Bull. Natl. Mus. Nat. Sci., Ser. C, 46, pp. 73–78, December 25, 2020

U–Pb age of the *Pravitoceras sigmoidale* Zone (upper Campanian, Cretaceous) in the Hidaka area, Hokkaido, northern Japan

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Abstract Radiometric age dating of zircons using the LA-ICP-MS method reveals that the U–Pb age of the tuff bed below the *Pravitoceras sigmoidale*-bearing bed in the Yezo Group in the Hidaka area, Hokkaido, northern Japan, is 72.4 ± 1.1 Ma (95% conf.), a span whose mean value infers a latest Campanian age. This result confirms the validity of previous biostratigraphic and magnetostratigraphic correlations. This age is nearly the same as that previously reported for the lower part of the *Didymoceras awajiense* Zone, suggesting that this zone may be of very short duration. Because the age of the Campanian/Maastrichtian boundary is 72.1 Ma, the time interval for the *D. awajiense*, *Pr. sigmoidale* and uppermost Campanian *Pachydiscus awajiensis* zones is estimated to be 0.3 million years.
Key words: Campanian, Cretaceous, *Pravitoceras sigmoidale* Zone, U–Pb age, zircon

Introduction

The Izumi Group is well known for its rich record of Campanian–Maastrichtian (Late Cretaceous) ammonoid faunas, and the following five upper Campanian ammonoid zones are recognized, in ascending order: *Baculites kotanii*, *Didymoceras* sp., *D. awajiense*, *Pravitoceras sigmoidale* and *Pachydiscus awajiensis* (Morozumi, 1985). However, these zones are characterized by endemic species restricted to Japan. The lack of more cosmopolitan taxa useful for international correlation and age assignment has long made it difficult to directly correlate these zones with other regions of the globe (Toshimitsu *et al.*, 1995).

Nevertheless, magnetostratigraphy and radiometric age determination of each zone have been shown to be effective methods for global correlation among the local zones. Kodama (1990) indicated that the *Baculites kotanii* and *Didymoceras* sp. zones correlate with the upper part of polarity chron C33n (= lower upper Campanian) and the *D. awajiense* through the *Pachydiscus awajiensis* zones correlate with the lower to middle parts of polarity chron C32.2n (= upper upper Campanian). The radiometric age of each zone has not been determined in the Izumi Group, but Shigeta *et al.* (2017) reported that the U–Pb age of the tuff bed in the lower part of the *D. awajiense* Zone of the Sotoizumi Group in the Aridagawa area, Wakayama, Southwest Japan, is 72.4 ± 0.8 Ma (95% conf.), a span whose mean value infers a latest Campanian age.

The late Campanian heteromorph ammonoid *Pravitoceras sigmoidale* Yabe, 1902 exhibits a unique shell morphology consisting of a nearly planispiral phragmocone and subsequent S-shape body chamber that forms a retroversal hook at the mature growth stage (Matsumoto *et al.*, 1981; Morozumi, 1985). The taxon had long been regarded as an endemic ammonoid restricted to the Izumi Group on Awaji Island and Naruto in Shikoku, Southwest Japan, but the discoveries of specimens assignable to this species from the Yezo Group in the Hidaka area, Hokkaido, northern Japan demonstrated that it was distributed throughout a relatively wide area of the northwestern Pacific realm (Matsunaga *et al.*, 2008; Shigeta and Izukura, 2018).

Recently, we discovered a tuff bed below the *Pravitoceras sigmoidale*-bearing bed in the Hidaka area, Hokkaido (Fig. 1). In this paper, we utilize zircon-based geochronology to calibrate the international correlation of the *Pr. sigmoidale* Zone in the Yezo Group, and establish a more precise global

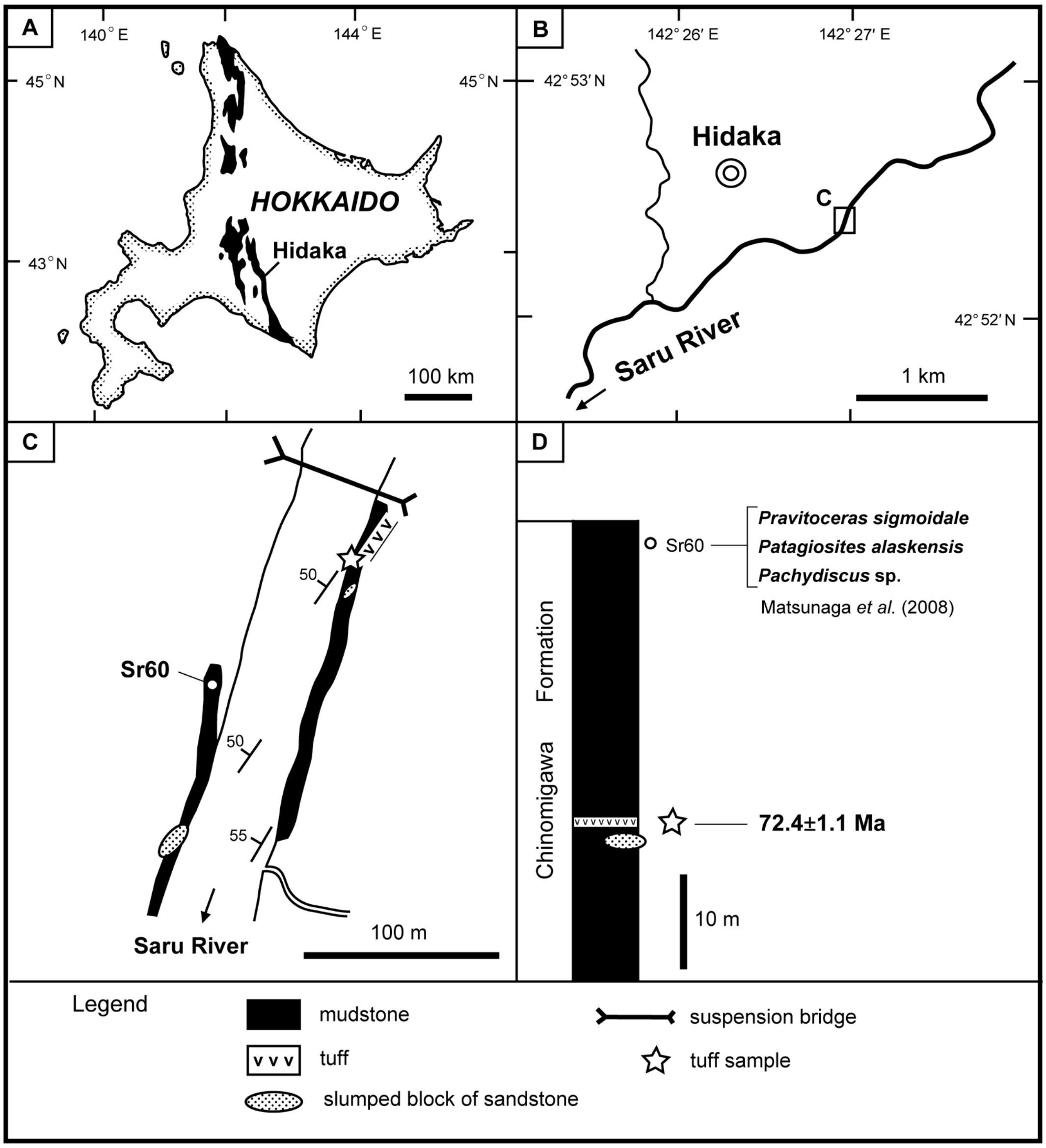

Fig. 1. Index map showing distribution of the Yezo Group (black areas) in Hokkaido (A), locality (B, C) and stratigraphic section (D) showing the occurrence of ammonoids and the position of the tuff bed that provided the U–Pb zircon age in the Chinomigawa Formation along the Saru River in the Hidaka, Hokkaido.

correlation of the zone.

Notes on stratigraphy

The Cretaceous Yezo Group is well exposed along the Saru River, southeast of Hidaka Town, south-central Hokkaido (Matsunaga *et al.*, 2008). Strata in the studied area, which strikes N30–40° eastward and dips 55–60° westward, consist mainly of dark grey, intensely bioturbated massive mudstone with thin white, vitric tuff and slumped blocks of sandstone (Fig. 1). The Yezo Group is lithologically similar to the Campanian Chinomigawa Formation in the Urakawa (Kanie, 1966; Sakai and Kanie, 1986) and Ribira River areas (Shigeta *et al.*, 2019). Matsunaga *et al.* (2008) reported various fossils from the Saru River section, including ammonoids (e.g. *Pravitoceras sigmoidale*, *Patagiosites alaskensis* Jones, 1963), inoceramids (e.g. *Inoceramus shikotanensis* Nagao and Matsumoto, 1940) and gastropods (e.g. *Gigantocapulus problematicus* (Nagao and Otatsume, 1938)).

Material and methods

Material: A tuff sample for zircon-based radiometric age analysis was taken from a 20 cm thick white, vitric tuff bed about 25 m below the *Pravitoceras sigmoidale*-bearing bed at Loc. Sr60 of Matsunaga *et al.* (2008) in an exposure on the left bank

Table 1. LA-ICP-MS analyzed data and calculated ages of zircons contained in the tuff sample from the Chinomigawa Formation. Errors are 1 sigma. Pb_c and Pb* indicate the common and radiogenic portions, respectively. "*" with label means the data are discordant.

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)
Saru_01	3.22	103	84	0.84	83.38 ± 4.40	0.0502 ± 0.0317	76.9 ± 4.0	76.5 ± 3.5
Saru_02	0.87	107	53	0.50	87.20 ± 3.82	0.0588 ± 0.0223	73.5 ± 3.2	72.4 ± 3.1
Saru_03	0.00	96	49	0.53	85.00 ± 4.18	0.0479 ± 0.0135	75.4 ± 3.7	75.3 ± 3.9
Saru_04	1.69	97	64	0.67	85.93 ± 4.31	0.0232 ± 0.0259	74.6 ± 3.7	75.8 ± 3.3
Saru_05	0.62	82	40	0.50	90.93 ± 5.10	0.0222 ± 0.0328	70.5 ± 3.9	70.9 ± 3.4
Saru_06	2.09	99	61	0.63	94.51 ± 5.94	0.0369 ± 0.0351	67.9 ± 4.2	68.7 ± 4.4
Saru_07	1.29	148	84	0.59	95.51 ± 4.46	0.0416 ± 0.0236	67.1 ± 3.1	67.6 ± 3.0
Saru_08	6.31	104	49	0.48	88.48 ± 4.99	0.0371 ± 0.0363	72.4 ± 4.1	73.4 ± 4.0
Saru_09	1.53	83	45	0.56	90.46 ± 5.19	0.0504 ± 0.0362	70.9 ± 4.0	70.6 ± 3.6
Saru_10	0.00	136	91	0.69	92.98 ± 3.57	0.0396 ± 0.0116	69.0 ± 2.6	69.0 ± 2.6
Saru_11	0.30	135	64	0.49	96.19 ± 4.50	0.0452 ± 0.0228	66.7 ± 3.1	66.8 ± 2.9
Saru_12	0.31	301	205	0.70	89.17 ± 2.36	0.0513 ± 0.0115	71.9 ± 1.9	71.6 ± 1.8
Saru_13	0.86	107	49	0.47	91.80 ± 4.25	0.0414 ± 0.0234	69.8 ± 3.2	70.4 ± 3.0
Saru_14	1.46	142	78	0.57	92.64 ± 3.96	0.0522 ± 0.0218	69.2 ± 2.9	68.8 ± 2.7
Saru_15	0.99	159	83	0.53	82.52 ± 3.15	0.0421 ± 0.0168	77.6 ± 2.9	78.2 ± 2.8
Saru_16	0.24	371	193	0.53	92.24 ± 2.47	0.0500 ± 0.0094	69.5 ± 1.9	69.3 ± 1.8
Saru_17	0.00	230	138	0.61	88.45 ± 2.89	0.0423 ± 0.0061	72.5 ± 2.4	72.5 ± 2.4
Saru_18	0.40	123	85	0.71	80.86 ± 3.57	0.0463 ± 0.0202	79.2 ± 3.5	79.4 ± 3.3
Saru_19	2.20	128	97	0.78	86.88 ± 4.11	0.0392 ± 0.0219	73.8 ± 3.5	74.6 ± 3.4
Saru_20	1.48	146	79	0.55	90.73 ± 3.86	0.0350 ± 0.0199	70.7 ± 3.0	71.7 ± 2.8
Saru_21	0.00	91	50	0.56	88.12 ± 4.56	0.0494 ± 0.0226	72.7 ± 3.7	72.6 ± 4.3
Saru_22	0.06	125	76	0.62	82.85 ± 4.08	0.0499 ± 0.0228	77.3 ± 3.8	77.1 ± 3.6
Saru_23	0.00	100	48	0.49	80.09 ± 3.36	0.0646 ± 0.0153	80.0 ± 3.3	78.3 ± 3.6
Saru_24	0.00	211	108	0.52	85.20 ± 2.66	0.0506 ± 0.0071	75.2 ± 2.3	74.9 ± 2.4
Saru_25	2.79	123	100	0.83	94.91 ± 4.62	0.0349 ± 0.0279	67.6 ± 3.3	68.6 ± 3.0
Saru_26	3.22	195	181	0.95	88.63 ± 3.49	0.0224 ± 0.0203	72.3 ± 2.8	74.6 ± 2.7
Saru_27	0.87	120	65	0.55	93.42 ± 3.86	0.0344 ± 0.0198	68.6 ± 2.8	69.2 ± 2.5
Saru_28	1.53	135	73	0.56	92.54 ± 4.01	0.0588 ± 0.0233	69.3 ± 3.0	68.3 ± 2.9
Saru_29	3.55	170	102	0.62	94.24 ± 3.69	0.0482 ± 0.0251	68.0 ± 2.6	68.0 ± 2.6
Saru_30	0.00	84	45	0.55	83.09 ± 3.80	0.0169 ± 0.0187	77.1 ± 3.5	77.2 ± 3.5
Saru_31	2.93	80	36	0.46	94.16 ± 6.11	0.0410 ± 0.0462	68.1 ± 4.4	68.7 ± 4.3
Saru_32	1.31	83	57	0.71	79.30 ± 5.51	0.0547 ± 0.0463	80.8 ± 5.6	80.1 ± 4.4
Saru_33	3.26	91	48	0.54	85.71 ± 5.04	0.0425 ± 0.0444	74.8 ± 4.4	75.3 ± 4.4
Saru_34	2.96	103	51	0.51	93.81 ± 4.94	0.0385 ± 0.0277	68.4 ± 3.6	69.2 ± 3.7
Saru_35	0.39	309	215	0.71	90.45 ± 2.32	0.0437 ± 0.0107	70.9 ± 1.8	71.2 ± 1.7
Saru_36	0.00	134	76	0.59	83.41 ± 3.07	0.0427 ± 0.0109	76.8 ± 2.8	76.9 ± 2.8
Saru_37	0.03	116	58	0.51	89.87 ± 3.89	0.0320 ± 0.0211	71.3 ± 3.1	71.4 ± 2.7
Saru_38	3.61	113	62	0.56	96.65 ± 4.26	0.0381 ± 0.0265	66.4 ± 2.9	67.2 ± 2.8

(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

of the Saru River (42°52′27.70″N, 142°26′59.09″E; Fig. 1).

Methods: Zircon grains were extracted by standard techniques: crushing, heavy liquid separation and handpicking. Then, the zircon grains were mounted in an epoxy disc with the FC1 zircon standard ($^{206}Pb/^{238}U = 0.1859$; Paces and Miller, 1993) and NIST SRM610 glass standard, and polished until the center of each grain was exposed. The backscattered electron and cathodoluminescence images of the zircon grains were used for selection of the analyzing spots. U–Pb dating of the sample was carried out using the LA-ICP-MS method, which consists of a NWR213, a 213 nm wave length Nd-YAG laser ablation system (Elemental Scientific Lasers) 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 approx. 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). For this paper, we have adopted 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

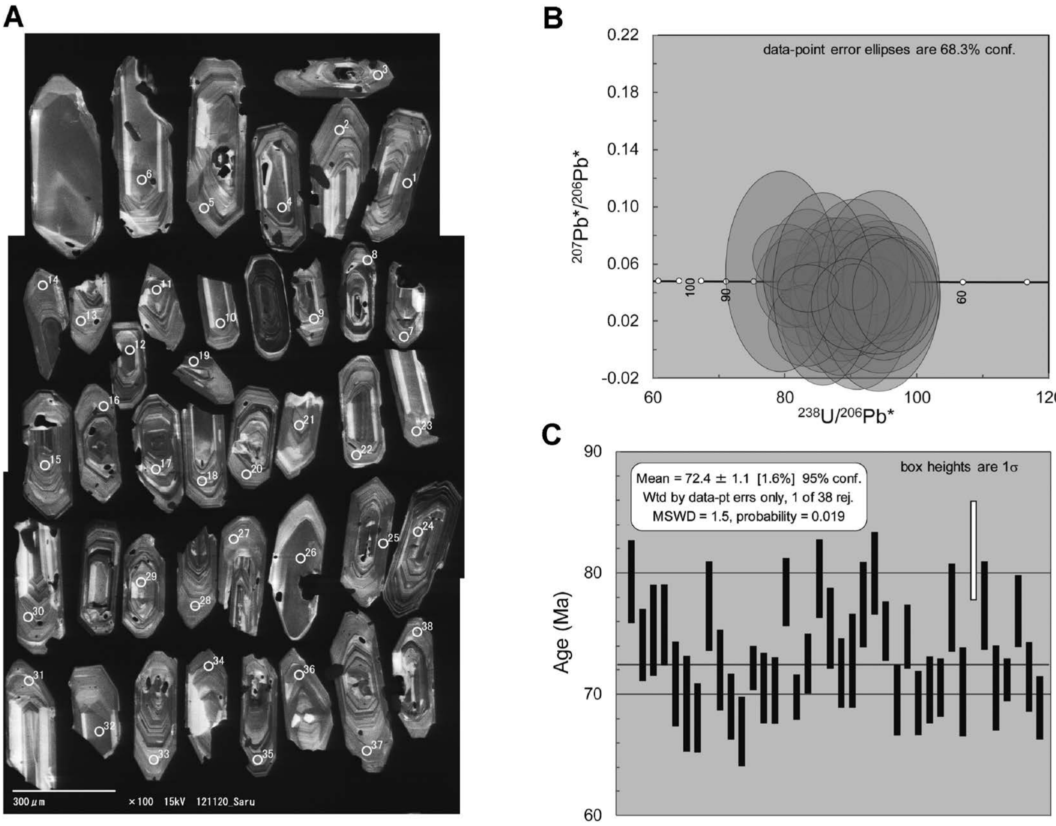

Fig. 2. U–Pb ages of zircon grains contained in a tuff sample collected from the Chinimigawa Formation. A, cathodoluminescence image (CL) of zircon grains from the sample. Circles on the grains represent spots analyzed by LA-ICP-MS. Spots are 25 μm across. B, Tera-Wasserburg U–Pb concordia diagram of zircons. C, ^{238}U–^{206}Pb age distribution plot of zircon grains in the tuff sample. ^{207}Pb* and ^{206}Pb* indicate radiometric ^{207}Pb and ^{206}Pb, respectively.

correction (e.g. Williams, 1998). The weighted mean age in this study is calculated using Isoplot/Ex software (Ludwig, 2012). 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 the zircons, including common ^{206}Pb content rate in total 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 level. All zircons in the samples show rhythmic oscillatory and/or sector zoning on cathodoluminescence images, 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). Figure 2 shows a Tera-Wasserberg concordia diagram and an age distribution plot for all analyzed spots of the sample. Data are obtained from 38 spots and all data are concordant. U–Pb ages cluster in the range 66.8–80.1 Ma. The weighted mean age of the data yield 72.4 ± 1.1 Ma (1 rejected; MSWD = 1.5; 95% conf.), which is thought to represent the magmatism/deposition age of the tuff sample.

Discussion

Morozumi (1985) reported the co-occurrence of *Solenoceras* cf. *texanum* (Shumard, 1861) and *Pravitoceras sigmoidale* on Awaji Island. Because *Solenoceras texanum* mainly occurs in the upper Campanian (Lewy, 1969; Klinger, 1976), he correlated the *Pr. sigmoidale* Zone with the upper part of the upper Campanian. This correlation was supported by a detailed magnetostratigraphic investigation by Kodama (1990), which indicated that the *Pr. sigmoidale* Zone correlates with the middle part of polarity chron C32.2n (= upper upper Campanian). Zircon geochronology in this study reveals that the U–Pb age of the tuff below the *Pr. sigmoidale*-bearing bed in Hokkaido is 72.4 ± 1.1 Ma (95% conf.), a span whose mean value infers a latest Campanian age, and thus confirms the validity of previous biostratigraphic and magnetostratigrahic correlations.

The *Didymoceras awajiense* Zone is established just below the *Pravitoceras sigmoidale* Zone on Awaji Island (Morozumi, 1985). Recently, Shigeta *et al.* (2017) reported that that the U–Pb age of the tuff in the lower part of the *D. awajiense* Zone in the Aridagawa area, Wakayama, Southwest Japan, is 72.4 ± 0.8 Ma (95% conf.), a span whose mean value infers a latest Campanian age. This value is almost the same as the age of the tuff below the *Pr. sigmoidale*-bearing bed of this study. Fragments assignable to *D. awajiense* were found below this tuff bed along the Saru River in the Hidaka area, Hokkaido (Shigeta unpublish data). This evidence suggests that the *D. awajiense* Zone may be of very short duration. The *Pachydiscus awajiensis* Zone, which is established just above the *Pr. sigmoidale* Zone on Awaji Island, has been correlated with the uppermost Campanian (Morozumi, 1985; Matsumoto *et al.*, 1986; Matsumoto and Morozumi, 1988; Kodama, 1990). Since the age of the Campanian/Maastrichtian boundary is 72.1 Ma (Ogg *et al.*, 2016), the interval of time for the *D. awajiense*, *Pr. sigmoidale* and *Pachydiscus awajiensis* zones is estimated to be 0.3 million years.

Acknowledgments

We are very grateful to Masataka Izukura (Sapporo, Hokkaido) and Tomohiro Nishimura (Hobestu Museum, Hokkaido) for their co-operation in the field. We thank Kentaro Nakada (Fukui Prefectural Dinosaur Museum, Katsuyama) for valuable comments on the first draft. Thanks are extended to Jim Jenks (West Jordan, Utah) for his helpful suggestions and improvement of the English text. This research was financially supported by the National Museum of Nature and Science project, Chemical Stratigraphy and Dating as a Clue for Understanding the History of the Earth and Life.

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