

## **<sup>14</sup>C DATING OF HOLOCENE SOILS FROM AN ISLAND IN LAKE PUMOYUM CO (SOUTHEASTERN TIBETAN PLATEAU)**

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**ABSTRACT.** Soil samples from an 85-cm-long continuous section (PY608ES) were collected from an island in Lake Pumoyum Co (southeastern Tibetan Plateau, ~5020 m asl) in August 2006. To estimate past environmental conditions of Lake Pumoyum Co during the Holocene, we analyzed radiocarbon ages, stable carbon isotope compositions, and total organic carbon/total nitrogen (TOC/TN) atomic ratios of the soil samples. The <sup>14</sup>C measurements were performed with the Tandemtron accelerator mass spectrometry system at the Center for Chronological Research, Nagoya University. The <sup>14</sup>C concentration in the surface layer (101 pMC; 5–10 cm soil depth) was nearly modern. A <sup>14</sup>C chronology of the sequence indicated that continuous soil development began on the island in Lake Pumoyum Co at ~5800 cal BP (at 63 cm soil depth, the top of a gravel layer). These results may reflect a decrease in the lake level in the middle Holocene. The age of the obvious lithologic boundary (~5800 cal BP) corresponds to the end of Holocene climate optimum.

### **INTRODUCTION**

Previous studies of soils, loess deposits, and lake sediments have documented environmental changes on the central, northern, and western Tibetan Plateau (Gasse and van Campo 1994; R Wang et al. 2002; Morrill et al. 2006; Thompson et al. 2006). However, the details of environmental changes on the southeastern Tibetan Plateau during the late Quaternary are still largely unknown because of its very high altitude (~5000 m asl) and severe environmental conditions. At present, the southeastern plateau is highly sensitive to monsoon variability because of its location near the limit of monsoonal influence (Wei and Gasse 1999; Shi 2002; Zhang et al. 2006). In addition, the Tibetan Plateau has played an important role in global climatic and environmental changes, especially Northern Hemisphere monsoon circulation during the Quaternary, because of its high altitude (R Wang et al. 2002). Therefore, paleoclimatic and environmental records from the Tibetan Plateau provide important clues for understanding the Asian paleoclimate.

Lake Pumoyum Co is located on the southeastern Tibetan Plateau (28°34'N, 90°24'E; ~5020 m asl; lake surface area 281 km<sup>2</sup>; present maximum water depth ~65 m). Limnological and geological investigations of Lake Pumoyum Co were performed by the China–Japan Scientific Research expeditions during 2001–2006 (Mitamura et al. 2003; Nishimura et al. 2003; Murakami et al. 2007; Watanabe et al. 2008, 2010; J Wang et al. 2009). During the expedition of August 2006, soil samples were collected from an 85-cm-long continuous section on an island in Lake Pumoyum Co. In this study, these soil samples were analyzed for <sup>14</sup>C age, stable carbon isotope composition ( $\delta^{13}\text{C}$ ), and total organic carbon/total nitrogen (TOC/TN) atomic ratios in order to estimate past lake-level changes and environmental conditions on the southeastern Tibetan Plateau.

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### SAMPLES AND ANALYTICAL METHODS

The “Big Island (Island No.1)” in Lake Pumoyum Co has a soil profile at least 58 cm thick, from an underlying gravel layer to the topsoil (Figures 1–3). Soil samples were collected at depths of 5–10, 25–30, 45–50, and 60–63 cm in August 2006 from an 85-cm-long continuous section (natural exposure, PY608ES) on this island (Figures 1 and 2). The samples were passed through a 20- $\mu$ m-opening sieve to remove the coarse fraction (gravel, sand, and roots).

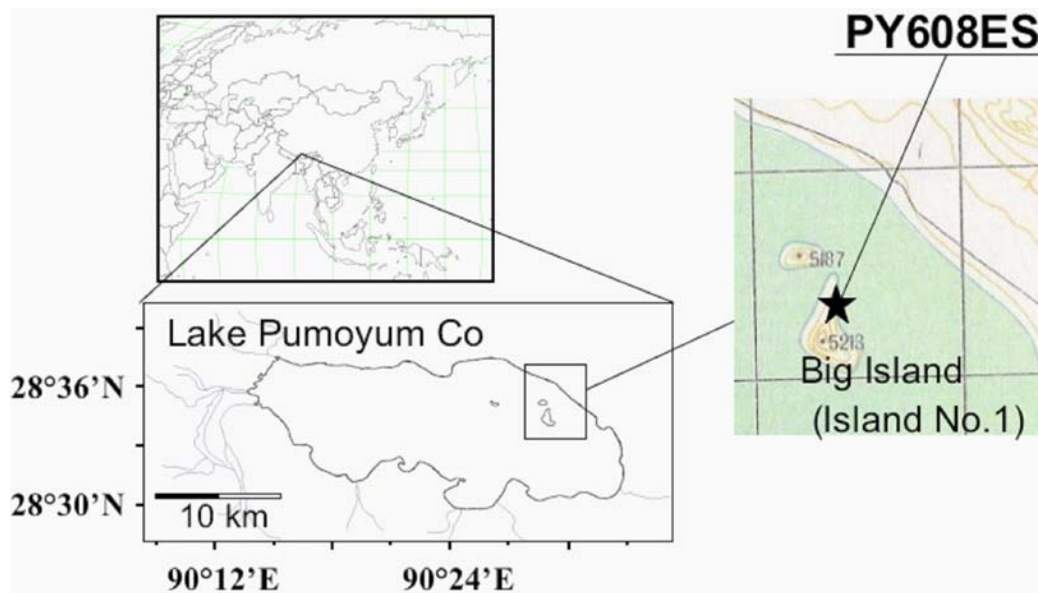


Figure 1 Map showing the location of Lake Pumoyum Co on the southeastern Tibetan Plateau, and the PY608ES sampling site on the “Big Island” (Island No.1, 28°34'52"N, 90°28'55"E) in the lake.



Figure 2 Photograph of the stratigraphy of the natural exposure on the “Big Island (Island No.1)” in Lake Pumoyum Co. The PY608ES sampling site is indicated.

The <20-μm fractions of the samples were dried and powdered and then treated with 1.2M HCl to remove carbonate. The decalcified soils were analyzed for total organic carbon (TOC) and total nitrogen (TN) concentrations with an elemental analyzer (EA) (NA-1500, FISONs). The δ<sup>13</sup>C values of the soil samples were measured using an EA interfaced to an isotope ratio mass spectrometer (IRMS) (MAT-252, Finnigan-MAT) via a ConFlo III split interface. δ<sup>13</sup>C values were expressed as per mil (‰) relative to the Peedee belemnite (PDB). Standard deviations of δ<sup>13</sup>C measurements of an organic compound standard (2,5-bis-(5-tert-butyl-benzoxazol-2-yl)-thiophene, δ<sup>13</sup>C = -26.7‰) were generally less than ±0.1‰.

For <sup>14</sup>C dating of TOC from the samples, the decalcified soils were combusted at 850 °C for 6 hr in evacuated quartz tubes with CuO and Ag wire. The resulting CO<sub>2</sub> was collected and purified in a vacuum line and subsequently reduced to graphite with an iron catalyst and hydrogen at 650 °C for 6 hr. <sup>14</sup>C measurements were obtained with the accelerator mass spectrometry (AMS) system (Model 4130-AMS, High Voltage Engineering Europe) at the Center for Chronological Research (CCR), Nagoya University. The <sup>14</sup>C ages were then converted to calendar years by using the IntCal04 data set (Reimer et al. 2004).

### RESULTS AND DISCUSSION

Vertical profiles of calibrated ages of TOC (<20 μm), TOC concentration, TOC/TN atomic ratios, and δ<sup>13</sup>C values of TOC of the PY608ES soil samples are shown in Figures 3 and 4. The calibrated ages indicate that the PY608ES soil sequence records at least the period since ~4000 cal BP. The TOC concentration of the topsoil horizon at 7.5 cm depth was higher (38 mg/g dry soil) than that of the lower soil layers (average of 14 mg/g dry soil, 25–63 cm in soil depth). TOC/TN atomic ratios (10.1 ± 0.8 on average) and the δ<sup>13</sup>C value of TOC (-23.9 ± 0.5‰ on average, Table 1) differed less among the soil layers (Figure 4a–c, Table 1) and are in agreement with those of modern soil samples (TOC/TN, ~8–9; δ<sup>13</sup>C, about -23‰, Nishimura et al. 2007) around Lake Pumoyum Co.

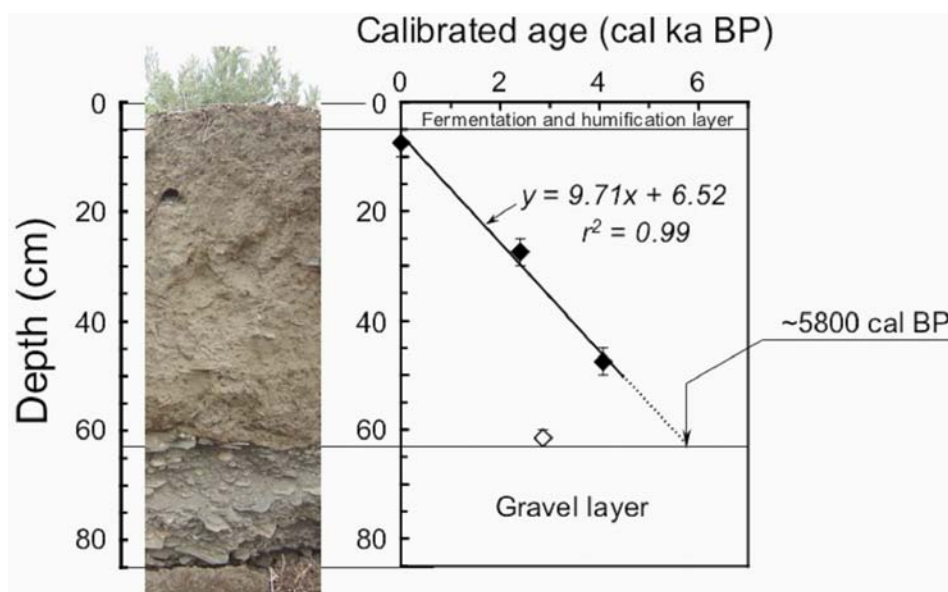


Figure 3 Calibrated ages of TOC (<20 μm) from the PY608ES section. The solid line is the regression line for the calibrated ages of TOC (filled diamonds) from the soil samples from 5–50 cm depth. The open diamond indicates the age obtained from the sample at the lithologic boundary (60–63 cm depth).

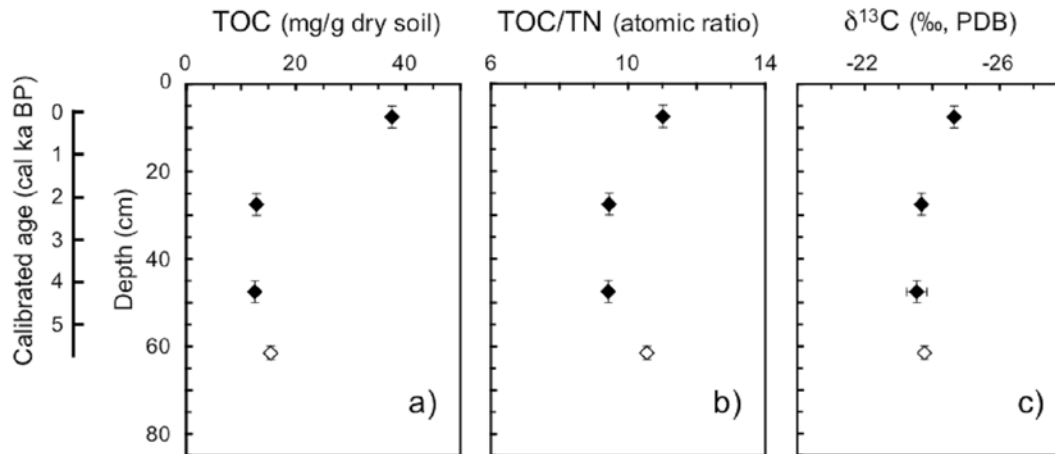


Figure 4 TOC concentrations, TOC/TN atomic ratios, and stable carbon isotope ratios ( $\delta^{13}\text{C}$ ) of the PY608ES soil samples. The filled diamonds show the results from 5–50 cm soil depth, and the open diamonds show the results from the lithologic boundary (60–63 cm soil depth).

Table 1 TOC contents, TOC/TN atomic ratios,  $\delta^{13}\text{C}$  values, conventional  $^{14}\text{C}$  ages, and calibrated ages for the PY608ES soil samples (grain size  $<20\ \mu\text{m}$ ).

Soil depth (cm)	TOC		$\delta^{13}\text{C}_{\text{organic}}$ (‰, PDB)	Conventional		Lab code (NUTA2-)
	(mg/g dry soil)	TOC/TN (atomic ratio)		$^{14}\text{C}$ age (BP, $\pm 1\ \sigma$ )	Calibrated age (cal BP, $1\ \sigma$ )	
5–10	3.75	11.0	$-24.7 \pm 0.1$	$-67 \pm 20$	modern	11622
25–30	1.28	9.5	$-23.7 \pm 0.1$	$2398 \pm 23$	2353–2456	11621
45–50	1.25	9.4	$-23.5 \pm 0.3$	$3734 \pm 24$	4003–4147	11620
60–63	1.54	10.6	$-23.8 \pm 0.1$	$2760 \pm 81$	2778–2946	12763

The calibrated ages of the TOC ( $<20\ \mu\text{m}$ ) in PY608ES become progressively older with depth (Figure 3), except for the sample from 60–63 cm in soil depth. The  $^{14}\text{C}$  concentration of the topsoil (101 pMC, 5–10 cm in soil depth) was near the modern value ( $\sim 108$  pMC, after Morril et al. 2006). In PY608ES (5–50 cm soil depth), calibrated ages of the TOC ( $<20\ \mu\text{m}$ ) ranged from 0 to 4200 cal BP, corresponding to the late Holocene (Figure 3).

The observed age reversal at the lithologic boundary (63 cm in soil depth, top of the gravel layer, Figure 3) might reflect contamination with modern carbon. Thus, the TOC fraction from 60–63 cm depth may contain a mixture of older carbon and fresh carbon from the overlying soil layer. For the  $^{14}\text{C}$  chronology of the PY608ES soil, this result (60–63 cm in soil depth) was ignored. In this research, the calibrated age at the lithologic boundary in the PY608ES section, obtained by extrapolating the regression line of the other 3 calibrated ages (5–50 cm in soil depth), was  $\sim 5800$  cal BP (Figure 3). However, soil organic matter is the product of an ongoing process (Y Wang et al. 1996; Fröberg et al. 2009). The residence time of the soil organic matter must also be considered (Y Wang et al. 1996). In this study, the estimated age ( $\sim 5800$  cal BP) at the lithologic boundary in the PY608ES section is interpreted as the minimum age for lake retreat and the start of pedogenesis.

A previous study reported a gradual decrease in the Indian monsoon intensity after  $\sim 8000$  cal BP, as determined from  $\delta^{18}\text{O}$  values from a stalagmite from southern Oman (Fleitmann et al. 2003). Gasse and van Campo (1994) also reported a decrease in monsoon precipitation between about 7000 and

5000 cal BP from pollen and lake records from the western Tibetan Plateau. In addition, the East Asian monsoon intensity was inferred to have decreased between about 6000 and 5000 cal BP from the  $\delta^{18}\text{O}$  record from Sanbao and Hulu caves in China (Y Wang et al. 2008). West African monsoon precipitation also decreased abruptly between about 6000 and 5000 cal BP (deMenocal et al. 2000). In these previous studies, the decrease in monsoon intensity/precipitation was associated with the end of the Holocene climate optimum. The estimated age of the obvious lithologic boundary (top of the gravel layer, ~5800 cal BP, Figure 3) in the PY608ES section thus corresponds to the end of the Holocene climate optimum. The beginning of soil formation at this time might have been caused by a decrease in the lake level of Lake Pumoyum Co in the middle Holocene.

## CONCLUSION

Calibrated ages of the TOC (<20  $\mu\text{m}$ ) in soil samples from the PY608ES section on an island in Lake Pumoyum Co, southeastern Tibetan Plateau, become progressively older with depth (0–4.2 cal ka BP), except for the soil sample from 60–63 cm depth. The results of this preliminary study indicate that soil sedimentation on this island began at ~5.8 cal ka BP (63 cm in soil depth, top of gravel layer), which corresponds to the end of the Holocene climate optimum.

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## REFERENCES

- deMenocal P, Ortiz J, Guilderson T, Adkins J, Sarnthein M, Baker L, Yarusinsky M. 2000. Abrupt onset and termination of the African Humid Period: rapid climate responses to gradual insolation forcing. *Quaternary Science Reviews* 19(1–5):347–61.
- Fleitmann D, Burns SJ, Mudelsee M, Neff U, Kramers J, Mangini A, Matter A. 2003. Holocene forcing of the Indian monsoon recorded in a stalagmite from southern Oman. *Science* 300(5626):1737–9.
- Fröberg M, Hanson PJ, Trumbore SE, Swanston CW, Todd DE. 2009. Flux of carbon from <sup>14</sup>C-enriched leaf litter throughout a forest soil mesocosm. *Geoderma* 149(3–4):181–8.
- Gasse F, van Campo E. 1994. Abrupt post-glacial climate events in West Asia and North Africa monsoon domains. *Earth and Planetary Science Letters* 126(4): 435–56.
- Mitamura O, Seike Y, Kondo K, Goto N, Anbutsu K, Akatsuka T, Kihira M, Qung T, Tsering, Nishimura M. 2003. First investigation of ultraoligotrophic alpine Lake Puma Yumco in the pre-Himalayas, China. *Limnology* 4(3):167–75.
- Morrill C, Overpeck JT, Cole JE, Liu K, Shen C, Tang L. 2006. Holocene variations in the Asian monsoon inferred from the geochemistry of lake sediments in central Tibet. *Quaternary Research* 65(2):232–43.
- Murakami T, Terai H, Yoshiyama Y, Tezuka T, Zhu L, Matsunaka T, Nishimura M. 2007. The second investigation of Lake Puma Yum Co located in the Southern Tibetan Plateau, China. *Limnology* 8(3):331–5.
- Nishimura M, Hasuike K, Kitagawa H, Zhu L, Nasu H, Chen Y. 2003. Climatic and environmental changes recorded in a sediment core from Lake Pumoyum Co during the past 18,000 yr in the southeastern Tibetan Plateau. In: Nishimura M, Takada M, editors. *Report on Scientific Research Expedition to Lake Pumayum Co on the Tibetan Plateau, 2001*. Hiratsuka: Tokai University Himalayan Expedition Committee. p 157–77. In Japanese with English abstract.
- Nishimura M, Matsunaka T, Kitagawa H, Zhu L, Hasuike K. 2007. Climatic and environmental changes recorded in a sediment core from Lake Pumoyum Co over the past 18,000 yr in the southeastern Tibetan Plateau—Part I—Based on new results from the PY104 core. In: Nishimura M, editor. *Report on Scientific Research Expedition to Lake Pumayum Co on the Tibetan Plateau, 2004*. Hiratsuka: Tokai University Himalayan Expedition Committee. p 127–57. In Japanese with English abstract.
- Reimer PJ, Baillie MGL, Bard E, Bayliss A, Beck JW, Bertrand CJH, Blackwell PG, Buck CE, Burr GS, Cutler KB, Damon PE, Edwards RL, Fairbanks RG,

- Friedrich M, Guilderson TP, Hogg AG, Hughen KA, Kromer B, McCormac G, Manning S, Bronk Ramsey C, Reimer RW, Remmele S, Southon JR, Stuiver M, Talamo S, Taylor FW, van der Plicht J, Weyhenmeyer CE. 2004. IntCal04 terrestrial radiocarbon age calibration, 0–26 cal kyr BP. *Radiocarbon* 46(3):1029–58.
- Shi Y. 2002. Characteristics of late Quaternary monsoonal glaciation on the Tibetan Plateau and in East Asia. *Quaternary International* 97–98:79–91.
- Thompson LG, Mosley-Thompson E, Davis ME, Mash-iotta TA, Henderson KA, Lin P-N, Tandong Y. 2006. Ice core evidence for asynchronous glaciation on the Tibetan Plateau. *Quaternary International* 154–155: 3–10.
- Wang J, Zhu L, Nishimura M, Nakamura T, Ju J, Xie M, Watanabe T, Matsunaka T. 2009. Spatial variability and correlation of environmental proxies during the past 18,000 years among multiple cores from Lake Pumoyum Co, Tibet, China. *Journal of Paleolimnology* 42(3):303–15.
- Wang Y, Amundson R, Trumbore S. 1996. Radiocarbon dating of soil organic matter. *Quaternary Research* 45(3):282–8.
- Wang Y, Cheng H, Edwards RL, Kong X, Shao X, Chen S, Wu J, Jiang X, Wang X, An Z. 2008. Millennial- and orbital-scale changes in the East Asian monsoon over the past 224,000 years. *Nature* 451(7182):1090–3.
- Wang RL, Scarpitta SC, Zhang SC, Zheng MP. 2002. Later Pleistocene/Holocene climate conditions of Qinghai-Xizhang Plateau (Tibet) based on carbon and oxygen stable isotopes of Zabuye Lake sediments. *Earth and Planetary Science Letters* 203(1):461–77.
- Watanabe T, Nakamura T, Nishimura M, Matsumaka T, Minami M, Kakegawa T, Nara FW, Zhu L. 2008. Radiocarbon chronology of a sediment core from Lake Pumoyum Co in the southeastern Tibetan Plateau. *Verhandlungen der Internationalen Vereinigung für Theoretische und Angewandte Limnologie* 30(4):611–4.
- Watanabe T, Matsumaka T, Nakamura T, Nishimura M, Nara FW, Kakegawa T, Zhu L. 2010. Changes of organic matter sources in sediment cores from a high-altitude lake (Pumoyum Co, southeastern Tibetan Plateau) over the last 19,000 years. *Nuclear Instruments and Methods in Physics Research B* 268(7–8):1070–2.
- Wei K, Gasse F. 1999. Oxygen isotopes in lacustrine carbonates of West China revisited: implications for post glacial changes in summer monsoon circulation. *Quaternary Science Review* 18(12):1315–34.
- Zhang W, Cui Z, Li Y. 2006. Review of the timing and extent of glaciers during the last glacial cycle in the bordering mountains of Tibet and in East Asia. *Quaternary International* 154–155:32–43.