J Radioanal Nucl Chem (2010) 284:211–219
DOI 10.1007/s10967-010-0472-1

Neutron activation analysis in geochemical characterization of Jurassic–Cretaceous sedimentary rocks from the Nordvik Peninsula

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Received: 27 January 2010 / Published online: 21 February 2010

Abstract As a part of geophysical, stratigraphical and paleontological study aimed at precise correlation of the Jurassic–Cretaceous (J/K) boundary interval in the Tethyan and Boreal Realms, detailed magnetostratigraphic and biostratigraphic profiles with well-calibrated J/K boundary have been selected from several localities for geochemical characterization. Instrumental and radiochemical neutron activation analyses (INAA and RNAA, respectively) were employed in the characterization of a vertical profile around the supposed J/K boundary in the Boreal Realm situated on the Nordvik Peninsula, Northern coast of Middle Siberia (Laptev Sea), Russia, for which an iridium anomaly has been reported in literature. INAA enabled determination of about forty major and trace elements. An RNAA procedure for assaying platinum group elements (PGE) has been tested consisting in sample decomposition by alkaline–oxidative fusion, reduction of PGE, and precipitation of their sulphides. Despite several difficulties, the procedure enabled to found Pt and Ir at ppb and sub-ppb levels, respectively, in several samples, namely in pyrite aggregates originated probably in diagenetic reductive processes following decomposition of burried organic matter. However, the existence of a pronounced PGE spike reported previously for the J/K boundary on the Nordvik Peninsula has not been confirmed.

Keywords Jurassic–Cretaceous boundary · Nordvik Peninsula · Iridium anomaly · INAA · RNAA

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Introduction

Biostratigraphies of sedimentary formations on the boundary of several geological stages characterized by distinct decrease in both marine and terrestrial biodiversity may be indicators of global climatic changes. These changes can be connected with large meteorite impacts or strong volcanic activity, which are, among others, indicated by significantly elevated levels of platinum group elements (PGE), particularly iridium, in related sediment beds [1]. In 1980s, Alvarez et al. [2–4] presented a hypothesis of extraterrestrial origin of the iridium anomalies ("spikes") discovered in deep-sea carbonate sediments on the Cretaceous-Tertiary (K/T) boundary (65.5 Ma), and their connection with global mass biotic extinction. In following years, iridium anomalies were reported from several other places worldwide also from sedimentary formations on the border between other geological stages (for a list see [5, 6], e.g.). Extremely high concentration of PGE have been found also for the Jurassic–Cretaceous boundary strata (J/K, 145.5±4.0 Ma) in sedimentary rocks on the Nordvik Peninsula, Northern coast of Middle Siberia (Laptev Sea), Russia (Fig. 1) in a 4–6 cm thick layer of phosphatized limestone deposited in a sedimentary profile of argillites (lithified silty-clayey sediments) (Fig. 2). This horizon shows a lithologic stability from the Nordvik Peninsula in the east to the Barents Sea in the west, and is supposed to

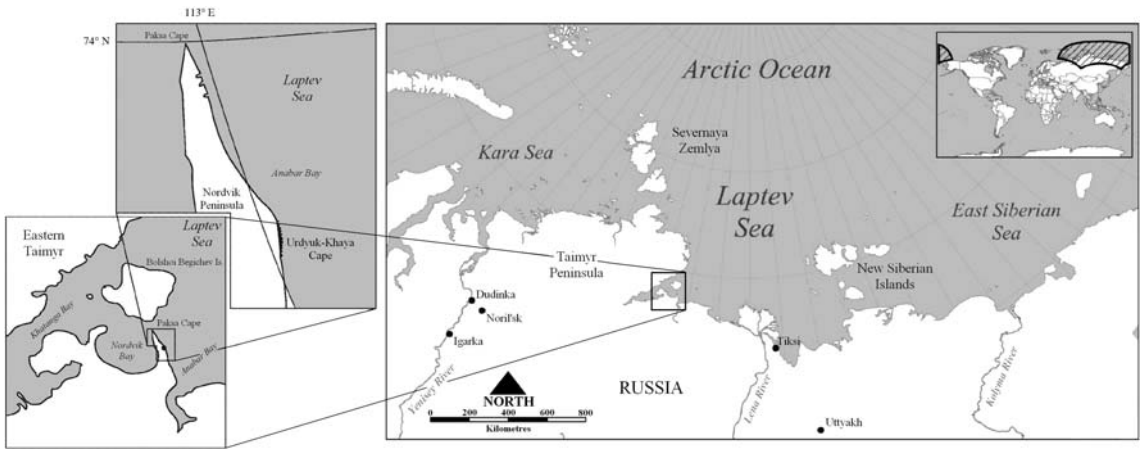

Fig. 1 Location of the studied sedimentary profile around the supposed J/K boundary on the Urdyuk-Khaya Cape, Nordvik Peninsula, west coast of the Anabar Bay in the Laptev Sea

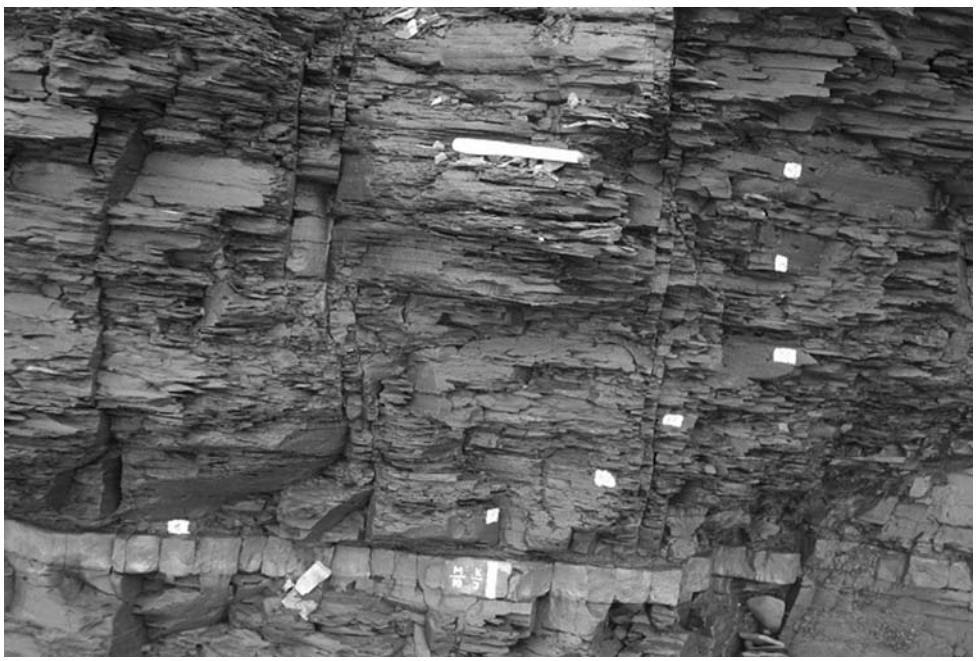

Fig. 2 Vertical profile of sedimentary rocks around the supposed J/K boundary (the distinctly lighter, ~4 cm thick interlayer of phosphatized limestone) on the Nordvik Peninsula

be a key sequence boundary located very close to the Boreal J/K boundary interval [7]. It originated during the maximum eustatic rise and represents the highstand systems tract in relation to the maximum flooding surface. Occurrence of the PGE anomaly has been explained by the presence of diagenetically sulphidized iron particles of cosmic dust deposited under conditions of drastically reduced clastic sedimentation rate [5]. Some authors have associated it with the isochronous marine impact Mjølnir in the Barents Sea [6]. This impact structure (~40 km diameter) lies 2500 km west of Nordvik, but its traces have been permitted even 3000 km away in possible tsunami deposits in northern France [8]. To the J/K boundary, also the Morokweng crater in South Africa (70–80 km diameter) is dated [9].

The J/K boundary was actually not connected with a major extinction in the Earth history. Less than 20% of biota became extinct during the Tithonian stage (uppermost Jurassic) and only $<5\%$ during the following Berriasian stage (lowest Cretaceous), compared to the ca. 45% K/T mass extinction. This rather suggests a continual biotic turnover caused partly by paleoceanographic changes and large paleogeographic events (i.e., opening of Middle Atlantic and connection of new migration ways) [1, 10, 11]. In the North-Siberian paleogeographical province the transition between Jurassic and Cretaceous was connected

with neither extinction nor reorganization in marine biota [5]. This has been supported by faunal analysis indicating an increase in ammonite diversity [12]. The belemnite diversity change has been recorded as well, and it shows partial decrease [13]. This fully corresponds with the above mentioned eustatic event, basin deepening and belemnite (as shallower water inhabitants) disappearing. However, on a global scale, the tectonic movements at the end of Jurassic initiated physicochemical, climatic, and temporarily biological differentiation of the Boreal (northern, cold) and Tethyan (southern, warm) provinces [14]. The faunistic barriers were strict and almost no Boreal–Tethyan faunal exchanges were observed. Resulting differences in the taxonomic composition especially in marine biota between both provinces complicate reliable determination and biostratigraphic correlation of a global J/K boundary. For a detailed and precise correlation of the J/K boundary interval in the Tethyan and Boreal regions based on high-resolution magnetostratigraphic, micro- and macropaleontological, and geochemical data, two key profiles in the Tethyan Realm (Brodno, Slovakia, and Puerto Escaño, Spain) have been selected besides the Nordvik profile in the Boreal Realm. The J/K boundary interval on the Nordvik Peninsula has been established basically by the presence of two important magneto-subzones, i.e., Kysuca (M20n) and Brodno (M19n) [7].

The present study has been aimed at geochemical characterization of sedimentary rock samples from the Urdyuk-Khaya Cape on the Nordvik Peninsula collected from the layer with the expected PGE anomaly (as reported in [5]) and its close under- and overlying beds. Geochemical characterization of limestone samples from the Brodno section based on neutron and photon activation analyses has been published recently [15], and analyses of the limestone samples from Puerto Escaño are underway in our laboratory.

Experimental

Samples were taken from the phosphatized limestone bed ("0" bed) and argillite beds 49 cm below and 55 cm above. Samples were crushed with an iron jaw crusher, milled and homogenized in a disc agate mill, pressed into pellets, and sealed in polyethylene capsules. These were irradiated with neutrons in the LVR-15 research nuclear reactor of the Nuclear Research Institute Řež plc. The multimode instrumental neutron activation analysis included short and long-time irradiation modes (ST and LT, respectively) with either reactor-pile neutrons, or with epithermal/fast neutrons under a Cd shielding (INAA and ENAA, respectively). For quality control, the USGS reference materials W-1 (diabase) and BCR-1 (basalt) were used. For group determination of Ir and other PGE, a radiochemical separation procedure (RNAA) has been designed and tested.

As calibration standards for data evaluation by the relative method, synthetic multielement standards (MES) prepared from pure elements or their stoichiometric compounds by pipetting their solutions on paper discs were used. In the ST mode, each sample or calibration standard was irradiated individually. In the LT mode, all samples and standards were irradiated together with multielement monitors of neutron flux gradient (Fe–Co–Mo foil) in a single irradiation can. In the ENAA mode, a special can with Cd lining (1 mm) was used. The LT-ENAA has been chosen mainly to explore possibility of instrumental determination of Ir via the reaction ^{191}Ir (n,γ)^{192}Ir, which should provide much higher sensitivity in the ENAA mode than in the INAA mode thanks to an extremely high value of the resonance integral (I_0 = 4500 b, σ_0 = 624 b [16]).

Gamma spectra of irradiated samples/standards were acquired using various coaxial HPGe detectors with standard parameters (20–53% relative efficiency, FWHM ~1.8 keV at 1332.5 keV) coupled to a Canberra Genie 2000 gamma-spectrometric system. Irradiation parameters and typical decay and counting times applied in the individual INAA analytical modes are presented in Table 1.

A radiochemical separation procedure has been employed to assay PGE in samples from selected beds of the profile and samples of pyrite concentrates (see below). The samples (bulk rocks ca. 250 mg, pyrites ca. 50 mg) were irradiated for 2 h with reactor-pile neutrons together with Ru, Pd, Os, Ir, and Pt standards prepared by pipetting diluted standard calibration solutions Astasol® (Czech Metrological Institute) on paper discs. After 4–7 day cooling, the samples were decomposed by alkaline–oxidative fusion with 2.5 g Na_2O_2 + 0.5 g NaOH + 0.5 g NaCl in the presence of Ru, Pd, Ir, and Au carriers (á 25 μg) at 900 °C. The melt, partially cooled down, was dissolved in a minimum volume of diluted HCl. The dissolved sample was boiled in concentrated HCl with Zn powder several times for PGE reduction and partial removal of As and Sb. After complete dissolution of the added Zn, dilution, addition of collectors (4 mg Cu and 10 mg Bi as nitrates) and heating, PGE/Au sulphides were coprecipitated with the collectors by 10% thioacetamide. The precipitate was filtered or centrifuged, dried and counted for 12 h on a 23% coaxial HPGe detector. Nuclear parameters of the PGE analytical radionuclides utilized in RNAA and gamma lines of interfering radionuclides are listed in Table 2. For quality control, the CCRMP (Canadian Certified Reference Materials Project) reference materials WMG-1 and UMT-1 with certified PGE contents were used.

Table 1 Parameters of the individual analytical modes of INAA used for analysis of sedimentary rocks from the Nordvik Peninsula

Analytical mode	Description	Sample weight	Irradiation–decay–counting times	Neutron fluence rate (cm^{-2} s^{-1})	Typically determined elements
ST-INAA	Short time activation with reactor-pile neutrons	~30 mg	1 min 12 min 12 min	3×10^{13} (thermal)	Na, K, Mg, Ca, Ba, Al, Ti, V, Mn, Dy, U
ST-ENAA	Short time activation with epithermal and fast neutrons (in Cd shielding)	~80 mg	45 s 15 min 15 min	5×10^{12} (fast)	Ba, Al, Si, I, V, U
LT-ENAA, 1st count	Long time activation with epithermal and fast neutrons (in Cd shielding)	250–300 mg	5 h 5 days 20 min		Ga, As, Br, Cd, W, Au, La, Sm, Lu, U
LT-ENAA, 2nd count			5 h 13 days 45 min	8×10^{13} (thermal) 2×10^{13} (fast)	Rb, Cs, Sr, Ba, Sb, Se, Sc, Cr, Fe, Co, Ni, Zn, Zr, Ag, Hf, Ta, Au, La, Ce, Nd, Sm,Tb, Tm, Yb, Lu, Th, U
LT-ENAA, 3rd (to 5th) count			5 h 28 (−80) days 1 h		Rb, Cs, Sr, Ba, Sb, Se, Sc, Cr, Fe, Co, Ni, Zn, Zr, Hf, Ta, Hg, Ce, Nd, Eu,Tb, Tm, Yb, Th

Table 2 Nuclear parameters of the analytical radionuclides of Pt group elements used in RNAA, and gamma lines of eventual interfering radionuclides [16]

Element	Analytical radionuclide	$T_{½}$	E_{γ} (keV)	Interference
Pt	^{197}Pt	18.3 h	77.3; 191.3	
	(^{199}Pt→) ^{199}Au	75.6 h	158.4; 208.2	^{197}Au (2n, γ) ^{199}Au; 159.4 (^{47}Sc); 208.2 (^{177}Lu); 209.75 (^{239}Np)
Ir	^{192}Ir	74.0 days	316.5; 308.4; 468.1	315.9 (^{239}Np)
Os	^{191}Os	15.4 days	129.4	
Ru	^{97}Ru	69.1 h	215.2	
	^{103}Ru	39.3 days	497.1	^{103}Ru from U fission; 496.3 (^{131}Ba)
Pd	^{109}Pd	13.5 h	88.1	

Results and discussion

Distribution of elements in a single vertical profile of sedimentary rocks around the supposed J/K boundary on the Nordvik Peninsula, which was determined by the INAA methods, is illustrated by Figs. 3 and 4. Significant geochemical difference between the "0" bed of the phosphatized limestone (41–48 wt% CaO, 17–28 wt% P_2O_5 according to [5]) and surrounding layers of clayey deposits is obvious. The "0" bed has much higher contents of Ca, Sr, Ba, and Mn than the neighbouring layers. On the other hand, Al, Si, and the elements typically associated with phyllosilicate (clay) minerals, as Fe, Ti, V, Hf, Rb, Cs, Mg, Th, etc., are depleted in the "0" bed, except for U and the rare earth elements (REE), which are significantly enriched. A significant difference between the profiles below and above the "0" bed can be observed. In the upper layers element concentrations vary slightly or mainly increase with increasing distance from the "0" bed, whereas in the lower part of the profile the concentrations pass through several extremes. Pronounced maxima occur for siderophile and chalcophile elements (here, e.g., Mn, Fe, Ni, Au, Zn, As, Sb, Se, Ag, Cd). They are often quite sharp, as in case of Au at the bed −32 cm, and are positioned roughly at each 10 cm of the profile, although there are slight variations (±2 cm) between individual elements.

Phosphatization of limestones, often accompanied by pyrite formation, is mostly diagenetic (postsedimentary) process connected with suboxic to anoxic conditions [17, 18]. With the highest probability, the pyrite abundance is connected to the burried organic matter coming from both benthic and planktonic (partly nekto-benthic and nektonic) biota. The high organic matter concentration has been evidenced also by occurrence of oil inclusions in

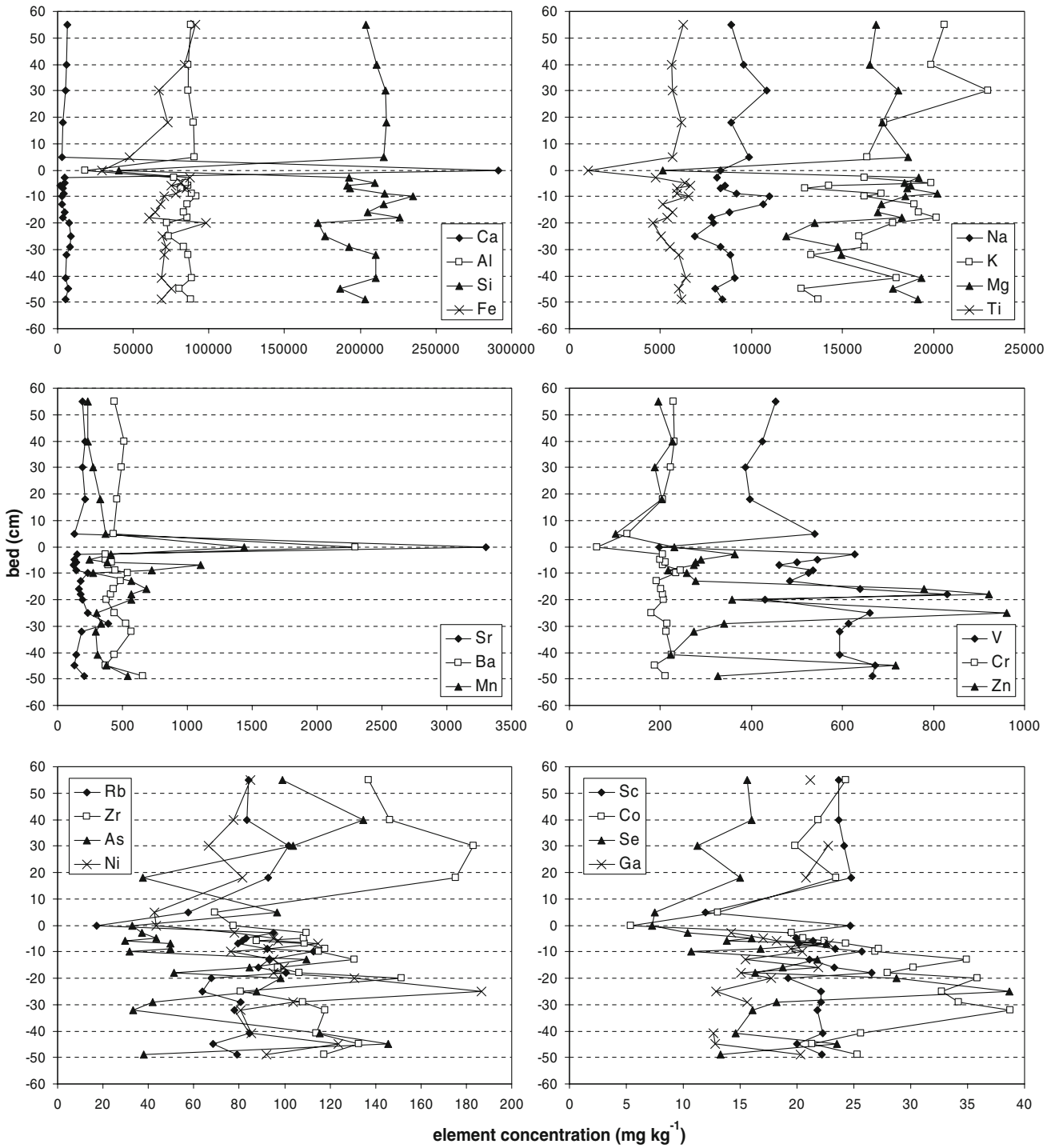

Fig. 3 Distribution of elements (concentration in mg kg^{-1}) in the vertical profile of sedimentary rocks around the supposed J/K boundary on the Nordvik Peninsula

some belemnite rostra. Some pyritized beds on the Nordvik section are generally supposed to be an equivalent of the oil-bearing "Bazhenov horizon" in the Central-West Siberia, where the uranium mineralization is characterized as an infiltration genesis type [19]. This could elucidate also the immobilization of U in the "0" bed from the highly soluble U(VI) to less soluble U(IV). The enrichment in REE in the "0" bed seems to be accompanied by their depletion in the neighbouring layers. This may indicate their postsedimentary migration and immobilization in the layer of biogenic phosphates. As the biogenic phosphate (skeletal apatite) is virtually free from REE, they probably

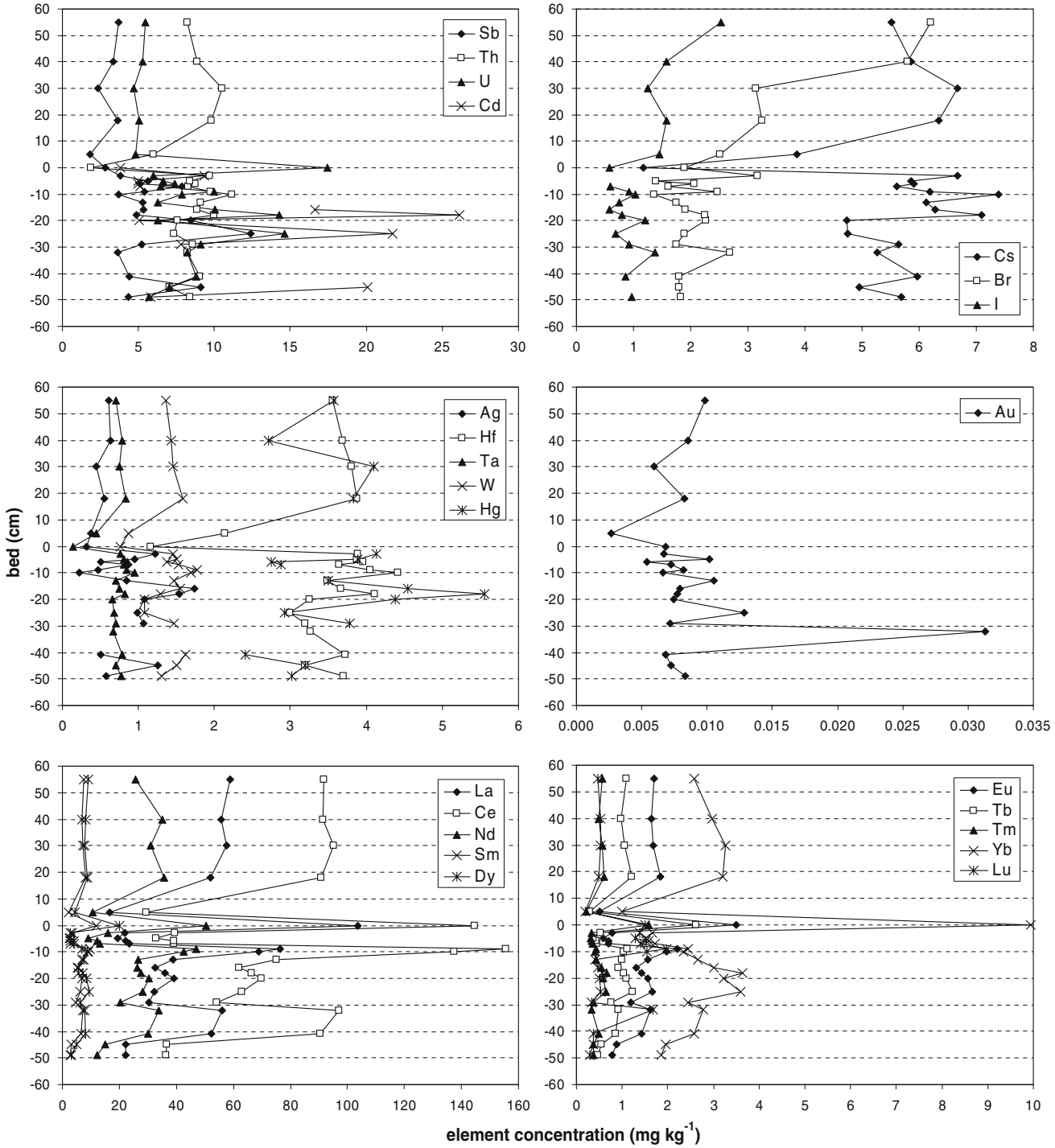

Fig. 4 Distribution of elements (concentration in mg kg^{-1}) in the vertical profile of sedimentary rocks around the supposed J/K boundary on the Nordvik Peninsula

precipitated on fish scales (vertebrate bones, crustaceans, etc.) from sea water during diagenesis. The REE pattern observed in the "0" bed shows a distinct negative Ce anomaly (see Fig. 5) pointing also to their origin in oxic sea water, thus the anoxic processes must have occurred in the sediment later during its diagenesis [20].

Dispersed sulphides can be found throughout the entire Nordvik profile. Pyrite microaggregates in the form of nodules and globules of a diameter about 50 μm have been separated from the layer of phosphatized limestone by Zakharov et al. [5]. In our samples, pyrites can be found in argillitic rock mainly as edging and filling of fossils

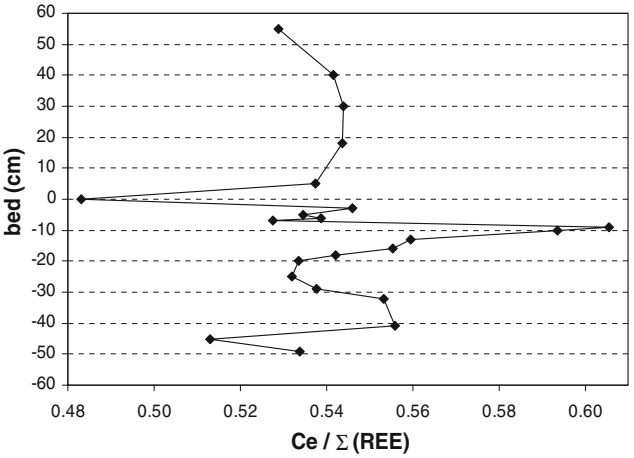

Fig. 5 The ratio of Ce content to the sum of contents of all REE determined throughout the vertical profile of sedimentary rocks around the supposed J/K boundary on the Nordvik Peninsula, illustrating a negative Ce anomaly in the layer of phosphatized limestone ("0" bed)

(belemnites—diagenetic replacement of calcitic, aragonitic and especially organic layers in the rostrum by pyrite) and their casts. Pyrite concentrates for RNAA of PGE were obtained by hand picking and flotation following calcite removal by HCl, however, only from the layers quite far from the profile analyzed by INAA (~3–7 m above and 1.5–4.5 m below the "0" bed).

Nor Ir neither any other PGE have been determined in the LT-ENAA mode above detection limit at a μg kg^{-1} level, even after 80 days of cooling. That is why the above RNAA procedure was designed and tested. With respect to the siderophile and chalcophile character of PGE and the fact that the most anomalous PGE levels on Nordvik are associated with pyrite microaggregates [5], besides the "0" bed sample also samples with increased levels of siderophile/chalcophile elements (Fe, Co, Ni, Sb, Se, etc.) from the underlying beds −7, −20, and −25 cm were taken for the analysis.

Unfortunately, the suggested RNAA procedure based on sulphide precipitation turned out to suffer from several shortcomings. The main flaw is its low selectivity. As sulphides, also cations of the 2nd analytical group are precipitated in acidic medium, from which mainly As, Sb and Cu contribute significantly to the Compton continuum enhancement. An attempt to remove As and Sb quantitatively by their reduction with nascent hydrogen into gaseous arsan/stiban (principle of the famous Marsh test) turned out to be quite insufficient. Several other elements (co-)precipitate or adsorb, e.g., Se, Sc, Mo, some REE and actinides. Their radionuclides produced by neutron capture or fission can, besides the Compton continuum contribution, interfere spectrally with the PGE analytical radionuclides (see Table 2). Determination of Ru is complicated by the presence of U, as the analytical radionuclide ^{103}Ru is produced also by fission with a high fission yield. On the other hand, in the case of Pt assay, the interference of ^{199}Au produced by double neutron capture from ^{197}Au is negligible with the irradiation conditions employed and relatively low Au content in the samples. Finally, Ir determination is obviously complicated by an insufficient separation yield, as evidenced by a tracer experiment with the reference material WMG-1 (yields: Pt 93%, Os 82%, Ru 81%, Ir 15%). Due to lack of a suitable Ir tracer different from the analytical radionuclide, the dubious yield precludes using this procedure for preactivation separation of PGE, more favourable for Ru determination thanks to U removal prior to irradiation.

Despite several analytical difficulties, the procedure enabled to find Pt and Ir at ppb and sub-ppb levels, respectively, in two of the samples (see Table 3). For comparison, the values reported by Zakharov et al. [5] in the phosphatized limestone layer and in the pyrite microaggregates separated from it are included. It is obvious from the quality control results that the values presented in Table 3 are of information character only. They approach a detection limit of the method and a faithful estimation of

Table 3 Results of RNAA of Pt group elements in two reference materials and selected Nordvik samples (in μg kg^{-1})

Sample	Pt	Ir	Os	Ru
CCRMP WMG-1 (Certified Reference Material for Mineralized Gabbro with Au and PGE)	820 (731 ± 35)[a]	14 (46.4 ± 4.1)[a]	20 (24.1 ± 3.5)[a]	118 (34.7 ± 5.1)[a]
CCRMP UMT-1 (Ultramafic Ore Tailings PGE Reference Material)	773 (129 ± 5)[a]	4.5 (8.8 ± 0.6)[a]	<4.1 (8.0)[a]	114 (10.9 ± 1.5)[a]
Nordvik—present values determined by RNAA				
Bed −20 cm, sediment	3.8	~10^{-2}	–	–
Bed 670 cm, pyrite concentrate	20	0.04		
Nordvik—published values [5]				
Phosphatized limestone ("0" bed)	3.1–16	0.05–75.0	–	–
Pyritic microaggregates from this bed	–	230–1200		

[a] Value given by the certificate

their uncertainty cannot be provided. The RNAA results indicate that the samples from a single vertical profile of sedimentary rocks around the supposed J/K boundary on the Nordvik Peninsula have not significantly elevated contents of PGE, and cannot support the reported existence of a PGE anomaly in this area. The Pt and Ir contents determined above the detection limit of RNAA do not exceed their background levels in the upper and even lower Earth's crust [21], and by far cannot be considered as a PGE spike. They are close to the bottom of the wide range of the Zakharov's values [5] and may thus represent background values for the profile, although Pt and Ir have not been evidenced in the "0" bed, but in a lower bed characterized by an increased Fe content. In agreement with the finding of Zakharov et al. [5], the elevated PGE contents must be associated with pyrites, as indicated by a significantly higher Pt content found in the pyrite aggregates formed near fossils. However, in our samples these aggregates have been found only in the beds up to several meters distant from the supposed J/K boundary and from each other. This obviously precludes their connection with a narrowly time limited event like a meteoritic impact dated to the transition between Jurassic and Cretaceous. With respect to the above discussed geochemical data for other elements (U, REE), it rather points to a connection with long-term diagenetic concentrating of PGE in the pyrite aggregates by other, probably biogeochemical redox process. Although Zakharov et al. [5] have not permitted correlation of the Nordvik PGE anomaly with biogenic processes due to a monotonous distribution of organic carbon along the J/K boundary, the association of synchronous PGE accumulation and formation of pyrites and phosphates with the decomposition of organic matter and possible reductive bacterial or algal activity has been suggested for other marine sediments ([17, 18, 20], e.g.). This interpretation is consistent with that applied in explanation of nickel and antimony anomalies, which we have found in the Brodno limestones near the beds assigned to the J/K boundary [15]. Yet, such an explanation does not necessarily preclude that the PGE spikes in the Nordvik sedimentary rocks had a distant source of extraterrestrial (e.g., Mjølnir) or volcanic origin, which supplied PGE for their subsequent concentration by the above processes [20].

Conclusion

Multimode INAA of a ca. 1 m vertical profile around the supposed J/K boundary in the Boreal Realm situated on the Nordvik Peninsula has shown a sharp geochemical distinction between the central layer of phosphatized limestone ("0" bed) and the surrounding argillite beds. The "0" bed is rich in Ca, Sr, Ba, and Mn (besides P, not determinable by INAA), whereas Al, Si, and the elements associated with phyllosilicate minerals (Fe, Ti, V, Hf, Rb, Cs, Mg, Th, etc.) are depleted, except for U and REE enriched in the bed. An RNAA procedure for PGE determination has been tested consisting in sample decomposition by alkaline–oxidative fusion, reduction of PGE, and precipitation of their sulphides. Despite several difficulties, the procedure enabled to found elevated levels of Pt and Ir in selected samples at ppb and sub-ppb levels, respectively. PGE are probably concentrated in pyrite aggregates inhomogeneously distributed throughout the profile, which may have originated in diagenetic reductive processes following decomposition of burried organic matter, similarly to Ni and Sb spikes found recently in limestones around the J/K boundary in Brodno, Slovakia. Ongoing analyses of samples from another J/K locality, Puerto Escaño in Spain, will hopefully provide further information for elucidation of these processes connected with the transition between Jurassic and Cretaceous.

Acknowledgement This work was supported by the grant GA 205/07/1365 of the Czech Science Foundation, and the grant MSM 0021620855 of The Ministry of Education, Youth and Sports of the Czech Republic.

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