Monazite geochronology of Pennsylvanian–Permian sandstones: Provenance and tectonic implications from the Zemplinic Unit, Western Carpathians

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Abstract: During its evolution, the Zemplinic basement underwent a polyphase of regional deformation. Distinctions between pre-Variscan and Variscan events have been widely discussed in previous studies. In order to assess the polyphase, detrital monazites from four Pennsylvanian–Permian sandstones, as well as one Permian vitroclastic tuff, were dated using the Th–U–Pb electron-microprobe method. In total, four monazite generations were recognized in the analyzed sediments and associated acid volcaniclastic rock: Cambrian–Ordovician, Mississippian, Pennsylvanian, and Cisuralian. All monazite phase analyses can be categorized as monazite-(Ce), with the extent of cheralite versus huttonite exchange. Detrital monazites from the Zemplinic Pennsylvanian–Permian sediments were determined by monazite dating and subdivided to several age populations. Early Paleozoic age population was recorded in a relatively large range, from 507 to 435 Ma, with a calculated average age of 461±6.7 Ma. These ages roughly correspond to previously published detrital zircon (SHRIMP) age determinations (Middle/Late Ordovician with the age peak ca. 459 Ma) from the same stratigraphic horizons. Nevertheless, the majority of detrital monazite age data span between 360 and 300 Ma, thus forming significant spikes at 320 and 330 Ma and resulting in a calculated average age of 327±1.6 Ma. This population of detrital monazites indicates the predominance of Bashkirian–Moscovian tectonic events in the assumed source area. Derivation of monazites from the Kasimovian–Gzhelian synsedimentary acid volcanism is indicated by ages in the range of 308–299 Ma. Interestingly, only a few monazite grains derived from the volcaniclastic rock yield the age indications for the Permian extensional thermal re-heating, which has long been associated with the manifestations of acid volcanism (270–290 Ma). The monazite age data from the Zemplinic Pennsylvanian-Permian sedimentary formations reflect the main stages of tectonothermal progress in the Zemplinic crystalline basement.

Keywords: detrital monazite, Th–U–Pb electron microprobe dating, Pennsylvanian–Permian sediments, Zemplinic basement, Western Carpathians

Introduction

The Pennsylvanian–Permian sequence of the Zemplinicum (Figs. 1, 2) was the principal object of the present study; thus, the subsequent text is focused only on this tectonic unit. The Zemplinic Upper Paleozoic sediments are the best stratigraphically-dated, pre-Triassic terrestrial sediments in the Western Carpathians. Age data are predominantly based on macroflora findings, which were gradually determined mainly in the works of Šusta (1931), Němejc (1946, 1953), Němejc & Obrhel (1958), and Sitár (in Planderová et al. 1981), including the microflora from Planderová in Planderová et al. (1981). This envelope of the Zemplinic crystalline basement is formed chiefly by clastic sediments, conglomerates, sandstones, and shales associated with acid volcanics and pyroclastic/volcaniclastic rocks. Lenses of semi-anthracite coal, as well as extremely rare lenses of limnic carbonates are important members, even though they are represented insignificantly by volume. From field and lithological studies, both on the surface and in numerous boreholes, a direct connection between the crystalline basement rocks and overlapping Carboniferous–Permian sediments is evident. This is evidenced primarily by the petrographic composition of the pebble material in the conglomerates, as well as the mineral composition of accompanying sandstones (e.g., Pantó 1965; Pantó et al. 1967; Grecula & Együd 1982; Baňacký et al. 1986; Vozárová & Vozár 1988; Faryad 1995; Vozárová & Šarinová 2024). In addition to rock-forming minerals (quartz, feldspars, and rock fragments), the Pennsylvanian–Permian sandstones contain heavy mineral association, among which zircon and rutile dominate. In comparison to the latter, fluorapatite, titanite, garnet, and monazite-(Ce) grains were found in smaller quantities. U–Pb (SHRIMP) detrital zircon ages from the Zemplinic Pennsylvanian–Permian sandstones were used to characterize the age of the source area, with the aim of clarifying the provenance of these sediments more closely, or their correlation with other Western Carpathian occurrences. The U–Pb detrital-zircon age spectrum demonstrates that the Neoproterozoic arc crust had been

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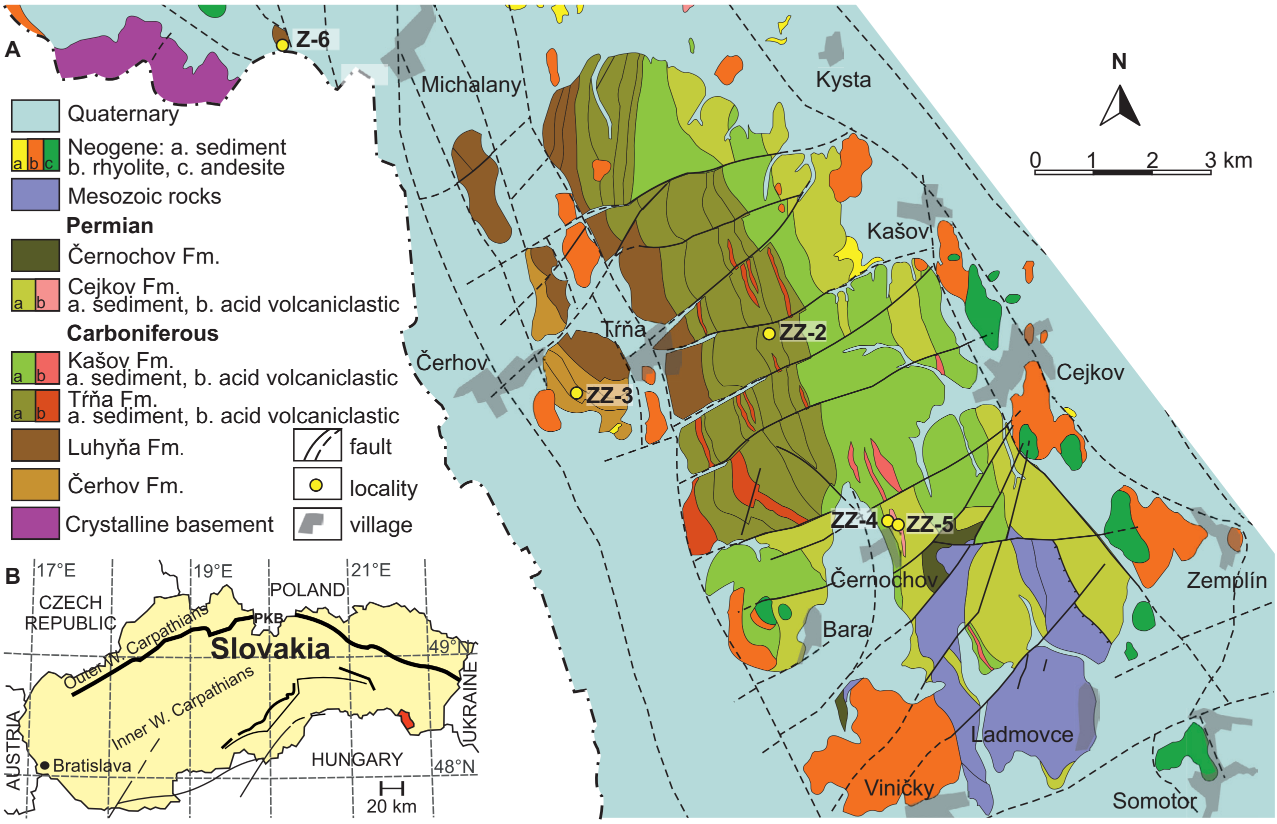


Fig. 1. A — Geological map of the Zemplínske vrchy Mts. showing the localities of magmatic and detrital monazite samples, modified according to the Geological map of the southern part of the East Slovak Lowland, 1:50,000, from Vozár in Baňacký et al. (1986); **B** — Studied area location in the simplified map of Slovakia.

affected by extensional thermal relaxation and melting during the Middle/Late Ordovician. The subsequent re-working had been connected with Mississippian events, followed by the Pennsylvanian/Permian extension (Vozárová et al. 2019a). At the same time, the age of the synsedimentary acid volcanics/volcaniclastics was dated to the Middle/Upper Pennsylvanian (308±3, 307±2 and 305±1 Ma; Vozárová et al. 2019a).

The occurrence of detrital monazites in the Zemplinic spectrum of heavy minerals enabled the determination of their chemical composition and Th–U–Pb EPMA dating. Monazite is more sensitive to tectonometamorphic events than zircon, thereby improving our knowledge on the succession of events in the source area. The focus of this study is to: (i) greater specify provenance for the Zemplinic Pennsylvanian–Permian sedimentary sequence and try to reconstruct the metamorphic and tectonic history of the source region, as well as refine the depositional age; (ii) characterize changes in sedimentary sources in relation to the particular stratigraphic levels and provide critical sequential constraints on the tectonic evolution of the Zemplinicum domain; (iii) confront the U–Pb ages of detrital zircons with the Th–U–Pb EPMA ages of detrital monazites, as well as try to document the tectonometamorphic events in the source area more precisely; (iv) date the age of volcaniclastic rocks in the Permian-Cejkov Formation.

Geological background

The Zemplinic basement rocks, together with their Late Paleozoic and Mesozoic cover sequence, occur within a tectonic horst striking NW–SE, uplifted from the basement of the Cenozoic filling of the East Slovak (Transcarpathian) Basin (Fig. 1). The Zemplinicum was defined by Slávik (1976) as an independent tectonic unit. The basement rocks are exposed in a small area in the south-eastern part of Slovakia, crossing the border with Hungary (ca. 6 km long and 2 km wide; Pantó 1965, Baňacký et al. 1986, 1989; Fülöp 1994). The presence of basement rocks under the Late Paleozoic and Neogene sedimentary cover have been confirmed by several boreholes in Slovakia, as well as in Hungary (Pantó 1965; Grecula & Együd 1982; Vozár et al. 1986). The Zemplinic basement consists of various paragneisses of the amphibolite facies regional metamorphism, locally intercalated with amphibolites, orthogneisses, and migmatites (Pantó at al. 1967; Magyar 1969; Kisházi & Ivancsics 1988; Vozárová 1991; Faryad 1995; Faryad & Vozárová 1997; Faryad & Balogh 2002; Vozárová et al. 2022). From their mineral composition, two groups of mica schists and paragneisses have been distinguished: kyanite-bearing and kyanite-free. The majority of these basement rocks suffered a variable degree of mylonitization: brittle and ductile that occurred under low- to middle greenschist facies

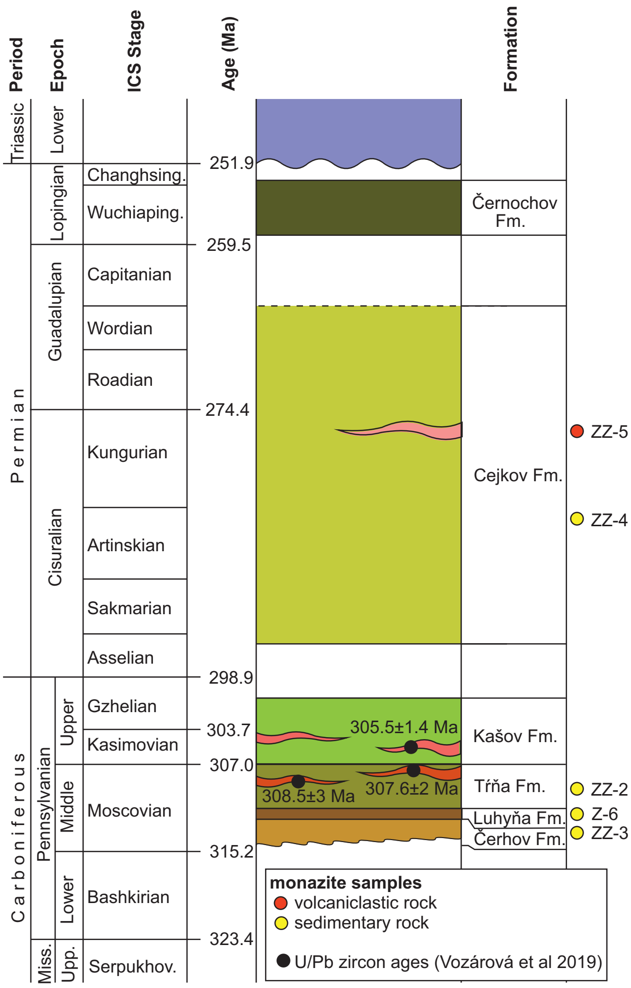


Fig. 2. Stratigraphic scheme of the Zemplinic Pennsylvanian–Permian sedimentary succession tabulated on the ICS Chart (2024). The former scheme (Baňacký et al. 1986) was modified based on magmatic zircon age data from Vozárová et al. (2019a).

conditions. The Cretaceous reworking was confirmed by the K–Ar ages (105±4 Ma and 127±5 Ma) on altered plagioclase and white mica from strongly-mylonitized amphibolite (Faryad & Balogh 2002). When taking into consideration the occurrences of blastomylonite pebbles within the Pennsylvanian/Permian conglomerates, the retrograde reworking most likely had been connected with both the Variscan and the Alpine processes. With regards to the lithology and metamorphic conditions of the Zemplinic basement rocks, a possible correlation with the other tectonic units of the Central Western Carpathians may be suggested (Andrusov 1968; Fusán et al. 1971; Vozárová & Vozár 1977, 1988; Mahel' 1986; Vozárová 1991; Faryad 1995). An entirely different opinion was presented by Grecula & Együd (1977), who correlated Zemplinicum with the Tisia Megaunit, mostly with the Villány and Mecsek Carboniferous–Permian sequences and with the Reşita zone of the Getic Nappe. However, having been inferred from the whole rock and muscovite Rb/Sr data from kyanite-bearing micaschists, the Zemplinic basement rocks were interpreted as Precambrian (984±108 Ma and 962±39 Ma, Pantó et al. 1967). This was one of the main reasons to tentatively correlate the Zemplinicum with the Eastern Carpathian basement (Grecula et al. 1981). However, the K/Ar age of amphiboles from amphibolites yielded an age of ca. 307 Ma and was thus interpreted as the age of overprint (Lelkes-Felvári et al. 1996). The age of 338±22 Ma was obtained on monazite from mica schist, thereby signifying a Variscan regional metamorphism event (Finger & Faryad 1999). Similarly, the 338±13 Ma and 313±13 Ma K–Ar ages of amphibole from amphibolite were interpreted as the Variscan age of metamorphism (Faryad & Balogh 2002). In the Hungarian part, low-grade metamorphic rocks (phyllites, "porphyroides") have been reported in boreholes by Pantó (1965). The age of metamorphism of these rocks was determined by the Rb–Sr method to 394±54 Ma on muscovite, while from the whole rock it was dated to 450±130 Ma (Pantó et al. 1967). However, these data are not credible due to their high standard deviation.

The Zemplinic Pennsylvanian–Permian sedimentary cover consists of six lithostratigraphic units (Figs. 1, 2). From the bottom upwards they are: Čerhov (Grecula & Együd 1982), Luhyňa (Vozárová 1986), as well as the Tŕňa, Kašov, Cejkov and Černochov formations (Bouček & Přibyl 1959). The Pennsylvanian and Permian stratigraphic interval has been determined on the basis of macro- and microfloral assemblages (Němejc 1946; Němejc & Obrhel 1958; Bouček & Přibyl 1959; Planderová et al. 1981). A characteristic feature of this sedimentary sequence is a decrease in mineral maturity in the upward trend. This is clearly documented by the changes in the mineral composition of the sandstones, as well as by relative representation of stable and unstable rock fragments in the associated conglomerates. It should also be emphasized that the structural maturity of sediments decreases in the upward trend.

The Pennsylvanian Čerhov, Luhyňa, Tŕňa, and Kašov formations consist of siliciclastic deposits with characteristic grey, black, and grey-black colours. Polymictic conglomerates with grain-supported structure are the dominant lithofacies of the Čerhov Formation (ČF). Their angular unconformity on the Zemplinic crystalline basement is visible on the surface, and occurs only in the northeastern part of Hungary (Pantó 1965; Pantó et al. 1967; Kisházi & Ivancsics 1988; Lelkes-Felvári et al. 1996). The Čerhov Formation was described on the surface occurrences east of the village of Čerhov as well as in the ZO-7 and ZO-8 borehole profiles (in Grecula & Együd 1982). Numerous fining-upward cycles consist of conglomerates and coarse-grained sandstones, mostly with erosive or sharp mutual contacts. They are interpreted as braided-river deposits. The age of the Westphalian D (upper Moscovian) was assumed by Bouček & Přibyl (1959) and later documented on the basis of microflora by Planderová et al. (1981).

The gradually changing Luhyňa Formation (LF) consists of fine-grained, sandy-shaly lacustrine sediments of a dark-grey and black colour that were interrupted by episodic distal-fan streams with a sandy-conglomeratic material. The dominant

LF lithofacies are shales and siltstones (ca. 55 %) associated with fine-grained sandstones, yet infrequently with medium- to coarse-grained sandstones and fine-grained conglomerates (ca. 6 %). A characteristic feature is a well-developed horizontal stratification, relatively stable lateral thickness of the layers, and mostly sharp or occasionally erosive contacts between them. Fine laminae is another defining feature with a concentration of cherry-red hematite that occur in dark shale. In addition, small pelosiderite concretions are present in the fine-grained sediments. The Stephanian A (Kasimovian) was proved mainly by macroflora (Sitár in Planderová et al. 1981).

Cyclothemes with coal seams are typical for the Tŕňa Formation (TF). The entire TF sequence is divided into two large sedimentary cycles with the seven limnic-fluvial cyclothemes that are present mainly within the lower cycle. Thickness of the coal seams is highly variable (from several cm up to 100 cm), with rapid thinning out along the strike (Együd 1982; Grecula & Együd 1982). In general, the sediments are rich in clastic mica, plant debris, and fragments of tree trunks. One typical feature is a distinct, fining-upward cyclicity with sets of black shales, thin coal seams, and/or lenses of dark clayey limestones in the upper part of the cycles. A second, large cycle is characterized by dominant sandstone lithofacies and the absence of inferior, coal-bearing strata. The relationship between total organic carbon, sulphur, and carbonate content (TOC/S and TOC/carbonate ratios) clearly indicates a continental depositional environment for the TF (Ebner et al. 2006). However, several horizons of acid pyroclastic rocks have also been found. The U–Pb magmatic zircon Concordia ages from the contemporaneous rhyolite volcaniclastics gave 308.5±3 Ma and 307.6±2 Ma, and dated the TF sedimentary sequences to the boundary between the uppermost Moscovian and Kasimovian (according to ICS Chart 2024/12), which is consistent with the cited biostratigraphic data (Planderová et al. 1981 and references therein).

Rhyolite–dacite volcanic/volcaniclastic rocks, including ignimbrites in some places accompanied with alluvial, riverbed, and flood plain deposits, create the standard lithofacies of the Kašov Formation (KŠF). Greenish-grey, mostly coarse-grained sandstones with conglomeratic interlayers, with only a smaller proportion of immature sandy shales (15–20 %), are accompanying sediments. They were included in the Kasimovian–Gzhelian on the basis of their occurrence in the overlier of the biostratigraphical documented Trňa Formation, as well as in the bedrock of the Cejkov Formation. Only the silicified remains of *Dadoxylon sp*. strains and the community of microflora classified by Planderová (in Planderová et al. 1981) into the Stefan B-C were found in it. The 305.5±1.4 Ma Concordia zircon age from the Kašov volcaniclastic rocks corresponds to the Kasimovian (Vozárová et al. 2019a), which is roughly consistent with the biostratigraphic data.

An abrupt transition from the humid to arid climate is characteristic for the Zemplinic Permian sediments. In general, the deposition proceeded in an alluvial environment with typical features of semi-arid and arid environments. A red-coloured sedimentary sequence of the Cejkov Formation (CJKF) is composed of proximal and distal alluvial facies, intercalating in places with flood plain and ephemeral lake deposits with calcrete horizon. Poorly-sorted mud-flow and debris-flow structures have also been detected. A part of this sequence comprises discrete, thin layers of rhyolite tuffs. The youngest lithostratigraphic unit, the Černochov Formation (ČNF), is represented by a monotonous complex of violet-red mudstones. This playa succession is unconformably overlain by the light-grey Early Triassic quartzose conglomeratic sandstones.

Regional metamorphism of the Pennsylvanian–Permian rocks was determined by vitrinite reflectance, DTA characteristic, and X-ray diffraction of carbonaceous matter. This corresponds well with the boundary between very low- and low-grade metamorphic conditions (Milička et al. 1991). Identification of ammonium illite in shales associated with the TF semi-anthracite seams clearly confirmed the anchimetamorphic conditions as well (Šucha et al. 1994). Evidence of the Lower Cretaceous anchizonal metamorphism (120–140 Ma) was provided based on the Illite crystallinity method and K–Ar geochronological dating from acid volcaniclastics at the loc. Šimonov Vrch (TF) (Varga et al. 2024).

Sample characteristics

Detrital monazites were dated in the thin-sections from five samples: three samples from the Pennsylvanian (ZZ-3, Z-6, and ZZ-2) and two samples from the Permian (ZZ-4, ZZ-5) of which sample ZZ-5 comes from volcaniclastic rocks (Fig. 1). Their location is shown in Table 1.

Sample ZZ-3: The sample comes from the upper part of the ČF, which contains layers of dark grey to rust-grey sandstones. Quartz grains are dominant among the clasts (85–90 %). Strongly-decomposed clasts of plagioclases are rare (~1 %). Clastic micas, muscovite, as well as decomposed biotite (~8 %) are quite often present. As the ČF sandstones are coarse-grained, some fragments of blastomylonite, weathered gneiss, and acid blastofelsite have also been identified. On the other hand, these sandstones are relatively well-sorted structurally. The ČF sandstones, represented by the sample *ZZ-3*, are the relatively mineralogical mature sediments belonging to the group of quartzose and sublithic arenite.

Sample Z-6: The fine-grained sandstones were taken from the LF, which is characterized by a very well-developed, horizontal stratification. Tabular sandstones bodies are graded-bedded or massive. They belong to the group of quartzose to sub-lithic greywackes according to their mineral composition. The studied sandstones contain a relatively high proportion of quartz grains (around 80 %) and clastic mica (around 8 %), and only a few feldspars (around 1 %), while compared to feldspars, rock fragments have a more significant presence (around 8 %).

Sample ZZ-2: The sandstone was taken in the lower part of the TF, which represents the higher thickness within the Pennsylvanian formations in the Zemplinicum. The studied

Table 1: List of analysed samples with location. The samples marked with an asterisk were dated by both methods, the U–Pb dating of detrital zircons (Vozárová et al. 2019a) as well as by EPMA dating of detrital monazites (present paper).

Sample	Lithostratigraphic unit	Age	Sample locality	GPS coordinates
*ZZ-3	Čerhov Formation	Upper Pennsylvanian	1 km SW from Veľká Tŕňa Village, slope of Čierna hora height, 216 m a.s.l.	N 48°27'941" E 21°40'133"
Z-6	Luhyňa Formation	Upper Pennsylvanian	SE from Kazimír Village, abandoned quarry, 206 m a.s.l	N 48°27'941" E 21°40'133"
*ZZ-2	Tŕňa Formation	Upper Pennsylvanian	S from Rozhľadňa height, NE from Veľká Tŕňa Village, 286 m a.s.l.	N 48°28'892" E 21°41'954"
*ZZ-4	Cejkov Formation	Permian	N margin of Černochov Village, 197 m a.s.l.	N 48°26'460" E 21°44'120"
ZZ-5	Cejkov Formation	Permian	northern margin of Černochov Village, 177 m a.s.l.	N 48°26'543" E 21°43'918"

sandstones have a black colour, caused by an admixture of organic matter in its matrix. In contrast to the ZZ-3 and Z-6 samples, the quartz content in the ZZ-2 sandstone significantly decreases, and the feldspar content increases. The mineral composition is as follows: quartz (53 %), plagioclase (7 %), potassium feldspar (11 %), detrital mica (16 %), rock fragments (13 %) together with volcanic clasts prevailing (8 %).

Sample ZZ-4: Coarse-grained reddish-brown sandstone of the Permian CJKF is characterized by a low degree of structural and mineral maturity. Its mineral composition is as follows: quartz (53 %), plagioclase (8 %), potassium feldspar (6 %), detrital mica (22 %), rock fragments (11 %), whereas the volcanic clasts dominate between them (9 %). Arid climatic conditions are signalled by carbonate cement, which is the cause of the disintegration of clastic sediments (its dissolution is due to recent weather conditions).

Sample ZZ-5: The sample was taken from a thin layer of acid volcaniclastic rock of the CJKF. The studied rock is well stratified, fine-grained and massive, with prevalent recrystallised vitroclastic texture. Small fragments of volcanic glass, spear-shaped volcanic quartz, and scarce plagioclase scraps have also been observed.

Thin section images of the Zemplinic Pennsylvanian–Permian studied samples are shown in Fig. 3.

Analytical technique

The chemical composition of monazites-(Ce) was acquired by the electron microprobe Cameca SX-100 at the Department of Microanalysis at the State Geological Institute of Dionýz Štúr (Geological Survey of Slovak Republic) in Bratislava.

Accelerating voltage of 15 KV is sufficient to excite Th, U, Pb, and REE lines. The following measured lines were used: P *Kα*, Pb *Mα*, Th *Mα*, U *Mβ*, Y *Lα*, La *Lα*, Ce *Lα*, Pr *Lβ*, Nd *Lα*, Sm *Lα*, Eu *Lβ*, Gd *Lα*, Tb *Lα*, Dy *Lβ*, Ho *Lβ*, Er *Lβ*, Tm *Lα*, Yb *Lα*, Lu *Lβ*, Fe *Kα*, S *Kα*, Ca *Kα*, Sr *Lα*, Al *Kα*, Si *Kα,* and As *Lα.* Counting time at peak position for Pb was 300 s, Th 35 s, U 80 s, Y 40 s, REE between 20–60 s with tendency for longer times for HREE, As 120 s, element S, P, Ca, Al and Si 10 s and finally Fe with Sr 20 s. High beam current 180 nA was used. For analytical calibration, the following natural minerals and chemical compounds were used: Al – Al_2O_3, Si – SiO_2, P – apatite, Ca – wollastonite, REE – (REE) PO_4, Pb – PbS, U – UO_2 and Th – ThO_2. Th, U, Pb, Y, P were measured with LPET, REE with LLIF, As – $GaAs_2$ and Si, Al with TAP analysing crystal. Peak interferences among Th–U–Pb–Y are harmful for monazite dating. Pb$M\alpha$ is overlapped with Th$M\zeta_1$, Th$M\zeta_2$ and Y$L\gamma_{2,3}$ and U$M\beta_1$ with Th$M\gamma_1$. Numerous interferences are present within REE group. Overlaps were resolved by empirically defined correction coefficients.

At the beginning of sample dating, a set of 7 so-called age standards was measured. Age of the monazite standards was determined with spot SHRIMP isotopic analyses. The deviation of the standard ages from the ages determined by microprobe is normally ±5 to 7 Ma. The age standards are used as additional proof that the microprobe is properly calibrated; the measurement conditions are stable and the analytical procedure can reproduce the monazite standard ages. Th–U–Pb concentrations were recalculated to ages by adopting the recalculation scheme developed by Montel et al. (1996), where single analysis gives an apparent age, and the resulting age corresponds to the weighted average of the apparent age's group. The errors are calculated as propagation of ±2 sigma of Th, U, and Pb through Montel's calculation scheme. The two most deviated values from the resulting age are averaged and taken as symmetric ±2 sigma. Monazite composition was processed with a program designed by P. Konečný (unpublished) called MONDAT. Outputs give apparent and resulting ages, histograms, isochrones, and statistical treatments. The more detailed information on measurement conditions and recalculation method is given in Konečný et al. (2004) and Konečný in Kusiak et al. (2014). A new procedure of "monazite age reference correction" (MARC) given in Konečný et al. (2018) was applied for all dating monazite grains.

Monazite features and chemical compositions

The monazites under investigation are present in two types of rocks: sandstones and acid volcaniclastic rock. Altogether, the detrital monazites from 5 samples of both genetic rock types were studied with electron microprobe (Supplementary Table S1). Monazite grains commonly create small anhedral

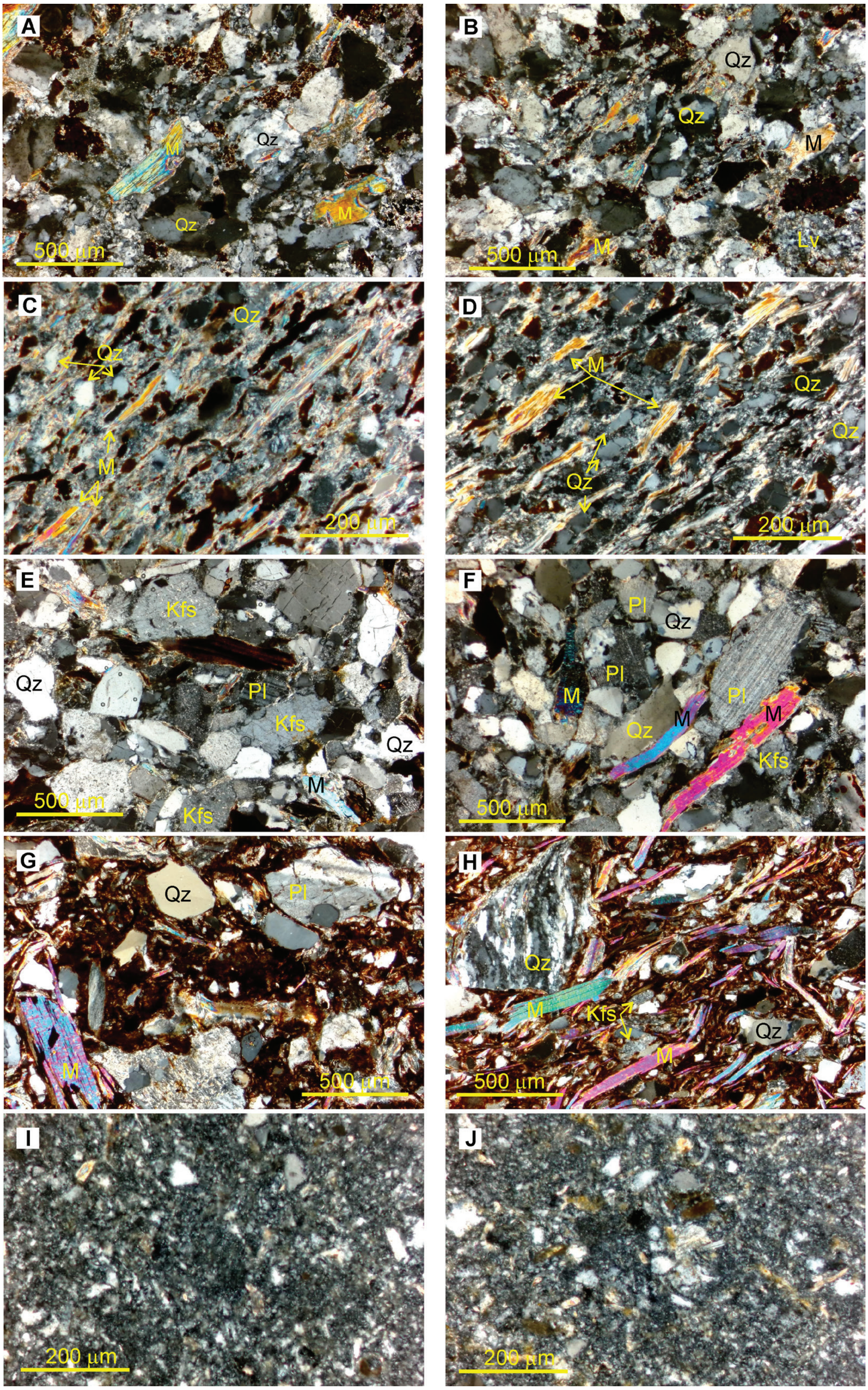


Fig. 3. Thin section images (crossed-polarised light) of the Zemplinic Pennsylvanian–Permian sandstones textures, with marking of basic diagnostic grains. **A, B** — Čerhov Formation sandstone, sample ZZ-3; **C, D** — Luhyňa Formation sandstone, sample Z-6; **E, F** — Trňa Formation sandstone, sample ZZ-2; **G, H** — Cejkov Formation sandstone, sample ZZ-4; **I, J** — vitroclastic tuff, sample ZZ-5. Abbreviations: Qz – quartz total; Kfs – potassium feldspar; Pl – plagioclase; Lv – volcanic lithic grains; M – detrital mica.

grains 20–80 μm in size. They are homogeneous in chemical composition (Figs. 4, 5). A significant part of them was found enclosed within the sandy grains of quartz and aggregates of detrital micas. They are less often scattered in the sedimentary matrix. Monazite grains, although they have an irregular shape, have preserved, rounded edges in places (Fig. 4). In addition, the edges of the grains show signs of partial corroding (Figs. 4, 5). Specific cluster microstructures were found in monazites from the sample ZZ-5 of Permian vitroclastic tuff. Cluster microstructures are interpreted to indicate a new monazite nucleation and growth (Schulz 2021). A total of 246 complete monazite analyses, including REE (Rare Earth Elements), S, and Sr were conducted (Supplementary Table S1).

The molar fractions of the monazite endmembers of the system $2CePO_4$–$2ThSiO_4$–$CaTh(PO_4)_2$ (Linhout 2007) were calculated and plotted in the ternary diagram (Fig. 6A, B). All measured monazites can be classified as monazite-(Ce). Monazites show two major compositional substitutions: cheralite substitution: $2REE^{3+} \leftrightarrow Ca^{2+} + Th^{4+}(+U^{4+})$, huttonite substitution: $P^{5+} + REE^{3+} \leftrightarrow Si^{4+}\ Th^{4+}(+U^{4+})$.

The cheralite versus huttonite exchange in monazite can be observed on the diagram Si+U+Th vs. REE+Y+P+As (Fig. 6C). Detrital monazites from the Pennsylvanian sandstones (samples ZZ-3, Z-6 and ZZ-2) create two groups roughly divided by a dashed line. Group 1 contains monazites from the samples ZZ-2 and ZZ-3, whose points lie very close to the vector representing cheralite substitution. Group 2 consists of the rest of ZZ-2 and ZZ-3 monazites and of Z-6 monazites. These monazites contain a higher proportion of huttonite molecule than Group 1, although huttonite substitution prevails over cheralite substitution only in a few of Z-6 monazites. Detrital monazites from the Permian sample ZZ-4 show the same pattern as monazites from the Pennsylvanian samples ZZ-2 and ZZ-3. The sample ZZ-5 from the Permian acid volcaniclastic rock contains monazites with the highest proportion of huttonite molecule. The huttonite exchange vector is clearly overlapped with cheralite in all monazite phases, indicating that the $Ca^{2+} + Th^{4+} \leftrightarrow 2REE^{3+}$ (cheralite: e.g., Förster 1998; Förster & Harlov 1999) and $Th^{4+} + Si^{4+} \leftrightarrow REE^{3+} + P^5$ (huttonite: e.g., Della Ventura et al. 1996) coupled substitutions may have taken place synchronously. In general, of all analysed monazites phases, the most significant proportion of the huttonite substitution is in the monazite from samples of the Pennsylvanian basal formations (Čerhov and Luhyňa) and in the youngest monazites phases of the Permian vitroclastic tuffs.

The relationship of the ∑REE overall content to the Th content and age of the analysed monazites is expressed in Fig. 7 A, B. In general, the relationship between the ∑REE and Th in the analysed monazite grains shows a negative correlation, indicating the LREE substitution with Th (Fig. 7A). Similar ∑REE characteristics of the majority of monazites from the ZZ-2, ZZ-3 and ZZ-4 samples may be observed with an age of about 300–350 Ma (Fig. 7B). There is a difference in the group of Cambrium–Ordovician monazites. These older monazites from the ZZ-2 sample have a visibly higher ∑REE content as compared to monazites from the ZZ-3 and Z-6 samples. Overall, the highest ∑REE contents are seen in the monazites from the Permian vitroclastic tuff in the ZZ-5 sample.

Dating results

Different monazite populations have been identified in the samples of the Zemplinic Pennsylvanian–Permian sandstones (Supplementary Table S2). The oldest monazite ages, which are preserved only occasionally (ca. 7 % of all), correspond to the Ordovician and only with exception to the Cambrian or Silurian ages. This oldest group of detrital monazite ages was found only in the Pennsylvanian sandstones, with most of them in the Čerhov, Luhyňa, and Tŕňa formations. They are preserved either as solitary crystals or as inherited relics, especially in the Mississippian aged monazites (Fig. 4). The distribution of apparent Cambrian–Ordovician ages in the age distribution histogram is non-uniform (Fig. 8A). For the most part, the age range is wide; the oldest belonging to the Lower/Middle Ordovician (7 ages in the range of 483–460 Ma) and the following 3 ages indicate Upper Ordovician (456–444 Ma). Later, one date for the Cambrian (507 Ma) and the Lower Silurian (435 Ma) were detected. The calculated average age corresponds to the 461±6.7 Ma (n=12), which roughly corresponds with the Middle Ordovician. Isochrone ages represent an old assemblage in the Pennsylvanian samples (Fig. 8B).

It must be emphasized, however, that most of the detrital monazite age data span between 360 and 270 Ma, thus forming the significant spikes at 320 and 330 Ma in the age distribution histogram, with the resulting calculated average age of 327±1.6 Ma (n=160) (Fig. 8C). Isochrone ages representing the dominant assemblages of detrital monazites from the Pennsylvanian–Permian sandstones are shown in Fig. 8D. Still, from the entire set of data, a significant part of them indicates Mississippian ages (ca. 38 % within the range of 355–324 Ma). The margin of apparent ages is dispersed between 389 and 358 Ma, thereby suggesting the Middle/Upper Devonian (ca. 3 % of all). The most significant group indicates Pennsylvanian ages (ca. 51 % of all), which represent the Bashkirian–Moscovian in the range of 323–309 Ma. In small quantities, younger ages have been detected in the range of 308–299 Ma, representing the Kasimovian–Gzhelian. Minute crystals of the Permian ages, ranging from 290 to 270 Ma, have also been found especially within the older Variscan detrital monazite grains (Fig. 8C).

Similarly, an extensive group of monazite ages was found in the ZZ-5 sample, which signifies the volcaniclastic rock. This proves contemporaneous volcanic activity associated with the sedimentation of the Permian red-beds. The nature of the volcaniclastic sediment implies that it may contain detrital monazite grains derived from surrounded sediments. Two groups of age data were found: an older one with a range of 380 to 310 Ma (calculated average age 339±5.8 Ma, n=20;

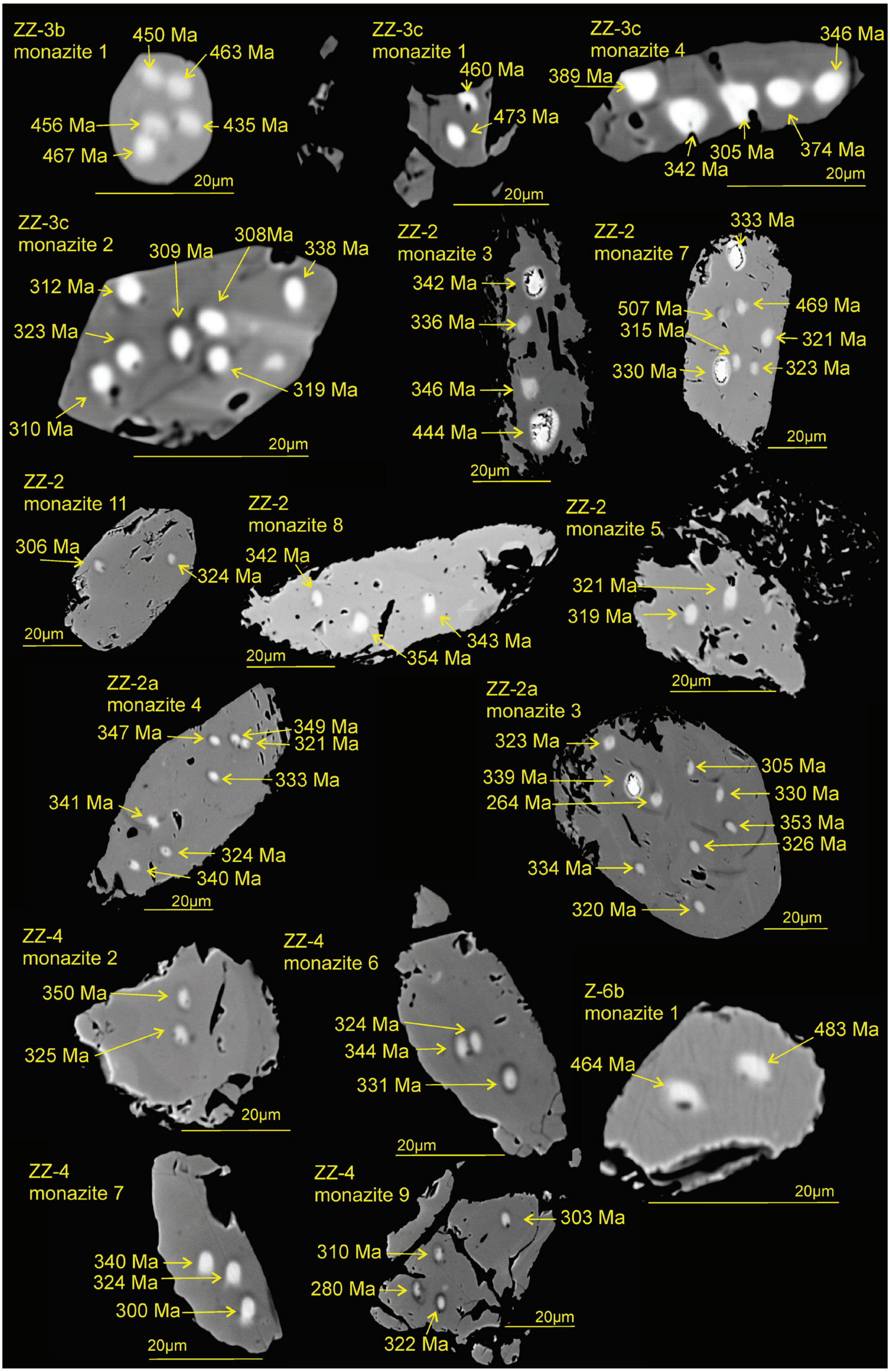


Fig. 4. BSE (Backscattered Electron) images of the detrital monazite from the sandstones and measured in situ age values in Ma.

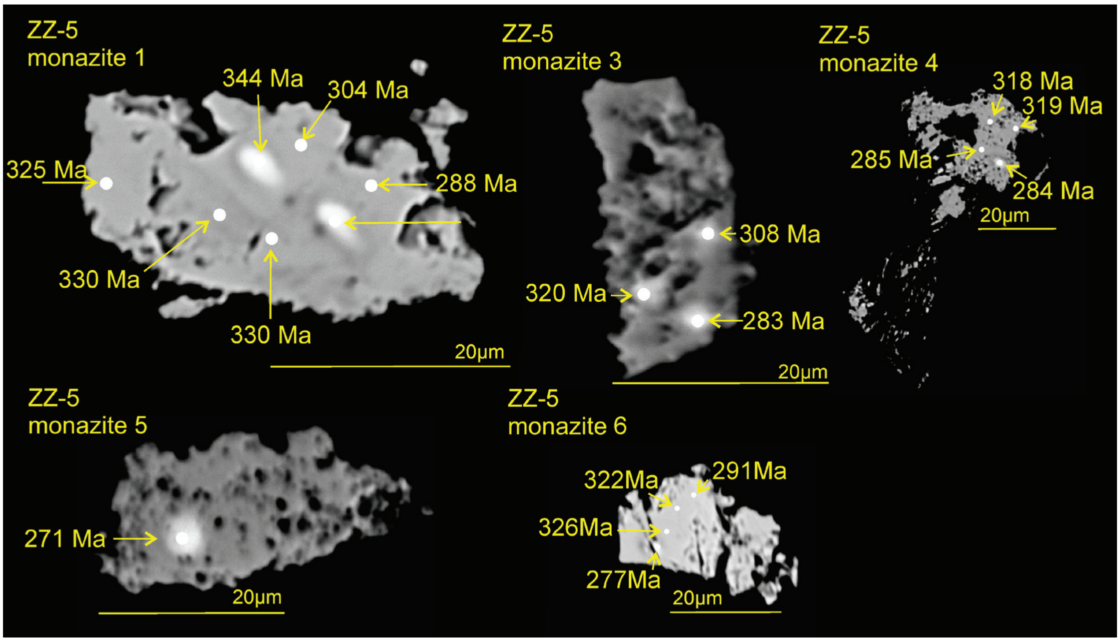


Fig. 5. BSE (Backscattered Electron) images of the monazite from the volcaniclastic rock (sample ZZ-5) and measured in situ age values in Ma.

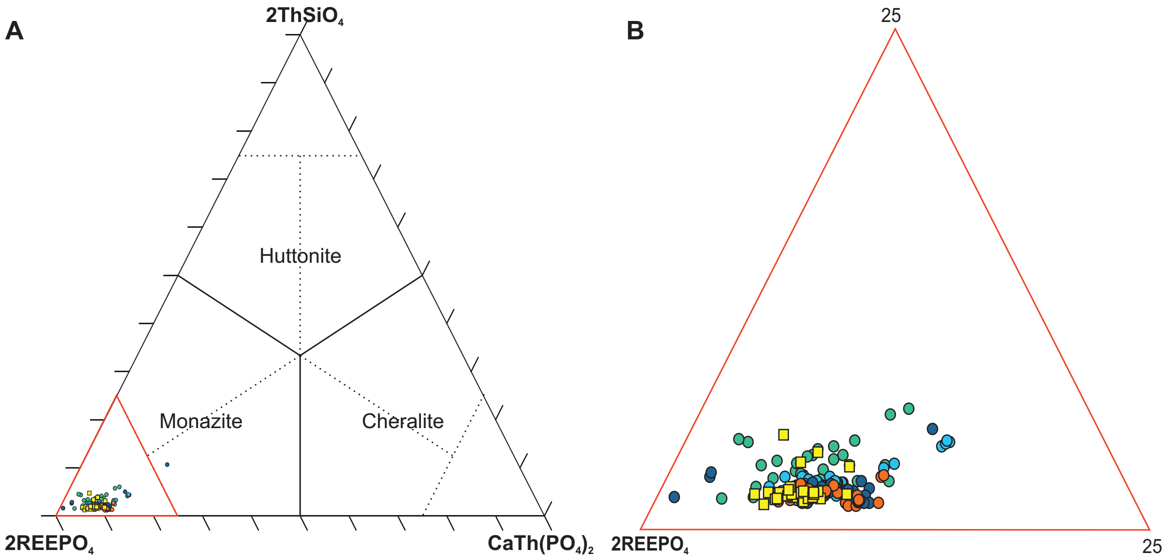
Fig. 9A, B) and a younger one with a range of 300 to 270 Ma (calculated average age 299±7.6 Ma, n=9; Fig. 9C, D). The first group of data indicates Carboniferous ages, completely identical to the age range of detrital monazites from the associated sandstones. The most interesting data are represented by age data ranging from 290 to 270 Ma, which could thus signal the Cisuralian age of the volcanic activity.

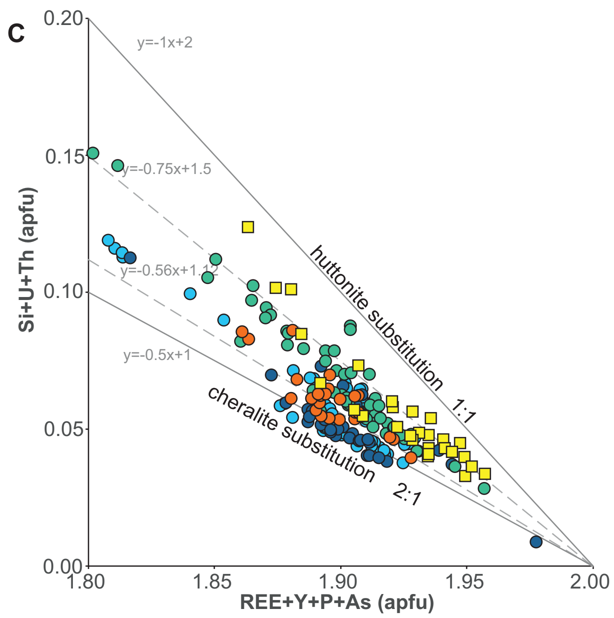
Discussion

Multiple monazite populations have been identified in the samples of the Pennsylvanian–Permian sediments of the Zemplinicum. Evidence for dissolution of monazite and reprecipitation of syntectonic monazite during regional metamorphism (e.g., Pyle et al. 2001), as well as recycling of detrital monazite, has been found in many studies (e.g., Rasmussen & Muhling 2007; Hietpas et al. 2010; Moecher et al. 2019). On the basis of chemical dating of detrital monazites, it was possible to identify the main tectono-thermal events in the source area, as well as interpret the subsequent development of the post-Variscan Pennsylvanian–Permian sedimentary basin.

The oldest Zemplinic detrital monazite ages correspond to the Middle Ordovician (calculated average age 461±6.7 Ma, n=12; Fig. 8A). Overall, it should be noted that these detrital monazite grains were found only in the Pennsylvanian formations. They fully correspond with the U–Pb detrital zircon ages, which were found in the same horizons of the Pennsylvanian sandstones (Vozárová et al. 2019a). Detrital zircon populations from the Zemplinic Pennsylvanian formations (samples ZZ-2 and ZZ-3) yielded identical age clusters, with dominant peaks at ca. 458 Ma and 461 Ma, thereby corresponding to the Middle Ordovician or straddle boundary between the Middle/Upper Ordovician (ICS Chart 2024). Isolated Cambrian or Lower Silurian U–Pb detrital zircon ages have also been sporadically identified, similarly to the detrital monazite population (in this paper). However, some differences can be observed in the distribution of detrital zircons and monazites. If the Middle Ordovician detrital monazites were found only in the Pennsylvanian sediments (samples ZZ-2, Z-6, and ZZ-3), detrital zircons of the same age would form a dominant group, or at least a significant part in all of the Zemplinic Late Paleozoic formations, including the Permian sequence (Vozárová et al. 2019a and the present paper). A relatively consistent detrital zircon spectrum dominated by Cambrian–Ordovician (ca. 497–450 Ma, with average age at ca. 460 Ma), is associated with less Neoproterozoic (ca. 807–560 Ma), Tonian–Stenian (ca. 1024–977 Ma), mid-Paleoproterozoic and early-Archean ages (ca. 2.6–1.75 Ga), since they confirm the Cambrian–Ordovician was dominated by Paleozoic orogenic events that were associated with the Cenerian orogeny (Vozárová & Šarinová 2024). The Middle Ordovician detrital monazite population, even though it has been preserved in a small amount, confirms this assumption.

The most essential group of data in the Zemplinic detrital monazite population is represented by Carboniferous ages in a relatively wide range from the Mississippian to the top of Pennsylvanian (Fig. 8C, D). The scattering of the age ranges from ca. 389 to 300 Ma results in the calculated average age of 327±1.6 Ma (n=160). The histogram distribution shows the most prominent spike at 320 Ma (Fig. 8C). This indicates the predominance of the Serpukhovian–Bashkirian ages, which can be compared to the younger phase of Variscan magmatic events and, at the same time, the continued phase of collision





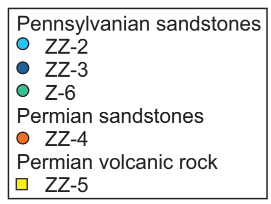


Fig. 6. Chemical composition of the Zemplinic detrital monazites–(Ce): **A** — Classification diagram $2CePO_4$–$CaTh(PO_4)_2$–$2ThSiO_4$ (after Linhout 2007); **B** — Detail of monazite classification from Fig. 6A. **C** — binary Th+U+Si versus REE+Y+P+As showing the cheralite vs. huttonite substitutions in monazites. *Abbreviation*: *apfu* – Atoms per Formula Unit.

orogen in the Inner Western Carpathians. The smaller age population in the range of 310–300 Ma (Fig. 8C) is identical to the U–Pb ages of magmatic zircons detected in synsedimentary volcanites within the Třňa and Kašov formations (308.5±3.0 and 307.6±2.0 Ma in the Třňa Fm.; 305,6±1.4 Ma in the Kašov Fm.; Vozárová et al. 2019a). This age range is consistent with the biostratigraphic data from the above Pennsylvanian formations and corresponds to the uppermost Moscovian/Kasimovian and Gzhelian. In addition, almost the same age, 307.9±12 Ma, was determined by K/Ar method from muscovite of pegmatite that cut the Zemplinic crystalline basement (Faryad & Balogh 2002). The smallest group of detrital monazite ages represent a range from 290 to 270 Ma, corresponding to the relatively wide stratigraphic range, from the middle Cisuralian to lower Guadalupian (according to ICS Chart 2024). They indicate the Permian extensional events which were accompanied by manifestations of calc-alkaline to slightly alkaline acid volcanism.

The textural relationship, mineral compositions, and the age data from the Zemplinic crystalline rock complex indicate Variscan and Alpine metamorphism (e.g., Vozárová 1991; Faryad 1995; Kisházi & Ivancsics 1988; Lelkes-Felvári et al. 1996; Finger & Faryad 1999; Faryad & Balogh 2002). Amphibolite facies metamorphism was dated on K/Ar measurements of amphibole from amphibolites, which gave the age of 338±13 Ma (Faryad & Balogh 2002). This corresponds with the age of 338±22 Ma obtained by Th–U–Pb dating of monazite from micaschists (Finger & Faryad 1999). These ages, with a relatively high standard deviation, do not exclude that the dominant Variscan phase in the Zemplinic basement was associated with the Serpukhovian-Bashkirian period, as is evidenced by ages in the spectrum of detrital monazites in

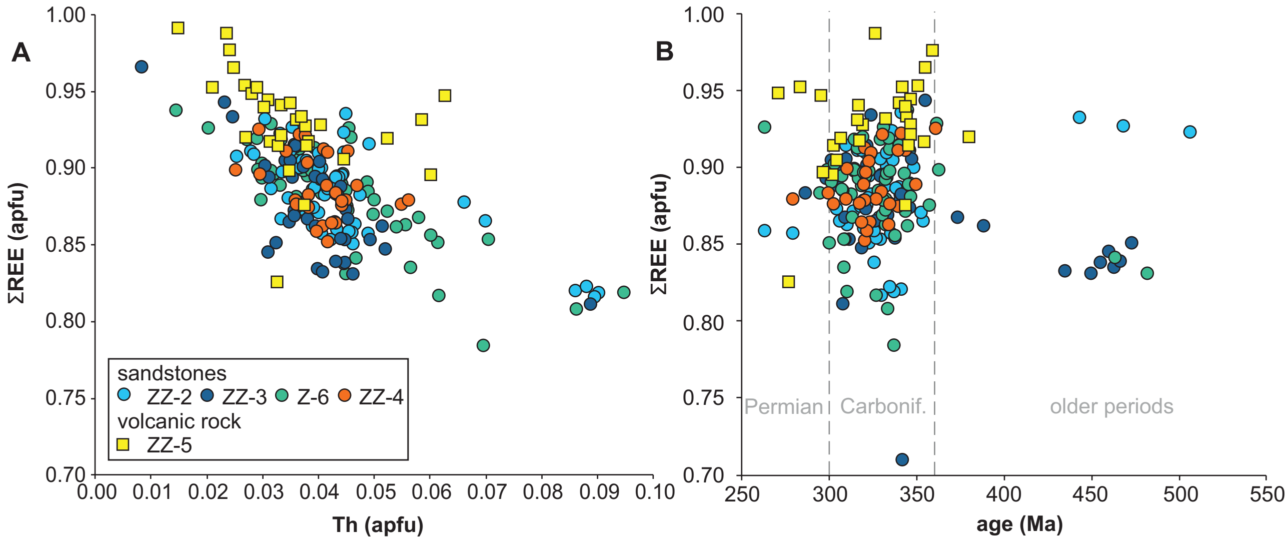


Fig. 7. **A** — Relationship between ∑REE content and Th; **B** — Diagram expressing the relationship of ∑REE and the age of analysed monazites. *Abbreviation*: *apfu* – Atoms per Formula Unit.

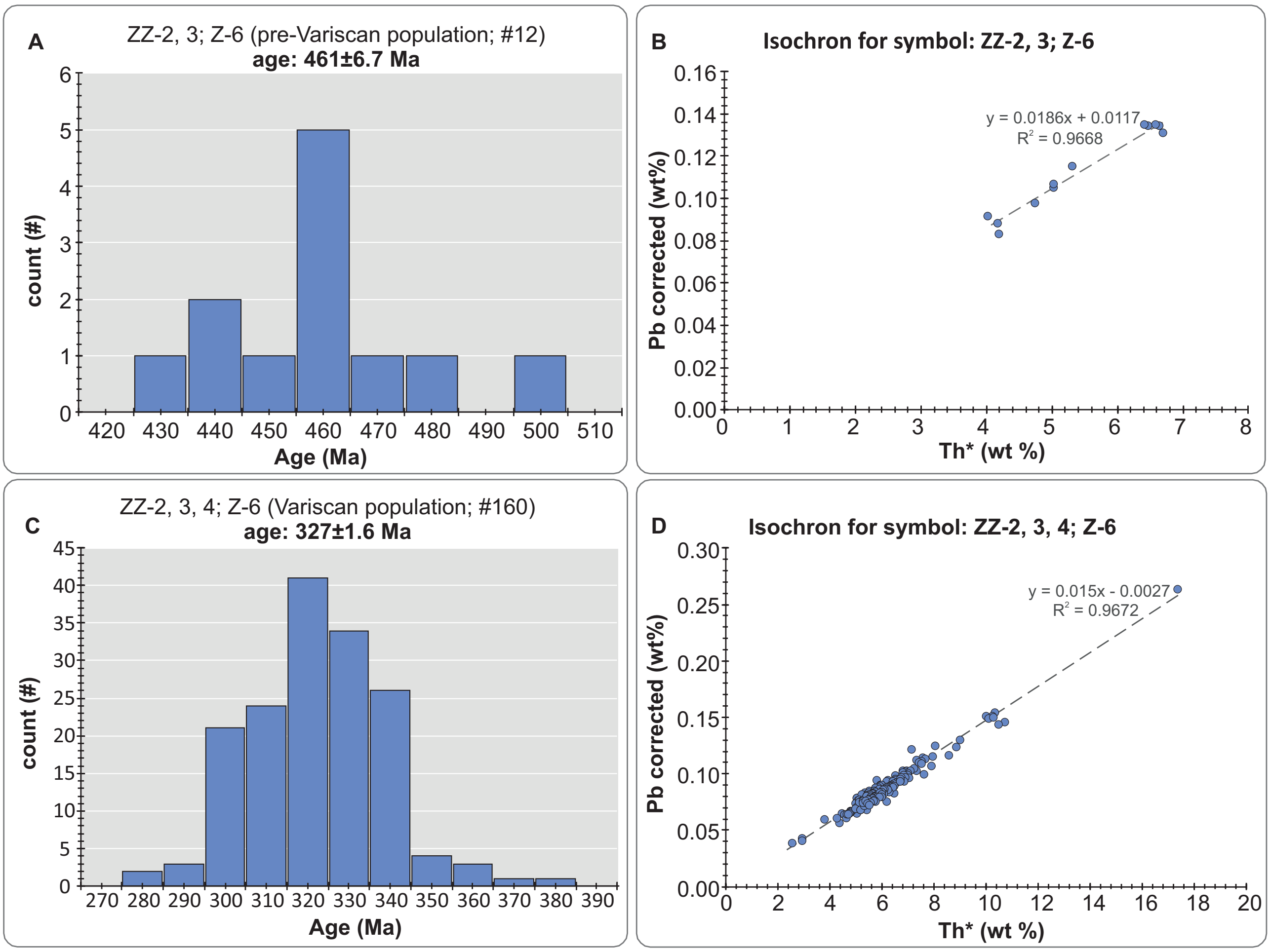


Fig. 8. A — Histogram showing the pre-Variscan detrital monazite assemblage with frequency spike at 460 Ma and calculated average age of 461.0±6.7 Ma (n=12); **B** — Isochrone of the detrital monazite ages for the pre-Variscan assemblage from the Pennsylvanian sandstones; **C** — Histogram of the Variscan detrital monazite assemblage from the Pennsylvanian–Permian sediments (ZZ-2, ZZ-3, ZZ-4 and Z-6 samples), showing one frequency spike at 320 Ma with calculated average age of 327.0±1.6 Ma (n=160); **D** — Isochrone ages representing the dominant Variscan assemblage of detrital monazites from the Pennsylvanian–Permian sediments.

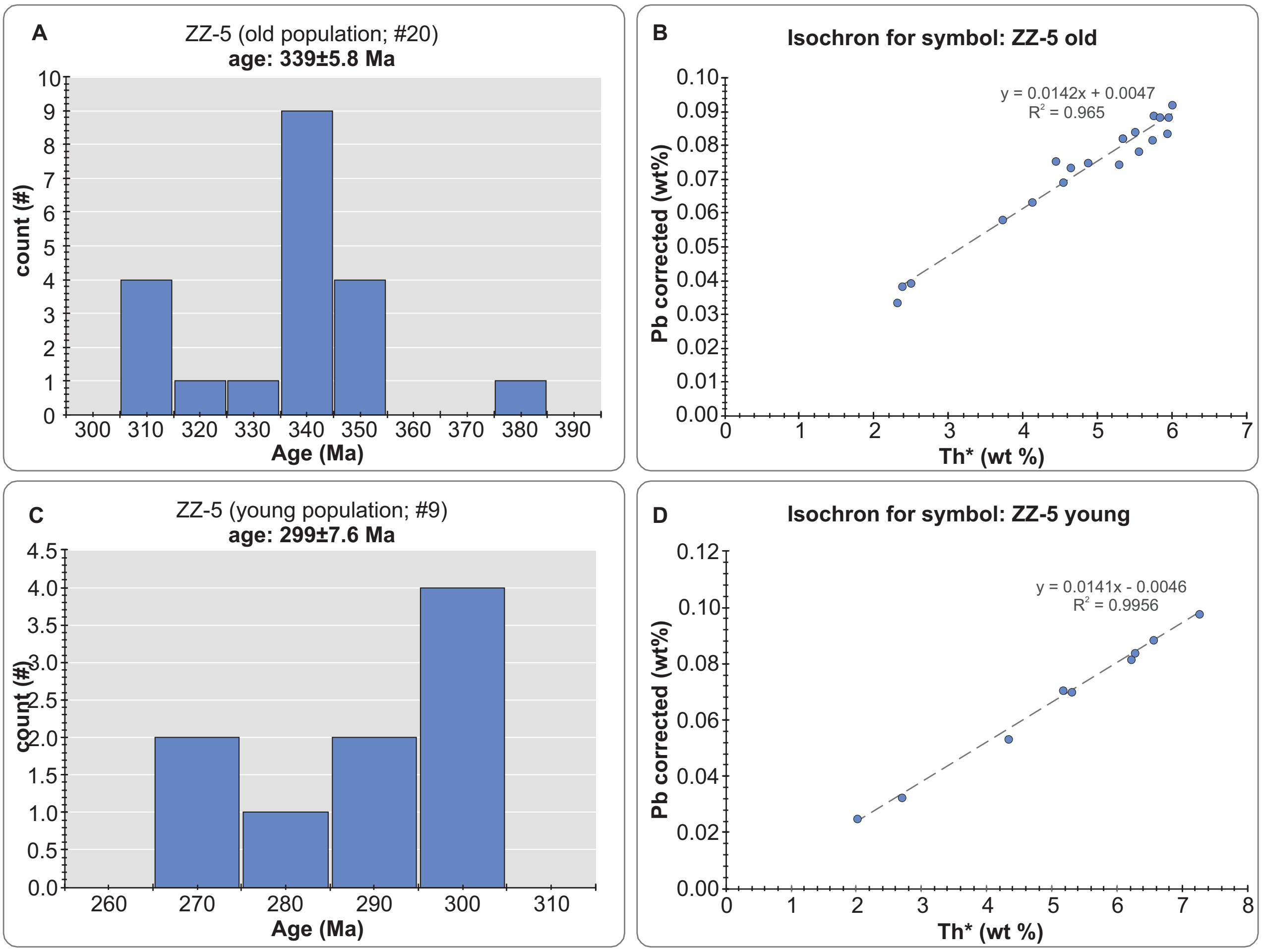


Fig. 9. A — Histogram showing the old, reworked monazite assemblage with frequency spike at 340 Ma and calculated average age of 339±5.8 Ma (n=20) from the volcaniclastic rock (sample ZZ-5); **B** — Isochrone illustrating old, detrital monazite ages; **C** — Histogram of the youngest assemblage of monazite ages from the volcaniclastic rock (sample ZZ-5) showing two frequency spikes at 270 and 300–310 Ma, with calculated average age of 299±7.6 Ma (n=9); **D** — Isochrone illustrating the youngest ages of monazite in the sample ZZ-5.

the Pennsylvanian–Permian sediments (in this article). Lower Cretaceous reworking has been confirmed by the 127±5 and 105±4 Ma K–Ar ages on altered plagioclase and white mica from strongly-mylonitized amphibolite (Faryad & Balogh 2002), as well as from the newly-formed illite in the Pennsylvanian volcaniclastic rock (140–120 Ma; Varga et al. 2024).

Electron microprobe Th–U–Pb dating reveals that the detrital monazite population consists of a mix of various age domains ranging from ca. 507 to 270 Ma. Combined evidence presented here suggests that instead of Pb loss by volume diffusion, the apparent resetting of the U–Th–Pb systematics in monazite rather involves new crystal growth or regrowth by recrystallisation and dissolution/reprecipitation. It should be pointed out that the U–Pb SHRIMP detrital zircon ages obtained from the Zemplinic Pennsylvanian–Permian sandstones are predominantly of Middle/Late Ordovician ages within the range of ca. 444–476 Ma, with a maximum peak at 459 Ma. The detrital zircons, when considering $^{232}Th/^{238}U$ values and their internal textures, point to a felsic magma. However, negligible grains indicate $^{232}Th/^{238}U$ values between 1 and 1.50 (~5 %) or even >1.50 (l.c.), which, according to Wang et al. (2011), can indicate mafic or intermediate melts and a possible contribution of juvenile mantle-derived magma during zircon evolution. The minority of detrital zircons has very low $^{232}Th/^{238}U$ ratios (ca. 0.01), which documents not only magmatic but also Ordovician metamorphic processes in the Zemplinic basement (Vozárová et al. 2019a). This Middle/Upper Ordovician tectonothermal event has also been preserved in the population of detrital monazites, specifically in the Pennsylvanian sandstones. However, the Variscan orogenic events of the Zemplinic basement is documented by the occurrence of the Visean/Serpukhovian and the dominant Bashkirian/Moscovian populations of detrital monazites.

In monazite, the relationship between Rare Earth Elements (REE) and Th is complex and influenced by various factors. REEs and Th can substitute each other in the crystal structure, however, their relative proportions vary depending on the specific geological environment and conditions. Th^{4+} incorporation depends on specific REEs showing negative correlation with Ce, Pr and Nd (Ni et al. 1995; Clavier et al. 2011).

The Th/U ratio is used as a criterion for the distinction between the hydrothermal and magmatic monazites (Janots et al. 2012 and references therein). Within the studied detrital monazites, the values of Th/U ratios varied within 3.5–17, only rarely reaching 30–81. This implies that the studied monazites should be primary, without secondary changing of their composition by the action of hydrothermal or metasomatic fluids. Hydrothermal monazite crystals are characterized by extremely high Th/U ratios up to 629 (Janots et al. 2012). High values of Th/U in the secondary monazites were also described by Seydoux-Guillaume et al. (2012), which arose at a low-temperature alteration of primary monazite. Extremely high Th/U ratios (up to 1670) were described in the authigenic monazites (Zeh et al. 2025). In the youngest group of ages that indicate Permian (sample ZZ-5), the Th/U ratios are low in three spots (in the range of 4–6–78), thereby indicating the primary volcanic monazites. Higher values of 132 and 195 in the two others analyses are likely to signal the secondary alteration of originally primary monazites.

Regarding the comparison of the detrital monazite ages from the Zemplinic Pennsylvanian/Permian sediments, some differences have been found in comparison to the monazite ages from the basement of Tatricum and Veporicum. Certainly, in granites and metamorphites, Variscan ages prevail in the range of approx. 360–340 Ma (e.g., Finger et al. 2003; Konečný et al. 2011; Uher et al. 2014; Potočný et al. 2020). Only scarce relics of older ages were found, mostly in the range of the Upper Silurian/Lower Devonian (Uher et al. 2014; Janák et al. 2022). Older ages in the range of 670–470 Ma were captured in the Veporic Fe-gneisses from the village of Kokava (Konečný et al. 2011). In the Hronic Permian sandstones, detrital monazite ages predominate in the range of 380–330 Ma and are associated with Permian ages ranging from 280 to 250 Ma in some of them (Olšavský 2008; Vozárová et al. 2014a). Monazites from metapelites and metavolcaniclastics of the Early Paleozoic Gelnica Group (GG) from the Southern Gemeric basement have provided a wider range of ages. In the GG acid metavolcaniclastics, the Lower to Upper Ordovician monazite ages prevail (from 477±7 to 444±13 Ma), which are associated with a small amount of recycled Precambrian ages (550, 660, 980, 1200 Ma) (Vozárová et al. 2014b). These Early Paleozoic magmatic monazites were partly overprinted by the low-Y Alpine monazites (133±5 and 184±16 Ma) at their rims. In the GG metapelites, newly-formed low-Y monazites of Alpine age are dominant, reflecting the polystage Alpine compression with the three distinct peaks at 100±8, 133±5, and 190±16 Ma, respectively. Recycled older ages (400–650 Ma) were only reflected in metapelites (Vozárová et al. 2014b).

The assemblage of detrital monazites found in the Zemplinic Pennsylvanian–Permian sediments cannot be compared to the spectrum of monazites detected in the CZK crystalline complexes. The main reasons are: (1) The presence of Cambro–Ordovician ages of detrital monazites in the Zemplinic sediments; (2) The prevalence of the Serpukhovian–Bashkirian population of detrital monazites in Zemplinic Pennsylvanian–Permian sediments compared to the Tournaisian–Visean monazite ages in the CZK crystalline basement. Similarities can be seen among the monazites found in the Southern Gemeric Gelnica Group, e.g., the presence of Cambro–Ordovician age in both parts. The truth is, however, that the Gelnica Group nearly lacks monazites of Variscan ages. Only traces of 310–340 Ma were detected. The Alpine polystage reworking of the Southern Gemeric basement is clearly documented by the dominant Lower Cretaceous monazite ages, which are likely followed only after the Jurassic events (Vozárová et al. 2014b). In contrast, the Alpine monazites are absent in the Zemplinic sediments, which is understandable considering the anchi-metamorphism degree of the Alpine reworking of Zemplinicum. However, the K–Ar dating of the newly-formed illite from the Pennsylvanian volcaniclastics also demonstrate Lower Cretaceous metamorphism (Varga et al. 2024).

In general, Famennian/Tournaisian and Visean/Serpukhovian ages (monazite and zircon) are typical for the Central Western Carpathians Zone (CWCZ) and Northern Gemericum (NGU). They are identical to the main magmatic events in the CWCZ (e.g., Poller & Todt 2000; Gaab et al. 2005; Kohút et al. 2009; Burda et al. 2013; Kohút & Larionov 2021; Broska & Svojtka 2020; Broska et al. 2022), as well as to the development of the Tournaisian–Visean syn-collisional sedimentary basin in the NGU (Vozárová 1996, 1998; Vozárová & Šarinová 2024 and cited references). However, the Bashkirian–Moscovian tectono-thermal events are characteristic for the external zone of Variscan orogeny of the Inner Western Carpathians. These events are also related to the origin of the Bashkirian turbidite trough in the Turnaic Unit (Vozárová & Vozár 1992). The population of detrital monazites found in the Zemplinic Pennsylvanian–Permian sediments indicates the paleoposition of their source area, to the external zone of the Variscan orogeny, which assumes their paleoposition close to units of the Inner Western Carpathians. The similarity of the detrital zircon age populations from the Southern Gemeric, Bôrka Nappe, Turnaic, and Zemplinic tectonic units has previously been shown in the study by Vozárová et al. (2019b). The Cambro-Ordovician age of detrital and magmatic zircons from the Southern Gemeric basement was also confirmed by the study of Soejono et al. (1924). A comparison of the detrital monazite ages between the Southern Gemericum and Zemplinicum confirmed this assumption. We therefore consider the Southern Gemeric and Zemplinic units to be a part of the basement nappes, which were originally situated on the northern margin of the Meliata Ocean and detached to the current position in connection with the closure of the Meliata Suture and the following Early Cretaceous orogenetic movements.

Conclusions

The established detrital monazite ages, which were obtained from the Zemplinic Pennsylvanian–Permian envelope sequence, clearly document the multistage origin of the Zemplinic

Crystalline Basement. The first stage, although recognised by a smaller number of detrital monazites grains, indicates the Middle Ordovician tectonothermal processes. This one is well-documented mainly by the previous dating of the detrital zircons from the same stratigraphic horizons. The predominantly representated groups of the Bashkirian–Moscovian detrital monazites reflect the main Variscan collision orogenic processes in the Inner Western Carpathian basement. These were connected with the development of the Pennsylvanian sedimentary basins. With these events, a significant thermal relaxation of the Zemplinic Crystalline Basement was connected. The assemblage of the youngest Pennsylvanian/Permian detrital monazites ages is linked with a post-collisional stage of Variscan orogeny and with the development of the Moscovian/Kasimovian sedimentary basin in the Zemplinicum, which was accompanied by Kasimovian/Gzhelian and Permian volcanism.

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Electronic supplementary material is available online:
Supplementary Table S1 at https://geologicacarpathica.com/data/files/supplements/GC-76-5-Vozarova_TableS1.xlsx
Supplementary Table S2 at https://geologicacarpathica.com/data/files/supplements/GC-76-5-Vozarova_TableS2.xlsx