Pergamon

Quaternary Science Reviews, Vol. 14, pp. 137–143, 1995.

0277–3791/95 $29.00

0277–3791(94)00123–5

AMS RADIOCARBON DATING OF ANNUALLY LAMINATED SEDIMENTS FROM LAKE HOLZMAAR, GERMANY

IRENA HAJDAS,*‡ BERND ZOLITSCHKA,†|| SUSAN D. IVY-OCHS,‡ JÜRG BEER,* GEORGES BONANI,‡ SUZANNE A.G. LEROY,§ JÖRG W. NEGENDANK,|| MATTHIAS RAMRATH† and MARTIN SUTER‡

**EAWAG-ETHZ, CH-8600 Dübendorf, Switzerland*
†FB VI/Geologie, Trier Universität, D-54286 Trier, Germany
‡Institut für Teilchenphysik, ETH Hönggerberg, CH-8093 Zürich, Switzerland
§Paléontologie et Paléogéographie, UCL, Pl. L. Pasteur 3, B-1348 Louvain-la-Neuve, Belgium. Present address: PAGES/CPO, Bärenplatz 2, CH-3011 Bern, Switzerland
||GeoForschungs Zentrum und Universität Potsdam, Telegrafenberg A26, D-14473 Potsdam, Germany

Abstract — AMS radiocarbon ages have been determined on terrestrial macrofossils selected from the annually laminated sediments of lake Holzmaar (Germany). The radiocarbon chronology of this lake covers the last 12.6 ka. Comparison of the radiocarbon dated varve chronology with tree ring data shows that an additional 878 years have to be added to the varve chronology. The corrected ^{14}C varve chronology of Holzmaar reaches back to ca. 13.8 ka cal. BP and compares favourably with the results from Soppensee (Switzerland) (Hajdas *et al.*, 1993). The corrected ages for the onset and the end of the Younger Dryas biozone are 11,940 cal. BP and 11,490 cal. BP, respectively. The ash layer of the Laacher See volcanic eruption is dated at 12,201 ± 224 cal. BP and the Ulmener Tephra layer is dated at 10,904 cal. BP.

INTRODUCTION

Most late Weichselian/Holocene lake chronologies are based on radiocarbon dating. For a long time, however, it has been difficult to obtain a correct radiocarbon time scale for lake sediments because of the difficulty in obtaining adequate sample size and the problems of hard-water contamination. Traditionally, because of the amount of material required for radiometric analysis, radiocarbon dating was usually done on bulk sediment which enhanced the susceptibility to the hard water effect (Pazdur *et al.*, 1987; Olsson, 1991; Aravena *et al.*, 1992). Due to the input of dissolved 'old' carbon from the surrounding bedrock, the radiocarbon activity of CO_2 in the lake water becomes lower than that of the atmosphere. Older ages of carbonate and authigenic organic matter reflect this disequilibrium. These problems can be resolved by dating material of terrestrial origin. As plant remains found in sediments are typically very small, precise dating of lake sediments became first possible with the help of Accelerator Mass Spectrometry (AMS) (Bonani *et al.*, 1987).

The possibility of precise radiocarbon dating of lake sediments, especially when they are annually laminated, presents an important method of extending the radiocarbon calibration curve. At present, a continuous curve based on tree ring dating reaches back to 10 ka BP. However, further extension of the radiocarbon calibration curve becomes difficult because fossil trees which grew during the Younger Dryas (Kromer and Becker, 1992), have not yet been found. A chronology for the Lateglacial has been derived from laminated sediments from the Swiss lake Soppensee (Hajdas *et al.*, 1993). A longer chronology has been obtained by U/Th and radiocarbon dating of corals (Bard *et al.*, 1992, 1993; Edwards *et al.*, 1993), but as far as can be determined at present, the chronologies do not agree and it is critical therefore that a comparison between different chronologies is necessary in order to derive an extension of the radiocarbon calibration curve.

In this study we present the results of radiocarbon dating on annually laminated sediments from lake Holzmaar (Eifel, Germany).

LAKE AND SEDIMENTS

Lake Holzmaar is a small (5.8 ha) volcanogenic lake located in the highlands of the Eifel, Germany (425 m a.s.l.; 50°7'N, 6°53'E; Fig. 1). This mesotrophic to eutrophic lake is 20 m deep and drains a catchment area of 2.0 km^2. There is a minor inlet as well as an outlet (Sammetbach). The lake basin has very steep-sided walls

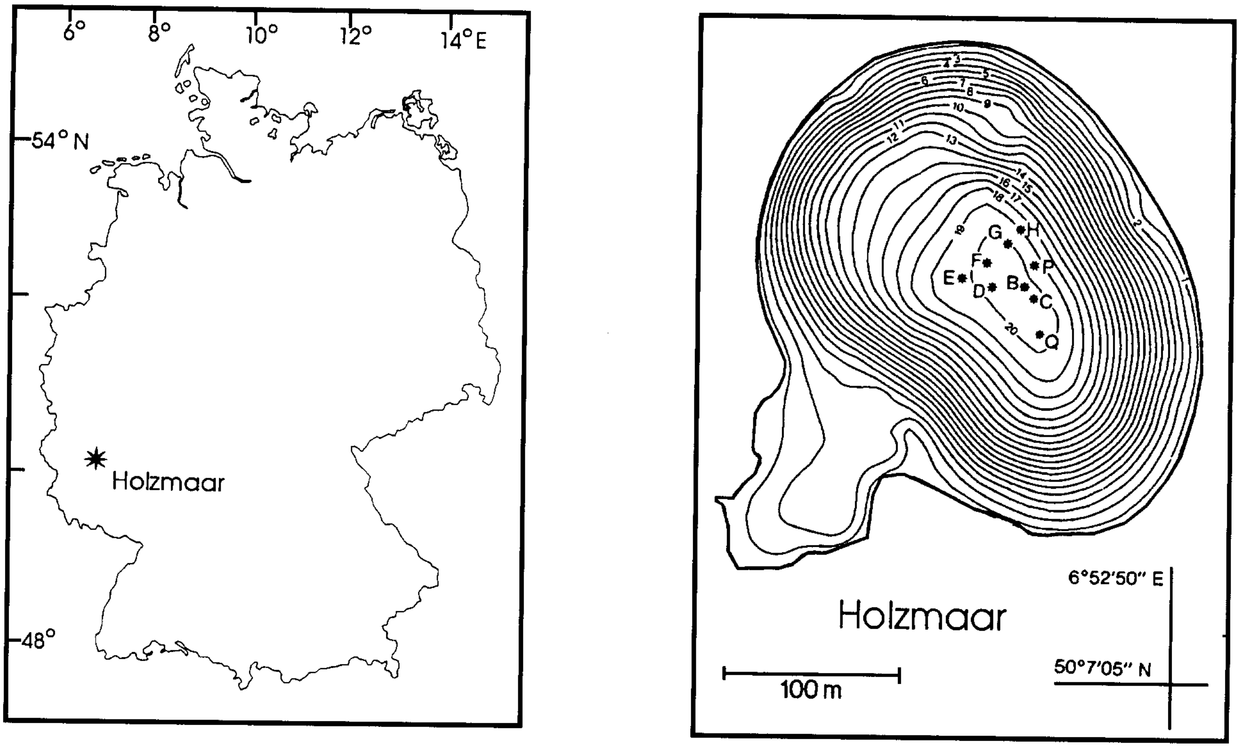

FIG. 1. Map of Germany showing the location of Holzmaar and bathymetric map of the lake with indication of coring sites. Depths of the lake are in metres.

and a flat central area (Fig. 1). The phreatomagnetic explosion which formed the Holzmaar crater occurred more than 25,000 years ago. The most recent studies indicate that laminations in Holzmaar sedimentary record go back to 22,500 varve years BP (Brauer, 1994).

Nine long sediment cores were recovered in 1984 and 1990 from the central area (Fig. 1). All sediment cores include the Lateglacial and the Holocene which comprise ca. 12 m of organic varves. In the lowest part of the Lateglacial sediment clastic varves occur. The records from cores HZM-P, HZM-E and HZM-F provide an additional 20 m of clastic, non-glacial (periglacial) varves which include the Last Glacial Maximum. These periglacial varves contain little organic matter, with total organic carbon (TOC) values of about 0.5% and therefore their radiocarbon dating is rather difficult. The annually laminated sediments of the Lateglacial and the Holocene are composed of pale diatom layers which alternate with dark layers of organic and minerogenic detritus (Zolitschka, 1991). The TOC values vary between 2 and 18%, biogenic opaline values range between 10 and 60%. The occurrence of autochthonous carbonates (precipitated calcite and siderite) are restricted to the Bølling/Allerød and the early Holocene with total inorganic carbon (TIC) values of less than 2 and 1%, respectively. Two tephra layers are present: 78 mm thick Laacher See Tephra (LST) of Allerød age and 1.5 mm thick Ulmener Maar Tephra (UMT) of Preboreal age (Zolitschka *et al.*, *in press*).

Two cores HZM-B and HZM-C were used for detailed sedimentological, palynological, palaeomagnetic and geochemical studies (Negendank *et al.*, 1990; Negendank and Zolitschka, 1993; Haverkamp and Beuker, 1993; Seret *et al.*, 1993). Detailed investigation of the microstructure and measurements of varve thickness were carried out on the composite profile HZM-B/C. Long thin sections (12 cm) were used for microscopic analysis (Zolitschka, 1990, 1991). A master varve chronology, which was established on the combined core HZM-B/C, gives a total of 12,900 varve years before 1950 (Varve Time = VT).

During the coring campaign in 1990, four sediment cores (HZM-E, F, G and H) were recovered from lake Holzmaar. Precise correlation between these cores and the core HZM-B/C was carried out with the help of distinct marker beds, like tephra layers, layers with a prominent colour, or turbidite deposits which were present and easy to recognize in every single core. Samples for AMS radiocarbon dating were selected from correlated depths of all four cores to provide a sufficient amount of terrestrial macrofossil (at least 1 mg).

MACROFOSSILS SELECTED FROM HOLZMAAR SEDIMENT

The sample preparation procedure and AMS measurements of the Holzmaar samples followed the procedure described by Hajdas *et al.* (1993). First the sediment samples were treated with acid and base in order to destroy the sediment structure and facilitate the washing out of the sediment. The residue was examined with a low-power binocular microscope. All fossils which could be identified as terrestrial (Birks, 1980) were picked out with a pair of fine tweezers. In order to remove contamination by carbonates and humic substances, the macrofossils were chemically cleaned using the acid–base–acid method (Olsson, 1986). Preparation of the samples for AMS dating followed procedures described by Vogel *et al.* (1984, 1987) and measurements as in Bonani *et al.* (1987). Conventional radiocarbon ages were calculated according to the procedure given by Stuiver and Polach (1977).

RESULTS

The results of the AMS dating of macrofossils selected from the Holzmaar sediments are presented in Table 1. Conventional radiocarbon ages have been corrected for the isotopic fractionation, $\delta^{13}C$, by measurement of the $^{13}C/^{12}C$ ratio of the dated organic matter.

Macrofossils from Holzmaar sediments containing ash from the Ulmener Maar eruption yielded a date of 9515 ± 75 BP (HZM9). Lake Holzmaar is situated 13 km to the southwest of Ulmener Maar. A sample of plant remains (bark), which was found at a site 400 m southwest of Ulmener Maar embedded in the terrestrial tephra, resulted in a radiocarbon age of 9650 ± 85 BP (ULM2). A piece of charcoal found in the terrestrial tephra, located 2000 m southwest of Ulmener Maar, was dated conventionally and a radiocarbon age of 9435 ± 70 BP was obtained by M. Geyh (*pers. commun.*, 1990; Zolitschka *et al., in press*).

The Laacher See Tephra (LST) occurs in core HZM-B/C at a depth of 964.7 cm. The sample HZM13 (100 years above the LST) contained abundant reworked ash particles. Two samples of macrofossils were selected and radiocarbon dated. Both of the obtained ages (11,210 ± 95 and 11,380 ± 95 BP) are close to the reported dates of the LST (Bogaard and Schminke 1985; Juvigné 1991). However, they are slightly older than 11 ka BP which sug-

TABLE 1. AMS radiocarbon dates from macrofossils selected from sediment of lake Holzmaar. The weight of organic material is given for each sample. Conventional ages were calculated following the procedure by Stuiver and Polach (1977), one sigma error includes statistical error of measurements, blank and standard corrections. $\delta^{13}C$ values were measured quasi-simultaneously

Sample	Depth in HZM-B/C cm	ETH number	Weight o.m. (mg)	^{14}C age ± 1 σ error (BP)	$\delta^{13}C$ ± 1 σ error (‰)
HZM1.1-a	218.5	7238-1	2.6	660 ± 55	–29.1 ± 1.0
HZM1.1-b		7238-1	3.2	710 ± 55	–28.8 ± 1.2
HZM1.2-a	219.5	7238-2	5.4	830 ± 60	–27.5 ± 1.5
HZM1.2-b		7238-2	2.8	755 ± 60	–27.4 ± 1.3
HZM1.3-a	220.4	7238-3	6.7	905 ± 60	–31.5 ± 1.3
HZM1.3-b		7238-3	1.2	1085 ± 75	–21.9 ± 2.6
HZM2.2+3	314.0	7239-2 + 3	ca. 2.0	1565 ± 55	–27.8 ± 1.6
HZM3.1	418.4	7240-1	1.3	2405 ± 60	–27.9 ± 1.0
HZM3.3†	420.9	7230-3	5.3	2750 ± 60	–23.4 ± 1.0
HZM23†	448.2	7260	9.0	2720 ± 60	–21.5 ± 1.0
HZM24	463.0	9500	5.1	2620 ± 65	–28.4 ± 1.7
HZM25	496.1	9501	ca. 3.0	3465 ± 70	–28.6 ± 1.4
HZM26	518.9	9502	ca. 1.0	4100 ± 90	–14.9 ± 1.4*
HZM4.1	556.1	7241-1	4.0	4575 ± 65	–26.6 ± 1.0
HZM4.2	557.2	7241-2	3.3	4730 ± 70	–29.6 ± 1.7
HZM4.3	558.2	7241-3	2.8	4675 ± 70	–25.2 ± 1.4
HZM5.3	676.8	7242-3	5.6	6455 ± 70	–29.2 ± 1.0
HZM6.1	783.0	7243-1	1.4	8800 ± 95	–27.0 ± 1.0
HZM6.3/AQ		7243-3	8.2	9295 ± 85	–21.9 ± 1.0
HZM7-a	837.0	7244	7.4	9450 ± 75	–30.1 ± 0.9
HZM7-b		7244	6.2	9440 ± 75	–28.8 ± 1.0
HZM7-c		7244	6.6	9510 ± 75	–29.4 ± 1.0
HZM8-a	851.2	7245	9.8	9490 ± 80	–28.2 ± 1.6
HZM8-b		7245	3.5	9500 ± 70	–26.0 ± 0.9
HZM9	868.8	7246	5.1	9515 ± 75	–29.3 ± 0.9
HZM10.1	897.5	7247-1	7.8	10,085 ± 80	–31.1 ± 1.6
HZM10.2/AQ		7247-2	4.9	10,670 ± 95	–28.9 ± 1.0
HZM11.3	914.6	7248-3	ca. 2.0	10,195 ± 85	–25.2 ± 1.3
HZM12	937.3	7249	4.2	10,520 ± 90	–28.5 ± 1.6
HZM13-a	956.7	7250	4.5	11,210 ± 95	–35.4 ± 1.6
HZM13-b		7250	5.5	11,380 ± 95	–28.5 ± 1.6
HZM14†	963.0	7251	5.3	11,780 ± 100	–25.9 ± 1.6
HZM16†	988.5	7253	4.0	13,140 ± 140	–31.7 ± 1.6
HZM17	1010.4	7254	3.2	12,100 ± 110	–31.9 ± 1.6
HZM18	1135.4	7255	3.3	12,430 ± 110	–29.6 ± 1.6
HZM19-a	1139.5	7256	5.4	12,590 ± 110	–31.0 ± 1.6
HZM19-b		7256	3.2	12,520 ± 110	–28.6 ± 1.0
ULM2		8156	10.2	9650 ± 85	–22.7 ± 1.0

*Possibly fractionated during graphitization, small sample.
†Turbidite, material could be reworked (see text).

gests that the radiocarbon age of the LST may be closer to 11.2 ka BP as proposed earlier by Hajdas (1993).

The radiocarbon age of 11,780 ± 100 BP obtained on the sample (HZM14), located 18 varve years above the LST, seems to be too old in comparison with other results. It is possible that the organic material used for dating, was reworked immediately following the Laacher See eruption. This is also suggested by pollen studies of this sediment which show the presence of reworked pollen grains. Unfortunately, the sample closest to the LST (HZM15) contained too little organic matter and therefore could not be dated.

An even older radiocarbon age was obtained for the sample HZM16 which was dated at 13,140 ± 140 BP (Table 1). Pollen analyses showed that the sample HZM16 contained reworked material. However, as this sample was made up of wood and seeds of *Potamogeton*, the very old age may be caused by dating of reworked material and/or due to the hard water effect. For further discussion, the ages from samples of aquatic and presumably reworked material are excluded. In Fig. 2, the ^{14}C results are plotted against sediment depth.

As a test of the hard water effect, macrofossils of aquatic plants were dated and their ages were compared with the ages of terrestrial macrofossils. Although the sediment of lake Holzmaar is poor in carbonates (Zolitschka, 1990), the samples of aquatic plants are older than the samples prepared from terrestrial macrofossils found in the same sediment layer. The age difference is 495 and 585 years for sample HZM6 and HZM10, respectively.

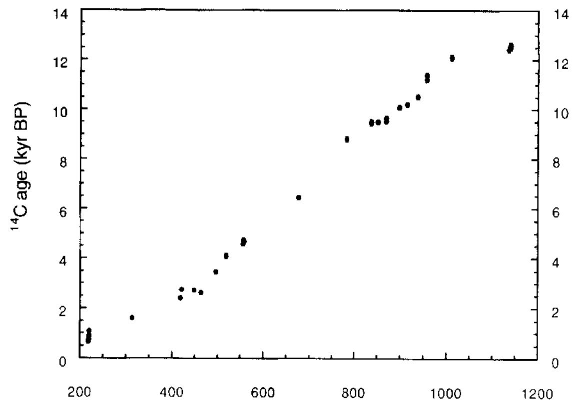

FIG. 2. Conventional radiocarbon ages of macrofossils plotted versus depth in the composite profile HZM-B/C. The one-sigma error for the radiocarbon ages is comparable to the size of the circles.

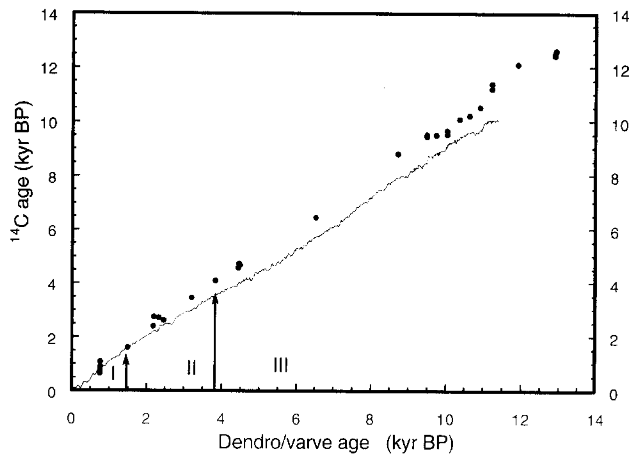

FIG. 3. Conventional radiocarbon ages of macrofossils plotted versus Varve Time (VT) based on varve counting. The shift of ca. 900 years (see text) between the tree ring curve and the varve chronology is observed between ca. 2000 to 4000 cal. BP.

HOLZMAAR RADIOCARBON-VARVE CHRONOLOGY

In Fig. 3 radiocarbon ages are plotted against Varve Time (Zolitschka, 1990, 1991). The first 1500 years of the varve chronology (0 to 1488 years VT, section I), follow the calibration curve perfectly. Beyond 3838 VT there is a shift of ca. 900 years (section III). The difference between VT and dendro time scale seems to occur within sediment section II (Fig. 3). Recent investigations showed that the interval between 3838 VT and 3200 VT is characterized by a very low sedimentation rate (0.39 mm/year). Also, over an interval of 24 cm, a complete lack of diatom-rich sublaminae is observed. Instead of diatom/organic detritus alternation in varve structure, which predominates throughout the Holocene, laminations are only composed of organic detritus and faint clay layers. These couplets have been interpreted as representing annual laminations. However, as suggested by results from radiocarbon dating, this led to an underestimated number of years (varves) counted.

Although shifted, the radiocarbon–VT curve for Holzmaar shows many similarities to the tree ring curve, i.e. the curves are clearly parallel (section III in Fig. 3), which indicates reliability of the radiocarbon dating of the Holzmaar sediment.

CORRECTED VARVE TIME IN HOLZMAAR VARVE CHRONOLOGY

In order to place the radiocarbon ages on the calibration curve (Kromer and Becker, 1993), the floating varve chronology (all beyond 3838 VT) was fitted to the dendro-calibration curve. The χ^2 statistical method was used to minimize the difference between both curves. In this way, the correction of 878 years was found and added to the original master chronology (Zolitschka, 1990, 1991) of sediment below 500 cm (Table 2).

The corrected varve chronology of lake Holzmaar follows the dendro-calibration curve (Fig. 4). The 9.6 ka BP plateau as well as the following abrupt change at 10 ka BP clearly show up in the Holzmaar radiocarbon-varve curve. This good agreement with the tree ring curve shows that the varve counting yields an appropriate calendar time scale for the 7.5 ka time span, from 4 ka cal. BP to 11.5 ka cal. BP. The resulting calendar ages of the LST and UMT in the adjusted VT are: 12,201 ± 224 cal.

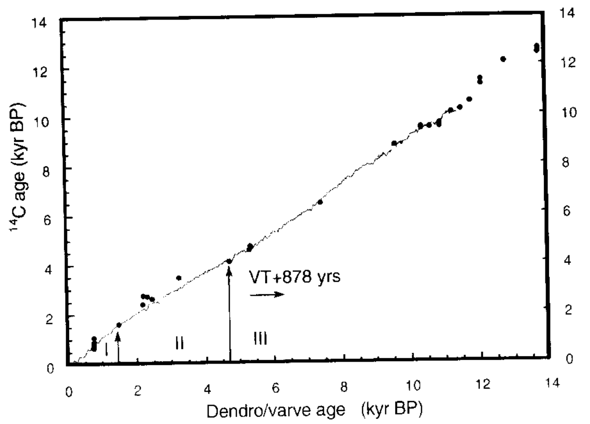

FIG. 4. Corrected varve chronology: a correction of 878 years was added to the VT of samples older than 3838 VT BP (section III). In the case of samples in segment II, correction between 0 to 878 years is probably required but not made here. In segment I no corrections are needed, i.e. varve time corresponds to the calibrated age of the dated samples.

BP and 10,895 ± 177 cal. BP, respectively. On the basis of this varve chronology, the time scale for the Holzmaar Lateglacial sediments must also be corrected. The boundaries of the Younger Dryas based on pollen analysis (Leroy, *in preparation*) and increased minerogenic sediment input (Zolitschka *et al.*, 1992) are now dated at 11,940 cal. BP (AL/YD transition) and 11,490 cal. BP (YD/PB).

The absolute age of 12,201 ± 224 cal. BP for the LST in Holzmaar is very close to the age of 12,350 ± 135 ca. BP obtained from the varve chronology of lake Soppensee (Hajdas *et al.*, 1993). This shows that both radiocarbon–varve chronologies of the Lateglacial agree very well. However, a disagreement exists between the varve chronology of lake Holzmaar and ^{14}C–U/Th dated corals (Edwards *et al.*, 1993; Bard *et al.*, 1993) as previously reported for the Soppensee varve chronology (Hajdas *et al.*, 1993). This offset between varves and corals is illustrated by the difference in the absolute dating of the LST. Calibration of the radiocarbon age of 11.2

TABLE 2. The corrected chronology of lake Holzmaar. The VT of samples older than sample HZM26 was corrected for additional 878 years (see Fig. 2). The shaded area marks the younger part of the varve chronology where no correction is applied.

Sample	Depth in HZM B/C (cm)	Varve time VT (BP) (Zolitschka 1991)	Corrected VT VT + 878 (BP) (this work)	^{14}C age (BP)
HZM1.1-a	218.5	747	747	660 ± 55
HZM1.1-b				710 ± 55
HZM1.2-a	219.5	751	751	830 ± 60
HZM1.2-b				755 ± 60
HZM1.3-a	220.4	757	757	905 ± 60
HZM1.3-b				1085 ± 75
HZM2.2+3	314.0	1488	1488	1565 ± 55
HZM3.1	418.4	2180	2180	2405 ± 60
HZM3.3	420.9	2196	2196	2750 ± 60
HZM23	448.2	2324	2324	2720 ± 60
HZM24	463.0	2464	2464	2620 ± 65
HZM25	496.1	3235	3235	3465 ± 70
HZM26	518.9	3838	4716	4100 ±90
HZM4.1	556.1	4433	5311	4575 ± 65
HZM4.2	557.2	4458	5336	4730 ± 70
HZM4.3	558.2	4484	5362	4675 ± 70
HZM5.3	676.8	6523	7401	6455 ± 70
HZM6.1	783.0	8705	9586	8800 ± 95
HZM7-a	837.0	9485	10,363	9450 ± 75
HZM7-b				9440 ± 75
HZM7-c				9510 ± 75
HZM8-a	851.2	9733	10,611	9490 ± 80
HZM8-b				9500 ± 70
HZM9	868.8	10,026	10,904	9515 ± 75
HZM10.1	897.5	10,367	11,245	10,085 ± 80
HZM11.3	914.6	10,632	11,510	10,195 ± 85
HZM12	937.3	10,908	11,786	10,520 ± 90
HZM13-a	956.7	11,223	12,101	11,210 ± 95
HZM13-b				11,380 ± 90
HZM17	1010.4	11,903	12,781	12,100 ± 110
HZM18	1135.4	12,874	13,752	12,430 ± 110
HZM19-a	1139.5	12,879	13,757	12,590 ± 110
HZM19-b				12,520 ± 110

Shaded area: not corrected VT.

ka BP, which is the radiocarbon age of the LST, by using coral data gives an age of ca. 13,000 cal. BP which is about 1000 years older than the age obtained by the varve chronologies.

SUMMARY

The results of the AMS ^{14}C dates obtained from terrestrial macrofossils from lacustrine sediments of Holzmaar illustrate that varve counting has the potential to be used for building an absolute time scale which is comparable to tree ring data. However, an application of varve counting as a dating method must always consider the possibility of a hiatus which may not be obvious in the sedimentary records. This shows the necessity for study of numerous sites and comparison of various independent varve chronologies. In this study, results of radiocarbon dating have shown that an additional 878 years has had to be added to the younger part of the varve chronology (at 3838 VT) from lake Holzmaar. Due to this correction, the LST layer in Holzmaar sediments is dated at 12,201 ± 224 cal. BP, the onset of the YD biozone is dated at 11,940 cal. BP and the YD/PB boundary at 11,490 cal. BP. The very good agreement with results from Soppensee (Hajdas *et al.*, 1993) suggests that after corrections both varve chronologies represent a real time scale.

ACKNOWLEDGEMENTS

Financial support was provided by Swiss National Science Foundation, the German Science Foundation (DFG Ne 154/21). The work of S. Leroy was enabled by the "Impulse Programme 'Global Change' supported by the Belgian State, Prime Minister's Services, Science Policy Office" and by PAGES (Past Global Change), a core project office of the International Geosphere-Biosphere Programme. Radiocarbon measurements were done at the ETH/PSI AMS facility at Zürich — we thank the tandem crew for technical support. We thank A. Brauer for many discussions and his help in selection of sediment samples for dating.

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