

First U–Pb LA-ICP-MS Radioisotopic Ages of the Devonian–Carboniferous Boundary Black Shales (Domanik Facies) in the Kama–Kinel Trough System, Volga–Ural Petroleum Province, East European Platform

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For the first time, zircons from thin volcanic ash layers contained within organic-rich black shales (Domanik facies) at the Devonian–Carboniferous boundary have been dated using the U–Pb LA-ICP-MS method. The studied material comes from core samples of two boreholes located in the axial and marginal zones of the Kama–Kinel Trough System within the Volga–Ural Petroleum Province. In the axial zone, the base of the *Siphonodella quadruplicata* conodont zone yielded a concordant U–Pb age of 357.6 ± 1.7 Ma. In the marginal zone, the upper part of the *Palmatolepis gracilis expansa* Zone provided a concordant U–Pb age of 360.0 ± 1.2 Ma. These ages agree, within analytical uncertainty, with the current conodont-based chronostratigraphic framework and refine the onset of the Hangenberg Event in the studied basin.

Keywords: East European Platform, Kama–Kinel Trough System, Devonian, Carboniferous, black shales, Domanik facies, U–Pb LA-ICPMS zircon dating

Recommended citation: Silantiev V.V., Nurgaliev D.K., Sungatullina G.M., Miftakhutdinova D.N., Kulikova A.V., Nourgalieva N.G., Saetgaleeva Ya.Ya., Vandin A.S. (2025). First U–Pb LA-ICP-MS Radioisotopic Ages of the Devonian–Carboniferous Boundary Black Shales (Domanik Facies) in the Kama–Kinel Trough System, Volga–Ural Petroleum Province, East European Platform. *Georesursy = Georesources*, 27(2), pp. 297–304. <https://doi.org/10.18599/grs.2025.2.22>

Introduction

Volcanic material in the Domanik facies of the Devonian and Carboniferous deposits of the Volga–Ural region occurs either as ash beds (up to 3 cm thick) dominated by volcanic glass, as greenish-grey clays, or as micro-lenses of tuffaceous material (Fortunatova et al., 2018, 2023). Previous studies have provided detailed microscopic, X-ray, and thermal analyses of tuff layers from the Upper Devonian and Tournaisian strata of the Mukhanovo–Erokhovo Depression within the Kama–Kinel Trough System (Shakirov et al., 2022).

During 2023–2024, laboratory studies were conducted to develop a method for extracting heavy-mineral

fractions from these tuff beds. The main difficulty is that these beds are extremely hard and only a few millimetres thick, so the sample weight typically does not exceed 3–5 grams. The materials analysed in this study come from one of the few localities where tuffs from the Devonian–Carboniferous boundary interval have been directly dated by radioisotopic methods (Fig. 1) (Trapp et al., 2004; Liu et al., 2012; Myrow et al., 2014; Ferri et al., 2021; Xu et al., 2024).

Geological setting

The Kama–Kinel Trough System (KKTS) was active from the early Frasnian to the late Tournaisian, developing along the margins of isolated carbonate platforms that existed on the edge of the East European Platform, adjacent to the Ural Ocean (Silantiev et al., 2024). The troughs were filled with clayey, siliceous, and carbonate sediments rich in organic matter.

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Figure 1. Main sections where the Devonian–Carboniferous boundary interval has been dated: 1 – Western Canada (Ferri et al., 2021), 2 – Germany (Trapp et al., 2004), 3 – Poland (Myrow et al., 2014), 4 – South China (Liu et al., 2012; Xu et al., 2024), 5 – East European Platform (this study). The palaeogeographic map has been adapted and simplified from free resources by R. Blakey (Deep Time Maps; <https://deeptimemaps.com/map-lists-thumbnails/global-series/>).

In Russian geological terminology, these organic-rich deposits are classified as “Domanikites” (5–25% total organic carbon) and “Domanikoids” (<5% total organic carbon), collectively referred to as Domanik-type rocks or Domanik facies (i.e., oil source rocks). The good preservation of ash layers within these rocks is due to their depositional environment – relatively deep-water settings (below storm wave base), located far from clastic sediment sources and with minimal clastic input.

Materials

Volcanic ash beds were encountered in two boreholes that penetrated the Devonian–Carboniferous boundary interval in the axial (Borehole 1) and marginal (Borehole 2) zones of the Lower Kama (Nizhnekamsk) segment of the KKTS. The stratigraphic subdivision of these boreholes is based on detailed studies of conodont assemblages (Sungatullina et al., 2025), while full descriptions of the sections and associated biofacies will be published separately (Silantiev et al., 2025, in press).

The ash beds, 3–4 mm thick, are composed of hard, compacted, and well-consolidated vitroclastic material (Fig. 2), which contrasts sharply with the dark host rock due to its light yellowish-grey colour and distinct ultraviolet fluorescence. A 3–5 g sample from each bed contained 60–100 euhedral zircon grains suitable for geochronological analysis.

Methods

Sample preparation. The tuff layer was extracted from drill core using a diamond saw. The resulting slab, weighing 3–5 g, was crushed into 3×5 mm fragments and immersed in dimethyl sulfoxide ((CH₃)₂SO) at 50 °C for 24 hours with constant stirring. The sample was then subjected to ultrasonic disintegration (25 kHz) using a point-probe sonicator for 8 hours. This procedure was repeated 20 to 26 times until the original rock had completely disintegrated. The resulting suspension was separated using the heavy liquid GPS-B (a concentrated aqueous solution of sodium heteropolytungstate; density

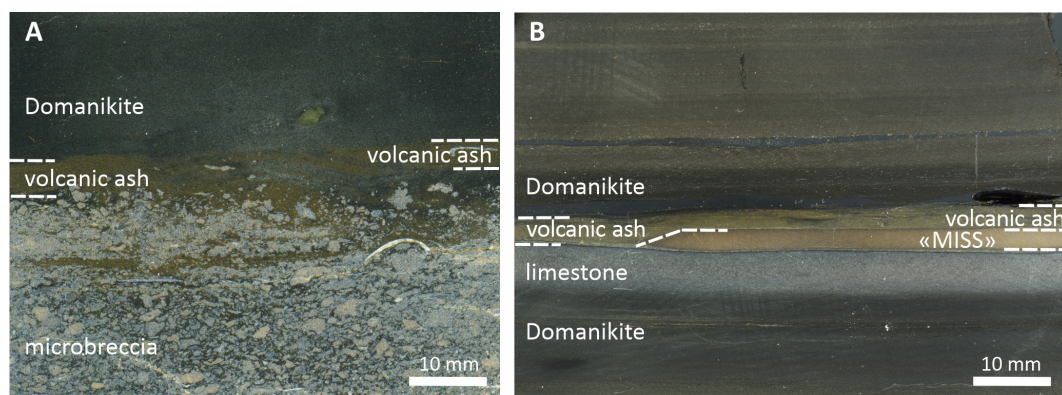


Figure 2. Volcanic ash layers: (A) – Borehole 2, within the *Palmatolepis gracilis expansa* Zone; (B) – Borehole 1, within the *Siphonodella quadruplicata* Zone. White dashed lines mark the boundaries of the ash beds; Abbreviation: MISS – microbially induced sedimentary structures.

3.00 g/cm³). Individual zircon grains were manually picked from the heavy fraction under a ZEISS Stemi DV4 binocular microscope (Germany).

U–Pb dating of zircon grains using LA-ICP-MS was performed at the Geothermochronology Research and Education Centre, Institute of Geology and Petroleum Technologies, Kazan (Volga Region) Federal University. Zircon dating employed a laser ablation system based on an excimer laser (wavelength 193 nm) – Analyte Excite (Teledyne CETAC Technologies, USA) – coupled to a quadrupole inductively coupled plasma mass spectrometer (ICP-MS), iCAP Qc (Thermo Scientific, Germany). Masses measured included ²⁰²Hg, ²⁰⁴(Pb+Hg), ²⁰⁶Pb, ²⁰⁷Pb, ²⁰⁸Pb, ²³²Th, ²³⁵U, and ²³⁸U. Laser beam diameter was 35 µm; pulse repetition rate was 5 Hz; and laser fluence ranged from 2.5 to 3.0 J/cm².

The analytical protocol included three measurements of both the primary (external, calibration) standard and the secondary (monitoring, validation) standard at the beginning of each session and two at the end. International reference zircons were used: Plešovice (337 Ma; Sláma et al., 2008) as the primary standard, and 91500 (1065 Ma; Wiedenbeck et al., 1995) as the secondary standard. Every ten unknown analyses were bracketed by one analysis of each standard. The primary standard (Plešovice) was used to correct for elemental fractionation, mass bias, and instrument drift. The secondary standard (91500) was used to monitor the accuracy of the analytical procedure. Additionally, the NIST SRM 612 synthetic glass was measured at the beginning, middle, and end of each session to monitor instrument sensitivity.

Mass spectrometry data reduction – including baseline correction, signal integration, isotopic ratio (²⁰⁷Pb/²⁰⁶Pb, ²⁰⁶Pb/²³⁸U, ²⁰⁷Pb/²³⁵U, ²⁰⁸Pb/²³²Th) calculation was carried out using Iolite 3.65 within the Igor Pro 7 platform (Paton et al., 2010). Calculation of age values from isotopic ratios and concordia diagram plotting were performed in IsoplotR (Vermeesch, 2018). Analyses with discordance values < 5% or > 5% were excluded from the final dataset (in the Supplementary file “Age of individual zircon grains determined by LA-ICP-MS” (<https://www.geors.ru/jour/article/view/562>) these values are crossed out)).

Results

The heavy fraction of light-coloured minerals in the studied tuff layers consists of 50–90% zircon (ZrSiO₄), of which 80–85% are euhedral crystals showing no signs of rounding or transport (Fig. 3). The zircon grains are represented by prismatic and short-prismatic crystals ranging from 50 to 175 µm in length. Cathodoluminescence images reveal oscillatory zoning, and Th/U ratios vary between 0.39 and 1.3, indicating a magmatic origin (Corfu et al., 2003; Wu, Zheng, 2004).



Figure 3. Zircon crystals from the tuff layer in Borehole 2 (Palmatolepis gracilis expansa Zone)

In the marginal zone (Borehole 2), a U–Pb age of 360.0 ± 1.2 Ma was obtained from a tuff layer in the upper part of the *Palmatolepis gracilis expansa* Zone (52 zircon grains dated). In the axial zone (Borehole 1), a concordant age of 357.6 ± 1.7 Ma was determined from a tuff layer at the base of the *Siphonodella quadruplicata* Zone (30 zircon grains dated) (Figs. 4, 5).

The obtained ages are consistent, within analytical uncertainty, with the current chronostratigraphic framework of conodont zonation. They constrain the onset of the Hangenberg Event in the studied basin to 360.0 ± 1.2 Ma, which is broadly in agreement with global data (Davydov et al., 2012; Becker et al., 2020; International Chronostratigraphic Chart, 2024).

Discussion

Chronological calibration of the Devonian relies on the integration of high-precision U–Pb CA-ID-TIMS zircon dating from tuff layers with detailed conodont zonal biostratigraphy. This approach not only refines the radioisotopic ages of stage boundaries but also estimates the durations of individual biozones with a resolution of up to 0.6 Ma. Dedicated studies (Kaufmann, 2006; Harrigan et al., 2022) have proposed detailed “biochronometric” frameworks that align each conodont zone with a numerical (radioisotopic) age.

The radioisotopic age of the Devonian–Carboniferous boundary was first estimated at 360.7 ± 0.7 Ma using interpolation based on a series of U–Pb ID-TIMS zircon dates from tuff layers in the Tournaisian part of the Hasselbachtal section (Germany) and the Upper Famennian Exshaw Formation (Western Canada) (Trapp et al., 2004). Since then, the boundary age has been progressively revised to younger values. According to the *Geological Time Scale 2020*, the boundary is dated at 359.3 ± 0.3 Ma (Becker et al., 2020), supported by additional data from South China (Liu et al., 2012; Xu et al., 2024). The most recent International Chronostratigraphic Chart (2024 edition)

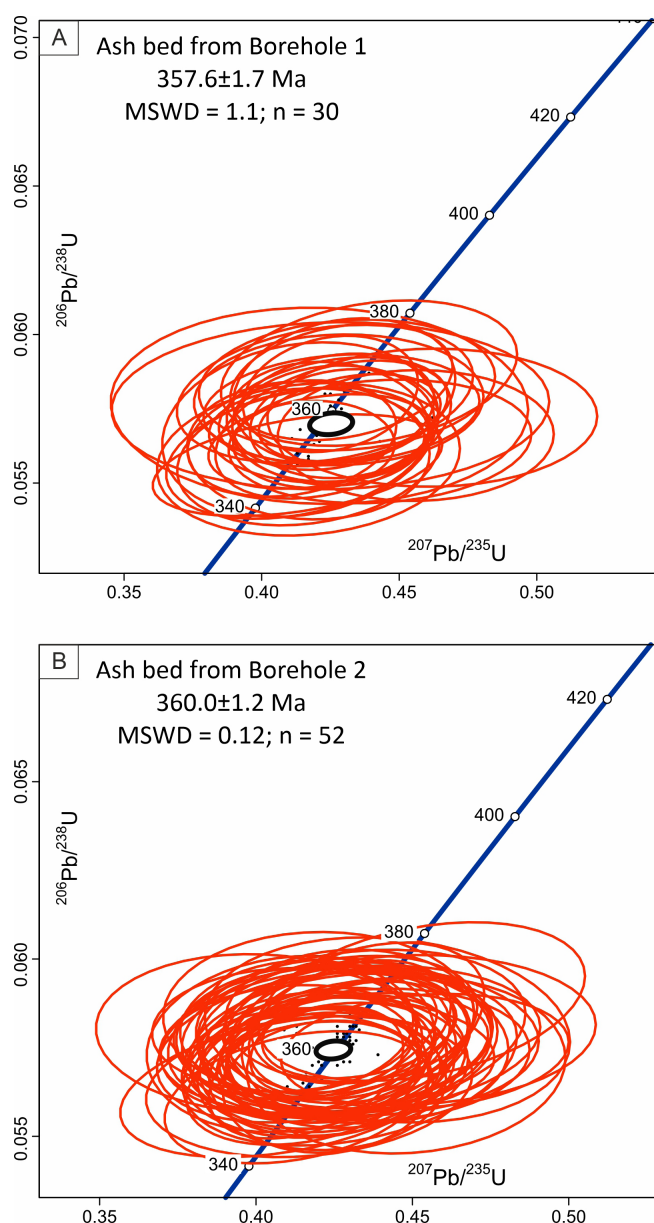


Figure 4. Concordia diagrams for zircon U–Pb analyses: (A) – sample from Borehole 1, (B) – sample from Borehole 2. Ellipses represent 2σ analytical uncertainties for individual zircon analyses. Abbreviations: MSWD – mean square of weighted deviates; n – number of dated zircon grains.

places the Devonian–Carboniferous boundary at 358.86 ± 0.19 Ma.

The stratigraphic position of volcanic ash layers in various regions worldwide demonstrates that they are preserved both in carbonate facies – for example, the Hasselbachtal section in Germany (Trapp et al., 2004) and the Muhua II section in South China (Xu et al., 2024) – and in Domanik-type facies, such as the Exshaw Formation (Fm) in Western Canada (Ferri et al., 2021; Kabanov, 2022), the Daposhang section in South China (Liu et al., 2012; Xu et al., 2024), and the Kowala section in Poland (Myrow et al., 2014; Davydov, 2020).

In Western Canada, the Devonian–Carboniferous boundary interval has been dated using tuff layers

identified within the Exshaw Fm. This formation consists of rhythmically laminated, dark grey, silt- and clay-rich shales with high total organic carbon content, closely resembling the rocks of the Kama-Kinel Trough System. In the Liard Basin (Ferri et al., 2021), three tuff horizons have been dated by the U–Pb CA-ID-TIMS method and yielded ages of 364.35 ± 0.26 Ma, 364.03 ± 0.31 Ma, and 363.07 ± 0.25 Ma, corresponding to the Late Famennian. In the Jura Creek section (Kabanov, 2022), where the Exshaw Fm reaches a thickness of approximately 9 m, a tuff layer (~ 360 Ma) occurs in the lower member, within an interval of intense anoxia interpreted as equivalent to the Hangenberg Event.

At Hasselbachtal, two tuffs (Beds 79 and 70) were dated within the *Siphonodella sulcata* and *S. duplicata* conodont Zones at 360.5 ± 0.8 Ma and 360.2 ± 0.7 Ma, respectively (Trapp et al., 2004). In the Daposhang section (South China), a 5 cm tuff bed – located just above the black shale unit known as Bed E and interpreted as the main phase of the Hangenberg Event – yielded a CA-ID-TIMS age of 360.47 ± 0.68 Ma (Xu et al., 2024). In the Kowala section (Poland), three tuff layers underlying and overlying the Hangenberg Black Shale unit yielded U–Pb ages of 359.97 ± 0.46 Ma, 358.97 ± 0.11 Ma, and 358.89 ± 0.20 Ma (Myrow et al., 2014), corresponding to the Late Famennian. In all regions discussed, researchers attempt to correlate radioisotopic ages with conodont zonation in order to establish stratigraphic boundaries using multiple lines of evidence.

An important feature of the volcanic ash layers encountered in the studied boreholes is their high content of euhedral zircon crystals per unit volume. This enables the extraction of sufficient numbers – several dozens to the first hundreds – of crystals suitable for radiometric dating from layers only a few millimetres thick, as preserved in core material. Similar features have been reported for hydrocarbon source rocks from the Ordovician–Silurian boundary interval in South China (Du et al., 2020, 2021), and from the Jurassic–Cretaceous boundary interval (Bazhenovo Formation) in Western Siberia (Rogov et al., 2023).

The radioisotopic age of 357.6 ± 1.7 Ma, obtained from the tuff layer at the base of the *Siphonodella quadruplicata* Zone, is consistent with global data (Aretz et al., 2020), supporting the short duration (approximately 0.5 Ma each) of the *S. sulcata* and *S. duplicata* Zones – the basal zones of the Tournaisian Stage.

It can be assumed that the *S. belkai* Zone has a similarly short duration, although its presence in the studied sections requires further justification.

Each of the conodont zones discussed above – *S. sulcata*, *S. duplicata* and *S. belkai* – corresponds to a regional stage: the Gumerovian, Malevian and Upian Stages, respectively (Fig. 5; Alekseev et al., 2022).

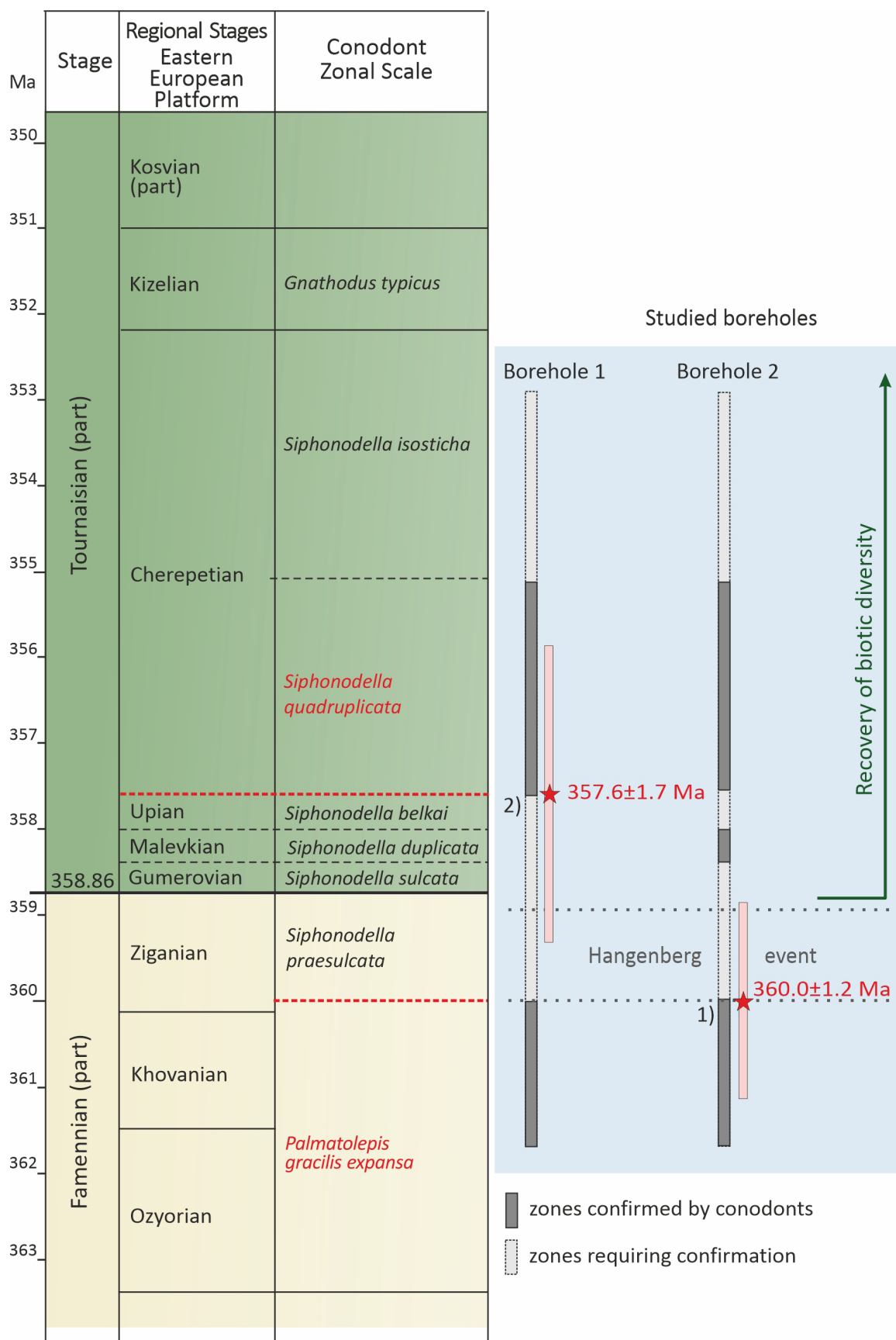


Figure 5. U–Pb LA–ICP–MS ages of the studied samples and their stratigraphic position within the boreholes; asterisks mark dated samples; pink rectangles represent the analytical uncertainty ranges; the position of the Devonian–Carboniferous boundary is given according to the International Chronostratigraphic Chart (2024); conodont zonation follows the schemes of Aretz et al. (2020), Becker et al. (2020), and Alekseev et al. (2022).

The durations of these stages are equal to those of the zones, which is approximately 0.5 Ma. Over such periods, sediment accumulation in deep-water anoxic environments could result in strata only 2.5 to 5 m thick after compaction and diagenesis (De Vleeschouwer et al., 2013). Accordingly, the entire stratigraphic interval corresponding to the Gumerovian, Malevkian, and Upiian regional Stages could be represented in KKTS by a succession of 7.5 to 15 m thick.

Conclusion

For the first time, radioisotopic ages have been obtained for tuff layers from the Devonian–Carboniferous boundary interval within Domanik facies of the East European Platform using the U–Pb LA-ICPMS method. The resulting ages – 360.0 ± 1.2 Ma and 357.6 ± 1.7 Ma – are in good agreement with the International Chronostratigraphic Chart and global data on the Hangenberg Event. These results refine the age constraints of conodont zones and demonstrate the applicability of LA-ICPMS for dating millimetre-scale tuff layers within black shale successions. The study opens up new opportunities for developing a high-resolution chronostratigraphic framework for Devonian and Early Carboniferous Domanik-type source rocks in Eastern Europe by integrating biostratigraphic (conodont) and radioisotopic methods.

Supplementary files

Supplementary file “Age of individual zircon grains determined by LA-ICP-MS” is located at: <https://www.geors.ru/jour/article/view/562>.

Funding/Acknowledgements

This work was partly funded by the subsidy allocated to Kazan Federal University for the state assignment project № FZSM-2023-0023 in the sphere of scientific activities.

We would like to express our sincere gratitude to Professor Alexander S. Alekseev (Lomonosov Moscow State University) and Corresponding Member of the Russian Academy of Sciences Alexander S. Biakov, whose comments and suggestions have significantly enhanced the scientific quality of this article.

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Manuscript received 16 May 2025;
Accepted 20 June 2025; Published 30 June 2025

Первые радиоизотопные U–Pb LA-ICP-MS датировки пограничных отложений девона и карбона в доманиковых фациях Камско-Кинельской системы прогибов (Волго-Уральская нефтегазоносная провинция, Восточно-Европейская платформа)

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Впервые радиоизотопным методом U–Pb LA-ICP-MS датированы цирконы из тонких прослоев вулканического пепла, содержащихся в высокоуглеродистых породах доманиковой фации (нефтематеринские отложения) пограничного интервала девонской и каменноугольной систем. Материал происходит из керна двух скважин, расположенных в осевой и бортовой зонах Камско-Кинельской системы прогибов Волго-Уральской нефтегазоносной провинции. В осевой зоне основание конодонтовой зоны *Siphonodella quadruplicata* имеет конкордантный U–Pb возраст 357.6 ± 1.7 млн лет. В бортовой зоне верхняя часть зоны *Palmatolepis gracilis expansa* имеет конкордантный U–Pb возраст 360.0 ± 1.2 млн лет. Полученные датировки соответствуют, в пределах погрешности анализа, современной хроностратиграфической конодонтовой шкале и позволяют уточнить начало Хангенбергского события в пределах изученного бассейна.

Ключевые слова: Восточно-Европейская платформа, Камско-Кинельская система прогибов, девон, карбон, доманиковые фации, U–Pb LA-ICP-MS датирование цирконов

Для цитирования: Силантьев В.В., Нурғалиев Д.К., Сунгатуллина Г.М., Мифтахутдинова Д.Н., Куликова А.В., Нурғалиева Н.Г., Саетгалеева Я.Я., Вандин А.С. (2025). Первые радиоизотопные U–Pb LA-ICP-MS датировки пограничных отложений девона и карбона в доманиковых фациях Камско-Кинельской системы прогибов (Волго-Уральская нефтегазоносная провинция, Восточно-Европейская платформа). *Георесурсы*, 27(2), с. 297–304. <https://doi.org/10.18599/grs.2025.2.22>