Radiocarbon, Vol 56, Nr 1, 2014, p 181–187 DOI: 10.2458/56.16920
© 2014 by the Arizona Board of Regents on behalf of the University of Arizona

TESTING THE ACCURACY OF ^{14}C AGE DATA FROM POLLEN CONCENTRATES IN THE YANGTZE DELTA, CHINA

Chunhai Li[1] • Yongxiang Li[2] • George S Burr[3]

ABSTRACT. In order to test the accuracy of accelerator mass spectrometry (AMS) radiocarbon dating of pollen, 8 samples of pollen concentrates and 4 bulk organic samples were collected and analyzed from trench T1041 at the Tianluoshan site, Yuyao city, Zhejiang Province. This site was chosen because a reliable chronology had been previously established there based on radiocarbon dates of plant materials. The pollen concentrate samples were measured using AMS ^{14}C and the 4 bulk organic samples were measured by liquid scintillation counting (LSC). The pollen concentrates and bulk organic samples yield ages that are a few hundred years to thousands of years older than those from plant materials, respectively. Contributions from reworked sediments can explain the older ages for the pollen concentrates and sediment organic dates. This study suggests that caution must be exercised when discussing millennial- or centennial-scale climate events based on chronologies that are controlled by age determinations of pollen concentrates.

INTRODUCTION

The Yangtze Delta is an important area for understanding human history and its relationship to paleoenvironmental change during the Quaternary. This area is heavily influenced by the Pacific Ocean and large rivers, especially the Yangtze (Changjiang). In addition, the East Asian monsoon exerts a strong influence here, affecting both the Yangtze Delta ecosystem and human activities. This area is one of the original sites of rice agriculture (Zheng et al. 2009) and is well known for its rich archive of Neolithic remains, as well as its central role in the economy of ancient China. During the past decades, an increasing number of studies have focused on the relationship between environmental change and human history in this area (e.g. Stanley and Chen 1996; Yu et al. 2000; Zhang et al. 2005; Yi et al. 2006; Atahan et al. 2007, 2008; Itzstein-Davey et al. 2007; Zong et al. 2011; Zheng et al. 2012). Elucidating the relationship between environmental changes and human activities requires high-resolution, reliable chronologies. However, few high-resolution, reliable chronologies have been available for this area, and chronologies in many studies have been based on either archaeological layers or radiocarbon dates from bulk sediments. In an assessment of 65 ^{14}C dates from 20 cores from the Yangtze Delta, Stanley and Chen (2000) found that nearly two-thirds of the ^{14}C dates were significantly older than expected due to incorporation of reworked carbon. In contrast, ages obtained from plant macrofossils from lake or swamp sediments are found to agree well with known ages. Therefore, it is always desirable to obtain ^{14}C dates from plant macrofossils. However, plant macrofossils are often very scarce in sediments and thus difficult to collect for accelerator mass spectrometry (AMS) ^{14}C dating. Under such circumstances, pollen concentrates may be used as alternative materials for AMS ^{14}C dating in order to build a chronology (Brown et al. 1989; Regnéll 1992).

The accuracy of AMS dating of pollen concentrates has been tested with sediments from lake (Brown et al. 1989; Kilian et al. 2002), marine (e.g. Mensing and Southon 1999), eolian (Zhou et al. 1997), and wetland (e.g. Vandergoes and Prior 2003; Neulieb et al. 2013) deposits using different extraction methods (Regnéll 1992; Long et al. 1992; Richardson and Hall 1994; Regnéll and Everitt 1996; Mensing and Southon 1999; Vandergoes and Prior 2003). In some cases, pollen concentrates yield much older ages than the actual deposition age. Kilian et al. (2002) used annual laminations of Lake Gościąż in Poland to test the reliability of ^{14}C dates of pollen concentrates and found that the pollen dating results were older than varve ages by many centuries. Vasil'chuk et al. (2005) also

1. State Key Laboratory of Lake Science and Environment, Nanjing Institute of Geography and Limnology, Chinese Academy of Sciences, 73 East Beijing Road, Nanjing, China.
2. School of Earth Sciences and Engineering, Nanjing University, Nanjing 210093, China.
3. NSF-Arizona Accelerator Mass Spectrometry (AMS) Laboratory, University of Arizona, Physics Department, Tucson, Arizona 85721-0081, USA.

https://doi.org/10.2458/56.16920 Published online by Cambridge University Press

reported that dates from pollen concentrates were too old because of the incorporation of redeposited pre-Quaternary pollen and spores. In some cases, younger-than-expected ages are obtained from pollen concentrates due to contamination by younger carbon (Vandergoes and Prior 2003; Neulieb et al. 2013). There are also cases where pollen concentrates produce ages that agree well with expected ages (e.g. Zhou et al. 1997; Neulieb et al. 2013), demonstrating the potential utility of pollen concentrates to construct a reliable chronology.

Since Stanley and Chen (2000) have shown that the majority of ^{14}C dates from the Yangtze Delta were significantly older than expected due to contamination of reworked older carbon, pollen concentrates have been used to reconstruct chronologies in the Yangtze Delta (e.g. Itzstein-Davey et al. 2007; Atahan et al. 2008; Zong et al. 2011). These chronologies are further used to discuss millennial- to centennial-scale climate events such as the 4.2ka event in south China (e.g. Zong et al. 2011). The claim of the existence of the 4.2ka climate event, however, conflicts with records from other areas in China (Liu and Feng 2012). This raises a pressing concern about the reliability of pollen concentrate-derived chronologies in the Yangtze Delta, particularly given the aforementioned uncertainties in terms of pollen dating accuracy. The reliability of AMS dating of pollen concentrates in south China has not been critically assessed. Could the ^{14}C dates of pollen concentrates from the Yangtze Delta also be affected by reworked sediments? Are these pollen-derived ^{14}C dates accurate enough to establish chronologies suitable for studying short-lived climate events?

To answer these questions, we have conducted a detailed analysis of samples from trench T-1041 in the Tianluoshan area of China (Figure 1). This trench has been intensively investigated previously in terms of its stratigraphy, pollen assemblages, phytoliths (Li et al. 2012), charcoal abundance, and archaeological significance (Zheng et al. 2009). The age control at this site is robust. The age model of Li et al. (2012) is deemed reliable because (1) ^{14}C ages were derived from macrofossils of upland and emerged plants including Yagara bulrush and Flatstalk bulrush, which are ideal candidates for determining deposition age; (2) 10 ^{14}C ages obtained from a ~1.5-m interval are stratigraphically compatible and show no age reversals; and (3) the ages agree well with archaeological layers in the study area. The availability of such a reliable chronology presents an excellent opportunity for testing the reliability of ^{14}C dating of pollen concentrates.

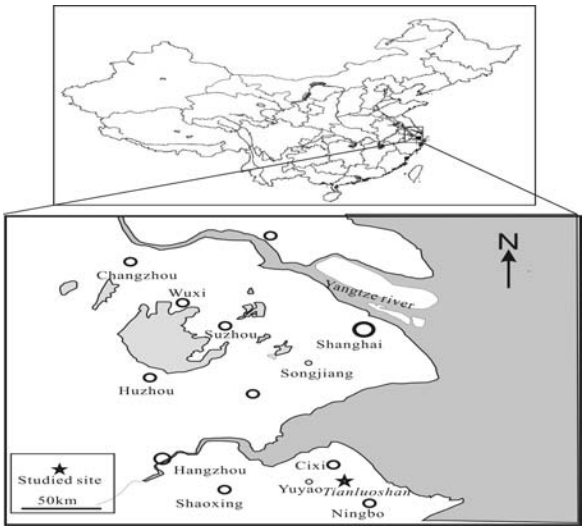


Figure 1 Map showing the location of the Tianloushan site.

https://doi.org/10.2458/56.16920 Published online by Cambridge University Press

MATERIALS AND METHODS

We collected 8 samples from trench T-1041 in the Tianluoshan site to extract pollen grains for AMS ^{14}C dating. Each sample was taken from a 5-cm-thick interval to be consistent with the 5-cm thickness of samples from which each AMS ^{14}C age of plant macrofossils at this site was determined in the previous study (Zheng et al. 2009). For comparison purposes, we also collected four 1-cm-thick bulk samples from this section to determine their ages.

Chemical Treatment and Separation

Thirty grams of material from each sample were used for the pollen extraction. The pretreatment procedure followed the commonly used, widely accepted, standard hydrofluoric (HF) acid method (Faegri and Iversen 1989). Carbonates were removed with 10 vol% HCl at 80°C for 30 min. Humic acids were removed by dissolution in 5 vol% KOH solution at 80°C for 10 min. Silica was removed by dissolution in HF at 80°C for 3 hr. The residues were then treated with 5 vol% HCl at 80°C for 20 min. Organic and inorganic matter larger than pollen and fine organic debris were separated by sieving using 100- and 10-μm mesh sizes. The pretreated samples were sent to Rafter Radiocarbon Laboratory, National Isotope Centre, GNS Science in New Zealand for AMS ^{14}C dating.

Four 1-cm-thick bulk samples were treated with 10 vol% HCl to remove carbonates, then dated using liquid scintillation counting (LSC) in the Institute of Geography and Limnology, Chinese Academy of Science. ^{14}C calibration was conducted using CALIB v 6.0.1 (Stuiver and Reimer 1993). Calibrated ages of plant macrofossil-derived ^{14}C dates were used to establish the age-depth model for trench T1041 by linear interpolation and extrapolation, and the established age-depth model is used as a reference for comparison because ages obtained from plant macrofossils are considered reliable. Based on the age-depth model, a 5-cm thickness for each pollen sample corresponds to an age uncertainty of ~150 yr and a 1-cm thickness interval for each bulk sample represents ~30 yr.

RESULTS AND DISCUSSION

We obtained ^{14}C ages of pollen concentrates from the Tianluoshan site for comparison with the well-established, plant macrofossil-based chronology of Li et al. (2012) to examine the reliability of pollen-derived ^{14}C ages in the Yangtze Delta. ^{14}C results and their calibrated ages are listed in Table 1 and illustrated in Figure 2. It is evident that ages from bulk sediments are much older than those from plant material. Sample KF06026 (93–94 cm) is ~520 yr older than the calculated age for the interval from the plant-based age model, and the other 3 ages are over 2600 yr too old for their corresponding depth levels (Figure 2). The results are consistent with the previous findings of Stanley and Chen (2000) that ^{14}C ages from bulk organics in sediments are typically much older than expected. Ages of pollen concentrates are younger than those of bulk organics and closer to the plant-based age model (Figure 2). Ages of pollen concentrates appear to systematically deviate from the plant-based age model by ~300 yr overall (Figure 2). Since the ~300-yr deviation is greater than the ~150-yr uncertainty of the 5-cm-thick sample used for dating, the pollen-based age model cannot be considered the same as the plant-based age model, i.e. pollen-derived ages are systematically older than those from plant macrofossils for the same depth levels. One possible cause for the systematic deviation of pollen-derived dates toward old ages is the hardwater effect. Pollen analyses in the previous study (Li et al. 2012) show that the pollen spectra of T1041 are predominately composed of upland and emerged swamp plants such as *Typha* and *Cyperaceae*, with almost no submerged plants, suggesting that either there is no hardwater effect or its effect is negligible. Another possible cause for the systematic deviation of pollen-derived ages is sediment storage and/or reworking of sediments by which old pollen grains were transported, redeposited, and incorporated into young sediments, leading to systematically older dates of these pollen concentrates.

https://doi.org/10.2458/56.16920 Published online by Cambridge University Press

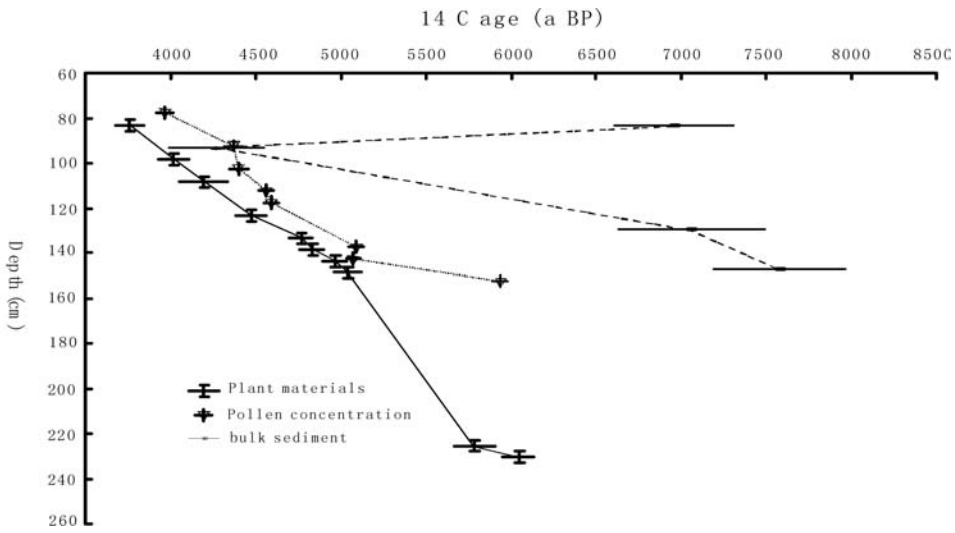


Figure 2 Age model of the studied section based on ^{14}C ages from plant materials, pollen concentrates, and bulk sediments. The mid-point depth of a 5-cm-thick AMS ^{14}C-dated sample and its calibrated median ages were used to construct the age-depth models.

Table 1 Summary of ^{14}C dates from the studied section in the Tianluoshan site. Ages of plant materials (*) are from Zheng et al. (2009) and Li et al. (2012).

Depth (cm)	Lab nr	Material	^{14}C age (yr BP)	Median age (cal yr BP)	$\delta^{13}C$ (‰)	Off value (yr)
81–86	BA07762	Plant remains*	3760 ± 40	4124	—	—
96–101	BA07761	Yagara bulrush*	4015 ± 45	4487	—	—
106–111	BA07760	Bulrush*	4195 ± 70	4715	—	—
121–126	BA08203	Bulrush*	4470 ± 45	5138	—	—
131–136	BA07758	Yagara bulrush*	4765 ± 35	5518	—	—
136–141	BA08895	Yagara bulrush*	4830 ± 35	5546	—	—
141–146	BA08894	Yagara bulrush*	4965 ± 35	5690	—	—
146–151	BA08893	Yagara bulrush*	5040 ± 40	5810	—	—
223–228	BA07764	Flatstalk bulrush*	5785 ± 60	6585	—	—
228–233	BA07763	Flatstalk bulrush*	6045 ± 45	6895	—	—
75–80	NZA-51187	Pollen	3965 ± 26	4438	–28.4	710
90–95	NZA-51186	Pollen	4368 ± 26	4925	–28.74	660
100–105	NZA-51185	Pollen	4402 ± 27	4965	–28.14	390
110–115	NZA-50973	Pollen	4559 ± 22	5266	–27.2	440
115–120	NZA-50972	Pollen	4589 ± 21	5312	–27.36	340
135–140	NZA-50971	Pollen	5090 ± 22	5807	–27.89	280
140–145	NZA-51085	Pollen	5072 ± 22	5814	–28.76	250
150–155	NZA-50970	Pollen	5937 ± 24	6760	–28.85	910
83–84	KF06027	Bulk organic	6960 ± 175	7801	–27.86	3860
93–94	KF06026	Bulk organic	4267 ± 140	4832	–28.13	530
129–130	KF06024	Bulk organic	7059 ± 215	7892	–27.13	2600
147–148	KF06025	Bulk organic	7576 ± 195	8388	–25.85	2600

https://doi.org/10.2458/56.16920 Published online by Cambridge University Press

Sediment storage and reworking of sediments can readily take place during rainstorms. The rainfall in southern China is mainly controlled by the East Asian summer monsoon and by typhoons. Figure 3 shows the average monthly precipitation (AD 1954–2005) of Cixi station, which is about 20 km from Yuyao. Precipitation occurs mainly from March to September. Because of typhoons and summer monsoons, there were 165 rainstorms (>50 mm per 24 hr) including 15 heavy rainstorms (>100 mm per 24 hr) during 1954–2005 in the Cixi area. Rainstorms often cause serious soil erosion (Pimentel and Kounang 1998), which promotes the transport of large amounts of sediments to lakes and the sea. Because of the incorporation of reworked sediments, the ^{14}C ages from bulk organics are typically much older than the actual deposition ages in southern China (Stanley and Chen 2000). Pollen concentrates account for only a small fraction of the bulk carbon in a sample, and they are less susceptible to reworking, but not immune to it, and reworked pollen grains can result in relatively older AMS ages. In some cases, we can identify reworked pollen grains in sediments from southern China.

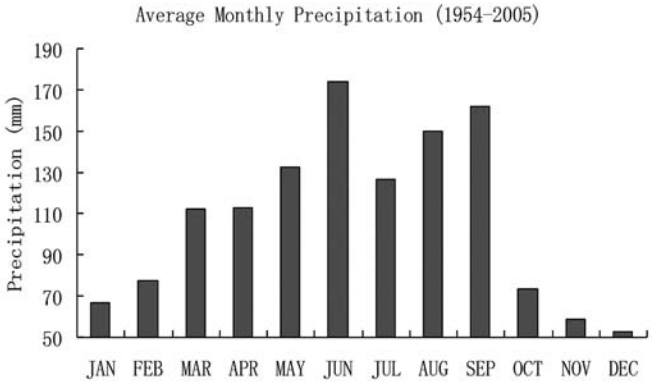


Figure 3 Averaged monthly precipitation (AD 1954–2005) of Cixi station near the study site.

Among the pollen concentrate ages presented here, samples NZA-50970 (150–155 cm) and NZA-51187 (75–80 cm) exhibit the largest deviations toward older ages, as compared to the plant-based age model (Table 1 and Figure 2), amounting to about 900 and 700 yr, respectively. The occurrence of the 2 anomalously old dates suggests that there may be additional factors affecting the ages besides sediment reworking. Zheng et al. (2012) demonstrated that the 150–206 and 20–81 cm intervals were affected by seawater and our 2 anomalous samples were collected from these 2 intervals. Also, marine dinoflagellates (<1%) were found in sediments at a depth of 150–175 cm. Thus, a hardwater effect may affect these particular intervals.

Because the results suggest that ages from pollen concentrates can potentially be several hundred years older than the actual deposition age, we must be very careful when using these data to discuss millennial- or centennial-timescale climatic changes, particularly regarding the relationship between environmental change and human activities in south China. We should reconsider the published sediment records that were based on pollen concentrate dates, and focus on records with reliable chronologies to study climatic and environmental changes. Our age data also imply that although pollen concentrates ages may be a few hundred years older than actual deposition ages, they are clearly superior to dates from bulk sediments in south China both in terms of internal consistency and absolute differences from plant-based chronologies.

https://doi.org/10.2458/56.16920 Published online by Cambridge University Press

CONCLUSIONS

Our age data from the Tianluoshan site in south China show that AMS ^{14}C dates from pollen concentrates appear systematically older than ^{14}C dates from plant materials, presumably due to sediment storage and reworking in the Yangtze Delta. Pollen concentrate dates should therefore be used with caution to study centennial- and millennial-scale climatic changes in south China. However, in the absence of datable plant material, pollen concentrates offer a useful alternative because pollen concentrates generally offer better reliability than bulk organics from sediments, and pollen concentrates provide meaningful age control within limits.

ACKNOWLEDGMENTS

This research is supported by National Basic Research Program of China (No: 2012CB956103), the New Century Excellent Talent in University Awards from the Ministry of Education, China (Awards to Yongxiang Li, NCET-09-0457) and the National Natural Science Foundation of China (41172151). We would like to thank Tim Jull, Mark McClure, and the reviewers for providing constructive comments that helped improve the manuscript greatly.

REFERENCES

Atahan P, Grice K, Dodson J. 2007. Agriculture and environmental change at Qingpu, Yangtze delta region, China: a biomarker, stable isotope and palynological approach. *The Holocene* 17(4):507–15.

Atahan P, Itzstein-Davey F, Taylor D, Dodson J, Qin J, Zheng H, Brooks A. 2008. Holocene-aged sedimentary records of environmental changes and early agriculture in the lower Yangtze, China. *Quaternary Science Reviews* 27(5–6):556–70.

Brown T, Nelson DE, Matthews RW, Vogel JS, Southon JR. 1989. Radiocarbon dating of pollen by accelerator mass spectrometry. *Quaternary Research* 32(2):205–12.

Faegri K, Iversen J. 1989. *Textbook of Pollen Analysis*. Chichester: John Wiley and Sons.

Itzstein-Davey F, Atahan P, Dodson J, Taylor D, Zheng H. 2007. Environmental and cultural changes during the terminal Neolithic: Qingpu, Yangtze delta, eastern China. *The Holocene* 17(7):875–87.

Kilian MR, van der Plicht J, van Geel B, Goslar T. 2002. Problematic ^{14}C-AMS dates of pollen concentrates from Lake Gosciaz (Poland). *Quaternary International* 88:21–6.

Li CH, Zheng YF, Yu SY, Li YX, Shen HD. 2012. Understanding the ecological background of rice agriculture on the Ningshao Plain during the Neolithic Age: pollen evidence from a buried paddy field at the Tianluoshan cultural site. *Quaternary Science Reviews* 35:131–8.

Liu FG, Feng ZD. 2012. A dramatic climatic transition at ~4000 cal. yr BP and its cultural responses in Chinese cultural domains. *The Holocene* 22(10):1181–97.

Mensing SA, Southon JR. 1999. A simple method to separate pollen for AMS radiocarbon dating and its application to lacustrine and marine sediments. *Radiocarbon* 41(1):1–8.

Neulieb T, Levac E, Southon J, Lewis M, Pendea IF, Chmura GL. 2013. Potential pitfalls of pollen dating. *Radiocarbon* 55(3):1142–55.

Pimentel D, Kounang N. 1998. Ecology of soil erosion in ecosystems. *Ecosystems* 1(5):416–26.

Qin J, Wu G, Zheng H, Zhou Q. 2008. The palynology of the first hard clay layer (late Pleistocene) from the Yangtze delta, China. *Review of Palaeobotany and Palynology* 149(1–2):63–72.

Regnéll J. 1992. Preparing of pollen concentrate for AMS dating—a methodological study from a hard-water lake in southern Sweden. *Boreas* 21(4):373–7.

Regnéll J, Everitt E. 1996. Preparative centrifugation—a new method for preparing pollen concentrates suitable for radiocarbon dating by AMS. *Vegetation History and Archaeobotany* 5(3):201–5.

Richardson F, Hall VA. 1994. Pollen concentrate preparation from highly organic Holocene peat and lake deposits for AMS dating. *Radiocarbon* 36(3):407–12.

Stanley DJ, Chen ZY. 1996. Neolithic settlement distributions as a function of sea level-controlled topography in the Yangtze delta, China. *Geology* 24(12):1083–6.

Stanley DJ, Chen ZY. 2000. Radiocarbon dates in China's Holocene Yangtze delta: record of sediment storage and reworking, not timing of deposition. *Journal of Coastal Research* 16(4):1126–32.

Stuiver M, Reimer PJ. 1993. Extended ^{14}C data base and revised CALIB 3.0 ^{14}C calibration program. *Radiocarbon* 35(1):215–30.

Vandergoes MJ, Prior CA. 2003. AMS dating of pollen concentrates—a methodological study of Late Quaternary sediments from South Westland, New Zealand. *Radiocarbon* 45(3):479–91.

Vasil'chuk A, Kim JC, Vasil'chuk Y. 2005. AMS ^{14}C dating of pollen concentrate from Late Pleistocene ice wedges from the Bison and Seyaha sites in Siberia. *Radiocarbon* 47(2):243–56.

https://doi.org/10.2458/56.16920 Published online by Cambridge University Press

Yi S, Saito Y, Yang D-Y. 2006. Palynological evidence for Holocene environmental change in the Changjiang (Yangtze River) Delta, China. *Palaeogeography, Palaeoclimatology, Palaeoecology* 241(1):103–17.

Yu SY, Zhu C, Song J, Qu WZ. 2000. Role of climate in the rise and fall of Neolithic cultures on the Yangtze Delta. *Boreas* 29(2):157–65.

Zhang Q, Zhu C, Liu CL, Jiang T. 2005. Environmental change and its impacts on human settlement in the Yangtze Delta, PR China. *Catena* 60(3):267–77.

Zheng YF, Sun GP, Qin L, Li CH, Wu XH, Chen XG. 2009. Rice fields and modes of rice cultivation between 5000 and 2500 BC in east China. *Journal of Archaeological Science* 36(12):2609–16.

Zheng YF, Sun GP, Chen XG. 2012. Response of rice cultivation to fluctuating sea level during the Mid-Holocene. *Chinese Science Bulletin* 57(4):370–8.

Zhou W, Donahue D, Jull AJT. 1997. Radiocarbon AMS dating of pollen concentrated from eolian sediments: implications for monsoon climate change since the Late Quaternary. *Radiocarbon* 39(1):19–26.

Zong YQ, Innes JB, Wang ZH, Chen ZY. 2011. Mid-Holocene coastal hydrology and salinity changes in the east Taihu area of the lower Yangtze wetlands, China. *Quaternary Research* 76(1):69–82.

https://doi.org/10.2458/56.16920 Published online by Cambridge University Press