

## RADIOCARBON AND STABLE CARBON ISOTOPE RATIO DATA FROM A 4.7-M-LONG SEDIMENT CORE OF LAKE BAIKAL (SOUTHERN SIBERIA, RUSSIA)

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**ABSTRACT.** A sediment core (VER99G12; core length, 4.66 m) was taken from the Buguldeika Saddle of Lake Baikal in 1999. Radiocarbon measurements of total organic carbon (TOC) and pollen concentrate fractions from the VER99G12 core were performed by a Tandemron accelerator mass spectrometry (AMS) system (Model-4130, HVEE) at Nagoya University. The AMS <sup>14</sup>C ages showed that the VER99G12 core spans the past ~30 cal ka BP (from the MIS 3 to present), and the average sedimentation rate of this core was calculated to be 13.6 cm/kyr based on the calibrated ages. This means that the time resolution of VER99G12 sediment samples in this study is better than ~70–80 yr/cm. Stable carbon isotope ratios of TOC ( $\delta^{13}\text{C}_{\text{TOC}}$ ) in the VER99G12 core varied widely from about 26.6‰ to 31.3‰ during the last glacial/post-glacial transition period (about 17–12 cal ka BP). Therefore, a rapid change in the carbon sources in Lake Baikal occurred in the last glacial/post-glacial transition period is concluded.

### INTRODUCTION

The sediments of Lake Baikal in southern Siberia (51–56°N, 104–110°E) constitute an excellent archive for understanding continuous paleoenvironmental changes in continental interiors. Lake Baikal is the largest lake in the world with 23,000 km<sup>3</sup> of water, and contains about 20% of the Earth's freshwater (Falkner et al. 1991). It is also the oldest freshwater lake, having originated about 35 million yr ago, and contains sediments of ~7500 m thickness dating back to the Middle Eocene (Hutchinson et al. 1992). Since Lake Baikal is located in the interior of the Asian continent, its limnological conditions are far removed from marine influences. As a result, the climate of Lake Baikal is one of the most continental on Earth and significantly reflects the global climate (Short et al. 1991). This climatic sensitivity and these geological features provide a unique opportunity to study long-term paleoclimate changes in continental regions.

To estimate the change of paleoproductivity, which is linked to climate change in Siberian region, the stable carbon isotope ratios of total organic carbon ( $\delta^{13}\text{C}_{\text{TOC}}$ ) profiles and radiocarbon (<sup>14</sup>C) dating of Lake Baikal sediments have been reported (Prokopenko et al. 1999; Horiuchi et al. 2000; Prokopenko and Williams 2004; Watanabe et al. 2004, 2009a). These studies have strongly linked productivity in Lake Baikal and its watershed to climate change in the Siberian region.

For Lake Baikal sediment cores, previous researchers mainly determined <sup>14</sup>C ages using total organic carbon (TOC). However, it has been difficult to estimate the lake reservoir effect, the hard-water effect, and the effect of terrestrial sources of dead organic carbon in the sediments (“old carbon effect”), such as materials from lake terraces, paleosols, or other strata with dead <sup>14</sup>C, which is necessary for accurate <sup>14</sup>C dating of TOC in sediment cores. To construct a new age model, we performed high-time-resolution <sup>14</sup>C measurements of TOC and pollen concentrate fractions from a sediment core from the Buguldeika Saddle of Lake Baikal (VER99G12).

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In addition, there are very few high-time-resolution profiles (<100 yr) of  $\delta^{13}\text{C}_{\text{TOC}}$  in Lake Baikal sediments.  $\delta^{13}\text{C}$  values have proved to be useful for deducing the carbon sources in lake and sea sediments (Meyers 1997; Huon et al. 2002; Watanabe et al. 2009b). In this study, changes in the carbon sources in Lake Baikal were evaluated in detail on the basis of high-time-resolution  $\delta^{13}\text{C}_{\text{TOC}}$  measurements and  $^{14}\text{C}$  data sets from a Lake Baikal sediment core (VER99G12).

## MATERIALS AND METHODS

A gravity core (VER99G12) was taken from the Buguldeika Saddle (52°31'36"N, 106°09'08"E; 365 m water depth; Figure 1) of Lake Baikal in 1999. The Buguldeika Saddle is geomorphologically separated by underwater uplifts in Lake Baikal formed mainly by the accumulation of terrestrial materials from the Selenga River (Kuzumin et al. 2000). The total length of the VER99G12 core is 466 cm. Just after retrieval, VER99G12 was divided into five 1-m-long sections. The cores were then sampled at 1-cm intervals for measurements.

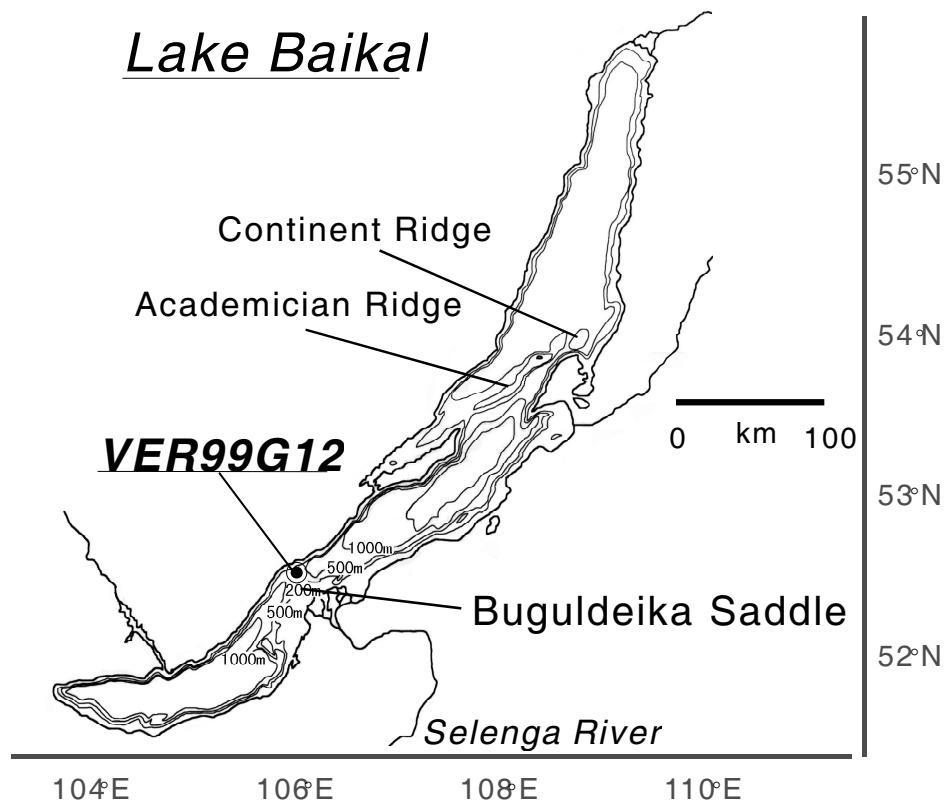


Figure 1 Map showing the location of the VER99G12 coring site on Buguldeika Saddle, Lake Baikal

Sediment samples for total organic carbon (TOC) analyses were treated with 6M HCl to remove carbonate prior to the measurements. The acid treatment was carried out as follows. Several drops of 6M HCl were added to ~100 mg of dried sample, which was left in HCl vapor for 2 days. Subsequently, the sample was transferred to a desiccator containing NaOH pellets for 5 days to remove HCl. Finally, the neutralized sample was freeze-dried for complete removal of water. TOC contents were determined by an elemental analyzer (FlashEA 1112, Thermo Electron).

$\delta^{13}\text{C}$  values of TOC were measured with an elemental analyzer (FlashEA 1112) interfaced to an isotopic ratio mass spectrometer (DELTA X Advantage, Thermo Scientific) via a Conflo III split interface. Carbon isotopic compositions were calibrated using a laboratory organic carbon standard (L-alanine;  $\delta^{13}\text{C} = -19.03\text{‰}$ ) and expressed in per mil (‰) relative to the VPDB standard. Standard deviations of the  $\delta^{13}\text{C}$  measurements in duplicate analyses were generally less than 0.1‰.

The pollen concentrate fractions for <sup>14</sup>C measurements were extracted by sieving and density gradient separation (Figures 2 and 3). The steps for the extraction of pollen concentrate fraction generally followed techniques used by Brown et al. (1989) and Piotrowska et al. (2004).

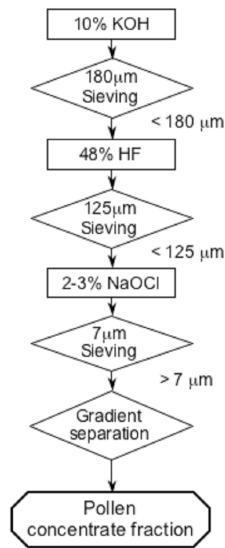


Figure 2 Extraction procedure to obtain the pollen concentrate fraction for <sup>14</sup>C measurement.

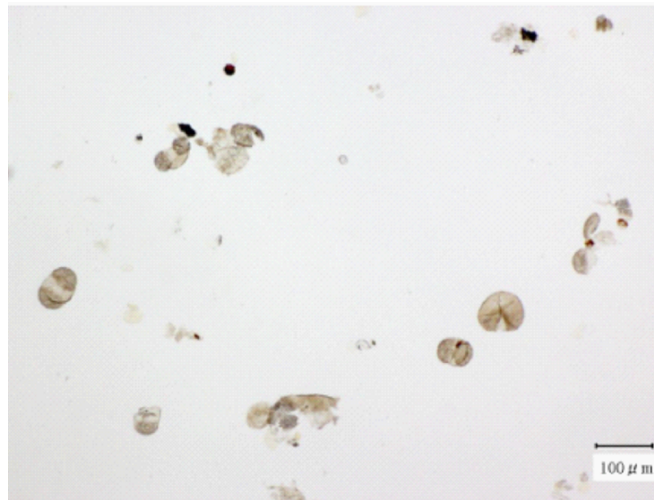


Figure 3 Macroscopic view of the pollen concentrate fraction (129–130 cm core depth, VER99G12).

The  $^{14}\text{C}$  measurements of TOC and pollen concentrate fractions were performed with a Tandemron accelerator mass spectrometry (AMS) system (High Voltage Engineering Europe, Model-4130 AMS, the Netherlands), at the Center for Chronological Research, Nagoya University, Japan. The analytical method of  $^{14}\text{C}$  measurements has been described previously (Nakamura et al. 2003; Nara et al. 2005). In this study, the  $^{14}\text{C}$  ages were calibrated by the program CALIB Rev. 5.0.1 (<http://intcal.qub.ac.uk/calib/>) using the IntCal04 data set (Reimer et al. 2004).

## RESULTS AND DISCUSSION

### Age Model of VER99G12 Based on Calibrated Ages of TOC and Pollen Concentrate Fractions

In the bottom layer of the VER99G12 sediment core (461–460 cm in depth), a conventional  $^{14}\text{C}$  age of TOC was determined to be  $\sim 27,200$  BP (Table 1), corresponding to marine isotope stage (MIS) 3.  $^{14}\text{C}$  ages of TOC are generally influenced by the reservoir effect in the lake and changes in organic matter sources (old carbon effect), such as materials from lake terraces, paleosols, or other strata with dead  $^{14}\text{C}$  (Lang and Hönscheidt 1999; Fuchs and Buerkert 2008; Watanabe et al. 2009c). On the Academician Ridge of Lake Baikal (Figure 1), Watanabe et al. (2009b) estimated the correction factor for these effects to be  $2100 \pm 90$  yr.

Table 1  $^{14}\text{C}$  age and calibrated age of total organic carbon and of the pollen concentrate fraction in the VER99G12 sediment core from Buguldeika Saddle, Lake Baikal.

Core depth (cm)	Material	Carbon content (dry wt%)	$\delta^{13}\text{C}$ (‰, VPDB)	Conventional $^{14}\text{C}$ age (BP, $\pm 1 \sigma$ )	Calibrated age, $-0.5$ kyr adjustment (cal BP, $1 \sigma$ )	Lab code (NUTA2-)
3–4 <sup>a</sup>	TOC	2.27	–27.3	1793 $\pm$ 28	1183–1278	8898
12–13 <sup>a</sup>	TOC	2.86	–27.6	3219 $\pm$ 30	2781–2845	8899
54–55 <sup>a</sup>	TOC	3.22	–28.4	4792 $\pm$ 32	4838–4865	8900
104–105 <sup>a</sup>	TOC	2.62	–28.2	7673 $\pm$ 37	7962–8010	8901
122–123 <sup>b</sup>	TOC	2.24	–29.1	8545 $\pm$ 27	8799–9015	11632
121–123	Pollen concentrate fraction	48.44	n.a. <sup>c</sup>	8684 $\pm$ 108	9009–9292	11629
124–126	Pollen concentrate fraction	51.34	n.a. <sup>c</sup>	8722 $\pm$ 115	9027–9396	11630
129–131	Pollen concentrate fraction	66.85	n.a. <sup>c</sup>	8814 $\pm$ 49	9280–9424	11631
130–131 <sup>b</sup>	TOC	2.87	–28.9	9131 $\pm$ 29	9540–9598	11633
139–140	TOC	2.43	–28.8	9688 $\pm$ 36	10,260–10,392	13990
150–151	TOC	2.09	–28.9	8975 $\pm$ 36	9478–9522	13991
154–155	TOC	2.34	–28.6	9882 $\pm$ 45	10,563–10,678	13254
156–157	TOC	2.15	–29.9	9210 $\pm$ 36	9562–9697	13992
159–160 <sup>b</sup>	TOC	2.25	–30.7	9453 $\pm$ 39	9946–10,201	10711
162–163	TOC	2.25	–29.1	9479 $\pm$ 36	9974–10,224	13993
164–165 <sup>a</sup>	TOC	1.41	–29.2	10,164 $\pm$ 50	10,876–11,186	9597
167–168 <sup>b</sup>	TOC	1.57	–26.7	11,105 $\pm$ 49	12,422–12,777	10723
174–175 <sup>a</sup>	TOC	1.26	–27.2	11,834 $\pm$ 53	13,164–13,262	9604
189–190	TOC	1.83	–27.0	12,571 $\pm$ 41	13,857–13,981	13985
198–199	TOC	1.72	–31.4	11,928 $\pm$ 42	13,239–13,321	13986
200–201	TOC	1.48	–31.3	11,785 $\pm$ 49	13,125–13,219	13255
202–203	TOC	1.41	–30.4	12,433 $\pm$ 65	13,724–13,731	13257
204–205	TOC	1.36	–29.7	12,289 $\pm$ 41	13,608–13,731	13989
214–215 <sup>a</sup>	TOC	1.57	–29.6	12,705 $\pm$ 54	13,996–14,132	9602
218–219 <sup>b</sup>	TOC	1.41	–29.2	13,810 $\pm$ 47	15,602–15,963	10716
222–223 <sup>b</sup>	TOC	1.18	–26.9	14,690 $\pm$ 49	16,729–17,129	10715
230–231 <sup>b</sup>	TOC	1.15	–26.1	15,154 $\pm$ 52	17,617–17,947	10712
240–241 <sup>a</sup>	TOC	1.13	–26.3	15,714 $\pm$ 56	18,577–18,697	8905
250–251 <sup>a</sup>	TOC	1.09	–25.9	15,964 $\pm$ 68	18,733–18,851	9601
265–266 <sup>a</sup>	TOC	1.00	–27.1	16,139 $\pm$ 69	18,839–18,947	9600

Table 1 <sup>14</sup>C age and calibrated age of total organic carbon and of the pollen concentrate fraction in the VER99G12 sediment core from Buguldeika Saddle, Lake Baikal. (Continued)

Core depth (cm)	Material	Carbon content (dry wt%)	δ <sup>13</sup> C (‰, VPDB)	Conventional <sup>14</sup> C age (BP, ±1 σ)	Calibrated age, -0.5 kyr adjustment (cal BP, 1 σ)	Lab code (NUTA2-)
267–268	TOC	0.92	-26.0	17,430 ± 71	19,950–20,149	13258
272–273 <sup>b</sup>	TOC	0.91	-25.7	17,551 ± 58	20,075–20,274	10718
277–278 <sup>b</sup>	TOC	0.87	-26.5	17,649 ± 72	20,162–20,379	10724
284–285 <sup>b</sup>	TOC	0.60	-25.9	18,173 ± 73	20,659–21,029	10719
303–304 <sup>a</sup>	TOC	0.60	-25.6	19,543 ± 71	22,443–22,613	8906
400–401 <sup>a</sup>	TOC	1.05	-26.3	23,654 ± 91	—	8907
460–461 <sup>b</sup>	TOC	0.94	-26.4	27,162 ± 130	—	12723

<sup>a</sup>Watanabe et al. 2007.

<sup>b</sup>Watanabe et al. 2009.

<sup>c</sup>n.a. = not analyzed.

In this study, the differences of calibrated ages between the pollen samples and TOC from the Buguldeika Saddle sediment core (VER99G12) were less than 500 yr (Figure 4a and Table 1). Boës et al. (2005) reported a time lag of about 500 yr during approximately the last 17 kyr between changes in δ<sup>18</sup>O in the GISP2 (Greenland) ice core (Stuiver et al. 1995) and grayscale fluctuations in the Continent Ridge core (Figure 1) associated with the pollen-based timescale. This time lag may result from specific properties of pollen grains in the Lake Baikal region, such as their sedimentation behavior. In addition, the <sup>14</sup>C age of a modern sediment trap sample (particles settling to Lake Baikal bottom) was 610 ± 40 BP (Prokopenko et al. 2007). Thus, in the VER99G12 sediment core, we adopted a correction factor for <sup>14</sup>C reservoir and old carbon effects of 500 yr (Table 1).

The estimated reservoir and old carbon effects (500 yr) for the VER99G12 sediment core from the Buguldeika Saddle was smaller than that of sediments from Academician Ridge (2100 yr, Watanabe et al. 2009b). This result could be caused by the large input of modern organic materials from the Selenga River. Calibrated ages of the pollen concentrate fraction and TOC showed that the VER99G12 core spans the past ~30 cal ka BP, indicating an average sedimentation rate of 13.6 cm/kyr (Figure 4a and Table 1). This means that the time resolution in the VER99G12 sediment core is less than ~70–80 yr/cm.

#### δ<sup>13</sup>C<sub>TOC</sub> Values and TOC Contents in the VER99G12 Sediment Core

Figure 4b shows the depth profiles of δ<sup>13</sup>C<sub>TOC</sub> in the VER99G12 sediment core. The δ<sup>13</sup>C<sub>TOC</sub> values in the core ranged from -31.4‰ to -25.1‰ (Figure 4b). In the lower part (from 30 to 24 cal ka BP, MIS 3) of the VER99G12 core, the δ<sup>13</sup>C<sub>TOC</sub> values gradually increased from about 28‰ to 25‰. During the MIS 2 (from 24 to 11.5 cal ka BP), the δ<sup>13</sup>C<sub>TOC</sub> values tended to decrease up to -31.4‰. In particular, δ<sup>13</sup>C<sub>TOC</sub> values varied greatly, ranging from -31.4‰ to -25.8‰ during the climate transition period from the last glacial to Holocene (~19–11.5 cal ka BP). From 14.3 to 13.5 cal ka BP, δ<sup>13</sup>C<sub>TOC</sub> values increased by up to 5‰ (Figure 4b). Similar positive shifts of δ<sup>13</sup>C<sub>TOC</sub> during the climate transition period from the last glacial to the Holocene have been reported from other sampling sites in Lake Baikal (Prokopenko et al. 1999; Prokopenko and Williams 2004; Watanabe et al. 2009b).

Watanabe et al. (2004) suggested that the δ<sup>13</sup>C<sub>TOC</sub> fluctuations in the Lake Baikal sediment cores may partly reflect changes in the organic carbon sources (allochthonous or autochthonous). The δ<sup>13</sup>C<sub>TOC</sub> fluctuations are also similar to changes in TOC/TN ratios and organic molecule composi-

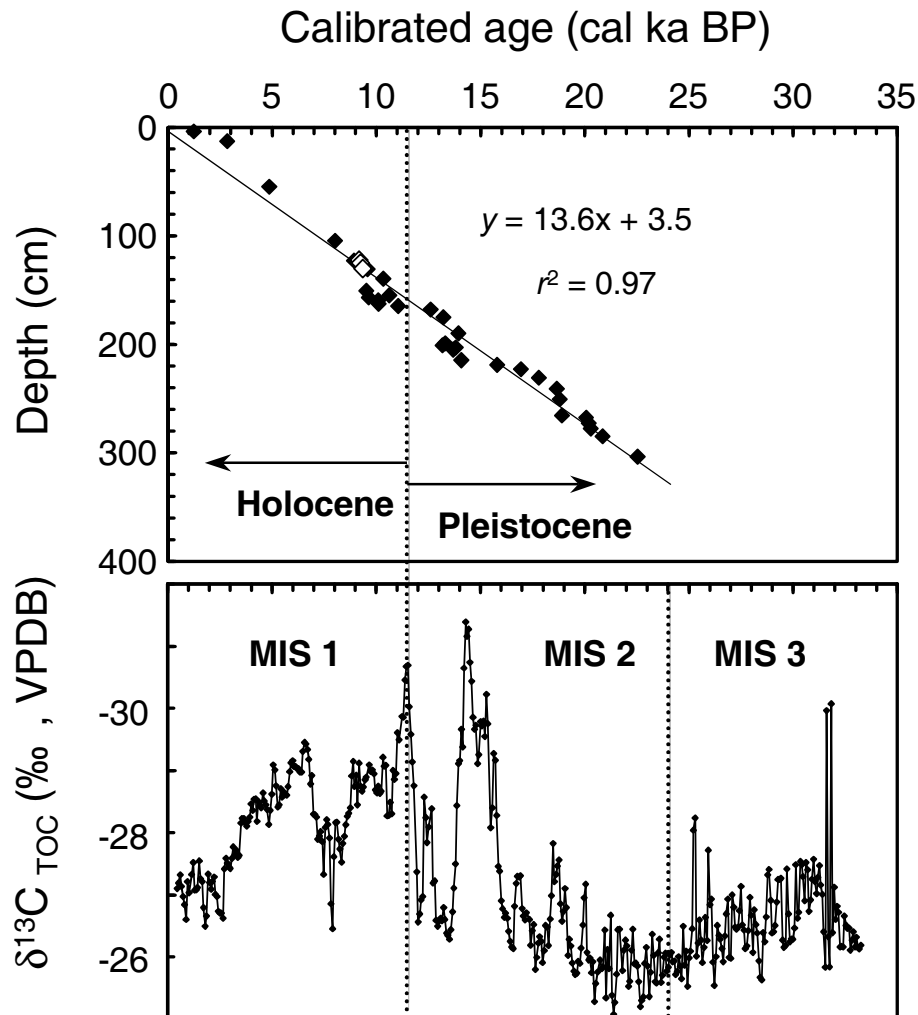


Figure 4 Calibrated ages of total organic carbon (TOC) and pollen concentrate fraction in the VER99G12 sediment core. Calibrated ages of the pollen concentrate fraction and TOC are shown as open and filled circles, respectively (Figure 4a).

tions (Yamamoto and Ishiwatari 1995; Matsumoto et al. 2000; Watanabe 2004). Another possible interpretation for the sources of  $^{13}\text{C}$ -depleted organic matter could be the high activity of methanogen and methane oxidizing bacteria in the lake sediment. For instance, a link between methane and depleted bulk TOC  $\delta^{13}\text{C}$  values was proposed by Prokopenko and Williams (2004) based on a parallel sedimentary record from Buguldeika Saddle.

Figure 5 shows the depth profiles of TOC content in the VER99G12 sediment core. TOC content gradually increased from 0.5 dry wt% near the core bottom to 3.5 dry wt% at ~4 cal ka BP (Figure 5a). Rapid increases in the abundance of TOC, biogenic silica, and chlorophyll-derivative compounds after ~15 cal ka BP have also been reported for other Lake Baikal sediment cores (Prokopenko et al. 1999; Prokopenko and Williams 2004; Watanabe et al. 2005; Soma et al. 2007; see also Figure 5b). A temporary decrease in TOC content occurred at ~12 cal ka BP (Figure 5a), perhaps

reflecting a depression of productivity at ~12 cal ka BP caused by the Younger Dryas cooling event (Hughen et al. 2000; Watanabe et al. 2004, 2009b).

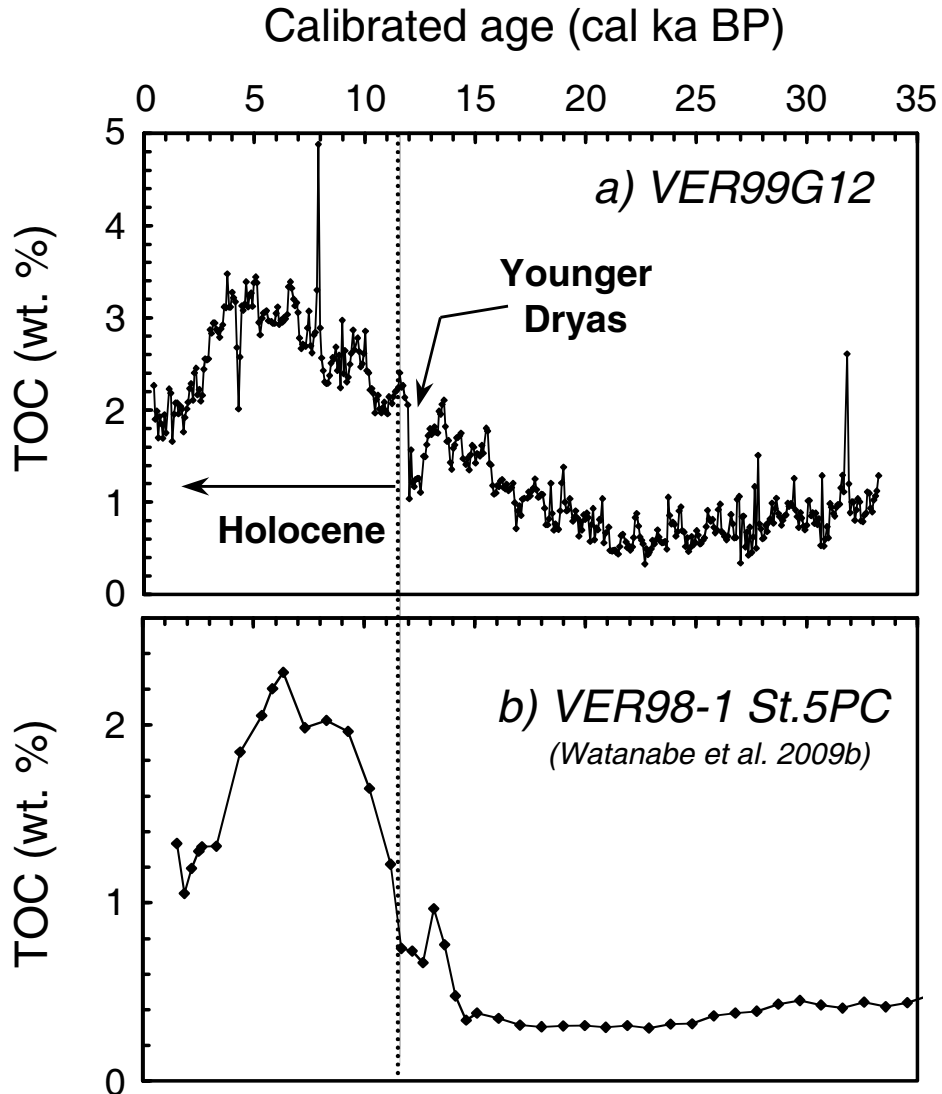


Figure 5 a) Depth profile of total organic carbon content (TOC, % of dry weight) in the VER99G12 sediment core. b) TOC contents of the VER98-1St.5 sediment core from the Academician Ridge, Lake Baikal, are also shown for comparison (Watanabe et al. 2009b).

## CONCLUSIONS

The VER99G12 sediment core from the Buguldeika Saddle in Lake Baikal was dated by AMS <sup>14</sup>C dating of pollen concentrates and TOC fractions. High-time-resolution analyses of  $\delta^{13}\text{C}_{\text{TOC}}$  in the VER99G12 core showed that the carbon sources changed abruptly during the climate transition from the last glacial to the Holocene.

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