-71A chronology, 2 overlapped the first two 10-yr groups of the TCSL-71A chronology, and two 10-yr groups immediately followed the end of the TCSL-71A chronology. An incomplete (5-yr) subsample on the outside of group TCIP-12A completed the sequence.

Samples were ground to 20-mesh and initially converted to holocellulose by the Jayme-Wise method modified after Leavitt and Danzer (1993). Samples were first extracted with toluene/ethanol and then with ethanol organic solvents in a soxhlet extraction apparatus, followed by boiling in deionized water. Samples were delignified in an acetic acid-acidified, sodium chlorite aqueous solution at 70 °C, and then thoroughly rinsed in deionized water. Samples were combusted to CO_2 on the vacuum lines at the Laboratory of Tree-Ring Research (LTRR), and then converted to graphite and analyzed via accelerator mass spectrometry at the Arizona NSF Accelerator facility.

After the initial round of analysis found all the Two Creeks samples to be about 400 yr younger than previously published ^{14}C dates (see Results section) and a few anomalous results in the other 2 sequences, we undertook a systematic test to determine if sample preparation—namely, the preparation of holocellulose, which lacks any processing with alkaline solutions—might be the source of problems. Two samples from each site were chemically treated to produce a set of samples for ^{14}C dating as follows:

1. The original holocellulose samples were treated with acid-alkali-acid (AAA = HCl-NaOH-HCl);
2. Raw wood from the samples was treated with AAA;
3. The original holocellulose samples were converted to α-cellulose by treatment in a 17% NaOH solution according to the methods described in Sternberg (1989).

Additionally, some of the original holocellulose samples were combusted using the vacuum lines at the NSF Accelerator facility to see if the anomalies might be related to combustion rather than preparation. The α-cellulose component was also separated from some holocellulose samples and combusted to CO_2 at the Arizona NSF Accelerator facility, where they were then converted to graphite and analyzed.

The TCIP-12A subsamples were generated late in the project, and the holocellulose component was not dated to avoid ambiguity. They were pretreated to α-cellulose, combusted to CO_2 at the LTRR, and converted to graphite and analyzed at the Arizona AMS facility.

RESULTS AND DISCUSSION

Radiocarbon Dates

^{14}C ages from the Liverpool East LSAS-49 samples (Table 1) fell between 10,068 and 10,395 BP with the exception of the final decade of 8111 BP. Sample LSIP-72 overlapped 3 decades of LSAS-49 and ranged from 9967 to 10,248 BP. The LSAS-49/17-18 value of 8111 BP is anomalous and considered a suspect outlier, perhaps related to greater degradation (low cellulose content) commonly observed in the outer sapwood of old wood samples. This may result from smaller recovered cellulose available for combustion and greater influence of minor laboratory contamination. The average age of the first 8 decades (excluding the outside decade) is 10,260 BP, which is quite similar to the mean of 10,264 BP obtained on wood and peat from the site (Schneider and Hansel 1990), although the 5 dates were widely scattered from 9080 BP (peat from top of deposit) to 11,290 BP (wood fragments in peat), with 1 date on a trunk in "growth position" of 9920 BP with a standard deviation error of ±200 ^{14}C yr.

The decadal subsamples of UPJH-144A from the Gribben Basin site fell between 8603 and 10,029 BP (Table 1). The final 3 decadal samples in the sequence, dating between 8728 and 8855 BP, are suspect. They were from sapwood, again suggesting some problems with the preparation and combustion of cellulose from the outer rings. However, the oldest sample (8603 BP) was in the middle of the sample cross-section, and combustion of the original holocellulose a second time produced an age of 9812 BP, much more consistent with the other ages. After eliminating the suspect ages above (along with UPJH-144A/19-20 with an age of 9210 BP), the average age of 9850 BP for the first 9 decades is within about 100 ^{14}C yr of the average of 9965 BP for 17 dates from 3 studies at 2 coexisting Gribben Basin forest sites (Hughes and Merry 1976; Lowell et al. 1999; Pregitzer et al. 2000).

At the Two Creeks site, the first results on the TCSL-71A sequence gave a range of ^{14}C ages from 9746 to 11,900 BP over the 170 yr represented, indicating very high interdecadal variability. Even the 9 values with ages of at least 11,000 BP averaged ~11,480 BP, which is ~330 yr younger than the average of 11,810 BP for 17 dates reported by Kaiser (1994) and Leavitt and Kalin (1992), and ~370 yr younger than the average of 11,850 BP reported by Broecker and Farrand (1963). It was this age offset between TCSL-71A and the preponderance of previous dates from the Two Creeks site, together with the high variability in ages in the sequence, that prompted the testing of preparation methods using samples from all 3 sites. The 6 oldest TCIP-71A α-cellulose samples run after the testing show a mean age of 11,740 BP, within 100 ^{14}C yr of the average age reported in the other studies. However, the average age of 2 of the outer 3 subsamples (excluding TCIP-12A/15-16 with an age of 10,920 BP) is 11,480 BP, ~250 ^{14}C yr younger than the inside decades, somewhat more than would be expected from the ~150 yr (rings) between the inner and outer decades that were dated.

Table 1 ^{14}C ages (with lab #s) corrected for $\delta^{13}C$ of sequences of decadal ring groups from 3 sites where combustions to CO_2 were done at LTRR. Holocellulose pretreatment was used on all samples except α-cellulose samples marked with "+". Two trees are represented at the Two Creeks and Liverpool East sites, and their line entries are positioned in relative time, e.g. "TCIP-12A 7-8" is the decade sample immediately preceding both "TCIP-12A 9-10" and "TCSL-71A 1-2," which are the same approximate age. *Denotes samples used in "wiggle-matching" in Figure 2.

Sample	1st date (age BP)	2nd date (age BP)	3rd date (age BP)	Sample	1st date (age BP)
TWO CREEKS					
				TCIP-12A 1-2	11,860 ± 63 (AA69236)+*
				TCIP-12A 3-4	11,689 ± 89 (AA69237)+*
				TCIP-12A 5-6	11,420 ± 59 (AA69238)+
				TCIP-12A 7-8	11,667 ± 89 (AA69239)+*
TCSL-71A 1-2	11,536 ± 73 (AA62039)			TCIP-12A 9-10	11,909 ± 73 (AA69240)+*
TCSL-71A 3-4	10,542 ± 75 (AA56926)	11,690 ± 91 (AA70119)+*		TCIP-12A 11-12	11,898 ± 71 (AA69241)+*
TCSL-71A 5-6	11,900 ± 110 (AA56927)*				
TCSL-71A 7-8	10,133 ± 63 (AA56928)	11,424 ± 63 (AA62030)	12,060 ± 110 (AA70111)+*		
TCSL-71A 9-10	10,910 ± 100 (AA56929)				
TCSL-71A 11-12	11,343 ± 77 (AA56930)	11,282 ± 90 (AA62031)	11,758 ± 86 (AA70118)+*		
TCSL-71A 13-14	9746 ± 72 (AA56931)	11,445 ± 63 (AA62032)			
TCSL-71A 15-16	10,263 ± 81 (AA56932)	11,919 ± 71 (AA70114)+*			
TCSL-71A 17-18	11,073 ± 66 (AA56933)				
TCSL-71A 19-20	10,993 ± 81 (AA56934)	11,588 ± 55 (AA70120)+*			
TCSL-71A 21-22	11,367 ± 74 (AA56935)				
TCSL-71A 23-24	11,458 ± 57 (AA56936)	12,112 ± 76 (AA70117)+*			
TCSL-71A 25-26	10,956 ± 64 (AA56937)				
TCSL-71A 27-28	11,559 ± 97 (AA56938)*				
TCSL-71A 29-30					
TCSL-71A 31-32	11,470 ± 91 (AA56939)				
TCSL-71A 33-34	11,618 ± 63 (AA56941)*				
				TCIP-12A 13-14	11,392 ± 71 (AA69242)+
				TCIP-12A 15-16	10,920 ± 68 (AA69243)+
				TCIP-12A 17	11,567 ± 73 (AA69244)+*

Table 1 ^{14}C ages (with lab #s) corrected for $\delta^{13}C$ of sequences of decadal ring groups from 3 sites where combustions to CO_2 were done at LTRR. Holocellulose pretreatment was used on all samples except α-cellulose samples marked with "+". Two trees are represented at the Two Creeks and Liverpool East sites, and their line entries are positioned in relative time, e.g. "TCIP-12A 7-8" is the decade sample immediately preceding both "TCIP-12A 9-10" and "TCSL-71A 1-2," which are the same approximate age. *Denotes samples used in "wiggle-matching" in Figure 2. *(Continued)*

Sample	1st date (age BP)	2nd date (age BP)	3rd date (age BP)	Sample	1st date (age BP)
LIVERPOOL EAST					
LSAS-49 1-2	10,302 ± 82 (AA62018)*				
LSAS-49 3-4	10,263 ± 59 (AA62019)*				
LSAS-49 5-6	10,155 ± 59 (AA62020)*				
LSAS-49 7-8	10,391 ± 61 (AA62021)*				
LSAS-49 9-10	10,395 ± 62 (AA62022)*				
LSAS-49 11-12	10,068 ± 58 (AA62023)*			LSIP-72 1-2	9967 ± 53 (AA65912)
LSAS-49 13-14	10,277 ± 60 (AA62024)*			LSIP-72 3-4	10,248 ± 54 (AA65913)*
LSAS-49 15-16	10,250 ± 100 (AA62025)*			LSIP-72 5-6	10,072 ± 61 (AA65914)*
LSAS-49 17-18	8111 ± 64 (AA62034)				
GRIBBEN BASIN					
UPJH-144A 1-2	10,029 ± 51 (AA56942)*				
UPJH-144A 3-4	9910 ± 100 (AA56913)*				
UPJH-144A 5-6	9716 ± 76 (AA56914)*				
UPJH-144A 7-8	10,028 ± 74 (AA56915)*				
UPJH-144A 9-10	9724 ± 56 (AA56916)*				
UPJH-144A 11-12	8603 ± 94 (AA56917)	9812 ± 63 (AA62033)*			
UPJH-144A 13-14	9959 ± 62 (AA56918)*				
UPJH-144A 15-16	9568 ± 63 (AA56919)*				
UPJH-144A 17-18	9915 ± 61 (AA56920)*				
UPJH-144A 19-20	9210 ± 130 (AA56921)				
UPJH-144A 21-22	8787 ± 55 (AA56922)				
UPJH-144A 23-24	8855 ± 59 (AA56923)	9874 ± 73 (AA70115)+*			
UPJH-144A 25-26	8728 ± 57 (AA56924)	10,132 ± 62 (AA70113)+*			

Tests of Preparation Methods

Among the 6 samples in the preparation (and combustion) tests (Table 2) were 2 Gribben Basin samples (UPJH-144A/11-12 and UPJH-144A/21-22) for which the original dates on holocellulose were younger by ~1000 ^{14}C yr than expected, although a second date on the UPJH-144A/11-12 was within ~100 ^{14}C yr of the expected age. One of the Two Creeks samples (TCSL-71/13-14) was ~2000 ^{14}C yr younger than expected, although a second combustion was only ~350 ^{14}C yr younger, and the second sample was ~450 ^{14}C yr younger than expected. The 2 Liverpool East samples (LSSL-49/5-6 and LSSL-49/9-10) were seemingly close to the average "expected" age, but were tested because of the great variability in the published ages and therefore uncertainty as to the age of the site (trees).

The results from dating different preparations of 6 samples show some evidence of consistency (Table 1, Figure 1), but a clear pattern that could explain the original ages did not emerge. With the exception of LSAS-49/9-10, all the tests produced ages that were usually significantly older than the original dates on holocellulose. For the Two Creeks samples, the various new pretreatments produced similar ages, with the α-cellulose tending to be ~100 ^{14}C yr older. This was not the case for the Liverpool samples, however, where the α-cellulose pretreatments yielded dates younger by ~150 ^{14}C yr than the other pretreatments, although the ages were still consistent with the dates in the previous study. AAA treatment of raw wood produced an age significantly younger than the other pretreatments only for the UPJH-144A/11-12 sample.

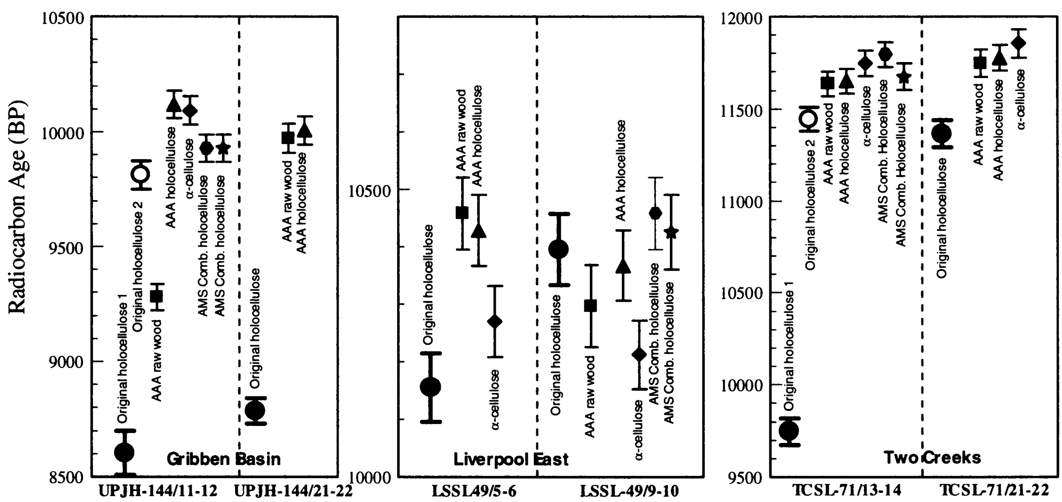

Figure 1 Original dates on holocellulose following combustion at the Laboratory of Tree-Ring Research (LTRR), and dates after various subsequent pretreatments (combustions at LTRR) and combustions of the original holocellulose at the AMS facility (Table 2).

Among the 3 samples for which the original holocellulose was combusted at the NSF Accelerator facility, the UPJH-144A/11-12 and TCSL-71A/13-14 ages were older by ~1500–2000 ^{14}C yr, but in both cases the ages were only about 100–250 ^{14}C yr older than the second LTRR combustions of those samples. However, combustion at LTRR could not be clearly identified as the sole source of anomalous values, because the 2 UPJH-144A/11-12 combustions at the AMS facility were ~200 ^{14}C yr younger than the AAA-treated holocellulose and α-cellulose combusted at the LTRR.

Table 2 Results of tests of preparation using 2 samples from each site. Entries include 1) the original ^{14}C ages (BP) on holocellulose; 2) subsequent dates on AAA (acid-alkali-acid) treatment of holocellulose, AAA-treated raw wood, α-cellulose combusted at the Laboratory of Tree-Ring Research (LTRR); and 3) dates on the original holocellulose combusted at the AMS laboratory for 1 sample from each site. The dates on AAA-treated holocellulose and α-cellulose marked with "*" are included in the wiggle-matching in Figure 2.

Sample	UPJH-144A 11/12	UPJH-144A 21/22	LSSL-49 5/6	LSAS-49 9/10	TCSL-71A 13/14	TCSL-71A 21/22
Original holocellulose	8603 ± 94 (AA56917)	8787 ± 55 (AA56922)	10,155 ± 59 (AA62020)	10,395 ± 62 (AA62022)	9746 ± 72 (AA56931)	11,367 ± 74 (AA56935)
	9812 ± 63 (AA62033)				11,445 ± 63 (AA62032)	
AAA on raw wood	9280 ± 56 (AA67759)	9972 ± 64 (AA67764)	10,458 ± 62 (AA67757)	10,297 ± 72 (AA67755)	11,636 ± 67 (AA67762)	11,748 ± 75 (AA67756)
AAA on holocellulose	10,119 ± 60* (AA67761)	10,006 ± 60* (AA67763)	10,429 ± 62* (AA67758)	10,367 ± 61* (AA67753)	11,651 ± 67* (AA67760)	11,778 ± 69* (AA67754)
α-cellulose	10,093 ± 60* (AA67768)		10,270 ± 62* (AA67766)	10,212 ± 60* (AA67769)	11,746 ± 68* (AA67767)	11,854 ± 75* (AA67770)
AMS combustion of holocellulose- 1	9929 ± 59 (AA67771)			10,458 ± 62 (AA67773)	11,795 ± 67 (AA67772)	
AMS combustion of holocellulose- 2	9986 ± 60 (AA67774)			10,425 ± 65 (AA67776)	11,675 ± 74 (AA67775)	

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Fit of the "Best" Results with the IntCal04 Calibration Curve

Excluding the suspect and questionable results from the initial dating of holocellulose in decade sequences, but including the ^{14}C ages measured during some re-analyses and in the preparation tests for the AAA-treated holocellulose and α-cellulose, we have visually placed these 3 sites into an absolute timescale based on calibrated calendar yr before 1950 (BP) (Figure 2).

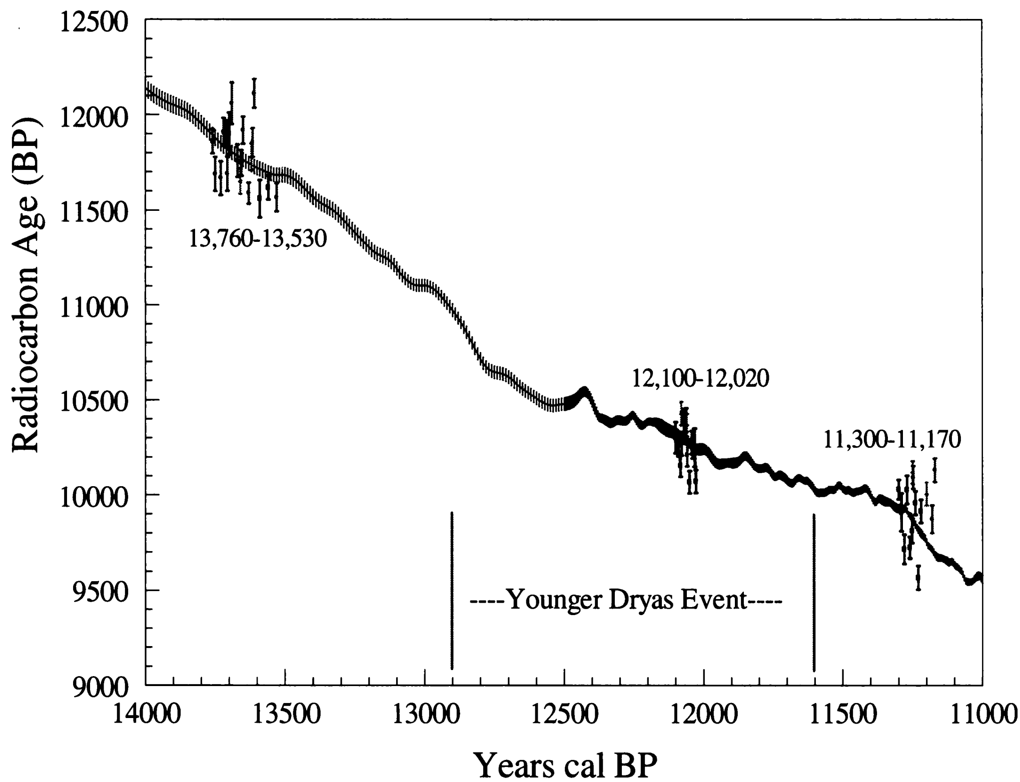

Figure 2 Suggested best fits of dates from this study onto the IntCal04 calibration curve (Reimer et al. 2004). Only dates judged most reliable as discussed in the text were plotted, and those dates came from some of the original holocellulose combustions (asterisks in Table 1) together with ages on the AAA-treated holocellulose and α-cellulose measured during systematic testing of preparation treatments (Table 2). The different shading of plotted ^{14}C ages reflects the pretreatment.

The Two Creeks ^{14}C ages suggest a good fit for the decadal sequence derived from 2 pieces of wood (TCSL-71A and TCIP-12A) is at 13,760–13,530 BP. The Liverpool East sequence seems to fit best at 12,100 to 12,020 BP, which would place it toward the middle of the Younger Dryas event. The Gribben Basin ages show a lot of scatter, but a case could be made for a good fit at 11,300–11,170 BP, which would make the Gribben Basin site about 700 yr younger than the Liverpool trees, and after the end of the Younger Dryas event at ~11,600 cal BP. However, if the 3 youngest ages plotted in Figure 2 are removed, the remaining samples could perhaps be repositioned 300 yr earlier, thereby bringing them within ~400 yr of the Liverpool East site and at the very end of the Younger Dryas event.

CONCLUSION

Ancient wood with many tree rings provides the potential to develop floating ^{14}C chronologies that can be matched with existing calibration curves. The confidence in such "wiggle-matching," however, depends on the internal consistency of ^{14}C results and the confidence we have in them. In this study, some of the original dates produced discrepancies that prompted tests of sample preparation and combustion.

The initial dates on holocellulose from the Two Creeks site were apparently erroneous for nearly all samples. This error does not appear to be an inherent bias introduced during sample combustions to CO_2 at the LTRR, however, because a similar pervasive error did not occur in the Liverpool East samples. Furthermore, the combustions of samples at the AMS facility did not produce ages that were consistently older than ages from LTRR combustions. Samples from the Gribben Basin site exhibited more age discrepancies than those from the Liverpool East site, but fewer than those from the Two Creeks sequence.

Although the error source(s) could not be precisely determined, the collective set of seemingly reliable original dates plus the new dates on AAA-treated holocellulose and α-cellulose were sufficient to visually fit them to the IntCal04 calibration curve to estimate calendar ages (before AD 1950) of the 3 sites. Lack of exact fits to the IntCal04 curve was probably a consequence of the noise in these data sets, but good fits to the calibration curve suggest ages of 13,760–13,530 cal BP for the Two Creeks wood, 12,100–12,020 cal BP for Liverpool East, and 11,300–11,170 cal BP for Gribben Basin. If correct, the Liverpool East trees would have grown near the middle of the Younger Dryas event and the Gribben Basin trees would have begun their growth about 700 yr later, after the Younger Dryas had ended. However, fairly large scatter within the ages of the Gribben Basin decadal samples could accommodate an age at the very end of the Younger Dryas, so the age difference between Gribben Basin and Liverpool East could be as small as ~400 yr.

Continued extension of floating tree-ring chronologies at these sites by means of crossdating of new samples can provide additional decadal tree-ring groups to expand the length of all 3 of these chronologies.

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