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U–Pb age of the *Didymoceras awajiense* Zone (upper Campanian, Cretaceous) in the Aridagawa area, Wakayama, southwestern Japan

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Abstract Zircons obtained from a tuff sample from the lower part of the *Didymoceras awajiense* Zone of the Sotoizumi Group, the Aridagawa area, Wakayama Prefecture, southwestern Japan, were subjected to radiometric age dating ($^{238}U/^{206}Pb$ ratios) using the LA-ICP-MS method. The results (72.4 ± 0.8 Ma, 95% conf.), which suggests a late late Campanian (Late Cretaceous) age, confirm the validity of previous biostratigraphic and magnetostratigraphic correlations. This age indicates that *D. awajiense* is the youngest *Didymoceras* species on either side of the Pacific Ocean as well as and Western Interior of the US.

Key words : Campanian, Cretaceous, *Didymoceras awajiense* Zone, U–Pb age, zircon

Introduction

Didymoceras awajiense (Yabe, 1901) is one of a few late Campanian heteromorph ammonoids characterized by helical whorls in contact during the early and middle growth stages followed by a long retroversal hook at the mature growth stage (Morozumi, 1985). It occurs abundantly in the upper Campanian in the Izumi Group on Awaji Island, southwestern Japan (Morozumi, 1985), the Sotoizumi Group in Wakayama, southwestern Japan (Misaki and Maeda, 2009) and the Nakaminato Group in Ibaraki, eastern Japan (Saito, 1958). Although *D. awajiense* has not yet been reported from Hokkaido, northern Japan, it is a possible that it will eventually be found there because of the common occurrence of many Campanian–Maastrichtian ammonoid species in southwestern Japan and Hokkaido (Matsunaga *et al.*, 2008; Shigeta *et al.*, 2010, 2012, 2016).

Morozumi (1985) erected the *Didymoceras awajiense* Zone based on a biostratigraphic interval ranging from the FAD (First Appearance Datum) of *D. awajiense* to the FAD of *Pravitoceras sigmoidale* Yabe, 1902 in the Izumi Group. This zone is characterized by endemic species restricted to Japan; thus, the lack of more cosmopolitan taxa has long made it difficult to directly correlate this local zone with other regions of the globe (Toshimitsu *et al.*, 1995). Effective methods for global correlation among the local zones include the integration of bio-, stable isotope- and magnetostratigraphy as well as the determination of radiometric ages of each zone. Indeed, a detailed magnetostraigraphic investigation conducted on the Izumi Group by Kodama (1990), indicated that the *D. awajiense*-bearing beds correlate directly with the lower part of polarity chron C32.2n, i.e., the upper upper Campanian. However, the radiometric age of this zone has not been determined until now.

In this paper, we calibrate the international correlation of the *Didymoceras awajiense* Zone with zircon-based geochronology in the Sotoizumi Group, Wakayama Prefecture, and establish an up-to date global correlation of the zone.

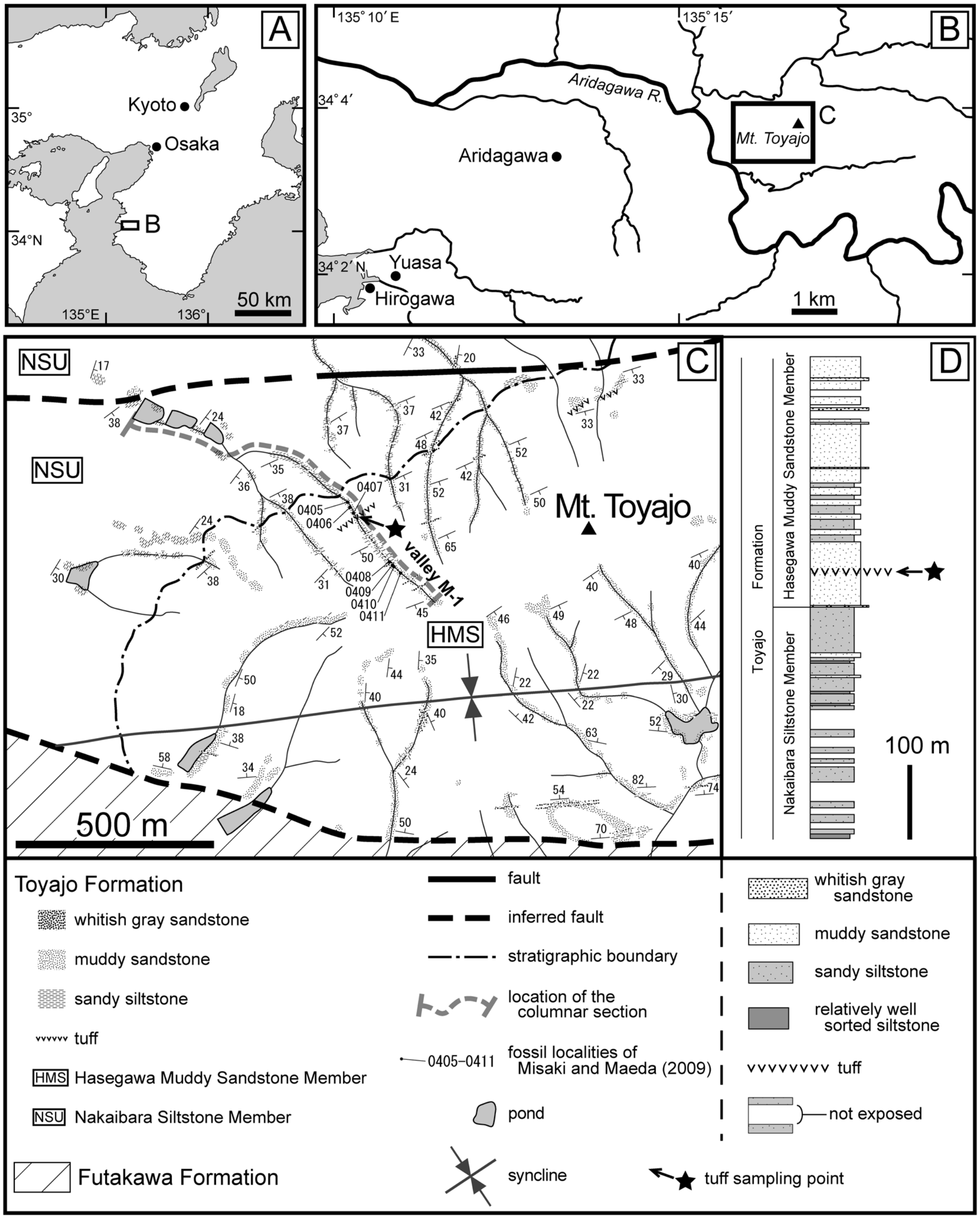

Fig. 1. Locality (A–C) and horizon (D) of the tuff bed providing the U–Pb zircon age in the Aridagawa area, Wakayama Prefecture, southwestern Japan.

Notes on Stratigraphy

The Cretaceous Sotoizumi Group is widely distributed in the Aridagawa area, Wakayama, southwestern Japan. Within the group, the Campanian–Maastrichtian Toyajo Formation, which is exposed in a narrow strip along the E–W trending ridge of Mt. Toyajo, yields numerous

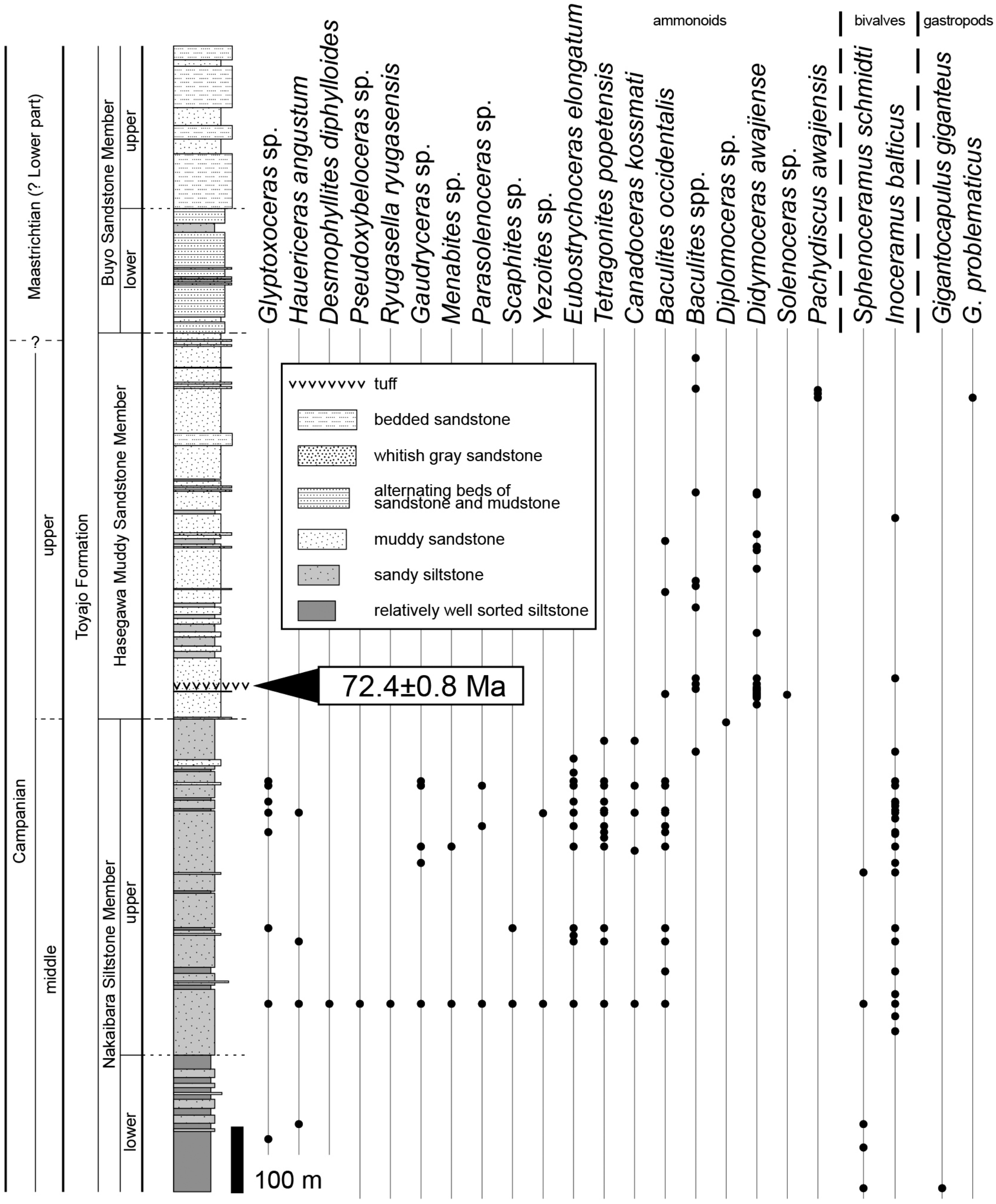

Fig. 2. Stratigraphic occurrence of molluscan fossils, and U–Pb zircon age of tuff in the Toyajo Formation.

relatively well-preserved megafossils from various horizons (e.g. Misaki and Maeda, 2009; Figs. 1, 2). The formation is divided into the Nakaibara Siltstone, Hasegawa Muddy Sandstone and Buyo Sandstone members in ascending order (Misaki and Maeda, 2009).

The 700-m thick Nakaibara Siltstone Member consists mainly of dark gray to gray, intensely bioturbated, massive siltstone and sandy siltstone with abundant plant remains. This member contains the early middle Campanian index inoceramid bivalve *Sphenoceramus schmidti* (Michael,

Table 1. LA-ICP-MS analyzed data and calculated ages of the tuff sample from the lowest part of the Hasegawa Muddy Sandstone Member in the Toyajo Formation. Errors are 1 sigma. Pb_c and Pb* indicate the common and radiogenic portions, respectively.

Labels	$^{206}Pb_c$ $^{(1)}$ (%)	U (ppm)	Th (ppm)	Th/U	$^{238}U/^{206}Pb^{*}$ $^{(1)}$	$^{207}Pb^{*}/^{206}Pb^{*}$ $^{(1)}$	$^{238}U/^{206}Pb^{*}$ age $^{(1)}$ (Ma)	$^{238}U/^{206}Pb^{*}$ age $^{(2)}$ (Ma)
Toya_01	0.43	276	135	0.50	91.55 ± 2.41	0.0513 ± 0.0113	70.0 ± 1.8	69.6 ± 1.8
Toya_02	2.40	128	67	0.54	91.33 ± 4.13	0.0442 ± 0.0224	70.2 ± 3.2	70.4 ± 3.1
Toya_03	0.00	463	204	0.45	82.36 ± 1.75	0.0343 ± 0.0032	77.8 ± 1.6	77.7 ± 1.6
Toya_04	0.89	283	169	0.61	89.52 ± 2.47	0.0401 ± 0.0116	71.6 ± 2.0	72.1 ± 1.8
Toya_05	0.00	114	55	0.49	86.44 ± 3.52	0.0550 ± 0.0135	74.1 ± 3.0	73.3 ± 3.2
Toya_06	0.42	388	353	0.93	90.78 ± 2.37	0.0447 ± 0.0123	70.6 ± 1.8	70.8 ± 1.6
Toya_07	0.17	186	136	0.75	88.44 ± 3.17	0.0488 ± 0.0180	72.5 ± 2.6	72.3 ± 2.4
Toya_08	0.67	127	80	0.65	88.57 ± 3.96	0.0223 ± 0.0232	72.4 ± 3.2	72.8 ± 2.7
Toya_09	0.44	464	458	1.01	88.33 ± 2.44	0.0408 ± 0.0113	72.6 ± 2.0	72.8 ± 1.8
Toya_10	0.52	346	206	0.61	85.73 ± 2.54	0.0420 ± 0.0103	74.8 ± 2.2	75.1 ± 2.1
Toya_11	0.64	237	122	0.53	89.42 ± 2.90	0.0497 ± 0.0124	71.7 ± 2.3	71.4 ± 2.3
Toya_12	0.86	204	121	0.61	94.44 ± 3.45	0.0492 ± 0.0152	67.9 ± 2.5	67.7 ± 2.4
Toya_13	0.15	255	114	0.46	88.64 ± 3.05	0.0302 ± 0.0134	72.3 ± 2.5	72.3 ± 2.3
Toya_14	0.08	569	209	0.38	86.71 ± 1.85	0.0476 ± 0.0058	73.9 ± 1.6	73.8 ± 1.6
Toya_15	0.11	188	100	0.54	95.64 ± 3.61	0.0604 ± 0.0168	67.1 ± 2.5	65.8 ± 2.5
Toya_16	0.00	214	86	0.41	81.33 ± 2.58	0.0547 ± 0.0075	78.8 ± 2.5	77.9 ± 2.6
Toya_17	2.05	178	124	0.72	90.50 ± 3.73	0.0425 ± 0.0185	70.8 ± 2.9	71.2 ± 2.7
Toya_18	0.79	159	107	0.69	93.74 ± 4.25	0.0476 ± 0.0249	68.4 ± 3.1	68.3 ± 2.7
Toya_19	0.03	825	285	0.35	85.27 ± 1.60	0.0475 ± 0.0050	75.2 ± 1.4	75.0 ± 1.4
Toya_20	2.38	248	178	0.73	92.82 ± 3.14	0.0413 ± 0.0164	69.1 ± 2.3	69.4 ± 2.2
Toya_21	0.92	690	300	0.45	89.82 ± 1.96	0.0424 ± 0.0061	71.4 ± 1.5	71.6 ± 1.5
Toya_22	2.28	215	124	0.59	93.10 ± 3.01	0.0399 ± 0.0141	68.9 ± 2.2	69.3 ± 2.2
Toya_23	0.00	153	81	0.54	86.15 ± 3.25	0.0509 ± 0.0114	74.4 ± 2.8	73.9 ± 3.0
Toya_24	0.09	772	269	0.36	87.34 ± 1.64	0.0458 ± 0.0046	73.4 ± 1.4	73.3 ± 1.3
Toya_25	0.05	1312	482	0.38	87.66 ± 1.14	0.0454 ± 0.0031	73.1 ± 0.9	73.2 ± 0.9
Toya_26	0.20	172	87	0.52	90.53 ± 3.78	0.0424 ± 0.0144	70.8 ± 2.9	71.0 ± 2.8
Toya_27	1.23	201	120	0.61	83.50 ± 2.85	0.0494 ± 0.0152	76.7 ± 2.6	76.6 ± 2.5
Toya_28	0.00	151	68	0.46	94.00 ± 3.73	0.0293 ± 0.0092	68.2 ± 2.7	68.2 ± 2.7
Toya_29	0.00	214	100	0.48	90.95 ± 3.27	0.0497 ± 0.0087	70.5 ± 2.5	70.3 ± 2.6
Toya_30	0.03	491	213	0.44	87.27 ± 2.15	0.0458 ± 0.0072	73.5 ± 1.8	73.5 ± 1.7

(1) Common Pb corrected by assuming $^{206}Pb/^{238}U$–$^{208}Pb/^{232}Th$ age-concordance.
(2) Common Pb corrected by assuming $^{206}Pb/^{238}U$–$^{207}Pb/^{235}U$ age-concordance.

1899) and ammonoid *Canadoceras kossmati* Matsumoto, 1954. Several other ammonoid forms including *Eubostrychoceras elongatum* (Whiteaves, 1903) are found in the upper part of this member (Misaki and Maeda, 2009).

The 590-m thick Hasegawa Muddy Sandstone Member is generally coarser grained than the Nakaibara Siltstone Member, and consists mainly of bluish gray, poorly sorted, massive, muddy very fine- to fine-grained sandstone, which includes a 2-m thick, whitish gray tuff bed about 50 m above from the base of the member. *Didymoceras awajiense* occurs throughout the lower to middle parts of the member and the latest Campanian index ammonoid *Pachydiscus awajiensis* Morozumi, 1985 is found in the upper part of the member.

The +490-m thick Buyo Sandstone Member consists of unfossiliferous bedded sandstone and sandstone-dominated alternating beds of sandstone and mudstone. Misaki and Maeda (2009) reported a fragment of an ammonoid shell from the lower part of the member.

Material and Methods

Material: A tuff sample taken from a 2-m thick whitish gray tuff bed about 50 m above from the base of the Hasegawa Muddy Sandstone Member of the Toyajo Formation was subjected to zircon-based radiometric age analysis. The tuff bed crops out along the valley M-1 in Misaki and

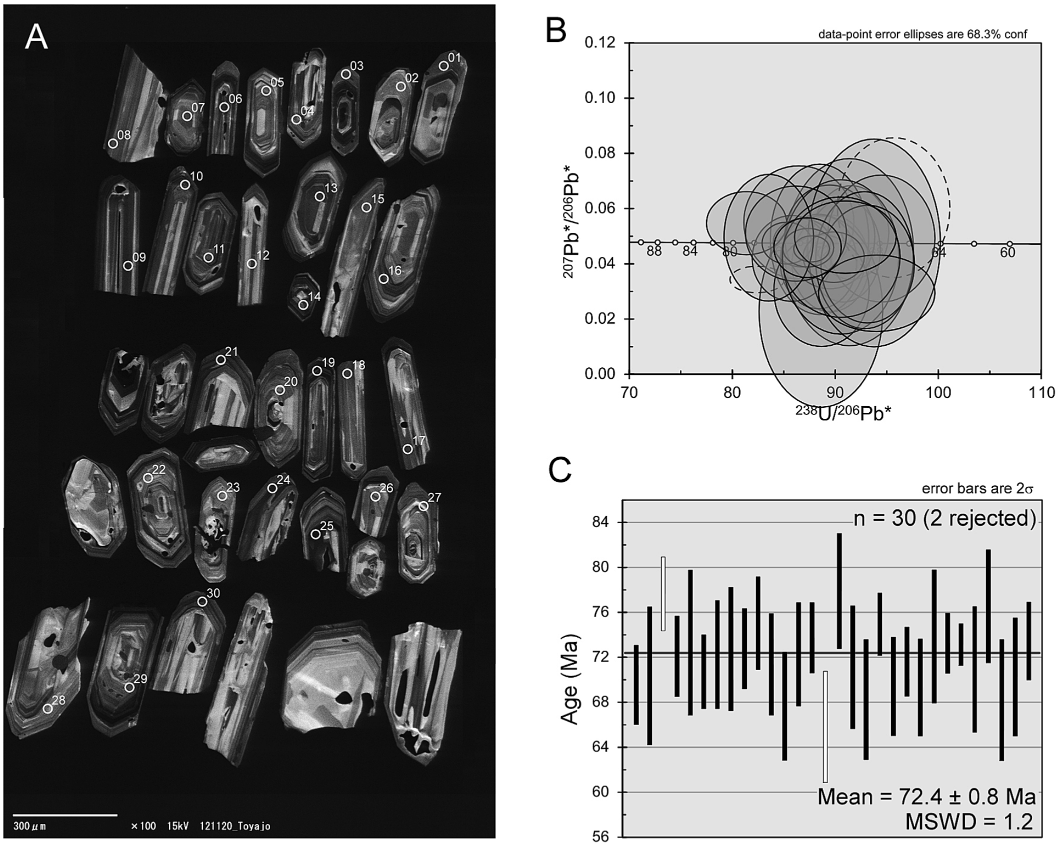

Fig. 3. U–Pb zircon ages of tuff sample collected from the lowest part of the Hasegawa Muddy Sandstone Member in the Toyajo Formation. A, cathodoluminescence image (CL) of zircon grains from the sample. Circles on the grain represent spots analyzed by LA-ICP-MS. Spots are 25 µm across. B, Tera-Wasserburg U–Pb concordia diagram of zircons. C, ^{238}U–^{206}Pb age distribution plot of tuff sample. $^{207}Pb^{*}$ and $^{206}Pb^{*}$ indicate radiometric ^{207}Pb and ^{206}Pb, respectively.

Maeda (2009).

Methods: The zircon grains were extracted by standard techniques: crushing, heavy liquid separation and handpicking. Then, the zircon grains, the FC1 zircon standard ($^{206}Pb/^{238}U = 0.1859$; Paces and Miller, 1993), and the SRM610 glass standard were mounted in an epoxy disc and polished until the center of each grain was exposed. The backscattered electron and cathodoluminescence image of the zircon grains was used to select the sites for analysis. U–Pb dating of these samples was carried out using the LA-ICP-MS method, which consists of a NWR213 Nd-YAG 213 nm wave length laser ablation system (Electro Scientific Industries) and an Agilent 7700x quadrupole ICP-MS (Agilent Technologies), installed at the National Museum of Nature and Science at Tsukuba, Japan. The experimental conditions and procedures for the measurements were based on the methods described in Tsutsumi *et al.* (2012). The spot size of the laser was 25 µm. Corrections for common Pb was made on the basis of the measured $^{207}Pb/^{206}Pb$ ratio (^{207}Pb correction) or $^{208}Pb/^{206}Pb$ and Th/U ratios (^{208}Pb correction) (e.g. Williams, 1998) as well as the model for common Pb compositions proposed by Stacey and Kramers (1975). In this paper, we adopt the ^{207}Pb correction for age discussion because it is more effective in calculating the Phanerozoic ^{238}U–$^{206}Pb^{*}$ age than the ^{208}Pb correction (e.g. Williams, 1998). The pooled ages presented in this study were calculated using Isoplot/Ex software (Ludwig, 2003). The uncertainties in the mean ^{238}U–^{206}Pb* ages represent 95% confidence intervals (95% conf.). ^{206}Pb* indicates radiometric ^{206}Pb.

Results

Analyzed data from zircons in terms of the fraction of common ^{206}Pb, U, and Th concentrations, Th/U, ^{238}U/^{206}Pb*, and ^{207}Pb*/^{206}Pb* ratios, and radiometric ^{238}U/^{206}Pb* ages are listed in Table 1. All errors state 1 sigma. All zircons in the samples show rhythmic oscillatory and/or sector zoning on cathodoluminescence images (Fig. 3A), which is commonly observed in igneous zircons (e.g. Corfu *et al.*, 2003), and their higher Th/U ratios (>0.1) also support their igneous origin (Williams and Claesson, 1987; Schiøtte *et al.*, 1988; Kinny *et al.*, 1990; Hoskin and Black, 2000). A Tera-Wasserberg concordia diagram and an age distribution plot for all analyzed spots of sample by LA-ICP-MS are shown in Figure 3B and C, respectively. All zircon age data cluster in the range 67–78 Ma. The weighted mean age, after the rejection of two data points, yield 72.4 ± 0.8 Ma (MSWD = 1.2; 95% conf.), which is thought to indicate magmatism/deposition age of the tuff sample.

Discussion

International correlation of the *Didymoceras awajiense* Zone

Based on biostratigraphic evidence, Morozumi (1985) assigned the *Didymoceras awajiense* Zone to the upper upper Campanian. Kodama (1990), who recognized three reverse and four normal magnetic chrons (C33r, C32n, C32r, C32.2n, C32.1r, C32.1n in ascending order) in the Izumi Group, correlated the portion of the Seidan Formation that yields *D. awajiense* to the lower part of polarity chron C32.2n, i.e., the upper upper Campanian. Zircon geochronology in this study reveals that the U–Pb age of the tuff in the lower part of the *D. awajiense* Zone is 72.4 ± 0.8 Ma (95% conf.), which infers a late late Campanian age, and thus confirms the validity of previous correlations.

Didymoceras Hyatt, 1894 is well known from the Western Interior of North America (Kennedy *et al.*, 2000), where all relevant ammonoid zones have been dated by K–Ar and/or other radioisotope methods (Obradovich and Cobban, 1975; Ogg and Hinnov, 2012). Various species of *Didymoceras* are found in zones ranging from the middle middle Campanian *Baculites gregoryensis* Zone to the middle upper Campanian *Didymoceras cheyennense* Zone (Kennedy *et al.*, 2000). Furthermore, on the west coast of North America, where all relevant ammonoid zones have been correlated to magnetic chrons (Ward *et al.*, 2012), the early late Campanian *D. nebrascense* (Meek and Hayden, 1856) and early–middle late Campanian *D. hornbyense* (Whiteaves, 1895) are known to occur. In Hokkaido, northern Japan, *D. hidakense* Shigeta in Shigeta *et al.* (2016) was recently described from the lower upper Campanian *Baculites subanceps* Zone, which has been radiometrically age dated (U–Pb ratios) by zircon-bearing tuff samples. This evidence suggests that *D. awajiense* is the youngest *Didymoceras* species on either side of the Pacific Ocean as well as the Western Interior of the US.

Stratigraphic gap or condensation in the Toyajo Formation

The upper part of the Nakaibara Siltstone Member of the Toyajo Formation contains the early middle Campanian index ammonoid *Canadoceras kossmati*, while the uppermost part includes *Diplomoceras* sp., which correlates with the middle middle Campanian or a higher zone, because *Diplomoceras* is known to range from the middle middle Campanian *Metaplacenticeras subtilistriatum* Zone to the Maastrichtian in Japan (Matsumoto, 1984; Maeda *et al.*, 2005; Shigeta *et al.*, 2015; Kurihara *et al.*, 2016). The lowest part of the overlying Hasegawa Muddy Sandstone Member includes a tuff bed, whose age is 72.4 ± 0.8 Ma (95% conf.), i.e., late late

Campanian. This evidence suggests that a stratigraphic gap or sedimentary condensation of about 4–5 Ma from a late middle to middle late Campanian age may be present at or around the boundary between the members. Therefore, it is necessary to consider this age gap in future isotopic- and magnetostratigraphy studies of the Toyajo Formation.

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