



# Geochemical fingerprinting of fossils with uncertain stratigraphic provenance: A case study from the Lower Jurassic Nishinakayama Formation (Yamaguchi, Japan)

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## ABSTRACT

A fossil without provenance data is problematic because it cannot be placed into meaningful paleoecological and paleobiogeographic contexts. This problem is particularly acute when the fossil is suspected or known to have originated from a formation within which a major interval of biotic and/or paleoenvironmental change has been recognized that could change the paleobiological implications of the taxon in question. Two reptile fossil specimens (a testudinate and a crocodylomorph) preserved within *ex situ* mudstone cobbles of the Lower Jurassic (Pliensbachian-Toarcian) Nishinakayama Formation in Yamaguchi, Japan exemplify this problem. Both specimens are preserved alongside associated ammonoid fossils that biostratigraphically constrain them to the Toarcian interval of the Nishinakayama Formation, but it is unclear whether their stratigraphic provenance is below, within, or above the interval of the formation that preserves the chemostratigraphic markers of the Toarcian Ocean Anoxic Event (T-OAE, ~182.5 Ma), which were first established at the nearby locality Sakuraguchidani. Herein, we used isotope ratio mass spectrometry and portable energy-dispersive X-ray fluorescence to investigate the geochemistry of sedimentary matrix sampled from each fossil and a new measured stratigraphic section of the Nishinakayama Formation close to where the fossil specimens were found. We interpret a ~2‰ positive shift in  $\delta^{13}\text{C}_{\text{org}}$  at the base of the section as the recovery of the negative carbon isotope excursion associated with the T-OAE, providing additional evidence of the event from a new locality within the Nishinakayama Formation. Linear discriminant analysis (LDA) of the total geochemical dataset was then used to explore the multivariate separateness of binned intervals of the composite section and predict the provenance of each fossil. The results suggest with 93.33% confidence that both fossils were derived from strata above the T-OAE interval. This predictive method can be applied to any fossil collected *ex situ* with preserved rock matrix and for which the general provenance is known or suspected.

## 1. Introduction

Field locality data such as stratigraphic and geographic provenance are routinely documented when fossil remains discovered *in situ* are collected. However, specimens collected *ex situ* inherently lack some precise provenance data. In this and other instances of specimens lacking provenance data—such as those acquired via illegal poaching, purchased from private sellers, or collected legally but without rigorous documentation—a lack of precise locality data can drastically affect

evolutionary interpretations of a specimen (e.g., Sereno et al., 2009; Fowler et al., 2011). Some case studies have implemented “geochemical fingerprinting” using rare earth elements (e.g., Trueman and Benton, 1997; Trueman, 1999; Trueman et al., 2006; Grandstaff and Terry, 2009; Kocsis et al., 2021a, 2021b; Botfalvai et al., 2021, 2022), laser ablation inductively coupled plasma mass spectrometry (LA-ICP-MS; e.g., Horwitz et al., 2011), and portable energy-dispersive X-ray fluorescence (pXRF; e.g., Fanti et al., 2018) to convincingly infer the provenance of fossil specimens lacking precise locality data. Whereas some of these methods

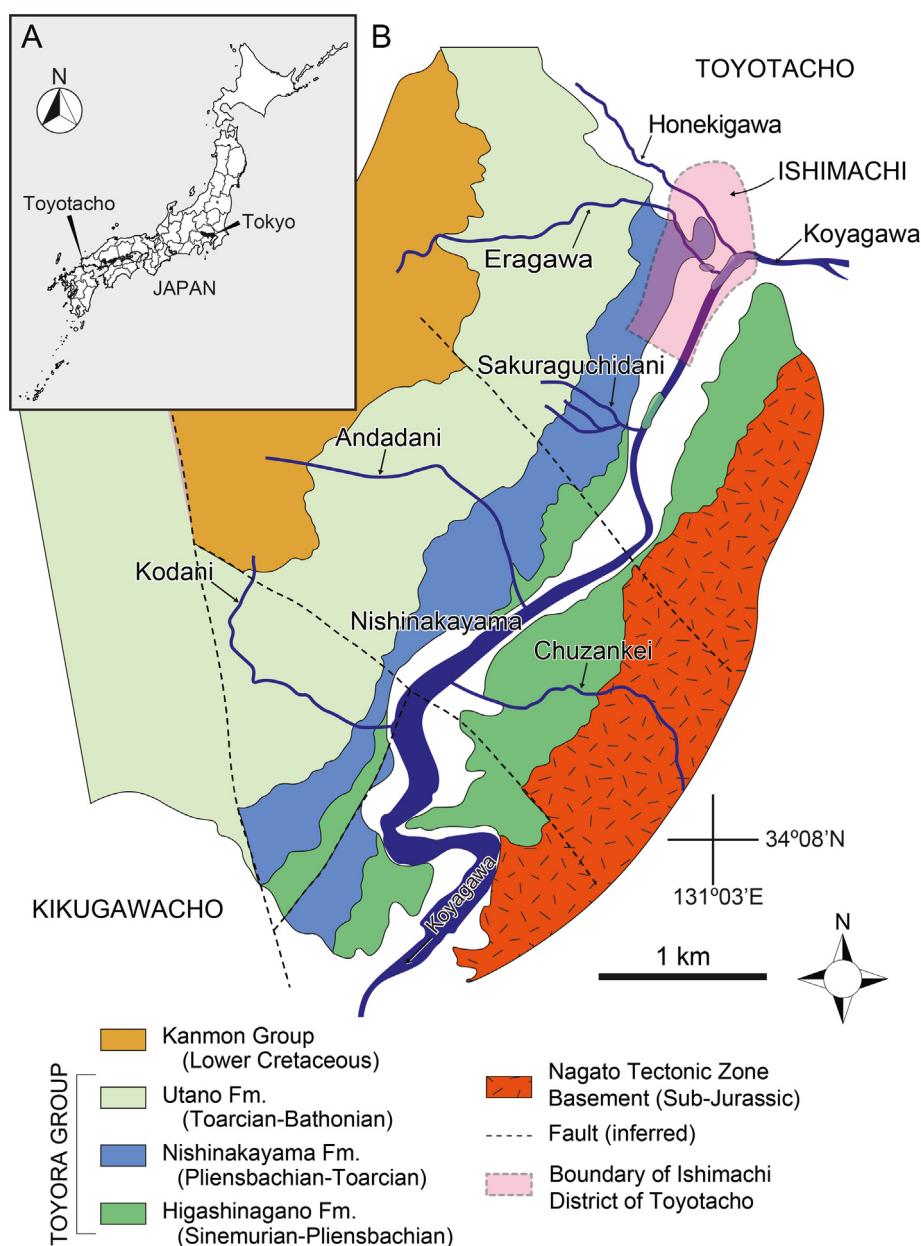
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are consumptive, expensive, and/or time-intensive, pXRF is quick, inexpensive, and nondestructive.

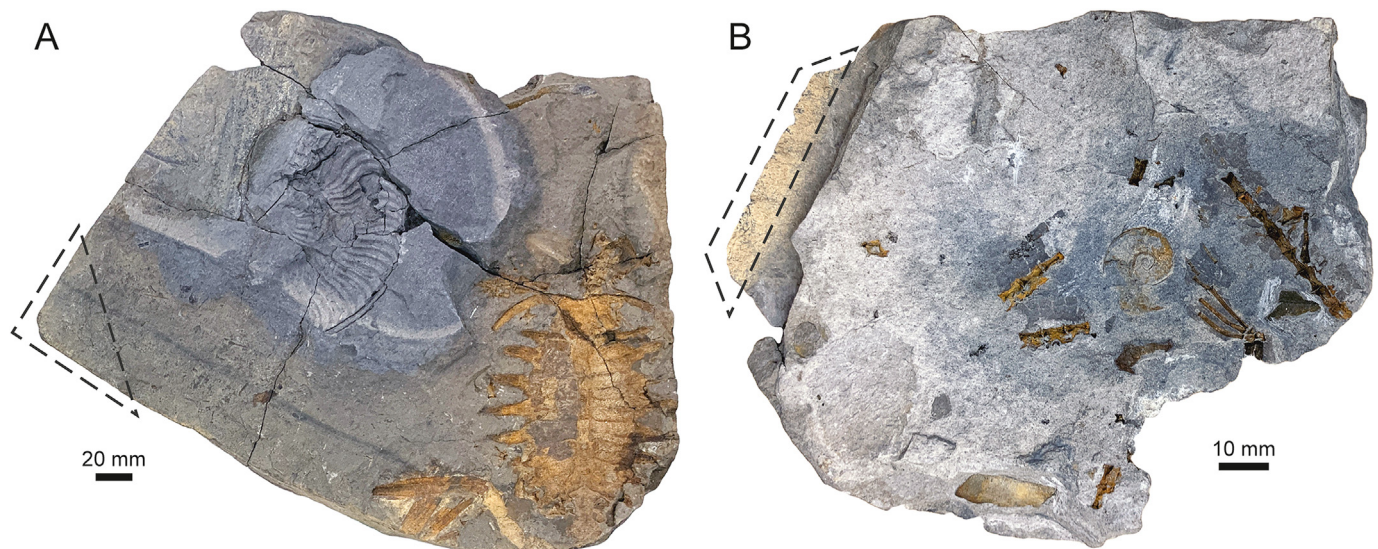
The Toarcian Ocean Anoxic Event (T-OAE, ~182.5 Ma) was an interval of global environmental change that was characterized by seawater deoxygenation and the widespread deposition of organic carbon-rich facies (Jenkyns, 1988). The sedimentary deposits of the T-OAE are also characterized by a marked negative carbon isotope excursion (NCIE), which is present in both marine and nonmarine sedimentary strata. As such, the NCIE is reflective of a global perturbation of the carbon cycle during that time, which has been linked to the release of  $^{12}\text{C}$ -enriched carbon from volcanism (Hesselbo et al., 2000, 2007; Them et al., 2017; Remírez and Algeo, 2020), methane release (Kemp et al., 2005; Ikeda et al., 2018), and decreased chemical weathering (Ikeda et al., 2018). The T-OAE NCIE also coincided with a faunal turnover, which remains relatively understudied. Some studies have documented a collapse in genus- and species-level marine biodiversity in ammonoids

and foraminifera during the early Toarcian coeval with the NCIE (e.g., Dera et al., 2010; Caruthers et al., 2013; Gorican et al., 2013), and others have proposed a link between the environmental perturbations that triggered the T-OAE and a nonmarine faunal turnover in dinosaurs, with relic taxa like coelophysoid theropods, non-eurypod thyreophorans, and non-sauropod sauropodomorphs going extinct during the Toarcian (Reolid et al., 2022).

The shallow marine Lower Jurassic (Pliensbachian–Toarcian) Nishinakayama Formation of the Toyora Group in Shimonoseki, Yamaguchi, Japan (Figs. 1 and 2) preserves the best record of the T-OAE from western Panthalassa (Izumi et al., 2012, 2018a, 2018b, 2020; Kemp and Izumi, 2014; Kemp et al., 2019), but its vertebrate fossil record remains relatively understudied among Early Jurassic vertebrate fossil assemblages. Although vertebrate fossils are neither as abundant nor as diverse as in some coeval strata such as the Lower Jurassic Kayenta Formation in the Navajo Nation, Arizona, USA (Tykoski, 2005) or the Lower Jurassic



**Fig. 1.** Regional geology of the study area in Toyotacho. (A) Geographic location of Toyotacho within Japan. (B) Simplified geologic map of southern Toyotacho highlighting the study area within Ishimachi. Maps redrawn after Me de Miru Furusato Toyota no Rekishi to Bunka Hensaniinkai (1999); Nakada and Matsuoka (2011); Izumi et al. (2012); Kawano (2014).



**Fig. 2.** Reptile fossil specimens from the Nishinakayama Formation in Toyotacho, Yamaguchi, Japan. (A) Testudinata, MMHF 5-00001. (B) Crocodylomorpha, MMHF 5-00002. Dashed lines indicate regions of matrix sampled for geochemical measurements.

Posidonia Shale in southern Germany (Etter and Tang, 2002), the Nishinakayama Formation has nevertheless yielded several articulated specimens of actinopterygian fish (Tanabe et al., 1982; Tanabe, 1991; Nakada and Matsuoka, 2012; Breeden, 2019), a single nearly complete turtle specimen (MMHF 5-00001; Fujinaga, 1990, 2022; Hasegawa et al., 1998), and a partially articulated but incomplete crocodylomorph postcranial skeleton (MMHF 5-00002; Manabe and Hasegawa, 1998). Because of their potential to elucidate reptile diversification throughout the T-OAE, this study focuses on the latter two specimens (Fig. 2), both of which were collected *ex situ* from cobbles in the river Eragawa (the Japanese suffix *-gawa*, which means ‘river,’ is transcribed as part of the proper names of rivers rather than translated herein; see supplementary materials for Japanese romanization strategies) in the town of Toyotacho (*Toyota-chō*) in northeastern Shimonoseki.

The crocodylomorph MMHF 5-00002 is possibly referable to Thalattosuchia, a clade with a fossil record beginning in the Pliensbachian but a ghost lineage extending into the Late Triassic (Wilberg et al., 2019, 2023). The age of this fossil therefore has the potential to constrain the early diversification and distribution of this major radiation of marine crocodylomorphs. Likewise, the Early Jurassic is a critical interval in turtle evolution as the Early Jurassic record of turtles is poor relative to the Late Triassic and later in the Jurassic when crown turtles evolved and diversified. Most of the Early Jurassic record of turtles is restricted to single localities (Joyce, 2007), and MMHF 5-00001 and an isolated *ex situ* costal plate from Hettangian–Toarcian strata within the Kuruma Group in Toyama, Japan (Sonoda et al., 2015) are the only specimens found in Laurasian Asia. Furthermore, Evers and Benson (2019) recognized three independent evolutionary transitions to marine life in non-pleurodiran turtles, only one of which (the non-testudinate Triassic taxon *Odontochelys* Li et al., 2008) lies outside of Testudines (i.e., crown turtles). MMHF 5-00001 may therefore represent a previously unrecognized transition to marine life among early turtles.

Herein, we use geochemical fingerprinting to infer the provenance of the two reptile fossils (MMHF 5-00001 and MMHF 5-00002) from the Nishinakayama Formation. The T-OAE NCIE has been well-documented in the Nishinakayama Formation at Sakuraguchidani (commonly translated or transcribed as Sakuraguchi-dani, Sakuraguchidani Valley, Sakuraguchi-dani Valley in earlier studies), which is a locality ~1 km southwest of Eragawa where the two reptile fossils were collected (Izumi et al., 2012, 2018a, 2018b; Kemp and Izumi, 2014); however, the T-OAE NCIE has not yet been established at Eragawa, and although the Toarcian age of the upper Nishinakayama Formation is now well established, it is

unclear how these reptile fossils relate to the major environmental changes throughout the T-OAE. Establishing the relative age of each fossil is therefore critical for interpreting its evolutionary implications.

The aim of this study is to estimate the approximate stratigraphic provenance of each fossil by characterizing the geochemistry of both the slabs of rock within which each specimen is preserved and the succession of strata in nearby outcrops of the Nishinakayama Formation from which they may feasibly have originated based on lithology and ammonoid biostratigraphy. We provide new geochemical data from a composite measured stratigraphic section of outcrops exposed along and near Eragawa, including stable isotope ratios of organic carbon ( $\delta^{13}\text{C}_{\text{org}}$ ), total organic carbon abundance (TOC), and major and trace elemental abundances measured using pXRF. We use these data to correlate this stratigraphic section with other Lower Jurassic successions regionally and globally using known geochemical phenomena (e.g., the NCIE associated with the T-OAE) and elucidate paleoenvironmental conditions. Finally, we use multivariate principal components (PCA) and linear discriminant (LDA) analyses of the total geochemical dataset to qualitatively test whether the geochemical data can statistically predict the probable stratigraphic provenance of the fossils and thus their ages relative to the T-OAE.

Institutional abbreviations: MMHF, Mine City Museum of History and Folklore (*Mine-shi Rekishi Minzoku Shiryōkan*), Mine, Yamaguchi, Japan.

## 2. Geological setting and stratigraphy

The Lower to Middle Jurassic (Sinemurian to Bathonian) Toyora Group is exposed across two districts to the north and south of the NW-SE trending Tabu Fault in Shimonoseki, Yamaguchi, Japan (Fig. 1). The study area is in the northern district, where the Nishinakayama Formation is exposed in southern Toyotacho, primarily along the river Koyagawa and its tributaries (Fig. 1B). Although the Nishinakayama Formation also crops out in the southern district within Kikugawacho (see Fig. S1E in supplementary materials), the biostratigraphy there is not well-established owing to a general lack of biostratigraphically informative macrofossils such as ammonoids (Kawamura, 2010), and no vertebrate fossil material has yet been reported from that region.

In many previous studies, Toyotacho was called the “Toyora” region or area (= *Toyoura-gun*, hereafter Toyoura; e.g., Izumi et al., 2012; Kemp and Izumi, 2014; Takeda and Tanabe, 2015; Izumi, Kemp and Breeden, 2019; Kemp et al., 2019); however, Toyoura was dissolved as an administrative district in 1926, and its toponym was finally retired from

both formal and informal use in 2005 (Shimonoseki–Toyoura Merger Council, 2005; Japan Post, 2005). Conversely, the names of Toyotacho and the three other towns that Toyoura once comprised remain in use as administrative divisions of the city of Shimonoseki (see Fig. S1E in supplementary materials). To avoid potential confusion with the town of Toyoura (*Toyouura-chō*) in western Shimonoseki (see Fig. S1E in supplementary materials), we therefore herein refer to Toyotacho by name rather than continuing to use the defunct name of the dissolved district of Toyoura (i.e., *Toyouura-gun*, “Toyora”). For a detailed historical geography of Toyotacho, see the supplementary online materials.

In the study area within Toyotacho, the Toyora Group unconformably overlies the metamorphosed Paleozoic rocks of the Nagato Tectonic Zone (Kawamura, 2010) and comprises, in ascending stratigraphic order, the Higashinagano Formation (*Higashinagano-sō*; Sinemurian–Pliensbachian), the Nishinakayama Formation (*Nishinakayama-sō*; Pliensbachian–Toarcian), and the Utano Formation (*Utano-sō*; Toarcian–Bathonian). These have been interpreted to represent transgressive, inundative, and regressive phases of a major sedimentary cycle, respectively (Hirano, 1971). The Nishinakayama Formation comprises silty shale, mudstone, sandstone, and volcanic tuff horizons which were deposited in a shallow marine environment with terrestrial influence (Kimura and Ohana, 1987; Izumi et al., 2012; Kemp and Izumi, 2014; Kemp et al., 2019). The Nishinakayama Formation in this region is approximately 300 m in maximum thickness and was divided into three members (Tanabe et al., 1982): Na (silty shale, ~90 m thick), Nb (dark silty shale with intercalated fine-grained sandstone and laminated black shales, ~160 m thick), and Nc (alternating sandstone and mudstone, ~20–60 m thick). The Nb member is relatively well-exposed in Toyotacho, primarily in valleys formed by both permanent rivers and ephemeral mountainside streams; however, continuous exposures are rare.

The biostratigraphic framework of the Toyora Group was first established by Hirano (1971, 1973a, 1973b) and more recently revised by Nakada and Matsuoka (2011) at Sakuraguchidani (34°10'N, 131°03'E; Fig. 1B). Nakada and Matsuoka (2011) recognized four ammonoid assemblage zones within the Nishinakayama Formation at Sakuraguchidani. In ascending stratigraphic order, those are the *Canavaria japonica*, *Paltarpites paltus*, *Dactyloceras helianthoides*, and *Harpoceras inouyei* zones. The *Canavaria japonica* Zone is exposed only along the southern branch of Sakuraguchidani and is characterized by the occurrence of *Canavaria japonica* and the Pliensbachian index taxon *Amaltheus margaritatus*. The same morphotype of *Amaltheus margaritatus* occurs in northwestern Europe, which allows correlation between the *Canavaria japonica* Zone of Japan with the *Pleuroceras apyrenum* Subzone of Europe (Meister, 1988). Nakada and Matsuoka (2011) placed the boundary between the Pliensbachian and Toarcian stages at the base of the *Paltarpites paltus* Zone; however, the contact between the *Canavaria japonica* and *Paltarpites paltus* zones is ambiguous owing to a scarcity of ammonoid fossils from that interval.

In the Sakuraguchidani section, a ~3.5‰ NCIE spanning ~30 m of the Nb member was recognized and interpreted as an expression of the T-OAE (Izumi et al., 2012, 2018b; Kemp and Izumi, 2014). That interpretation was supported by ammonoid biostratigraphy (Izumi et al., 2012). The negative carbon isotope excursion observed in the Sakuraguchidani section occurs within the *Dactyloceras helianthoides* Zone of Nakada and Matsuoka (2011) in the lower Toarcian and can be chemostratigraphically correlated to lower Toarcian sections in Europe and North America (Kemp and Izumi, 2014; Izumi et al., 2018b).

Another significant but understudied series of outcrops of the Nb member of the Nishinakayama Formation is exposed in the Ishimachi district of Toyotacho. The Nishinakayama Formation discontinuously crops out along the bed of Eragawa, which flows generally eastward into Koyagawa approximately 1 km north of the Sakuraguchidani section (34°10'N 131°03'E). At the mouth of Eragawa, the lowest outcrop of the Nishinakayama Formation extends into Koyagawa and can be correlated to another stratigraphically lower outcrop of the Nishinakayama Formation approximately 100 m to the northeast at the mouth of Honekigawa. Within these collective outcrops (hereafter, the ‘Ishimachi

section’), the Nishinakayama Formation generally strikes NE-SW and dips to the northwest at 20°–40°. Whereas the Sakuraguchidani section has recently been the subject of rigorous stratigraphic, sedimentological, geochemical, ichnological, and paleontological study (e.g., Nakada and Matsuoka, 2009, 2011; Izumi et al., 2012, 2018a, 2018b, 2020; Kemp and Izumi, 2014; Kemp et al., 2019; Kitabatake et al., 2019), the outcrops exposed along the rivers Eragawa and Honekigawa have received little attention since the early 1990s. It was within the Eragawa interval that the reptile fossils MMHF 5-00001 and MMHF 5-00002 were found *ex situ* (Fujinaga, 1990, 2022; Hasegawa et al., 1998; Manabe and Hasegawa, 1998; Breeden, 2019). Additionally, the biostratigraphy of the Ishimachi section has not been well-established owing to limited outcrop and a relative lack of abundant biostratigraphically informative marine fossils compared to the Sakuraguchidani section, and the Nb member of the Nishinakayama Formation varies lithologically over short distances, hindering local lithostratigraphic correlation. As a result, the stratigraphic context of those reptile fossils relative to the T-OAE and the associated Toarcian biotic turnover has not been systematically established.

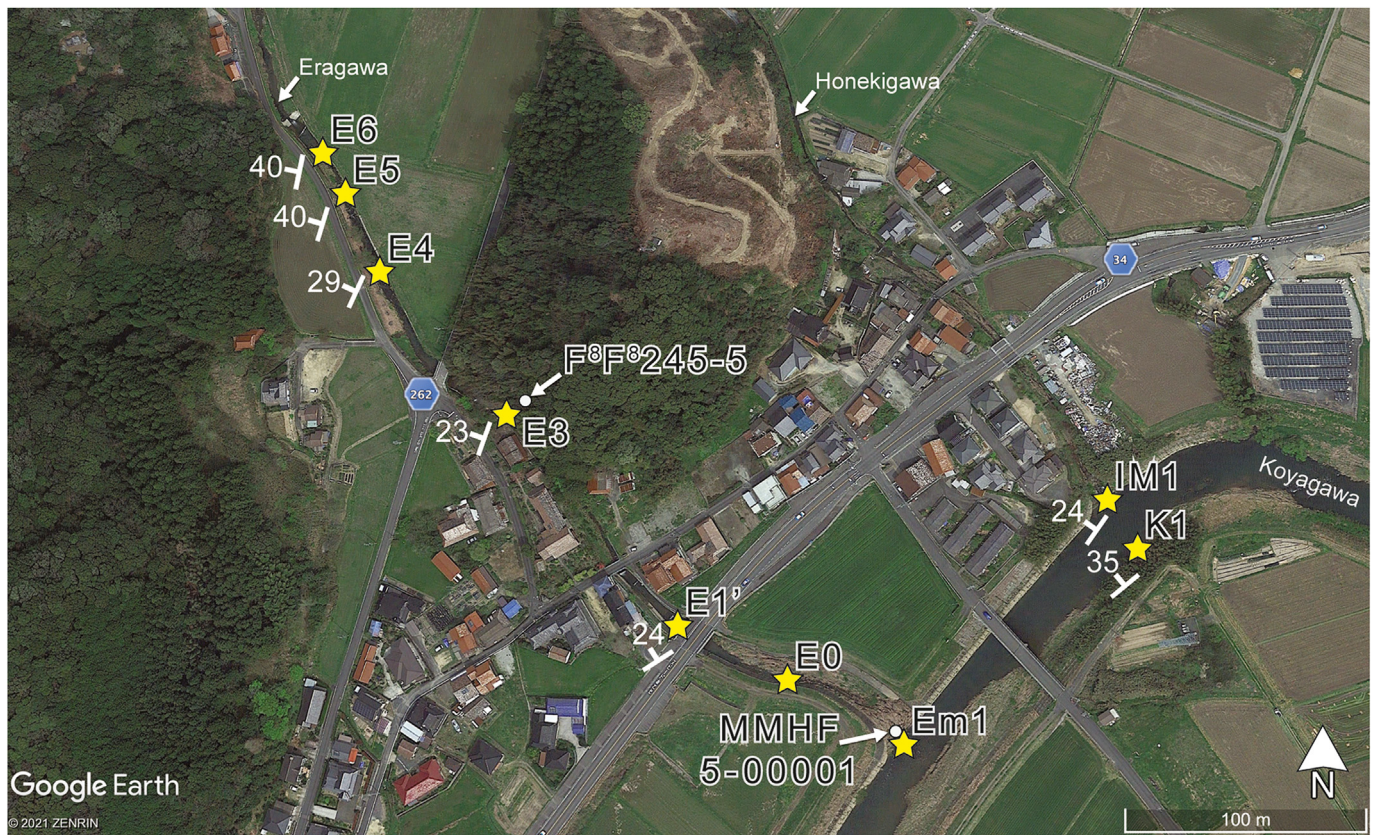
Hirano, 1971, 1973b, 1973a recognized five ammonoid fossil localities within the Nb member of the Nishinakayama Formation in the Ishimachi district of Toyotacho. However, the only ammonoid species that they observed were *Fucinoceras nakayamense* and *Dactyloceras helianthoides* in the first locality and *Dactyloceras helianthoides* in the second and third, stratigraphically lower localities. This constrains both localities to either the *Dactyloceras helianthoides* Zone or *Harpoceras inouyei* Zone of Nakada and Matsuoka (2011) within the lower Toarcian Stage (see supplementary materials for summary of ammonoid occurrences noted by Hirano [1971] at these localities).

Additionally, the shale slabs preserving the reptile fossils also preserve individuals of the hildoceratid ammonoid taxa *Cleviceras chrysanthemum* (MMHF 5-00001; Hasegawa et al., 1998) and *Harpoceras inouyei* (MMHF 5-00002; Manabe and Hasegawa, 1998), both of which have a stratigraphic range correlated to the lower Toarcian Stage. Based on the ammonoid biostratigraphy of Nakada and Matsuoka (2011), MMHF 5-00001 can thus be placed in the *Harpoceras inouyei* Zone, and MMHF 5-00002 can be placed in any of the *Paltarpites paltus*, *Dactyloceras helianthoides*, and *Harpoceras inouyei* zones. In the absence of useful biostratigraphic specimens, carbon isotope stratigraphy and elemental abundances provide an alternate means of both establishing the stratigraphic context of the reptile fossil specimens and correlating the Ishimachi section of the Nishinakayama Formation both regionally and globally.

### 3. Materials and methods

#### 3.1. Fossil specimens

The specimens were collected *ex situ* from the bed of Eragawa by Mr. Tadashi Fujinaga (MMHF 5-00001; Fig. 2A) in 1989 and the late Mr. Hideo Ishida (MMHF 5-00002; Fig. 2B) in 1986, and subsequently donated to the Mine City Museum of History and Folklore (MMHF). In addition to these two specimens, MMHF and its companion museum the Mine City Fossil Museum (*Mine-shi Kasekikan*) in Mine, Yamaguchi, Japan house remarkable collections of early Mesozoic plant (e.g., Naito, 2000), invertebrate (Fujiyama, 1973, 1974, 1991; Oyama et al., 2020, 2023; Oyama and Maeda, 2020), and vertebrate (Uyeno et al., 1996, 2003; Jinnouchi et al., 2018) fossils from western Yamaguchi, which have mostly been overlooked by researchers outside of Japan. Each of the two reptile fossils is preserved mostly as molds on and within split part and counterpart shale slabs with little original bone, hindering manual preparation and thus anatomical and systematic interpretation of the specimens when initially described. However, each will be re-evaluated in a modern phylogenetic context in separate studies.



**Fig. 3.** Map of outcrop localities within the Ishimachi section of the Nishinakayama Formation in Toyotacho, Yamaguchi, Japan. This map includes the geographic locations of each outcrop; the locality of the fossil turtle specimen MMHF 5-00001; and the Shimonoseki cadastral survey marker F<sup>8</sup>F<sup>8</sup>245-5 designated as stratigraphic zero. Strike and dip values are averages of measurements taken from multiple bedding planes within each outcrop. General outcrop sites are indicated by yellow stars, and individual points are designated by white circles.

### 3.2. Geological field methods

A composite stratigraphic section of the Lower Jurassic Nishinakayama Formation was measured and sampled for geochemical analysis between 2016 and 2020 along the rivers Eragawa, Honekigawa, and Koyagawa in Toyotacho Ishimachi, Shimonoseki, Yamaguchi, Japan (Fig. 4). Nine sites with accessible exposures of outcrop were recognized, which are designated Em1, IM1, K1, E0, E1', E3, E4, E5, and E6 in stratigraphic order (Figs. 3 and 4). The stratigraphic interval measured at K1 overlaps with the interval measured at IM1, so these are treated as discrete sites geographically but as the single stratigraphic interval IM1/K1 for the purposes of discussion. These sites are separated either by water, vegetation, or detrital overburden, and stratigraphic distance between outcrops was estimated using strike, dip, and geographic surface distance measured with a Jacob's staff monopod surveying instrument. The composite section (hereafter, the 'Ishimachi section') begins at lat. 34.176391°N, long. 131.063483°E and continues upsection to lat. 34.178131°N, long. 131.059557°E, and the Shimonoseki cadastral survey (*Shimonoseki-shi Toyota Sōgō Shisho Kensetsuka Chiseki Chōsakakari*) marker F<sup>8</sup>F<sup>8</sup>245-5 located at lat. 34.17702°N, long. 131.06063°E was designated as stratigraphic zero (Fig. 4F). Bulk rock samples (n = 215) were collected from unweathered shale, sandstone, and tuff beds throughout the Ishimachi section. Because the Nishinakayama Formation is not continuously exposed throughout the Ishimachi section, it was not possible to sample at a consistent interval, but a 2-decimeter sampling resolution was attempted where possible. Few *in situ* ammonoids were recovered from the Ishimachi section throughout the course of sampling for chemostratigraphy.

### 3.3. Carbon isotope ( $\delta^{13}C_{org}$ ) and total organic carbon (TOC) analysis

Seventy-nine samples from the Ishimachi section with fine-grained lithologies (i.e., siltstones and mudstones) were selected for the measurement of total organic carbon (TOC) and  $\delta^{13}C_{org}$  along with matrix sampled from both MMHF 5-00001 and MMHF 5-00002. Samples were prepared for analysis at the Department of Geology & Geophysics, University of Utah. Approximately 100 mg of each sample was powdered using a Dremel® Model 800 cordless rotary tool with a McCaster-Carr SA-41 1/8" double cut carbide flat cylinder burr drill bit cleaned between samples with ethanol. To remove carbonates and any other acid-soluble minerals, powdered samples were then treated with ~5 mL of 6 M hydrochloric acid (HCl) until no reaction was observed and left for 4 h total in 15 mL polypropylene centrifuge tubes. After acidification, the decalcified residues were centrifuged at 3000 rpm for 5 min using an Eppendorf Centrifuge 5804, and any HCl remaining after decarbonation was decanted. Sample residues were then rinsed with deionized and ultra-pure Milli-Q® water and mixed using a VWR Fixed Speed Vortex Mixer to ensure saturation. Sample residues were then centrifuged at 3000 rpm for 5 min, after which the supernatant of each was decanted and discarded. This process was repeated until the decanted supernatant of each sample reached a neutral pH. Residues were then dried in an oven at 50 °C for 48 h, and each was re-homogenized into a powder.

TOC and  $\delta^{13}C_{org}$  were both measured using a EuroVector Elemental Analyzer (EA) coupled to an Elementar Isoprime Isotope Ratio Mass Spectrometer (IRMS) at the Paleoclimate Colaboratory, University of Maryland, College Park, USA. 1.5–5 mg of each sample residue was weighed into a 4 mm × 6 mm tin capsule. Capsules were then dropped



**Fig. 4.** Representative photographs of select outcrops from the Ishimachi section of the Nishinakayama Formation in Toyotacho, Yamaguchi, Japan. (A) David Kemp (left) and Kentaro Izumi (right) standing on outcrop IM1 within the river Koyagawa with outcrop K1 visible along opposite bank in background. (B) Outcrop IM1 exposed in the mouth of the river Honekigawa at its confluence with Koyagawa. (C) Outcrop Em1 exposed in the mouth of the river Eragawa at its confluence with Koyagawa. (D) Tadashi Fujinaga standing at the point where he discovered the turtle fossil MMHF 5-00001 *ex situ* in Eragawa immediately upstream from its confluence with Koyagawa. (E) Outcrop E1' exposed along the southwestern bank of Eragawa. (F) Shimonoseki cadastral survey marker F<sup>8</sup>F<sup>8</sup>245-5 staked into outcrop E3 in Eragawa at the point designated as stratigraphic zero in the Ishimachi section. (G) Outcrop E5 exposed in Eragawa. (H) Outcrop E6 exposed in Eragawa.

sequentially by an autosampler into the top of a quartz combustion column packed with chromium oxide and silvered cobaltous/cobaltic oxide and heated to 1040 °C to ensure the complete oxidation of carbon to CO<sub>2</sub>. CO<sub>2</sub> released in the column via flash combustion was then passed through a magnesium perchlorate water trap and separated by a gas chromatograph (GC) column prior to elemental and isotopic analysis. TOC is reported in percentage by weight percent (%), and  $\delta^{13}\text{C}_{\text{org}}$  is

reported relative to the Vienna Pee Dee Belemnite standard (VPDB). Uncertainty was determined by comparison to the in-house urea ( $\delta^{13}\text{C} = -29.39\%$ ; TOC = 20%) and LECO 062 ( $\delta^{13}\text{C} = 0.25\%$ ; TOC = 0.04%) standards.

Additionally, 34 samples were analyzed for TOC and  $\delta^{13}\text{C}_{\text{org}}$  at Geo-Science Laboratory (*Chikyū Kagaku Kenkyūsho*), Nagoya, Aichi, Japan and 14 samples were analyzed for  $\delta^{13}\text{C}_{\text{org}}$  at Elementex, Ltd., Cornwall,

England, UK using equivalent methods of sample preparation and analysis to those described above. The instruments used for these analyses were a Thermo Finnigan FLASH2000 EA coupled to a Thermo Finnigan DELTAplus Advantage IRMS at Geo-Science Laboratory and a Thermoquest EA1110 EA coupled to Europa Scientific 20-20 IRMS at Elementx.

### 3.4. X-ray fluorescence

Fresh unweathered surfaces of ninety representative mudstone samples from the Ishimachi section and slabs of mudstone matrix from MMHF 5-00001 and MMHF 5-00002 (Fig. 2) were analyzed for major and trace element abundances using a Bruker AXS TRACER III-V energy-dispersive portable X-ray fluorescence (pXRF) unit (Bruker, Billerica, MA, USA) at the University of Utah. Relative elemental abundances were measured at 15 kV and 25  $\mu$ A for low mass elements ('majors') and 40.6 kV and 15  $\mu$ A for heavy elements ('traces') using S1PXR software. A helium flush was used for measurements of major elements, and a green Bruker filter composed of 12 mil Al, 1 mil Ti, and 6 mil Cu was used for measurements of trace elements. Raw data (see supplementary materials) were collected as spectrograms. In the absence of internationally accepted mudstone standards with adequate elemental ranges covering the geochemical diversity of mudstones, raw measurements were calibrated to weight percent for major elements and ppm for trace elements using a proprietary matrix-specific mudstone instrument calibration (Rowe et al., 2012). The pXRF instrument used for this study was calibrated using a suite of five internationally accepted commercial standards and a suite of 85 mudstone standards developed in-house by Rowe et al. (2012).

pXRF data are not as precise as other methods of determining elemental abundances, but multiple studies have demonstrated that pXRF is accurate for most elements of interest (e.g., Rowe et al., 2012; Saker-Clark et al., 2019). Although pXRF measurements are most useful for first-order observations to guide decisions to make more accurate measurements using other techniques, in using these data for geochemical fingerprinting, accuracy is less important as long as any bias in measurement of particular elements is systematic across samples. Nonetheless, to be conservative we have eliminated some elements from

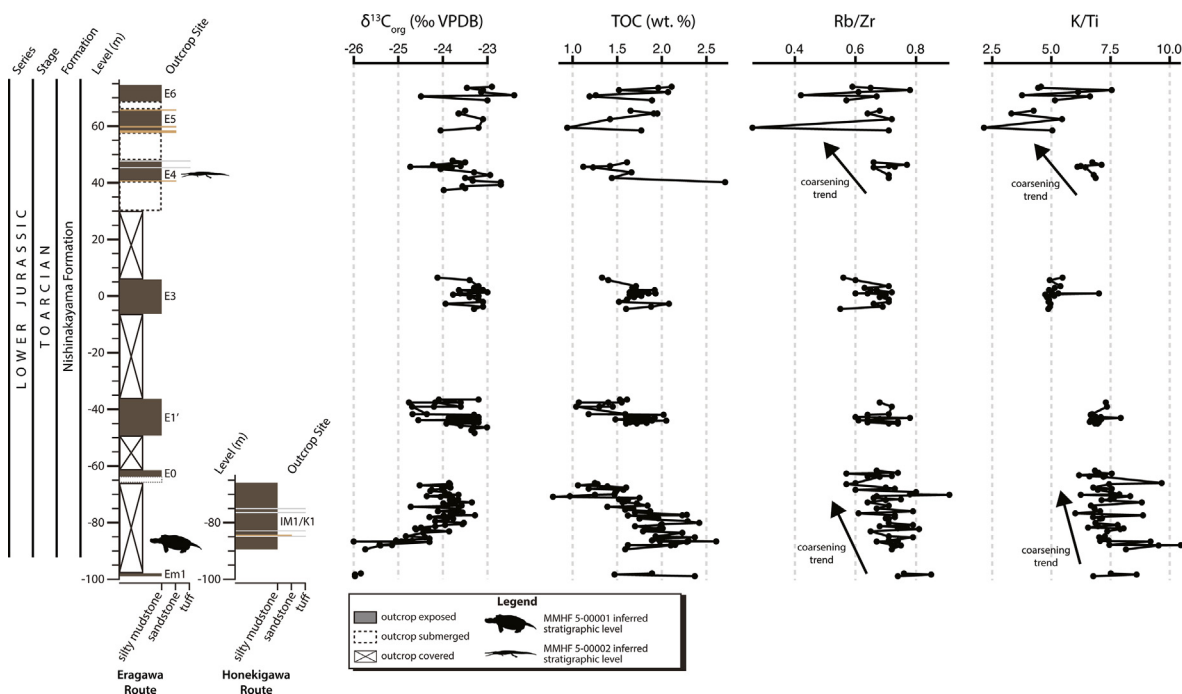
the dataset that are demonstrated to be less accurate with this calibration process (Rowe et al., 2012). Some remaining elements still showed negative calibrated values, but their inclusion in the multivariate analyses strengthens the confidence of the results, so they still seem to capture useful chemostratigraphic information, and we have retained all but Mo (for which all calibrated values were negative) for the LDA. In total, thirteen major elements (Mg, Al, Si, P, S, K, Ca, Ba, Ti, V, Cr, Mn, Fe) and ten trace elements (Ni, Cu, Zn, Th, Rb, U, Sr, Y, Zr, Nb) were retained in the final calibrated dataset.

### 3.5. Multivariate statistical analyses

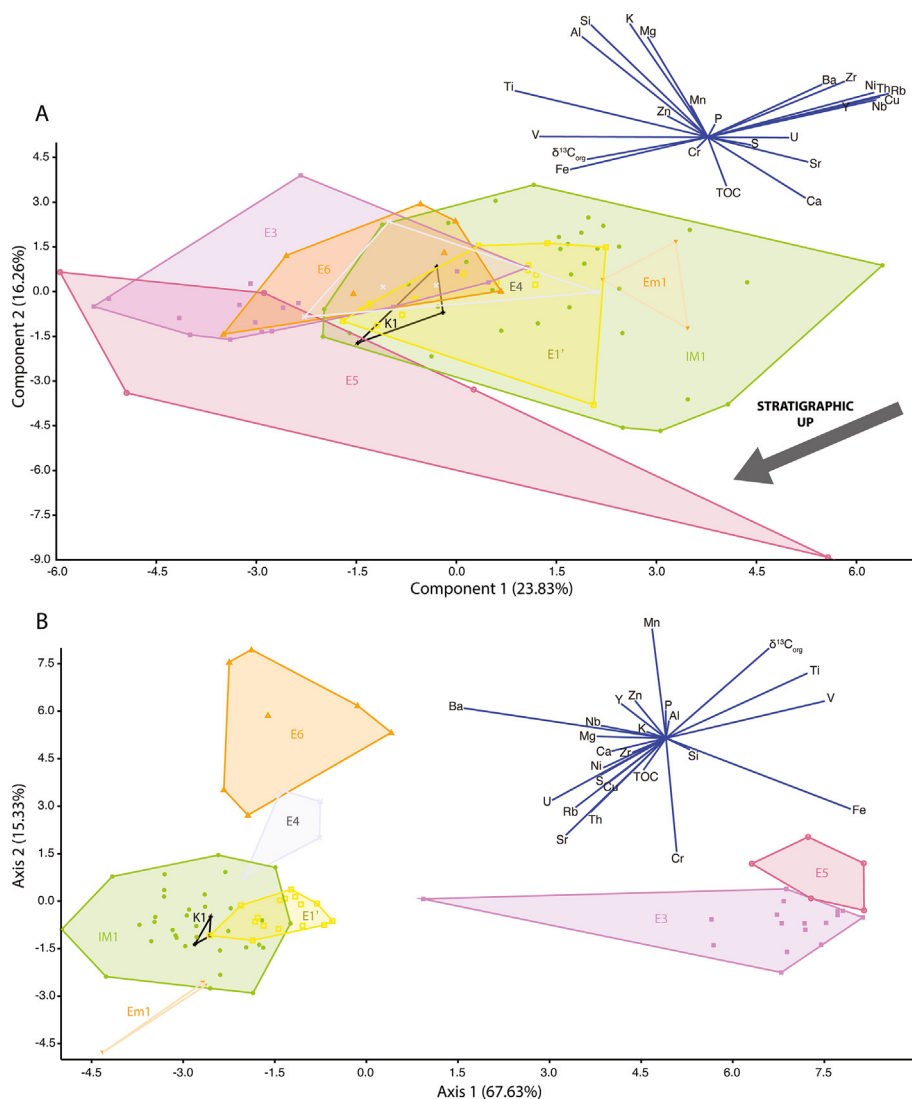
A dataset comprising  $\delta^{13}\text{C}_{\text{org}}$  values, TOC values, and calibrated elemental abundance pXRF data for a suite of ninety samples plus the matrix from the two reptile fossil specimens MMHF 5-00001 and MMHF 5-00002 ( $n = 92$ ) was compiled to statistically predict the stratigraphic provenances of the fossil specimens. A principal component analysis (PCA) was first conducted to qualitatively assess whether the dataset was stratigraphically informative, and a linear discriminant analysis (LDA) was performed to predict the outcrop-level stratigraphic provenance of the fossil specimens using the samples from the known outcrop sites as a training dataset. Statistical analyses were performed using the software PAST4.04 for Mac (Hammer et al., 2001).

## 4. Results

$\delta^{13}\text{C}_{\text{org}}$  values in the Ishimachi section ranged from  $-26.00\text{‰}$  to  $-22.40\text{‰}$  (Fig. 5; see supplementary materials for sample list and IRMS data).  $\delta^{13}\text{C}_{\text{org}}$  ranged from  $-25.97\text{‰}$  to  $-25.84\text{‰}$  at Em1,  $-25.74\text{‰}$  to  $-23.28\text{‰}$  at IM1,  $-26.00\text{‰}$  to  $-24.30\text{‰}$  at K1,  $-24.68\text{‰}$  to  $-23.01\text{‰}$  at E1',  $-24.12\text{‰}$  to  $-23.00\text{‰}$  at E3,  $-24.73\text{‰}$  to  $-22.70\text{‰}$  at E4,  $-24.05\text{‰}$  to  $-23.10\text{‰}$  at E5, and  $-24.49\text{‰}$  to  $-22.40\text{‰}$  at E6 (Fig. 5). The  $\delta^{13}\text{C}_{\text{org}}$  profile can be divided into two main intervals throughout the Ishimachi section. The first ( $-98.9$  to  $-84.8$  m) spans Em1 through the lower sequence of IM1/K1 and is characterized by a steady increase of  $\sim 1.20\text{‰}$  from  $-26.00\text{‰}$  to  $-24.8\text{‰}$ . The second of which ( $-83.6$  to  $74.4$  m) spans the upper sequence of IM1/K1 through E6 and shows a range of



**Fig. 5.** Ishimachi section stratigraphy. Lithostratigraphy, organic carbon ( $\delta^{13}\text{C}_{\text{org}}$  and TOC) chemostratigraphy, and stratigraphic distribution of detrital proxies (Rb/Zr and K/Ti) in the Ishimachi section. Note the relationship between the Rb/Zr and K/Ti grain size proxies and the distribution of sandstone horizons.



**Fig. 6.** Results of principal components and linear discriminant function analyses. Samples are grouped and color-coded by outcrop site. (A) Principal components 1 and 2 of known samples from the Ishimachi section showing that stratigraphically higher samples are characterized by relatively lower PC1 and PC2 values. (B) Discriminant axes 1 and 2 from the LDA. Eigenvector plots for each analysis are provided in blue.

–24.76‰ to –22.40‰ without any clear positive or negative trends. TOC values ranged from 0.78% to 2.71% throughout the entire section (Fig. 5), 1.47%–2.37% at Em1, 0.78%–2.42% at IM1, 1.89%–2.61% at K1, 1.18%–2.02% at E1', 1.33%–2.08% at E3, 1.12%–2.71% at E4, 0.94%–1.95% at E5, and 1.19%–2.11% at E6. The TOC profile is characterized by a steady decrease from average values > 2.0% to average values < 2.0% throughout the IM1/K1 interval, mirroring the  $\delta^{13}\text{C}_{\text{org}}$  trend (Fig. 5). The  $\delta^{13}\text{C}_{\text{org}}$  and TOC values of the matrix from the two fossil specimens are –25.22‰ and 0.68% for MMHF 5-00001 and –24.16‰ and 0.59% for MMHF 5-00002. The  $\delta^{13}\text{C}_{\text{org}}$  values for the fossil specimens are within the range observed in the Ishimachi section. However, the TOC values of both are curiously lower than any observed throughout the Ishimachi section.

The PCA of the geochemical profiles (i.e.,  $\delta^{13}\text{C}_{\text{org}}$ , TOC, and pXRF data) from the suite of known samples (Fig. 6A; see supplementary materials for results) shows that differences are concentrated primarily along the first two principal components and reveals two main clusters of samples. The first comprises samples from outcrop sites Em1, K1, IM1, and E1' and is characterized by relatively higher PC1 and PC2 values, and the second comprises samples from outcrop sites E3, E4, E5, and E6 and is characterized by relatively lower PC1 and PC2 values. A weak stratigraphic signal was thus captured by the carbon and pXRF data,

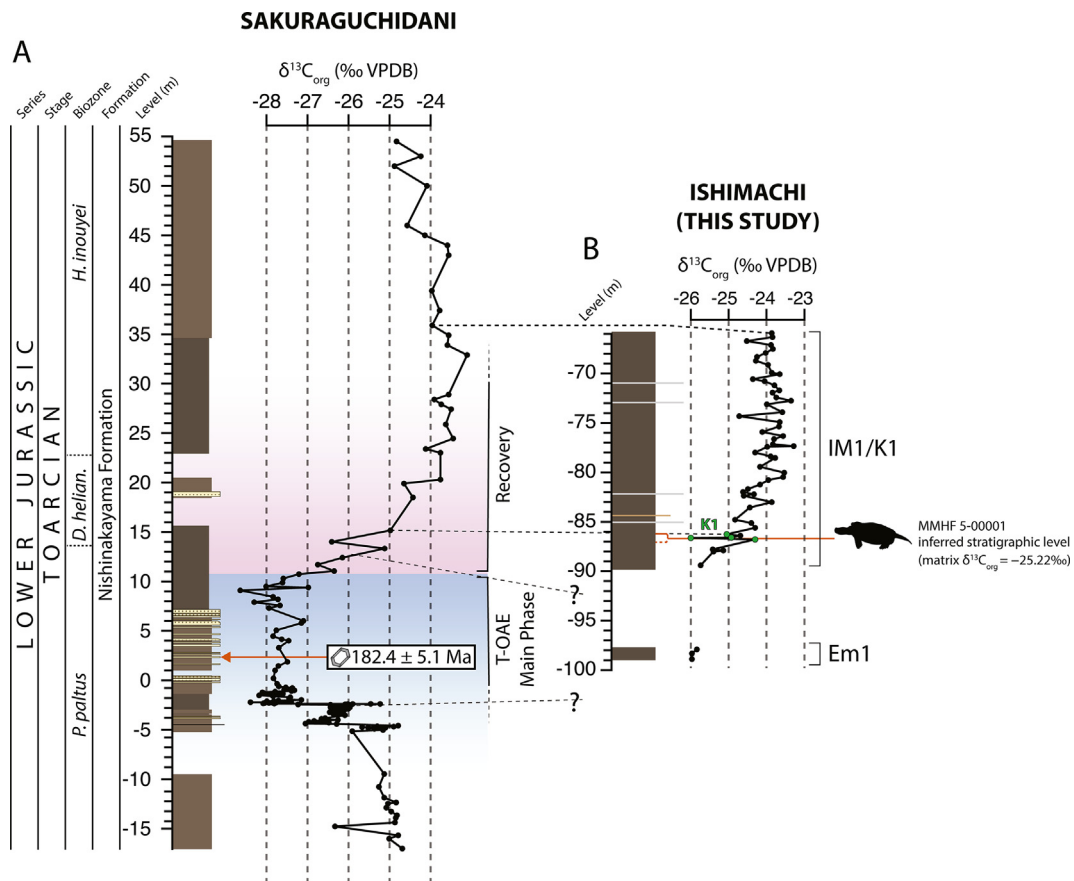
qualitatively indicating that it is possible to quantify the likely provenance of the *ex situ* fossil specimens using a linear discriminant analysis.

The LDA (Fig. 6B; see supplementary materials for results) correctly identified 84 out of 90 (93.33%) samples from known outcrop sites. Jackknifing the training dataset of known samples led to more mis-identifications, with only 57 out of 90 (62.22%) samples correctly identified. Axis 1 is most strongly influenced by Fe and Ba, whereas axis 2 is most strongly influenced by Cr and Mn. Using this training dataset, the LDA identified the *ex situ* fossil specimens MMHF 5-00001 and MMHF 5-00002 as originating from outcrop sites K1 and E4, respectively (Figs. 6 and 7).

## 5. Discussion

### 5.1. Correlation of the Ishimachi and Sakuraguchidani sections

For correlation with the Sakuraguchidani section of the Nishinakayama Formation, we used the Sakuraguchidani  $\delta^{13}\text{C}_{\text{org}}$  profile of Izumi et al. 2018b; see Fig. 2) as the primary reference. The  $\delta^{13}\text{C}_{\text{org}}$  profile of the lower Ishimachi section at outcrop sites Em1, IM1, and K1 compares favorably with the interval between –3 m and 35 m of the Sakuraguchidani section (Izumi et al., 2018b), as these intervals are both



**Fig. 7.** Chemostratigraphic correlation of the Ishimachi and Sakuraguchidani sections. Proposed correlation of the early Toarcian  $\delta^{13}\text{C}_{\text{org}}$  excursion interval within the Sakuraguchidani and lower Ishimachi sections of the Nishinakayama Formation in Toyotacho, Yamaguchi, Japan, and the inferred stratigraphic provenance of the fossil turtle specimen MMHF 5-00001 (B). Dashed lines show correlation based on changes in  $\delta^{13}\text{C}_{\text{org}}$ . Sakuraguchidani data (A) from Izumi et al. (2018b) and Izumi et al. (2020).

characterized by the presence of a distinct positive shift in  $\delta^{13}\text{C}_{\text{org}}$  (Fig. 7). Whereas the NCIE is preserved from its onset negative shift through the main phase to the recovery positive shift to background  $\delta^{13}\text{C}_{\text{org}}$  values at Sakuraguchidani, we interpret the positive shift at Ishimachi to represent only the uppermost interval of the main phase and the recovery. As such, at Sakuraguchidani, the lowest measured  $\delta^{13}\text{C}_{\text{org}}$  value is  $-28.64\%$  at 9.09 m (Izumi et al., 2018b), but the lowest value measured at Ishimachi is  $-26.00\%$  at  $-86.6$  m. The  $\delta^{13}\text{C}_{\text{org}}$  values at site Em1 are  $\sim 26\%$ , and the gap between the Em1 and IM1/K1 stratigraphic intervals may therefore span the main phase of the T-OAE NCIE with the interval characterized by the lowest  $\delta^{13}\text{C}_{\text{org}}$  values below  $-28\%$  at Sakuraguchidani covered and inaccessible—and therefore unsampled—at Ishimachi, or it may simply span the recovery interval. Between  $-89.4$  m and  $-83$  m of the Ishimachi section, there is a gradual positive shift in  $\delta^{13}\text{C}_{\text{org}}$  of  $\sim 2\%$ , which we correlate with the upper interval of the recovery phase of the main T-OAE negative excursion at Sakuraguchidani as  $\delta^{13}\text{C}_{\text{org}}$  never reaches values below  $-25\%$  again in either section above these intervals. However, it is clear that the entire negative excursion was not sampled at Ishimachi given the observed  $\sim 2\%$  change in  $\delta^{13}\text{C}_{\text{org}}$  at Ishimachi compared to the  $\sim 3.5\%$  change revealed at Sakuraguchidani (Izumi et al., 2018b).  $\delta^{13}\text{C}_{\text{org}}$  values ranging above  $-25\%$  and below  $-23\%$  are observed above the excursion interval in both the Sakuraguchidani and Ishimachi sections.

Both the ammonoid biostratigraphy of the Sakuraguchidani section and a U–Pb date of  $182.4 \pm 5.1$  Ma from a detrital zircon grain from 2.35 m of that section (Izumi et al., 2020) are consistent with the interpretation of this negative carbon isotope excursion interval as representing the T-OAE in the Ishimachi section, and we therefore infer that the entire Ishimachi section (including both reptile fossil specimens) is early

Toarcian in age and that none of the Pliensbachian interval of the Nishinakayama Formation was sampled at any of the Ishimachi section outcrop sites.

## 5.2. Stratigraphic provenance of *ex situ* fossils

The LDA predicted with 93.33% confidence that the turtle fossil MMHF 5-00001 originated from the stratigraphic interval represented by outcrops at site K1, which is consistent with what is known of the specimen. The  $\delta^{13}\text{C}_{\text{org}}$  value measured from the matrix of MMHF 5-00001 (Fig. 2A) is  $-25.22\%$ , and as noted above,  $\delta^{13}\text{C}_{\text{org}}$  never reaches a value below  $-25\%$  above the IM1/K1 stratigraphic interval. The geographic provenance of MMHF 5-00001 is also known to be between outcrop sites Em1 and E0 within Eragawa at a point roughly equivalent to  $-95$  m of the Ishimachi section (Figs. 5D and 7B; T. Fujinaga Aug. 3, 2019, personal communication). Although the IM1/K1 stratigraphic interval was too covered to sample above Em1 in Eragawa, we infer that MMHF 5-00001 originated from a point just upstream from where it was ultimately discovered *ex situ* from a stratigraphic interval equivalent to between  $-87$  m and  $-85$  m of the Ishimachi section sampled in Honekigawa and Koyagawa at site K1 based on the results of the LDA and  $\delta^{13}\text{C}_{\text{org}}$  analyses. MMHF 5-00001 is therefore considered early Toarcian in age, which is consistent with a previous age constraint based on ammonoid biostratigraphy (Hasegawa et al., 1998). The inferred stratigraphic provenance from the interval between  $-87$  m and  $-85$  m of the Ishimachi section also indicates that MMHF 5-00001 was deposited during the recovery interval of the T-OAE.

The LDA predicted with 93.33% confidence that the crocodylomorph fossil MMHF 5-00002 originated from the stratigraphic interval

represented by outcrops at site E4. The precise geographic provenance of MMHF 5-00002 within Eragawa is unknown, but as the specimen likely originated from E4 above the T-OAE interval, it is considered early Toarcian in age. This is consistent with a previous age constraint based on ammonoid biostratigraphy (Manabe and Hasegawa, 1998). It should be noted that it is possible that either fossil could have originated from a stratigraphic horizon that was exposed prior to its discovery during the 1980s that has since been covered. However, the results of the LDA are congruent with biostratigraphic interpretations, and the interpretations presented above are the best supported by the available data.

Our interpretations together place the western Panthalassan faunal assemblage of the Nishinakayama Formation coeval with other early Toarcian sites in Tethyan and eastern Panthalassan strata. In addition to the turtle and thalattosuchian crocodylomorph reptiles and abundant ammonoids, the total faunal assemblage of the Nishinakayama Formation is characterized by insects (Fujiyama, 1974), bivalves (Tanabe et al., 1982; Tanabe, 1991), belemnoids (Tanabe et al., 1982; Tanabe, 1991), crinoids (Tanabe et al., 1982; Tanabe, 1991; Hunter et al., 2011), decapod crustaceans (Karasawa, 2002), bony fish (Tanabe, 1991; Nakada and Matsuoka, 2012; Breden, 2019), and invertebrate trace fossils (Izumi et al., 2012; Izumi, 2014). Among vertebrates, fish remains are the most abundant in the Nishinakayama Formation (BTB, pers. obs.), and MMHF 5-00001 and MMHF 5-00002 are the only unambiguous records of tetrapods. Although no locality within the Nishinakayama Formation could be considered a *Konservat-Lagerstätte*, it is useful to compare its faunal assemblage with those of *Konservat-Lagerstätten* that also occur within the T-OAE where coeval marine communities are well preserved. Among these, the best known are the Strawberry Bank *Lagerstätte* (UK; Pierce and Benton, 2006; Williams et al., 2015), the Posidonia Shale *Lagerstätte* (Germany; e.g., Hauff, 1921; Williams et al., 2015), and the Ya Ha Tinda *Lagerstätte* (Canada; Martindale and Aberhan, 2017; Martindale et al., 2017; Maxwell and Martindale, 2017; Muscente et al., 2019). The faunal assemblage of the Nishinakayama Formation is largely comparable to these Toarcian *Lagerstätten* despite the relative scarcity of vertebrate fossils and the poor overall preservation of both invertebrate and vertebrate fossils.

### 5.3. Paleoenvironmental implications

Major and trace element data measured using pXRF can also serve as paleoenvironmental proxies (e.g., Calvert and Pedersen, 2007). Titanium and zirconium are proxies for coarse grain size, as terrigenous mineral phases rich in those elements (e.g.,  $ZrSiO_4$ ,  $TiO_2$ ) are typically transported with silt-to sand-sized siliciclastic grains (Calvert and Pedersen, 2007; Kemp and Izumi, 2014). Potassium and rubidium are typically enriched in clays. Thus, K/Ti and Rb/Zr can be used to track relative changes in grain size, and we use both here to characterize detrital flux changes through the Ishimachi section. Both proxies follow observed changes in facies throughout the Ishimachi section. There is a slight overall decrease in values reflecting a coarsening trend throughout the Em1 and IM1/K1 stratigraphic intervals in which some sandstone and tuff horizons are present between approximately 90 and 70 m. Above this, values are relatively consistent throughout E1' and E3, which are dominated by mudstones. Another coarsening trend characterized by the lowest observed ratios of both K/Ti and Rb/Zr occurs throughout the E4, E5, and E6 intervals where sandstone horizons are more abundant (Fig. 5), possibly indicating that these outcrops are from the "Nc member" of the Nishinakayama Formation in which sandstones are more common (Tanabe et al., 1982; Izumi et al., 2012). However, ammonoid fossils from the *Harpoceras inouyei* Zone are known from strata at site E4, whereas the "Nc member" is considered a "barren interval" for ammonoid fossils at Sakuraguchidani (Tanabe et al., 1982; Nakada and Matsuoka, 2011). We therefore interpret that the contact between the "Nb" and "Nc" members lies somewhere between the strata of E4 and E5. The Ishimachi section is thus characterized by generally increased grain size (although all measurements are from mudstones and siltstones) similar to

Sakuraguchidani (Izumi et al., 2012). The increase in grain size throughout the Ishimachi section could reflect falling sea level or simply increased terrestrial flux associated with increased weathering. Regardless, the depositional environment was relatively proximal. These interpretations are largely consistent with those of the upper Sakuraguchidani section, where the paleoenvironmental record of the Nishinakayama Formation before, during, and after the T-OAE interval has been thoroughly documented (Izumi et al., 2012, 2018a, 2018b; Kemp and Izumi, 2014; Kemp et al., 2019). At Sakuraguchidani, K/Ti and Rb/Zr also decrease throughout sandstone-rich intervals of the section.

## 6. Conclusions

We have presented a novel means of predicting the stratigraphic provenance of fossils collected *ex situ* that can be applied to any fossil preserved with matrix, and for which the general provenance is known or suspected. Although this method may have the broadest applications for fossils preserved on slabs of matrix such as MMHF 5-00001 and MMHF 5-00002, it could be applied to any 3-dimensionally preserved fossils such as vertebrate skulls if matrix has filled any cavities in or to unprepared field jackets from historical collections that have been separated from field locality data. This method could therefore also have applications in repatriating illegally collected fossils specimens.

Both MMHF 5-00001 and MMHF 5-00002 are interpreted to have originated from the Toarcian interval of the Pliensbachian-Toarcian Nishinakayama Formation in the river Eragawa. We have also presented additional evidence of the T-OAE within the Nishinakayama from a new locality in Toyotacho Ishimachi by correlating the  $\delta^{13}C_{org}$  profile of the Ishimachi section of the Nishinakayama Formation with that of the nearby Sakuraguchidani section based on  $\delta^{13}C_{org}$  data from each section. This provides additional support that the T-OAE was a global event that is now documented from multiple localities in western Panthalassa, albeit within the same formation. For a broader sample of localities in western Panthalassa, additional Lower Jurassic strata in Japan deposited on the eastern margins of the north and south China cratons and biostratigraphically correlated to the Pliensbachian-Toarcian stages should be targeted.

### Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper. Benjamin T. Breden III reports financial support was provided by the American Philosophical Society and the Geological Society of America.

### Data availability

Data are attached.

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SA 3.0 (<https://creativecommons.org/licenses/by-sa/3.0/>) licenses, respectively, and no changes were made to either. This is a contribution to IGCP 739.

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## Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.eve.2023.100004>.

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