Testing significance of total organic carbon isotope vlues for chronostratigraphy : Application to the Cretaceous Yezo Group in Hokkaido, Japan

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Testing significance of total organic carbon isotope values for chronostratigraphy: Application to the Cretaceous Yezo Group in Hokkaido, Japan

Yoshito TOMINAGA[1], Takashi HASEGAWA[2]

[1]Graduate School of Natural Science and Technology, Kanazawa University
Kakuma, Kanazawa 920-1192, Japan
Email: tominaga@stu.kanazawa-u.ac.jp

[2]School of Natural System, Institute of Science and Engineering, Kanazawa University
Kakuma, Kanazawa 920-1192, Japan
Email: jh7ujr@staff.kanazawa-u.ac.jp

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Abstract To realistically interpret chronostratigraphic correlation based on carbon isotope stratigraphy, we clarified deviation of total organic $\delta^{13}C$ values within a single sample, deviations of total organic $\delta^{13}C$ values within identical horizons, and the relationship of $\delta^{13}C$ values between total organic matter and wood fragments. The deviation (2σ) of $\delta^{13}C$ values of total oraganic matter ($\delta^{13}C_{TOM}$ values) within a single sample was 0.4‰. Maximum differences of $\delta^{13}C_{TOM}$ values from identical horizons were smaller than 0.4‰. Although cross-plots of $\delta^{13}C_{TOM}$ and $\delta^{13}C$ values of wood fragments ($\delta^{13}C_{wood}$ values) certify their proportional relationship, generally negative values of $\delta^{13}C_{TOM}$ relative to the $\delta^{13}C_{wood}$ were also evident. Differences between $\delta^{13}C_{wood}$ and $\delta^{13}C_{TOM}$ were between 0.2 and 1.9‰. Regarding this character of $\delta^{13}C_{TOM}$ distribution, its stratigraphic variations are applicable for stratigraphic and correlation purposes. This result provides important criteria for application of $\delta^{13}C$ stratigraphy to the Yezo Group.

Keywords: Cretaceous, carbon isotope, chronostratigraphy, organic carbon

1 Introduction

The Cretaceous period (145.5-65.5 Ma) is generally considered warmer than other periods, compared with recent climate in particular (Huber et al., 2002). Knowledge of Cretaceous ocean-climate systems provides important information about forthcoming global warming. To improve knowledge of temporal and spatial dimensions of

environmental changes in the Cretaceous world, international correlation for the Cretaceous System is prerequisite. The Cretaceous is currently subdivided into 12 stages and 32 substages (Ogg et al., 2004; 2008). Global Boundary Stratotype Sections and Points (GSSPs) of Cretaceous stages and substages are located in Europe and America. The GSSPs provide primary and secondary criteria for interregional correlation of the stages (Kennedy et al., 2005).

International chronostratigraphic correlation of the Upper Cretaceous Yezo Group, especially the Cenomanian through Coniacian, has been extensively addressed through the occurrence of marine macro-micro fossils (Matsumoto et al., 1991; Toshimitsu et al., 1995; Nishi et al., 2003). However, the occurrence of international zoned marker fossil species or supplementary age-indicative species is generally rare or absent in Upper Cretaceous sequences of Japan.

On the other hand, stratigraphic fluctuations of carbon isotope values observed in carbonate sequences, as well as secular variation of the isotope value of marine and terrestrial organic carbon, have been used for global correlation (Jenkyns et al., 1994; Jarvis et al., 2006; Voigt, 2000). Therefore, carbon isotope stratigraphy of total organic matter has been established for the Yezo Group over the past decade (Hasegawa and Saito, 1993; Hasegawa, 1997, 2003a; Hasegawa et al., 2003; Uramoto et al., 2009). Because organic matter in each sample was derived predominantly from terrestrial plants in the previous studies, carbon isotope fluctuations of total organic matter have been interpreted as those of terrestrial plants. However, a minor part of the organic carbon appeared to be marine algal and/or bacterial in origin, and an analytical result from bulk rock did not necessarily show carbon isotopic fluctuations of terrestrial plants. Recently, Takashima et al. (2010) reported carbon isotope values of the Yezo Group, but using a different technique. They extracted and picked wood fragments from each mudstone sample, and analyzed them instead of bulk mudstone. However, the results occasionally showed large deviations when some adjacent horizons were compared, suggesting the potential predominance of signals without correlational importance.

The objective of this study is to clarify how to discriminate δ^{13}C global events in the Yezo Group from those representing regional, local or other sedimentological processes, for use in global correlation. Therefore, deviation of total organic δ^{13}C values within a single sample, deviations of total organic δ^{13}C values within identical horizons, and the relationship of δ^{13}C values between total organic matter and wood fragments were clarified.

2 Geologic setting

The Yezo Group is exposed along the Horotate River in the Kotanbetsu area of Hokkaido, Japan, and is interpreted as a forearc basin sequence (Okada, 1983). The group is mainly composed of hemipelagic and shallow marine mudstone and sandstone, and it yields well-preserved, abundant marine macro and microfossils. The kerogen composition in the sediments of this group shows a remarkable predominance of terrestrial woody kerogens over marine algal ones (Hasegawa, 2001).

Samples used for analyses were collected from the Saku and Haborogawa Formations of the middle-upper part of the Yezo Group exposed along the Horotate River section (Figs. 1-3). The Saku Formation consists of alternating beds of sandstone and siltstone. The Haborogawa Formation is mainly characterized by dark grey massive siltstone, with intercalated sandstone. See Wani and Hirano (2000) for details of the lithology of the study area. The ages of the studied interval range from Cenomanian to Coniacian, based on macrofossil biostratigraphy and carbon isotope stratigraphy (Wani and Hirano, 2000; Tsuchiya et al., 2003; Hasegawa, 2003a; Takashima et al., 2004). Occurrences of global age-diagnostic macrofossils such as *Collignoniceras woollgari* Mantell and *Inoceramus pictus minus* Matsumoto characterize the Cenomanian-Turonian sequences (Wani and Hirano, 2000; Tsuchiya et al., 2003).

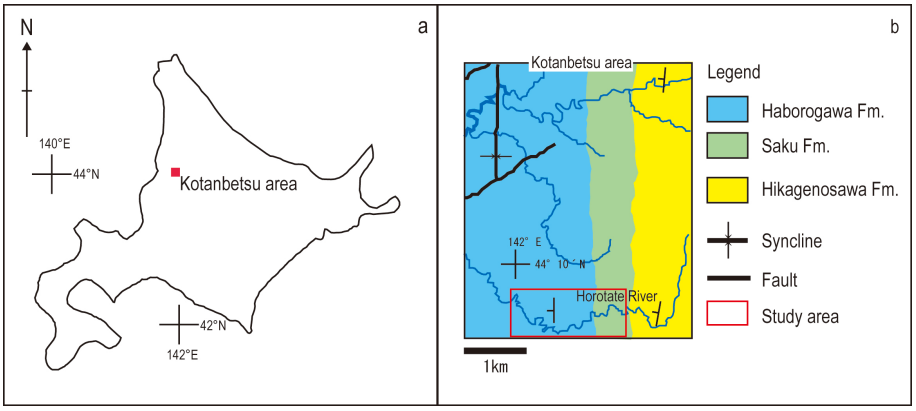

Figure 1: (a) Index map showing Kotanbetsu area of Hokkaido, Japan. (b) Location and geologic map of study area along Horotate River, Kotanbetsu. Boxed area shows sampling area. Geologic map is modified from Wani and Hirano (2000).

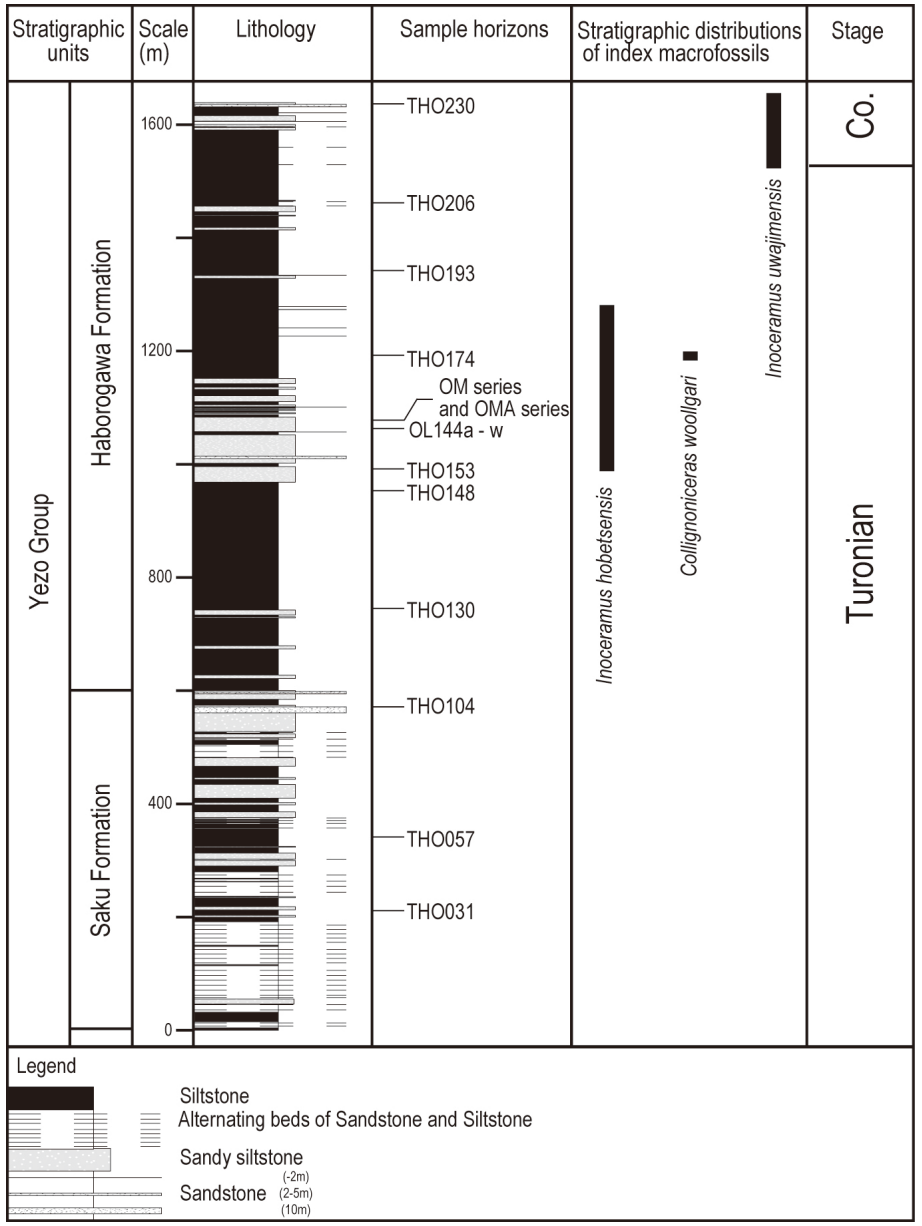

Figure 2: Geologic column and horizons of samples used for carbon isotope analyses and visual observation of kerogen. Assignment of stratigraphic units follows Wani and Hirano (2000). Macrofossil data and stage boundary follow Wani and Hirano (2000) and Tsuchiya et al. (2003).

3 Samples

Fresh siltstone and sandy siltstone samples were collected from 39 horizons for the Horotate section (Figs. 1-3). All studies of stable carbon isotope stratigraphy of the Yezo Group have used siltstone and sandy siltstone samples (e.g., Hasegawa and Saito, 1993; Hasegawa, 1997, 2003a; Hasegawa et al., 2003; Uramoto et al., 2009; Takashima et al., 2010).

For clarifying deviation of total organic δ^{13}C values within a single sample, an OL144 sample was collected (Fig. 2). Most samples were siltstones. Those sediment grain sizes are similar. However, the OL144 sample was sandy siltstone. Those sediment sizes are mixed. Therefore, sandy siltstones like the OL144 sample are considered to have the largest variation in total organic δ^{13}C values. Therefore, stable carbon isotopic values of total organic matter ($\delta^{13}C_{TOM}$) within the OL144 (about 20 cm $\times$ 40 cm) sample were measured every about 2 cm (Fig. 4).

To make known deviations of total organic δ^{13}C values within identical horizons, 28 samples (OMA010, OMA031, OMA041, OMA055, OMA061, OMA071, OMA080, OMA090, OMA098, OMA104, OMA110, OMA123, OM051, OM063, OM072, OM082, OM093, OM102, OM111, OM121, OM131, OM152, OM162, OM171, OM182, OM191, OM201 and OM213) were collected (Fig. 3). The outcrops from which these samples were extracted have the stable dip and strike of geologic stratum. Therefore, it is possible to pursue identical horizons on both banks of the Horotate River. Those identical horizons are about 6 m away horizontally from 4 m.

In order to clarify the relationship of δ^{13}C values between total organic matter and wood fragments of terrestrial plants, 10 samples (THO031, THO057, THO104, THO130, THO148, THO153, THO174, THO193, THO206 and THO230) were collected at 40 to 200 m stratigraphic intervals (Fig. 2). Moreover, nine of these samples (THO057, THO104, THO130, THO148, THO153, THO174, THO193, THO206 and THO230) were used to show clearly that the organic matter in each sample was derived predominantly from terrestrial plants.

4 Methods

To obtain the δ^{13}C value of total organic matter, each siltstone sample was powdered with a micro grinder, avoiding contamination from ichnolite and turbidite units. Then, the powdered samples were acidified by 1N and 5N solution of HCl to remove carbonates, then neutralized and dried. A small aliquot (3-5 mg) of the powder was weighed in a tin capsule and put in an elemental analyzer connected with a mass spectrometer, to analyze the δ^{13}C value in the same manner described below for analysis of wood fragments. Some samples were analyzed with a mass spectrometer (Delta plus Advan-

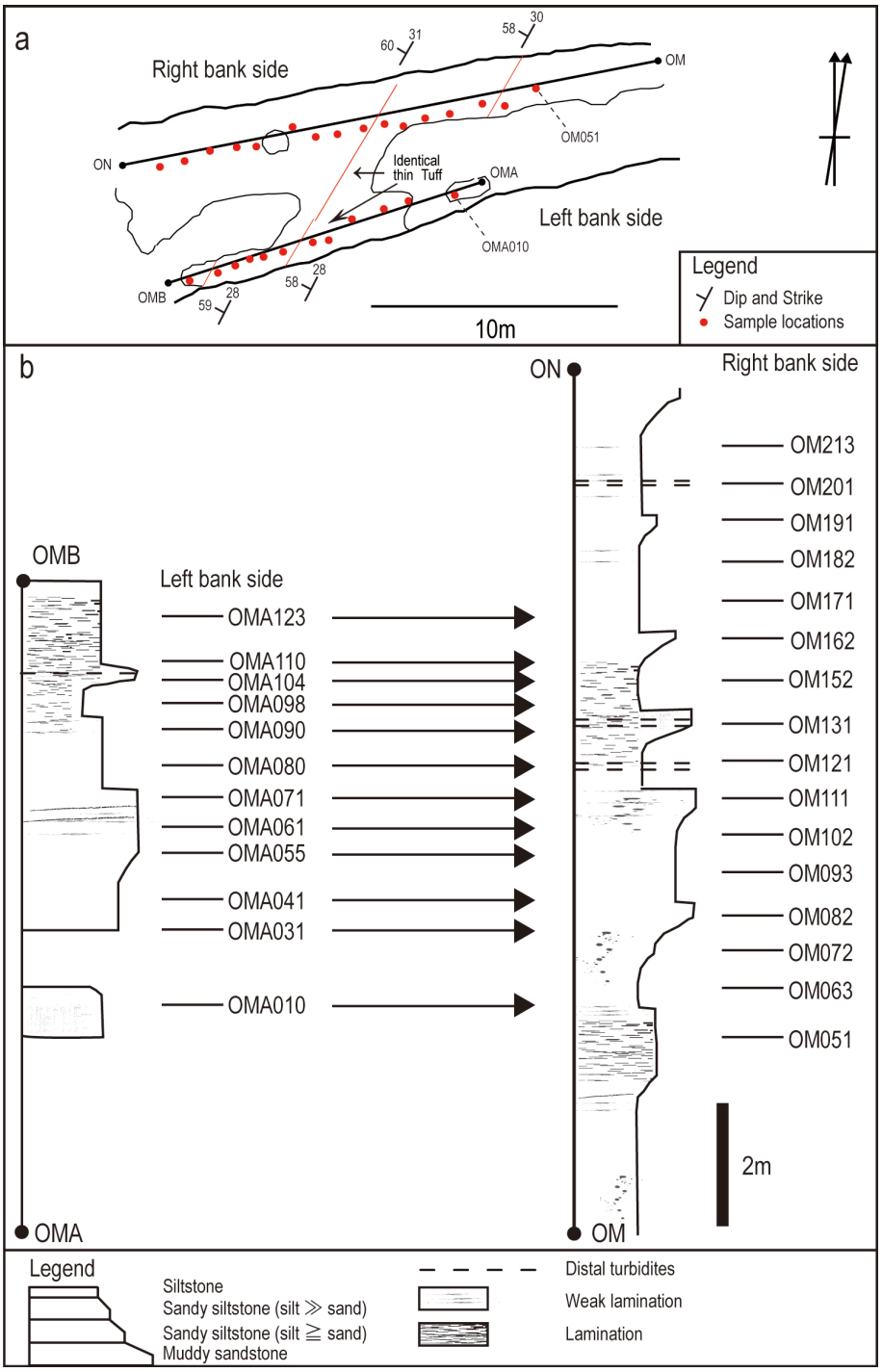

Figure 3: (a) Map showing locations of samples used for clarifying lateral deviation of δ^{13}C values within a single horizon. (b) Geologic column and horizons of samples used for lateral deviation of δ^{13}C values within a single horizon.

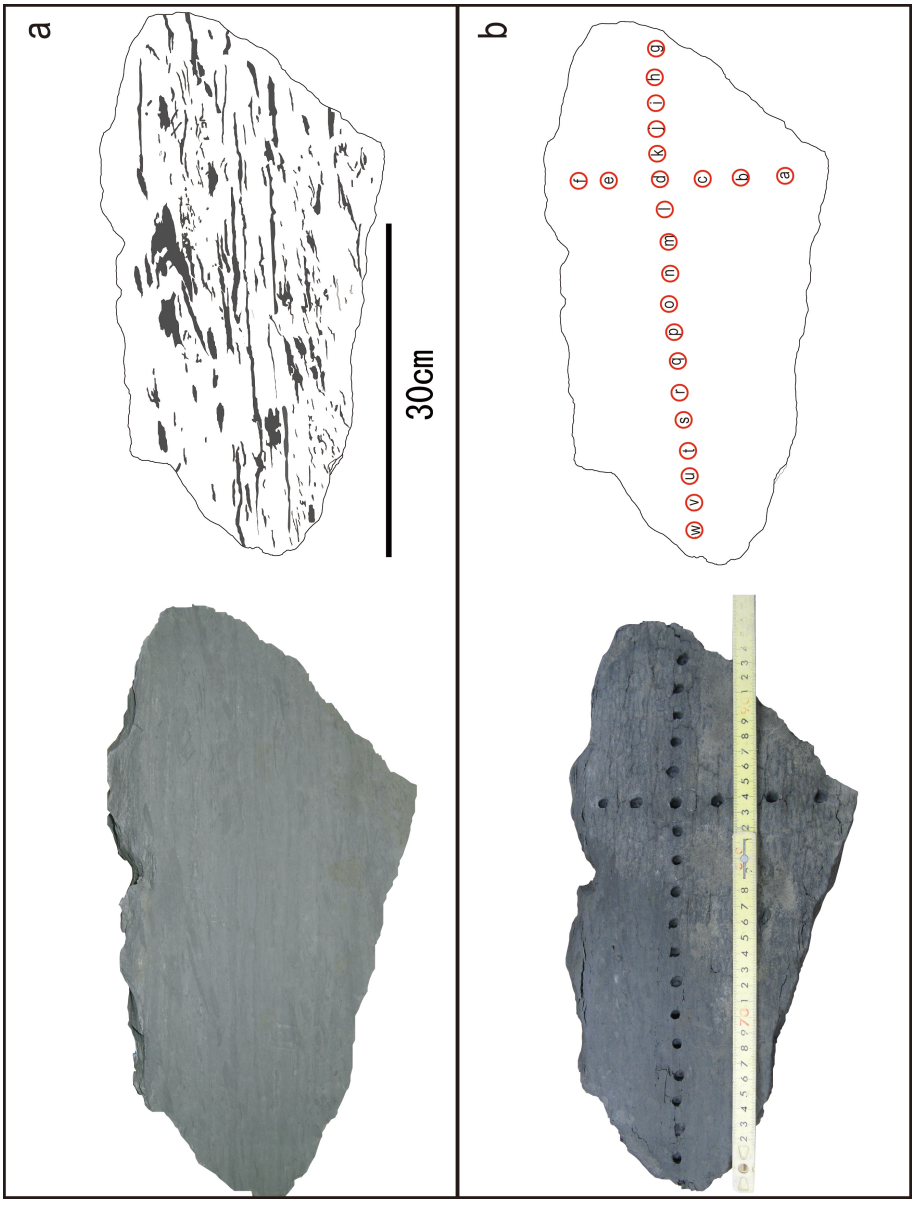

Figure 4: (a) Photograph and line drawing of OL144 used for clarifying deviation of δ^{13}C values within single sample. (b) Photograph and line drawing of OL144 showing localities of subsamples for analyses.

tage, Thermo Finnigan) in line with an elemental analyzer (Flash EA1112, Thermo Finnigan) at the Center for Advanced Marine Core Research of Kochi University.

Each selected sample of about 150 g was treated to extract wood fragments, following the method of Takashima et al. (2010). All wood fragments larger than 63 μm were handpicked under a microscope. Then the wood fragments of each sample were weighed in a tin capsule, put in the furnace of an elemental analyzer (NCS2500, Thermo Quest at Kanazawa University) and burned at 1000°C under helium flow, to convert organic carbon to CO_2. The CO_2 was transferred to a mass spectrometer (Delta V advantage, Thermo Electron at Kanazawa University) to analyze the δ^{13}C value.

Both mass spectrometers used were inter-calibrated with a lab standard (triphenylamine of Indiana University) and international standard (ANU-sucrose). The results reported herein were obtained using a reference CO_2 gas calibrated by ANU-sucrose directly and NBS-19 indirectly. Each data point was an average of multiple (more than three) analyses for each sample, and expressed in the conventional delta notation with respect to the VPDB standard, where δ^{13}C (‰) = [(^{13}C/^{12}C) sample/(^{13}C/^{12}C) standard $-$ 1] $\times$ 1000. Isotopic values were checked by isotopically well-characterized laboratory standards. Repeated analysis of laboratory standards indicated instrumental reproducibility to be $\pm$0.1‰ for both mass spectrometers.

Organic compositions of selected samples were checked by visual observation of kerogen, under reflected light and in fluorescent light. Crushed mudstone material was made into polished blocks following standard preparation procedures (Bustin et al., 1983). Polished pellets were examined under the microscope to identify organic particles.

5 Results

5.1 Deviation of δ^{13}C values within a single sample

All δ^{13}C values obtained are summarized in Table 1. Stable carbon isotopic values of total organic matter ($\delta^{13}C_{TOM}$) within a single sample (OL144) were from $-$25.0 to $-$23.9‰, with an average of $-$24.5‰ (Fig. 5). No systematic difference associated with subsample localities within a lump of OL144 siltstone (Fig. 3) was observed. Eighty-three percent of all data points from the OL144 sample were within $-$24.5‰ $\pm$ 0.2‰. If those values are based on the D'Agostino and Pearson K^2 test (K^2 = 5.3044 $<$ 5.991), it may be a normal distribution. The standard deviation (1σ) of a normal distribution is 0.2.

Sample name	$\delta^{13}C_{TOM}$ (‰ vs. VPDB)	$\delta^{13}C_{wood}$ (‰ vs. VPDB)	Stratigraphic level (m)
THO031	-23.4	-22.8	211.4
THO057*	-22.5	-21.5	343.0
THO104*	-23.0	-21.1	571.5
THO130*	-24.8	-23.9	745.3
THO148*	-24.7	-23.5	953.6
THO153*	-24.6	-23.2	991.2
THO174*	-25.1	-24.6	1194.2
THO193*	-26.3	-26.1	1341.3
THO206*	-24.2	-23.4	1462.1
THO230*	-25.0	-23.9	1636.9
OL144a	-24.8	-	-
OL144b	-24.7	-	-
OL144c	-24.6	-	-
OL144d	-24.6	-	-
OL144e	-24.7	-	-
OL144f	-24.6	-	-
OL144g	-24.4	-	-
OL144h	-24.7	-	-
OL144i	-24.5	-	-
OL144j	-24.5	-	-
OL144k	-24.4	-	-
OL144l	-23.9	-	-
OL144m	-24.3	-	-
OL144n	-24.2	-	-
OL144o	-24.5	-	-
OL144p	-24.4	-	-
OL144q	-24.6	-	-
OL144r	-24.7	-	-
OL144s	-24.7	-	-
OL144t	-24.4	-	-
OL144u	-24.7	-	-
OL144v	-24.4	-	-
OL144w	-25.0	-	-
OMA010	-25.0	-	-
OMA031	-25.1	-	-
OMA041	-25.2	-	-
OMA055	-24.9	-	-
OMA061	-24.9	-	-
OMA071	-24.8	-	-
OMA080	-24.9	-	-
OMA090	-24.9	-	-
OMA098	-25.1	-	-
OMA104	-24.7	-	-
OMA110	-24.5	-	-
OMA123	-24.1	-	-
OM051	-24.9	-	-
OM063	-25.04	-	-
OM072	-25.07	-	-
OM082	-24.95	-	-
OM093	-24.87	-	-
OM102	-24.61	-	-
OM111	-25.06	-	-
OM121	-24.65	-	-
OM131	-24.84	-	-
OM152	-24.61	-	-
OM162	-24.82	-	-
OM171	-24.09	-	-
OM182	-24.33	-	-
OM191	-23.97	-	-
OM201	-24.28	-	-
OM213	-24.57	-	-

Table 1: Carbon isotope values of studied samples from Horotate River, Kotanbetsu, Hokkaido, Japan. Sample names with asterisks indicate samples that are microscopically observed for organic composition.

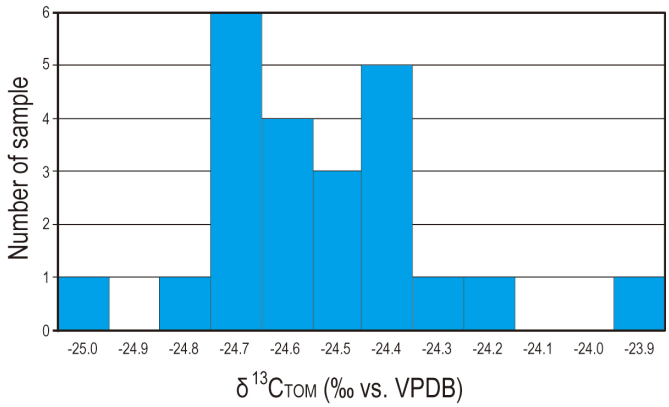

Figure 5: Deviation of δ^{13}C values within a single sample (OL144).

5.2 Lateral differences of δ^{13}C values between identical stratigraphic range

Two sequences of δ^{13}C value of total organic matter within a stratigraphically limited ($\sim$10 m) but identical range showed variations between -25.2 and -24.0‰ (Fig. 6). Stratigraphic trends of the two sequences were positive upwards. This was especially clear in upper parts of the successions (from the 5 to 10 m stratigraphic level). The sections showed a trend of 1‰ increase from bottom to top.

However, in lower parts of the successions (from the 2 to 5 m stratigraphic level), lateral differences of δ^{13}C values were conspicuous. The maximum lateral difference of δ^{13}C values observed within a single horizon (about 3 m stratigraphic level from OM051) was about 0.3‰.

5.3 Visual observation of total organic matter

The composition of kerogens in all selected samples shows the predominance of semi-fusinite, vitrinite or non-fluorescent amorphous kerogens of terrestrial origin. Alginite of possible marine origin with weak yellow fluorescence was observed in all samples, although it represented a small amount (5% or less of total organic matter).

5.4 δ^{13}C values of total organic matter and handpicked wood fragments

Stable δ^{13}C values of total organic matter ($\delta^{13}C_{TOM}$) from 10 selected samples were between -26.3 and -22.5‰, with an average of -24.4‰. On the other hand, δ^{13}C values of bulk wood fragments ($\delta^{13}C_{wood}$) from the same sample set were between -26.1 and -21.1‰, with an average of -23.4‰. $\delta^{13}C_{wood}$ were generally heavier than $\delta^{13}C_{TOM}$ in the studied samples.

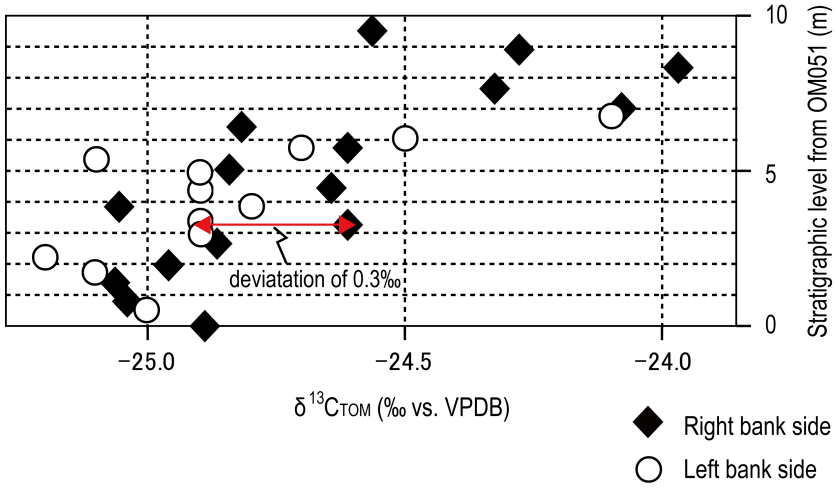

Figure 6: Lateral differences of δ^{13}C values within identical horizons.

6 Discussion

6.1 Relationship of δ^{13}C values between total organic matter and wood fragments

Previous studies have clearly documented that the majority of kerogens in siltstone of Cenomanian and Turonian sequences in the Upper Cretaceous Yezo group originated from terrestrial higher plants; a high marine organic content has not been reported (Hasegawa, 1997; Tsuchiya et al., 2003; Yamamoto et al., 2006; Uramoto et al., 2009; Hasegawa et al., 2010). The organic matter in each sample was derived predominantly from terrestrial woody plants, but contributions from other sources were also observed. Total organic matter contains minor amounts of cutinite, sporinite and amorphous kerogen that are derived from higher plant tissues, but not from lignin, a source of woody particles in the samples. This is a possible reflection of the fact that $\delta^{13}C_{TOM}$ values are generally lower compared with $\delta^{13}C_{wood}$ values, since organic matter other than wood tissues (such as leaves) is known to have lighter δ^{13}C values (Tyson, 1995). Differences between $\delta^{13}C_{wood}$ and $\delta^{13}C_{TOM}$ were between 0.2 and 1.9‰.

Nevertheless, stratigraphic carbon isotope variations from the bulk analysis technique are believed to be applicable for stratigraphic/correlation purposes, since the carbon isotope values of wood fragments and total organic matter in the same sample are highly correlated ($r = 0.96$) (Fig. 7). The slope of the regression line is 1.2. The standard error of the slope is 0.13. The intercept of the regression line is 6.43. The

standard error of the intercept is 3.17. This indicates that δ^{13}C stratigraphies based on both bulk organic matter and wood fragments are controlled by identical factors. As a result, it is believed that carbon isotope fluctuations of total organic matter have been interpreted as those of terrestrial plants. These findings demonstrate that the bulk analysis technique is adequate for carbon isotope stratigraphy of the Yezo Group. In addition, the tested variation of $\delta^{13}C_{TOM}$ within a single rock lump and single horizon was very stable in the Horotate River section (most $\delta^{13}C_{TOM}$ differences from an identical horizon were smaller than 0.4‰). This deviation is much smaller than that reported for wood fragments. For example, Takashima et al. (2010) indicated deviations as large as 0.5-1.1‰ in δ^{13}C values of wood fragments ($>$ 120 m in size) in mudstones collected from the identical horizons in the Kotanbetsu River section, 4 km from the Horotate River section. Similar results are reported in Yans et al. (2010). Moreover, differences of δ^{13}C values from neighboring horizons (vertically conjunct) obtained with the bulk rock analysis technique in this and previous studies (Uramoto et al., 2009; Nemoto and Hasegawa, 2011) are considerably smaller than those produced by the method of picking wood fragments (Takashima et al., 2010). This is considered to likely originate from total organic matter becoming mixed; compared with wood fragments (of only huge sizes, especially $\geq$ 120 μm), $\delta^{13}C_{TOM}$ values are stable compared with $\delta^{13}C_{wood}$ values. The wood fragment technique is also reasonable. However, the bulk analysis aids acquisition of a stable dataset, and is especially advantageous when closely-spaced, high-resolution samples are analyzed.

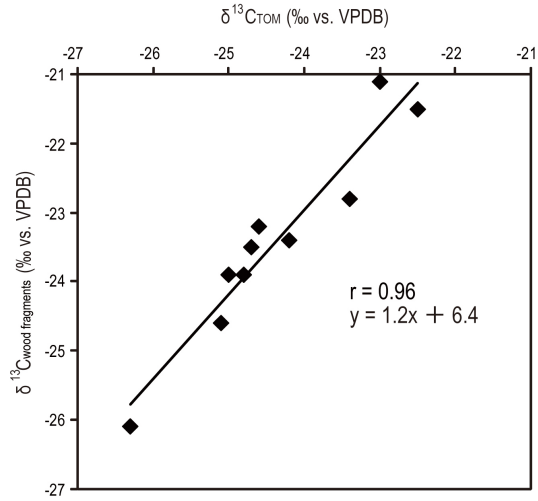

Figure 7: Cross-plots of δ^{13}C values of total organic matter (TOM) and wood fragments from selected samples.

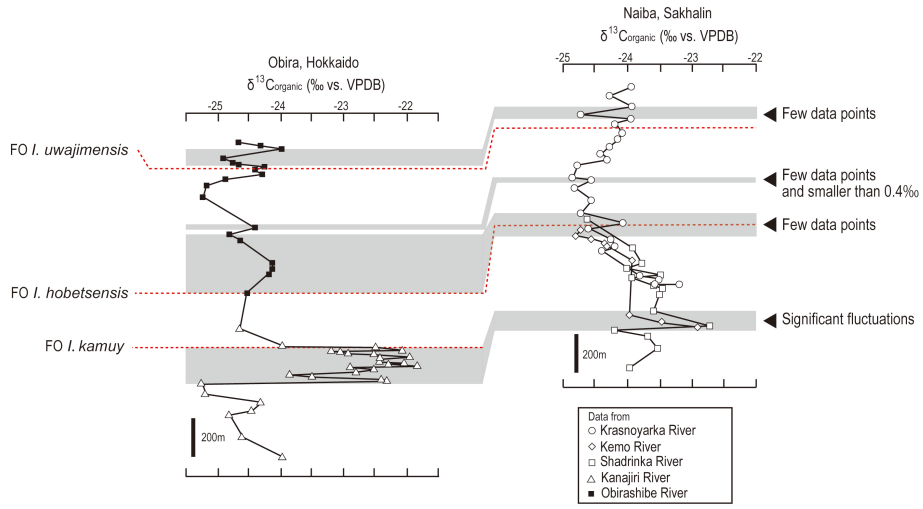

Figure 8: Comparison of Upper Cretaceous δ^{13}C profiles within Yezo Group. Data of the Obira area are from Uramoto et al. (2007) and Uramoto et al. (2009). Data of Naiba area are from Hasegawa et al. (2003). Correlative carbon isotope fluctuations recognized by Uramoto et al. (2009) are connected with grey bands. Macrofossil data for Obira area are after Tanabe et al. (1977), Matsumoto et al. (1981), Sekine et al.(1985) and Funaki and Hirano (2004). Red dashed lines are first occurrences of regional marker inoceramids of the Yezo Group.

6.2 δ^{13}C events in the Yezo Group available for correlation: A perspective based on results of this study

Based on comparison of δ^{13}C curves established with total organic matter, Uramoto et al. (2009) selected some δ^{13}C events for correlation of Cenomanian and Turonian sequences of the Yezo Group (and its equivalent of the Russian Far East) (Fig. 8). They evaluated their "tie zones" with intrabasinal biostratigraphy of regional marker inoceramids. However, most of the events employed for the correlation were represented by few data points. As our study shows that correlation cannot be safely made unless the events are larger than 0.4‰, up-dating of the correlation proposed by Uramoto et al. (2009) is required. Carbon isotope stratigraphy based on $\delta^{13}C_{TOM}$ should be applied with caution about this deviation size (0.4‰) that is potentially non-correlative use.

7 Conclusions

To realistically interpret chronostratigraphic correlation based on carbon isotope stratigraphy, we tested potential uncertainty sequestered in a dataset of organic carbon isotope values with the bulk rock analysis method. The size of the potential signal derived

from factors other than "long-term secular variation of carbon isotope values of atmospheric CO_2 reservoir" was determined to be 0.4‰ or smaller, for mudstone from the Yezo Group of Hokkaido, Japan. Such "noise" does not affect recognition of long-term secular variation of Late Cretaceous carbon isotope values, based on bulk rock analysis of organic carbon. Caution is required for event correlation when the magnitudes of δ^{13}C events are 0.4‰ or smaller. The data here lead to the recommendation that only δ^{13}C events larger than 0.4‰ be used for rigid chemostratigraphic correlation.

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