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Age of the coal accumulation in the Irkutsk Basin based on accessory zircon dating in the Azeisk deposit tonstein (LA-ICP-MS)*

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The Irkutsk Coal Basin is located in the south of the Siberian Platform. The deposits of the basin are represented by Jurassic continental sediments. Since the 1970s, tuffs and tuffaceous rocks have been found in sediments of the Irkutsk Coal Basin, but so far only fragmentary information has been available on them. Previously, the age of sediments in the Irkutsk Basin was determined by the biostratigraphic method. We studied a section of the Cheremkhovo Fm., the most ancient formation of the basin, at the Azeisk coal deposit (in the area of the town of Tulun) with the aim of obtaining a geochronological constraints on the timing of sedimentation. The paper shows that tonsteins, which are transformed to clay ash-fall layers within coals, are suitable for radioisotope dating by accessory zircons. U-Pb dating of accessory zircons in tonstein from an industrial coal seam was performed using the LA-ICP-MS method. The obtained U-Pb age of $187.44 \pm 0.45 - 1.60$ Ma, falling into the Pliensbachian Age, indicates the period of maximum coal formation in the Irkutsk Basin. This is the first radiometric age determination for the timing of accumulation of coal in the Irkutsk Basin. In the context of paleogeographic reconstructions, this age determines the upper limit of the tectonic quiescence period, which created conditions for lacustrine-boggy sedimentation in the vast territory of the Irkutsk Basin.

Keywords: Jurassic Period, volcanism, tonstein, Irkutsk coal Basin, accessory zircons, U-Pb dating.

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1. Introduction

The Irkutsk Coal Basin is located in the south of the Siberian Craton (Fig. 1). The deposits of the basin are represented by Jurassic continental-type sediments. A valid regional stratigraphic scale was adopted in 1981, with the distinguishing of the Cheremkhovo (Pliensbachian — early Toarcian), Prisayan (Toarcian — Aalenian) and Kuda (conditionally Aalenian) Fms. (Saks et al., 1981). According to the valid scheme, the studied Cheremkhovo Fm. is divided into three members: Member I, Member II, and Member III. However, according to other studies, the Cheremkhovo Fm. should be considered an indivisible macro-rhythm to the only exclusion of Member III (Panaev, 1968; Panaev and Nikitina, 1970; Skoblo et al., 2001). Skoblo et al. (2001) also propose that members of the Cheremkhovo Fm. should be considered sub-formations. In this paper, we consider the Cheremkhovo Fm. as consisting of two sub-formations — the lower and the upper (Fig. 2).

Despite the large number of publications devoted to studies of the Irkutsk Basin (Adamenko et al., 1971; Akulov et al., 2015; Feinstein, 1971; Kiritchkova et al., 2017; Odintsov, 1967; Skoblo et al., 2001; Timofeev, 1970), questions related to the age and position of the stratigraphic units still remain undetermined (Fig. 2). The age of the Irkutsk Basin sediments was determined by the biostratigraphic method, which gives a rather wide time interval. While the lower limit of the sedimentation onset is determined in the Pliensbachian Age (Akulov et al., 2015; Kiritchkova et al., 2017; Saks et al., 1981; Skoblo et al., 2001), the upper one drifts from the Aalenian to the Bajocian Age (Akulov et al., 2015;

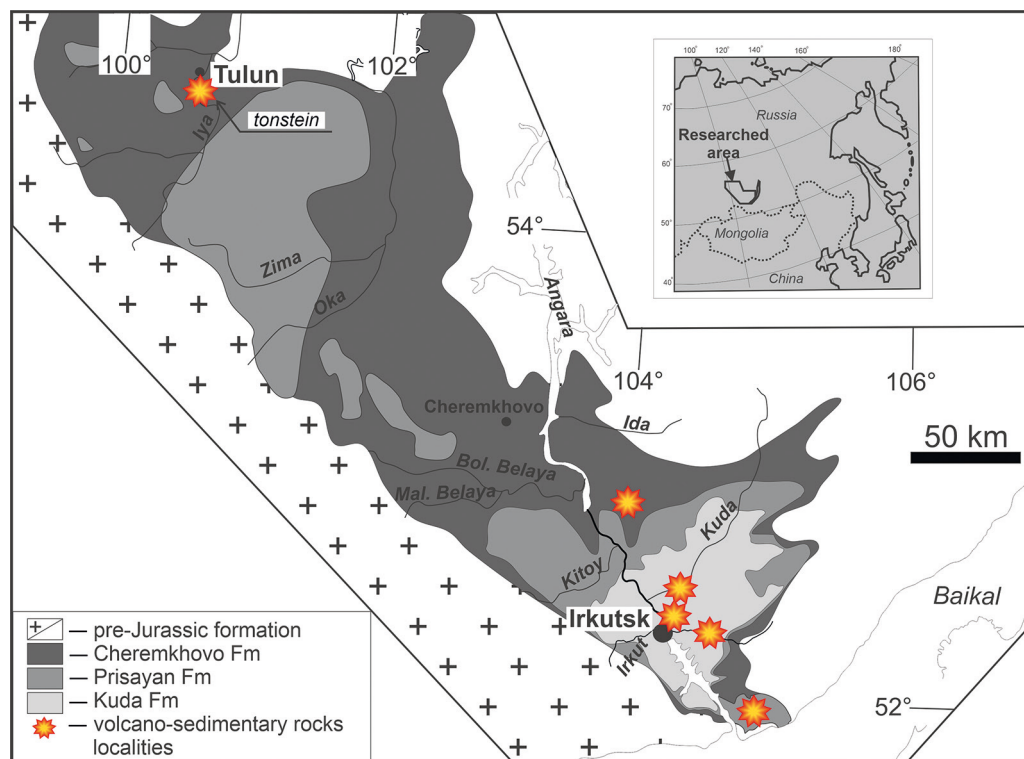


Fig. 1. Simplified geological map of the Irkutsk Basin and volcano-sedimentary rocks localities. Author's drawing

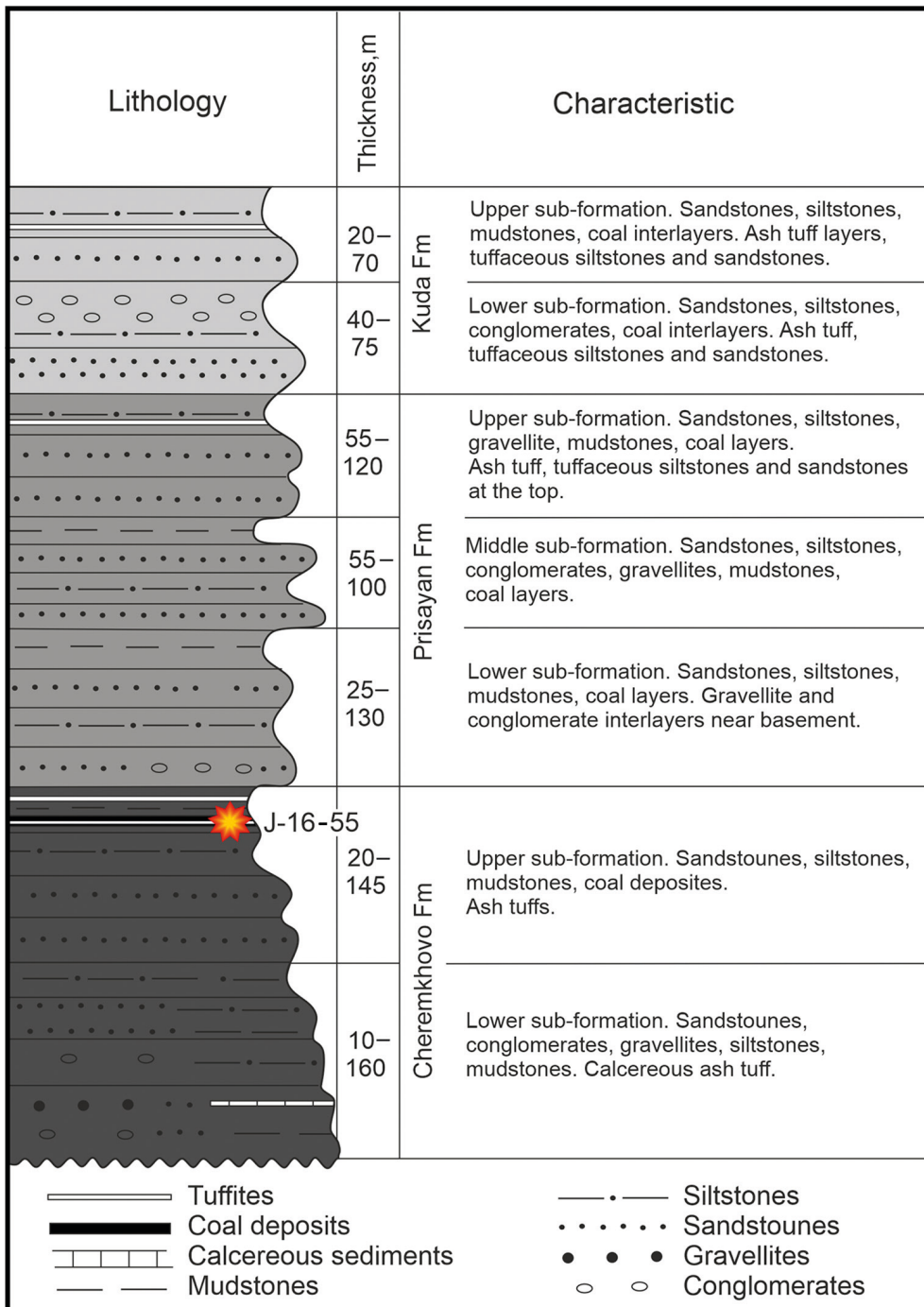


Fig. 2. Composite stratigraphic column for the Irkutsk Basin, modified after (Anonymous, 1981; Skoblo et al., 2001)

Kirichkova et al., 2016; Saks et al., 1981; Skoblo et al., 2001). The age of the sediments was determined mainly by plant residues (Frolov and Mashchuk, 2014; 2018; Kirichkova and Travina, 2000); fauna findings are rare and are mainly related to the lower sub-formation of the Prisayan Fm. (Saks et al., 1981; Skoblo et al., 2001).

At different times, tuffs and tuffaceous rocks were found in the sediments of the Irkutsk Coal Basin (Admakin and Portnov, 1987; Arbuzov et al., 2016; Kiziyarov and Meshalkin, 1978; Meshalkin et al., 1983; Rybakov and Hobotova, 1999; Skoblo et al., 2001). They were first discovered in the 1970s according to drilling results (Kiziyarov and Meshalkin, 1978; Meshalkin et al., 1983). Tuff and tuffaceous rock horizons are observed in the sediments of all formations (Fig. 2). Usually, they occur in the form of lenses, interlayers, and horizons with a maximum thickness of up to 18 m (Skoblo et al., 2001) and are most widespread in the southeastern part of the Irkutsk Basin (Fig. 1). They are represented by vitric tuffs, tuff siltstones, and tuff sandstones; according to geological survey data, there are found mainly in drill cores (Saks et al., 1981). Such rocks attain their greatest thicknesses in the upper part of the Jurassic section, namely, in the Prisayan and Kuda Fms. There is evidence of the presence of tuffaceous interlayers in the Cheremkhovo Fm. sediments (Arbuzov et al., 2016; Kiziyarov and Meshalkin, 1978).

This work investigates an ash interlayer (tonstein) in an industrial coal seam of the Azeisk deposit in the area of Tulun (Fig. 1). The research by Arbuzov et al. (2016) substantiated the volcanic origin of clay layers in coal, which were formed due to the transformation of felsic pyroclastic material (Arbuzov et al., 2016). This paper presents the data of U-Pb dating of accessory zircons from tonstein. The LA-ICP-MS U-Pb dating method was used to estimate the tonstein age, because felsic volcanic ash contains accessory zircon grains in addition to detrital ones. This method makes it possible to analyze a large number of grains, which is important for obtaining a sufficiently large cluster in calculating the age and can be used for dating of ash-fall.

2. Geological description

The Cheremkhovo Fm. is known throughout the limits of the Irkutsk Coal Basin. The formation is composed of terrigenous deposits with coal seams of industrial thickness; the thickness of the formation varies from 30–100 to 180–240 m. Within the basin, three groups of facies are distinguished for the Cheremkhovo Fm.: the alluvial, the alluvial-deluvial, and the floodplain (Odintsov, 1967; Timofeev, 1970). Most of the basin is encompassed by alluvial facies, which are represented in the southeast by foothill alluvium facies (the lower sub-formation). By the end of the accumulation of the lower sub-formation, the distribution contours of foothill alluvium conglomerates become significantly narrower. Alluvial-deluvial facies take the central part of the basin and give place to lacustrine-boggy facies by the end of the upper sub-formation accumulation. Floodplain facies are typical in the northern and northwestern parts of the basin and alternate with channel ones. All industrial coal deposits known in the Irkutsk Basin are associated with the upper sub-formation of the Cheremkhovo Fm. The number of coal seams and interlayers there varies from 1 to 16 individual seams and attains a thickness of 14 m (Cherepovskij, 2002; Skoblo et al., 2001).

In this work, we investigate a section of the Cheremkhovo Fm. at the Azeisk coal deposit located in the area of the town of Tulun (Fig. 1). The deposits here are represented

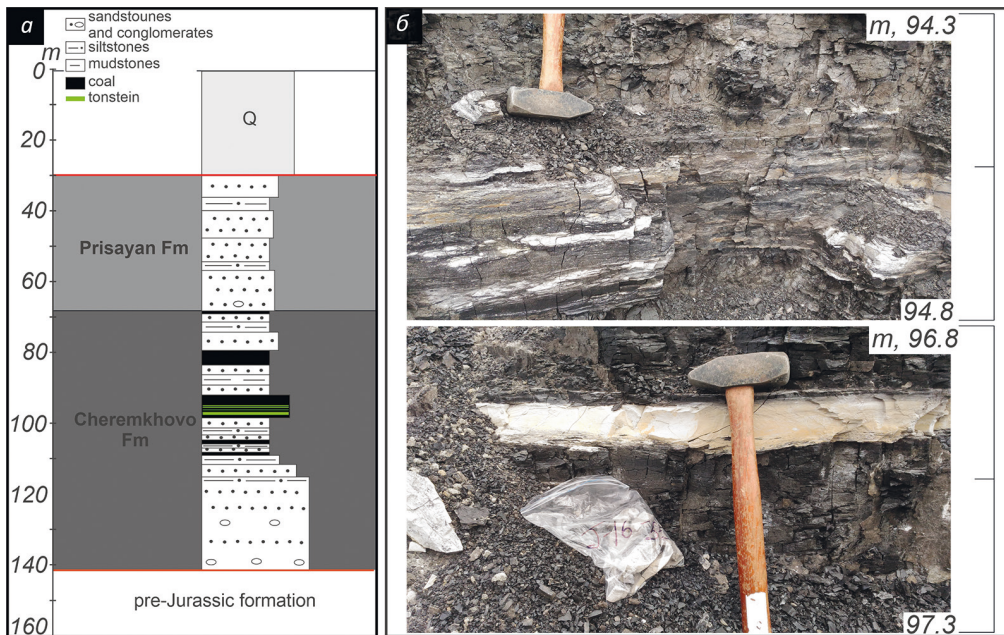


Fig. 3. (a) Composite stratigraphic column for the Jurassic sediments in the area of the Azeisk deposit (Cherepovskiy, 2002; Arbuzov et al., 2016); (b) Photographs of the tonsteins at the Azeisk deposit

by poorly consolidated fine- to medium-grained sandstones, siltstones, mudstones, and coals of the Cheremkhovo Fm. upper sub-formation (Fig. 3, a). Two mature, productive coal seams (II and I) are distinguished in the studied section. Seam II, which is pervasive, is the most mined (its average thickness is ~6 m) (Arbuzov et al., 2016; Cherepovskij, 2002). In Seam II, clay interlayers from white to grayish-white stand sharply apart against the background of coal (Fig. 3, b); they are less than 0.3 m thick and more than 4 km long. These interlayers were studied in detail in the paper by Arbuzov et al. (2016), and it was found that they almost entirely consisted of kaolinite with the participation of quartz, cristobalite, illite, and feldspars; accessory minerals are represented by zircon, monazite, and sulfides. Based on the occurrence conditions, composition, and geochemical features, the authors call these interlayers tonsteins and confirm their pyroclastic nature (Arbuzov et al., 2016).

Deposited in marine conditions, volcanic ash usually transforms into clay deposits known as bentonites (consisting mainly of montmorillonite or illite), whereas the Irkutsk Basin deposits are continental type sediments. The most suitable environment for the conservation of volcanic ash-fall material under continental conditions is shallow lakes, ponds, and swamps (Bohor and Triplehorn, 1993; Spears, 2012), i. e., the coal-forming environment of the Cheremkhovo Fm. is the most favorable. In coal-bearing rocks, layers of volcanic ash mainly form clays known as tonstein, which are characterized by a high content of kaolinite formed as a result of devitrification of volcanic glass fragments (Bohor and Triplehorn, 1993; Spears, 2012). It is generally accepted that tonsteins form as a result of volcanic ash transformation in situ (in swamp or lake environments), which was deposited when air masses transported ash emissions of powerful eruptions over long distances

with the participation of felsic magma (Triplehorn, 1990). It should be noted that at the present time there is a large number of papers, both on identification of tonsteins — in the understanding of their igneous nature (Anggara et al., 2018; Arbuzov et al., 2016; Dai et al., 2011; Jurigan et al., 2019) — and on dating such layers (Ducassou et al., 2019; Guerra-Sommer et al., 2008; Jurigan et al., 2019).

3. Analytical procedures

Material from the tonsteins was studied by X-ray powder diffraction analysis using a DRON-3.0 diffractometer (Burevestnik, Russia) at the Centre for Geodynamics and Geochronology (CGG) of the Institute of the Earth's Crust (IEC) of the Siberian Branch of the Russian Academy of Sciences (SB RAS) (analyst — M. N. Rubtsova). To determine the mineralogical composition, we selected six clay layers from the coal seam being mined that potentially correspond to tonsteins. To determine the mineral composition, the samples were triturated in an agate mortar with alcohol and studied by powder diffraction technique. Acquisition conditions: emission — $\text{CuK}\alpha$, Ni — filter, $V = 25$ kV, $I = 20$ mA, step angle — 0.05° . To identify clay minerals, oriented sample material was prepared by depositing a clay fraction on a glass plate, heating at 550°C for 3 hours, and saturation with ethylene glycol (Brown, 1965). The radiographs were identified using a phase search program. The semiquantitative component ratio was calculated using corundum numbers (RIR values) (Hubbard and Snyder, 1988). The phase proportions obtained are approximate.

Zircon grains were extracted from the most representative tonstein sample (J-16-55) following a classical mineral separation procedure and handpicked under a binocular microscope. After embedding the grains in an epoxy resin, the pucks were polished down to reveal the grain through their centers. The U-Pb zircon dating was performed by LA-SF-ICP-MS in the Analytical Center for Mineralogical, Geochemical, and Isotope Research of the Geological Institute SB RAS (Ulan-Ude). Zircon grains were extracted from the tonstein sample, following a classical mineral separation procedure. Analyzed crystal size varied between 60 and 180 μm . U-Pb dating was performed using ICP-MS Element XR (ThermoFisher Scientific) coupled to an UP-213 laser ablation system (New Wave Research). The laser beam was 40–50 μm in diameter, the fluency was about 3.5 J/cm^2 , the total time of laser radiation was 55 s. Helium was used as a carrier gas; it was mixed with argon. The 91 500, Plešovice and GJ-1 were used as external standards. Primary ages were calculated relative to the 91 500 standard. The measurement data were processed using the GLITTER (Griffin, 2008), IsoplotR (Vermeesch, 2018) and ISOPLOT (Ludwig, 2012) software. The instrumental parameters and measurement technique are described in more detail by Buyantuev et al. (2017) and Khubanov et al. (2016).

4. Results

Among six clay layers analyzed by XRD, only one sample corresponded to “pure” tonstein, mainly composed of kaolinite (up to 95%, Fig. 4). In addition, five clay layers are considered as “mixed” tonsteins, consisting of a mixture of volcanic ashes (represented by kaolinite) and detrital particles (15–70%). These layers present traces of quartz, feldspars

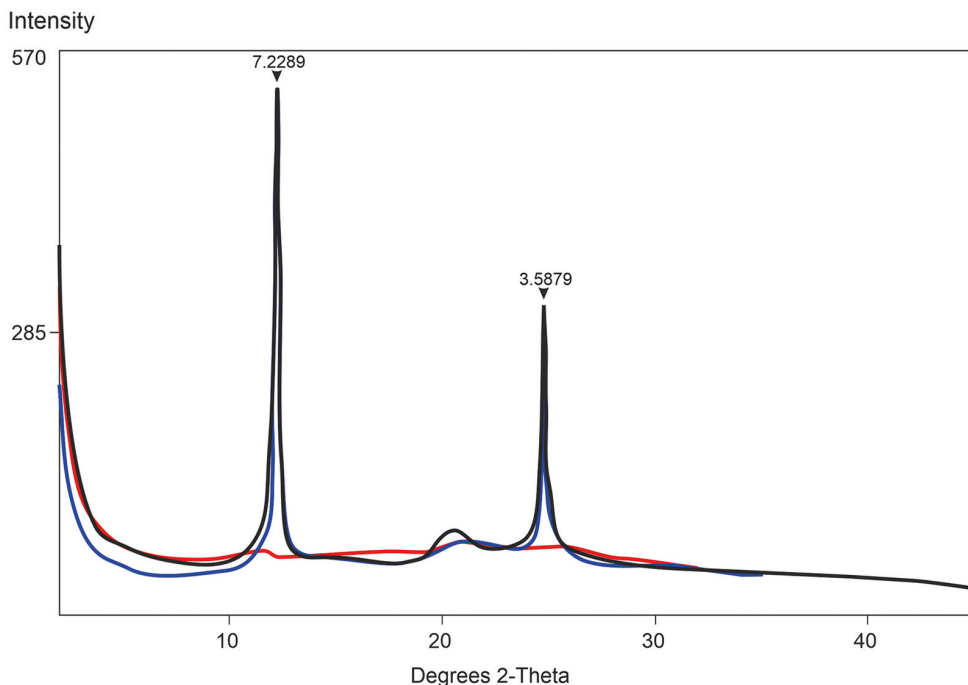


Fig. 4. XRD diffractogram showing a pure tonstein (J-16-55). Black, blue and red lines are for air dry, glycolated solvation and heated conditions, respectively

and illite. The “pure” tonstein was selected for geochronological analysis. Its location is reported in Figs. 1 and 2. In total, 110 zircon grains were analyzed. The size of the zircon grains is small (50 μm on average, up to 150 μm). Most of the grains are euhedral and elongated, less than 30 % are isometric and represented by fragments of elongated grains. Zircon grains are colorless and non-rounded.

Results of U-Pb dating of zircon are shown in Fig. 5. Data from zircon analyses are provided in Supplement 1¹. The first step consists in filtering the data based on their probability of concordance, calculated using IsoplotR (Vermeesch, 2018), by comparing $^{206}\text{Pb}/^{238}\text{U}$ and $^{207}\text{Pb}/^{235}\text{U}$ ratios including the decay constant errors (Ludwig, 1998; Nemchin and Cawood, 2005). The cut-off level applied to filter the data was 10 %. For the next step, only concordant data were considered. Then were calculated concordant age, reduced chi-squared (MSWD), and p-value for concordance for each analysis to assess of the data using IsoplotR (Vermeesch, 2018). Depositional age was calculated as the concordia age using IsoplotR (Vermeesch, 2018).

We used the TuffZirc algorithm (Ludwig and Mundil, 2002), implemented as the “zircon age extractor” tool in Isoplot/Ex 4.15 (Ludwig, 2012). This algorithm was developed to minimize the effects of both inheritance and Pb loss, by isolating the largest cluster of analyses yielding a probability of fit > 0.05, and calculating the median age of this cluster. The uncertainties are asymmetric and provided a 95 % confidence limit. The TuffZirc algorithm was applied to the concordant ages (i. e., obtained using the “concordia” function).

¹ Supplement 1 (supplement are given in the original edition) can be found at the website: <https://escjournal.spbu.ru/article/view/6556/5691>

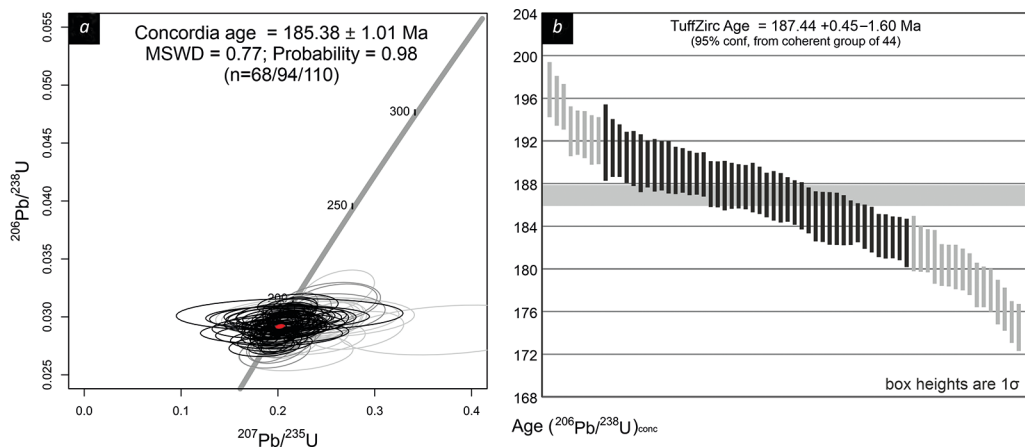


Fig. 5. U-Pb data for zircon grains of the sample J-16-55

(a) Concordia diagram. Age, MSWD and probability are for the concordant grains with discordance of less than 10%. N is for the number of filtered zircon analyses from which concordia age was calculated (shown by black ellipses) / the number of concordant data with the discordance of > 10% (shown by dark grey ellipses) / total number of zircon analyses (light grey ellipses for all other analyses).

(b) Results provided by the TuffZirc algorithm applied to the concordant zircon ages: the data selected and rejected to calculate the TuffZirc dates are shown in black and light grey respectively; the grey area correspond to the TuffZirc date and its calculated error.

In the dated sample, 68 out of 110 analyzed zircon grains are used for calculation (Fig. 5, a). For example, the data spread along the concordia curve and conform to a single population. The depositional age is 185.38 ± 1.01 Ma (MSWD=0.77; Fig. 5, a). The dispersion of data along the concordia curve shows that either zircon grains represent a mixture of different ages or were affected by minor radiogenic lead loss or both and therefore that the depositional age might be slightly older. The TuffZirc algorithm provides a date of $187.44 +0.45 -1.60$ Ma (Fig. 5, b). This value $187.44 +0.45 -1.60$ Ma is taken for the time of a distant volcanic eruption, which leads to formation of an ash-fall layer later transformed into tonstein. Within an error of the age determination, this marks the timing of accumulation of coal-bearing sediments of the upper sub-formation of the Cheremkhovo Fm. of the Irkutsk Basin.

5. Discussion

The pyroclastic nature of the studied clay interlayers is determined, first of all, by their appearance — thin, well lithified layers, almost white (Fig. 3, b), and of great length, up to 4 km. To ensure that the obtained U-Pb age corresponds to the age of sedimentation, it must be ascertained that the volcanic ash was not reworked and no detrital zircon was introduced. The mineral and chemical composition of Azeisk tonsteins has been studied in detail (Arbuzov et al., 2016; Ilyenok, 2018). The rock-forming minerals in tonsteins are represented by kaolinite, quartz, cristobalite, illite, and feldspars, and the accessory minerals are represented by zircon, monazite, baddeleyite, bastnasite, uranium oxide minerals, as well as by native minerals of gold, silver, nickel, zinc, tungsten, silicon, and intermetallic compounds in insignificant concentrations (Arbuzov et al., 2016). Thus, the authors

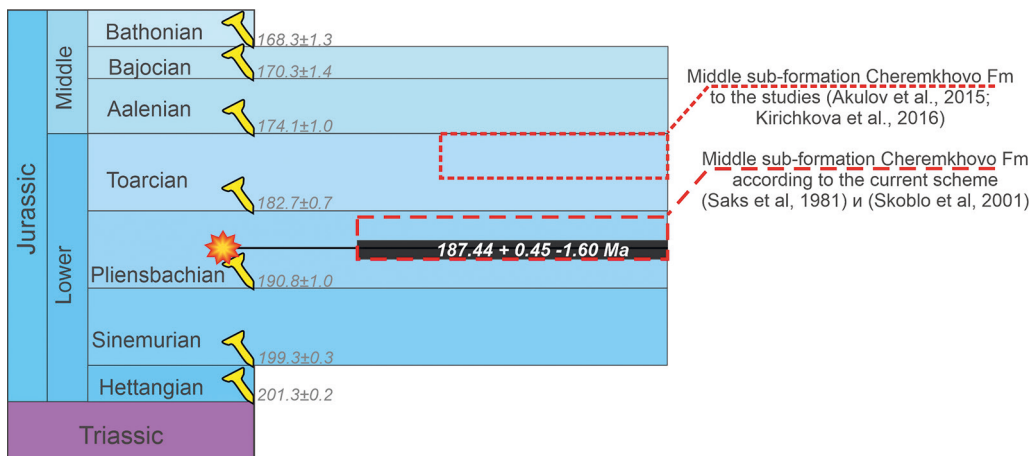


Fig. 6. U-Pb age of the accumulation of the coal-bearing sediments in the Irkutsk basin based on accessory zircon dating in the Azeisk deposit tonstein in comparison with recent studies (Skoblo et al., 2001; Akulov et al., 2015; Kirichkova et al., 2016). Preliminary data on calculated U-Pb age for the lower and upper limits of sedimentation in the Irkutsk Basin are indicated

confirm the pyroclastic nature of the initial matter of the tonsteins, and, based on the data obtained, indicate the felsic (rhyolite) composition of the initial igneous material (Arbuzov et al., 2016).

The upper Cherekhovo Fm. marks the lacustrine-boggy sedimentation in the vast territory of the Irkutsk Basin. It was established that there was coarsening of sediments upward through the Prisayan and Kuda Fm. Such upward coarsening, together with an increase of $^{143}\text{Nd}/^{144}\text{Nd}$ ratios and an increase of the Jurassic-age population of detrital zircon grains, was associated with the closure of the Mongol-Okhotsk Ocean, the collision of two continental blocks and an orogenic belt formation (Arzhannikova et al., 2020; Demonterova et al., 2017). Within this geodynamic scheme, the age of the upper Cherekhovo Fm. places the upper limit of the tectonic quiescence period before the intensification of tectonic processes in the southern mobile framing of the Siberian Platform.

In the context of regional stratigraphy, the obtained age of 187.44 Ma is consistent with the valid stratigraphic scheme (Saks et al., 1981) and the research of Skoblo et al. (2001) that supplements it (Fig. 6). An alternative age for the Cherekhovo Fm. was proposed by Akulov et al. (2015) and Kirichkova et al. (2016; 2017). In these papers, the age of the upper sub-formation of the Cherekhovo Fm. falls into the Toarcian (Fig. 6). However, this contradicts the U-Pb dating of tonstein provided in this study. It should be noted that the stratigraphy of the Jurassic Age of the Irkutsk Basin is mainly based on palynological data (Saks et al., 1981; Skoblo et al., 2001), and recent papers (Akulov et al., 2015; Kirichkova et al., 2016; Kirichkova et al., 2017) are the result of phytostatigraphic research. Thus, palynological data provide more robust constraints on the age of the Jurassic continental deposits, at least for the Irkutsk Basin.

6. Conclusions

The age of coal-bearing deposits of the Cheremkhovo Fm. represented at the Azeisk coal deposit was established as ~187 Ma using the LA-ICP-MS U-Pb method on accessory zircons from tonstein. This is the first radiometric age determination for the timing of accumulation of coal-bearing sediments in the Irkutsk Basin.

In the context of paleogeographic reconstructions, this age determines the upper limit of the tectonic quiescence period, which created conditions for lacustrine-boggy sedimentation in the vast territory of the Irkutsk Basin. The coarsening of sediments in the overlying Prisayan and Kuda Fm. indicates the intensification of relief formation processes on the border with the Siberian Craton associated with the formation and development of the Mongol-Okhotsk orogenic belt.

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Возраст угленакопления в Иркутском бассейне по данным датирования акцессорных цирконов из тонштейна Азейского месторождения (LA-ICP-MS)*

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Иркутский угольный бассейн расположен на юге Сибирской платформы. Отложения бассейна представлены юрскими континентальными осадками. Еще начиная с 1970-х годов в отложениях бассейна обнаружены вулканогенно-осадочные породы, по которым в настоящее время имеются лишь обрывочные сведения. Ранее возраст отложений Иркутского угольного бассейна устанавливали по биостратиграфическим данным, которые дают довольно широкий временной интервал. Нами изучен разрез черемховской, самой древней юрской свиты Иркутского бассейна на Азейском угольном месторождении (район г. Тулун) с целью получения геохронологических ограничений времени осадко-накопления. В работе показано, что тонштейны Иркутского бассейна, преобразованные в каолинит слои вулканического пепла в углях, пригодны для радиоизотопного датирования по акцессорным цирконам. Датирование U-Pb-акцессорных цирконов в тонштейне из промышленного угольного пласта было выполнено с использованием метода LA-ICP-MS. Полученный возраст $187.44 \pm 0.45 - 1.60$ млн лет, приходящийся на плинсбах, указывает на период максимального накопления угля в Иркутском бассейне. Это первое радиометрическое определение возраста для времени накопления промышленных угольных пластов в Иркутском бассейне. В отношении региональной стратиграфии полученный возраст согласуется с действующей с 1981 года стратиграфической шкалой и дополняющими ее исследованиями В.М. Скобло, основанными на палинологических данных. Работы, базирующиеся на фитостратиграфических исследованиях, предлагают более молодые оценки (тоар) времени максимального угленакопления в Иркутском бассейне. В контексте палеогеографических реконструкций возраст тонштейна определяет верхнюю границу тектонического периода покоя, что создало условия для озерно-болотных условий седиментации на обширной территории Иркутского бассейна.

Ключевые слова: юрский период, вулканизм, тонштейн, Иркутский угольный бассейн, акцессорные цирконы, U-Pb-датирование.

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