High Resolution ID-TIMS Redefines the Distribution and Age of the Main Mesozoic Lacustrine Hydrocarbon Source Rocks in the Ordos Basin, China

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Abstract: Using high-precision zircon U-Pb ID-TIMS geochronology, tuffs from the Chang 9 shale and the Chang 7 shale were dated. The tuff in the Chang 9 shale is 241.47 ± 0.17 Ma, which falls between the top tuff age of 241.06 ± 0.12 Ma and the bottom tuff age of 241.558 ± 0.093 Ma in the Chang 7 shale. These reveal that the Chang 9 and Chang 7 shales are contemporaneous, belonging to the Ladinian stage of the Middle Triassic. This insight expands the region of the main source rock of Chang 7 to the northeast and will inform the search for the deep Chang 9 shale petroleum system, increasing the scope for exploring the Chang 7 shale system in northern Shaanxi. The research results clarify the relationship between the two sets of shale in the Yanchang Formation and redefine the distribution range of the Chang 7 shale in the Ordos Basin. At the same time, it shows that there is a cross-layer problem in the stratigraphic division of the Yanchang Formation in different regions, the high-precision U-Pb dating technology providing a reference for the fine stratigraphic correlation of other continental basins in the world.

Key words: ID-TIMS, high precision age, redefining distribution, Chang 7 shale, Chang 9 shale, Ordos Basin

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1 Introduction

The Ordos Basin, as the second largest petroliferous basin in China, contains rich oil and gas resources, oil shale and sandstone-type uranium resources. Since 2013, the Ordos Basin has achieved both high and stable oil and gas production of more than 50 Mt for four consecutive years, becoming the largest oil and gas producing basin in China (Yang et al., 2013; Zhu et al., 2019). The crude oil of the Triassic Yanchang Formation and the Jurassic strata in the Ordos Basin is primarily derived from the main source rocks of the Chang 7 member (Zhang et al., 2006; Hanson et al., 2007). Since the upper member of the Chang 9 oil formation was first revealed as a black shale with high-quality source rock attributes, it has been generally believed that the Chang 7 shale is distributed in the central and southern part of the basin, while the Chang 9 shale is only locally developed in Ansai–Zhidan, in northern Shaanxi (Fig. 1) (Zhang et al., 2007; Yao et al., 2018). Recently, because it is difficult to completely distinguish the petrology, trace element geochemistry and organic biomarkers of the source rocks, the accumulation contribution and hydrocarbon supply range of the Chang 9 and Chang 7 source rocks have been challenging researchers. Although Chang 9 shale is obviously different from Chang 7 oil shale (shale with abnormal enrichment of TOC, usually up to 20%), it has similar characteristics to the black mudstone associated with Chang 7 oil shale (also a high-quality hydrocarbon source rock, with TOC usually close to 6%). For example, they all have the characteristics of abundant rearranged hopanes, low normal C_{30} hopanes, high clay mineral content and low strawberry-like pyrite content. Therefore, the Chang 7 and Chang 9 source rocks and the generated petroleum are difficult to distinguish mineralogically and geochemically (Zhang et al., 2007; Duan et al., 2009; Li et al., 2012). The present authors put forward a hypothesis: if the characteristics of the two sets of source rocks are very similar, is the existing stratigraphic correlation incorrect? In fact, unlike marine strata, stratigraphic correlation in terrestrial strata is a world-class challenge (Galloway, 1989; Tong et al, 2019). Fortunately, both Chang 7 and Chang 9 source rocks in the Ordos Basin developed zircon -rich layered tuffs, which provided dating materials for determining their development times (Qiu et al., 2009; Zhang et al, 2009; Cui et al., 2019a).

However, the age test results of the Chang 7 shale range from 220 to 241 Ma. The dating methods included ICP-MS, SIMS and ID-TIMS. For example, Deng et al. (2009) used LA-ICP-MS to carry out in situ zircon U-Pb dating and obtained the ages of the upper–middle and bottom

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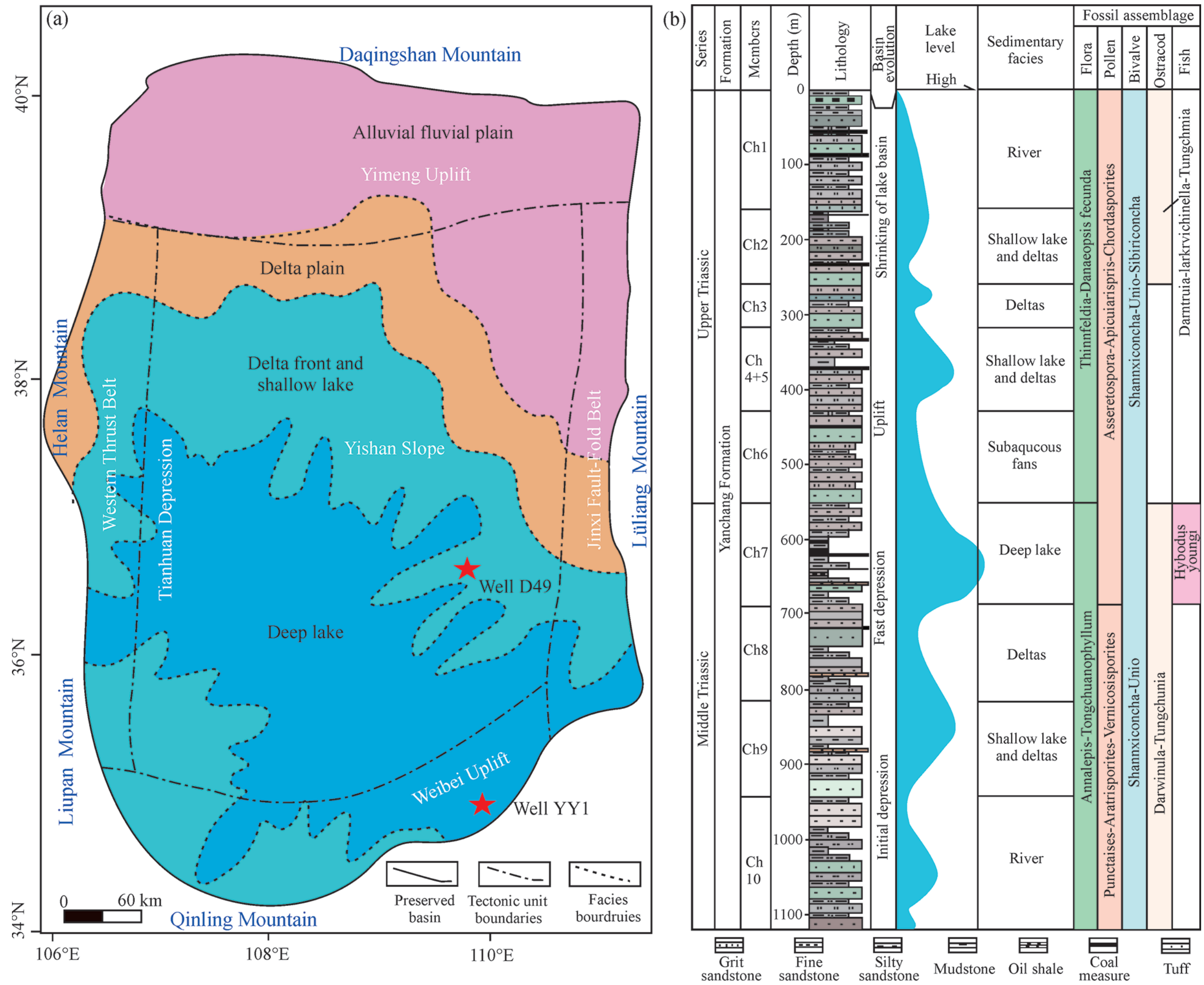

Fig. 1. (a) Extent of the Ordos Basin and sedimentary facies distribution of the Ch7 Member of the Yanchang Formation (modified from Zou et al., 2012); (b) representative stratigraphic column of the Yanchang Formation in the Ordos Basin (modified from Deng et al., 2018).

parts of the Chang 7 member as 221.8 ± 2.0 Ma and 228.2 ± 2.0 Ma, respectively. Zhang et al. (2009) used LA-ICP-MS to carry out in situ zircon U-Pb zircon dating on the thin-layer tuff of the Chang 7 oil shale, obtaining ages of 242–220 Ma and 220–205 Ma. Zhang et al. (2014) used LA-ICP-MS in the same laboratory to carry out zircon dating of the Chang 7 member tuff, the results revealing an age of 234–236 Ma. Wang et al. (2014) used SHRIMP to determine that the zircon U-Pb age of Chang 7 is 239–241 Ma. Zhu et al. (2019) used ID-TIMS to carry out zircon dating on the outcrop of the Chang 7 member tuff, which is located in Tongchuan, obtaining an age of 241.558 ± 0.093 Ma for the bottom and 241.06 ± 0.12 Ma for the top. Zhao et al. (2020) used ID-TIMS to date three tuff outcrops, located in Tongchuan, as 240.1–242.1 Ma.

This study has three research objectives. Firstly, through the ID-TIMS high-precision dating method, to accurately determine the development age of the Chang 9 source rock in northern Shaanxi and the Chang 7 source rock in the southeast of the basin. Then, to discuss the relationship of Chang 9 source rocks and Chang 7 source rocks. Finally, to establish the Triassic stratigraphic age in the Ordos Basin, which will provide an age scale for studying the formation and evolution of the basin, the evolution of its climate and the interaction between organisms and the environment.

2 Geological Background

The Ordos Basin as the second largest sedimentary basin in China, is a typical superimposed basin with an area of 25 × 10^4 km^2. The Paleozoic component is a marine and marine–continental interaction craton basin, the Mesozoic component being a continental lake basin. It is adjacent to Lüliang Mountain to the east, Qinling Mountain to the south, Liupan Mountain and Helan Mountain to the west and Daqingshan Mountain to the north (Fig. 1a).

The Triassic sediments have been transformed from Carboniferous–Permian marine–continental transitional facies to continental facies. The Yanchang Formation records the evolutionary history of the Ordos Lake Basin from occurrence and development to extinction. The strata were divided into 10 oil-bearing formations, referred to as Chang 1 (Ch1) to Chang 10 (Ch10), from top to bottom (Yang et al., 2013; Cui et al., 2019b). The lake basin began to develop in the Chang 10 period and expanded in the Chang 9–8 period. Chang 7 was the peak period of lake basin development. The lake basin began to shrink in the Chang 6 period, expanded briefly in the Chang 5–4 period, then shrank again during the Chang 3–1 period (Fig. 1b). It is generally considered that the sedimentary period of the Chang 7 oil formation is the largest lake flooding period in the lake basin. In the south of the Ordos Basin, a sedimentary system dominated by deep lake and semi-deep lake facies was formed, a large area of high-quality source rocks becoming developed. The lithology is mainly black shale and dark mudstone, known as ‘Zhangjiatan shale’. The Chang 9 member was formed in a small-scale basin lake. The thick, dark mudstone, known as ‘Lijiapun shale’, is only developed in Yingwang, Zhidan and other local areas (Zhang et al., 2007) (Fig. 2). During the development of the lake basin, regional tectonic activity was very strong, with earthquakes, volcanoes and hot water having substantial effects (Zhang et al., 2009, 2010; Zou et al., 2019). In the Yanchang Formation of the Ordos Basin, the tuff interlayers are widely distributed, with a generally NW trend. The petrochemical composition analysis showed that the content of SiO_2 is high (53%) and they are mainly neutral to acidic (Qiu et al., 2009; Zhang et al., 2009; Cui et al., 2019a). This provides good conditions for high-precision stratigraphic dating.

The tuffs are present in cores from the most developed mudstones of the Chang 7 and Chang 9 members, which are located to the southwest and northeast of the basin, respectively. The authors studied the tuff in the Chang 7 member at well YY1 in the southwest of the Ordos Basin, and at well D49 in Zhidan (Fig. 2a). YY1 is a parameter well drilled by the authors, as part of the project titled ‘Formation Mechanism and Enrichment Law of Continental Tight Oil (shale oil) in China’. The coring length of Chang 7 in well YY1 is about 60 m, the TOC value is 3%–30% and the thickness of the tuff is 15 cm. The detailed geochemical histogram is shown in Fig. 2b. The coring length of the Chang 9 shale in well D49 is about 24 m and the thickness of the tuff is 10 cm. The detailed geochemical histogram is shown in Fig. 2c. Vertically, the organic matter abundance and hydrocarbon generation potential of the Chang 9 black shale in well D49 are both high, the geochemical parameters having certain differences. However, TOC is distributed in the range 1.19%–8.64%. On the basis of asphalt ‘A’, rock pyrolysis analysis S1, pyrolysis hydrocarbon generation potential (S_1 + S_2), rock pyrolysis T_{max} and measured kerogen vitrinite reflectance (*R*o: 0.92%–1.05%), it is considered to be a high-quality source rock (Zhang et al., 2007; Zhang et al., 2008).

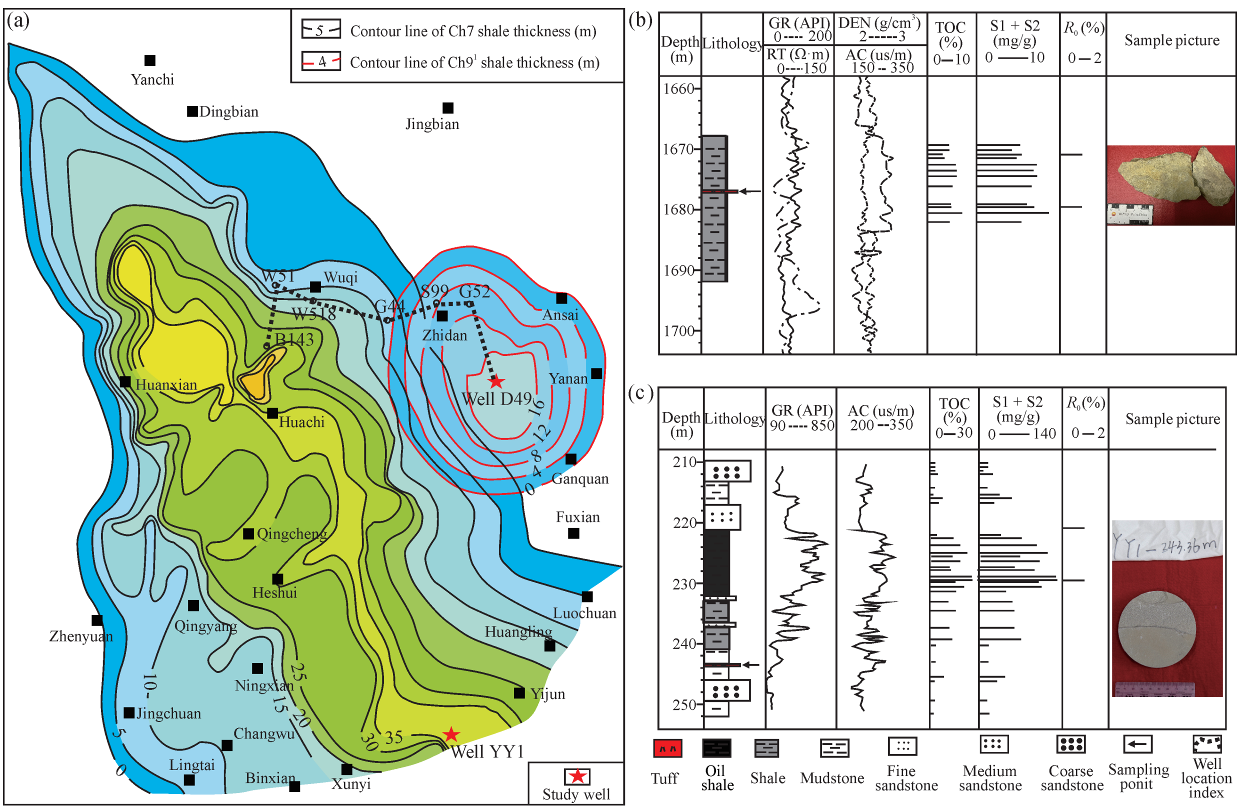

Fig. 2. Locations and information from the study wells.

(a) Current distribution thickness of Ch7 and Ch9 oil shale in the Ordos Basin and location of the study wells; (b) lithology and source rock geochemical parameters of well D-49; (c) lithology and source rock geochemical parameters of well YY1. Distribution thickness of Ch7 and Ch9, and geochemical parameters of well D-49, are modified from Zhang et al. (2008, 2009).

3 Experimental Methods

This experiment was performed at the MIT Isotope Laboratory (Massachusetts Institute of Technology, USA). The zircon and other U-bearing silicates were separated from bulk rock samples by standard crushing, heavy liquid and magnetic separation techniques, subsequently being handpicked under a binocular microscope, the selection being made on the basis of their relative clarity and crystal morphology. To overcome the effects of radioactive decay -induced crystal defects and associated lead loss, which would result in discordant analyses, the zircon grains were pretreated by using the thermal annealing and chemical leaching method, or by CA-TIMS (Mattinson, 2005). This method involves heating zircon in a furnace at 900°C for 60 h. The annealed grains were subsequently loaded into FEP Teflon® microcapsules and leached in concentrated HF at 210°C in high-pressure vessels for 12 h. The partially dissolved samples were then transferred into Savillex® FEP beakers for rinsing. The leached material was decanted with several milliliters of ultra-pure water and flushed successively with 4 mol/L HNO_3 and 6 mol/L HCl on a hot plate and/or in an ultrasonic bath. After a final rinse with ultra-pure water, the zircon grains were loaded back into their microcapsules, spiked with a mixed ^{205}Pb-^{233}U-^{235}U tracer solution and dissolved completely in concentrated HF at 210°C for 48 h. In essence, the process preferentially removes the high-U parts of the zircon crystals that are associated with Pb-loss, leaving a residue of relatively low U content (Jaffey et al., 1971). After extensive testing, it has been concluded that this method is the best way to obtain the most concordant analyses.

4 Results

Scanning electron microscopy and CL imaging showed that the zircon sizes were in the range 200–250 µm, with a completed crystal form, the ring band being uniform and obvious. In this study, high-precision zircon ID-TIMS U-Pb age determination of the tuff in the lower member of the Chang 7 shale at well YY1 resulted in a date of 241.36 ± 0.12 Ma (Fig. 3a). The breakthrough result is that the zircon age of the tuff (1676.5 m) in the lower member of the Chang 9 shale at well D49 was 241.47 ± 0.17 Ma (Fig. 3b).

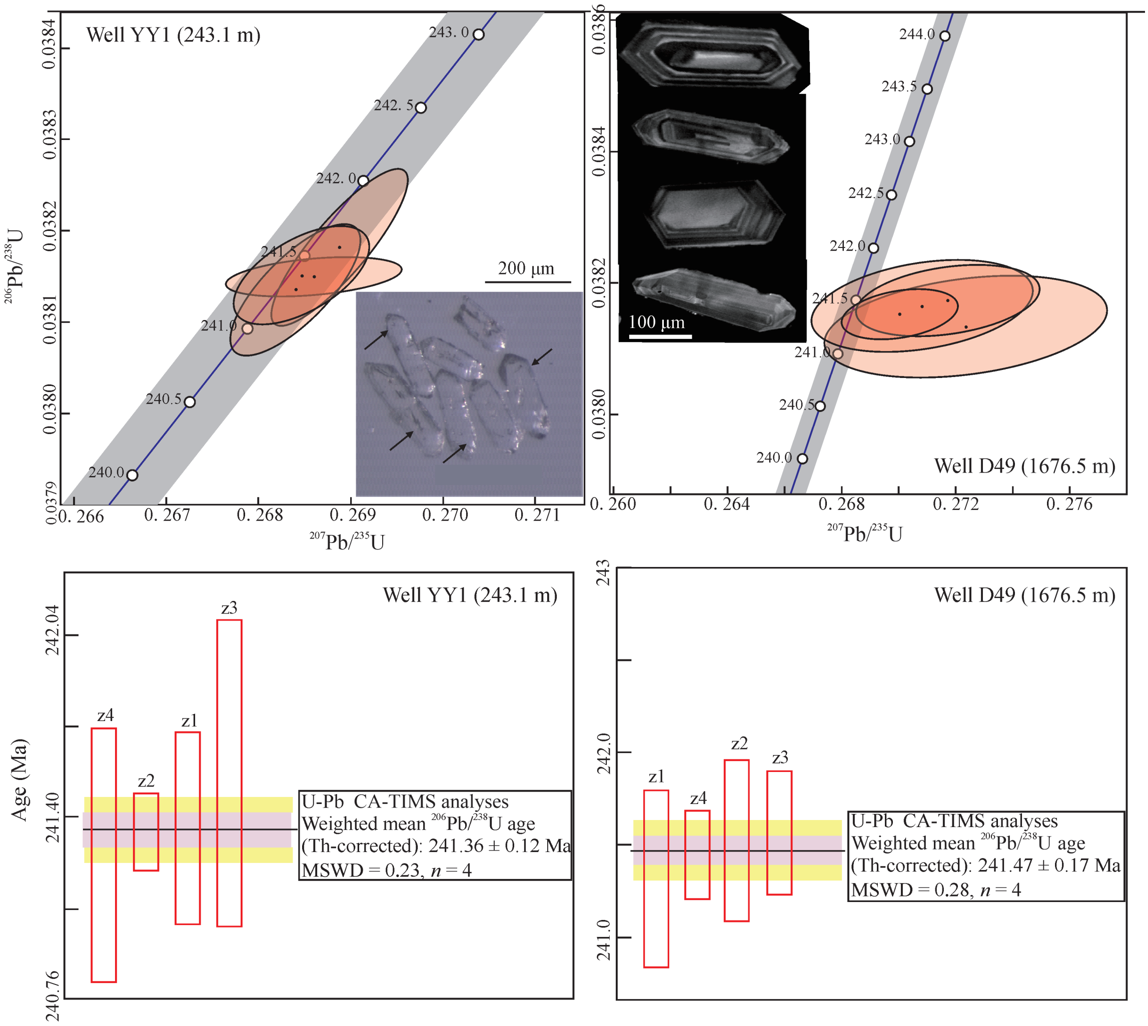

Fig. 3. Left: data of tuff zircon U-Pb ID-TIMS from the lower part of Ch7 (243.1 m), well YY1; right: data of U-Pb ID-TIMS from tuff zircon in central Ch 9 (1676.5 m), well D49.

5 Discussion

5.1 Age assignment of Chang 7 and Chang 9

These two ages are very similar, both being in the range of 241.06 ± 0.12 Ma–241.558 ± 0.093 Ma (Zhu et al., 2019). This implies that the traditional division of strata, including Chang 9 in northern Shaanxi, is wrong. In fact, the development time of the Chang 9 shale in northern Shaanxi is completely consistent with that of the Chang 7 shale in Tongchuan and they should be considered as syndepositional. The real Chang 9 source rock should be deeper and may be classified into either the lower part of the Chang 9 or the Chang 10 Formation. Therefore, it is considered that the upper and lower strata of Chang 7 may have the wrong allocation of age, the stratigraphic correlation in different regions thus having a risk of dislocation. It is suggested that further dating tests and evidence relating to petrology, paleontology, stratigraphy and sedimentary facies should be gathered, in order to improve stratigraphic correlation in the different areas.

According to the chronology and literature published by the International Commission on Stratigraphy (Cohen et al., 2022), the ages of the Chang 9 and Chang 7 shales in northern Shaanxi and Tongchuan should belong to the Middle Triassic Ladinian stage (237–242 Ma) (Table 1). In fact, most of the data in recent years have been clustered around 240 Ma, with paleontology also supporting the above division results (Wang et al., 2014; Deng et al., 2018; Zhu et al., 2019). Recently, dating of tuffs in three outcrops of Chang 7 in Tongchuan has also revealed a range of 240.1–242.1 Ma (Zhao et al., 2020), which further supports the age consensus here.

5.2 Redefinition of Chang 7 source rock distribution

According to the current stratigraphic division results of the Yanchang Formation in northern Shaanxi, the Chang 7 source rock is mainly developed in the Huachi region in the central part of the lake basin, showing high natural gamma characteristics (Fig. 4). Although Chang 7 becomes thinner near well Gao 44 in the Wuqi region, the Chang 7 source rock is still developed. In northern Shaanxi, at well Shun 99 in the Shunning region, together with wells Gao 52 and D49 in the Xihekou region, a set of mudstones developed at the top of Chang 9. According to the dating of tuff in the Chang 9 Formation at well D49, the shale at the top of Chang 9 is classified to be the same as the shale at the bottom of Chang 7 (Chang 7^3). After redefinition, it has been determined that the Chang 7 source rocks are also distributed in northern Shaanxi, consistent with the Huaqing and Wuqi regions in the central part of the lake basin; this also confirms the rationale of the stratigraphic re-division. Therefore, according to the high-precision ID-TIMS dating of tuff in the Chang 9 source rock at well D49, the distribution area of the Chang 7 source rock can be extended to northern Shaanxi and it is considered that the current stratigraphic division in northern Shaanxi is wrong.

The strata of the Yanchang Formation in northern Shaanxi needs to be re-correlated in light of this information and the sedimentary facies need to be rebuilt. This new insight not only moves the Chang 7 deep lake and semi-deep lake facies to Jingbian and Zhidan, but also transforms the northeast sedimentary environment from river and delta to semi-deep lacustrine and lacustrine facies during the development of the Chang 7 shale (Fig. 5). The lake area expands to the northeast, increasing the distribution area of the Chang 7 shale by 20% and expanding the oil and gas exploration scope of the Chang 7 source rock (Fig. 5). At the same time, it will guide exploration for the real Chang 9 source rock, along with evaluation of its effective distribution area and source rock characteristics. The outstanding question as to whether or not there is a real Chang 9 source rock in northern Shaanxi also requires further study.

The re-dating of the Chang 9 shale in northern Shaanxi will therefore become the focus of hydrocarbon exploration in this region. In addition, the new stratigraphic attribution and division will change our understanding of the prototype basin, sedimentation and paleoenvironment in northern Shaanxi in the Chang 7 period. It will also play an

Table 1 Division Scheme of the Chang 7 Stratum of the Yanchang Formation in the Ordos Basin

System	Series	International stage[a]	Age[a] (Ma)	Formation[b]	Member[b]	Oil reservoir formation[b]	Thickness[b] (m)	(ID-TIMS) dating (Ma)	This paper
Triassic System	Upper Triassic	Rhaetian	201.4 208.5	Yanchang Formation	T_3y^5	Chang 1	0–240		Rhaetian
		Norian	208.5 227.0		T_3y^4	Chang 2	120–150		Norian
						Chang 3	90–110		
		Carnian	227.0		T_3y^3	Chang 4 and Chang 5	80–90		Carnian
						Chang 6	110–130		
			237.0			Chang 7	100–120	241.06 ± 0.12–241.558 ± 0.093[c] 241.36 ± 0.12 (YY1)	Ladinian
	Middle Triassic	Ladinian	237.0	Tongchuan Formation	T_3y^2	Chang 8–Chang 9	75–90–80–110	241.47 ± 0.17 (D49)	
			242.0		T_3y^1	Chang 10	210–350		Anisian
		Anisian	242.0 247.2	Zhifang Formation					
	Lower Triassic	Olenekian	247.2 251.2	Heshanggou Formation					Olenekian
		Induan	251.9 251.902	Liujiagou Formation					Induan

Note: (a) international stage and absolute age are based on the ICS International Chronostratigraphic Chart 2022/10; (b) formation and thickness are based on Deng et al. (2009) and Wang et al. (2014); (c) data from Zhu et al. (2019).

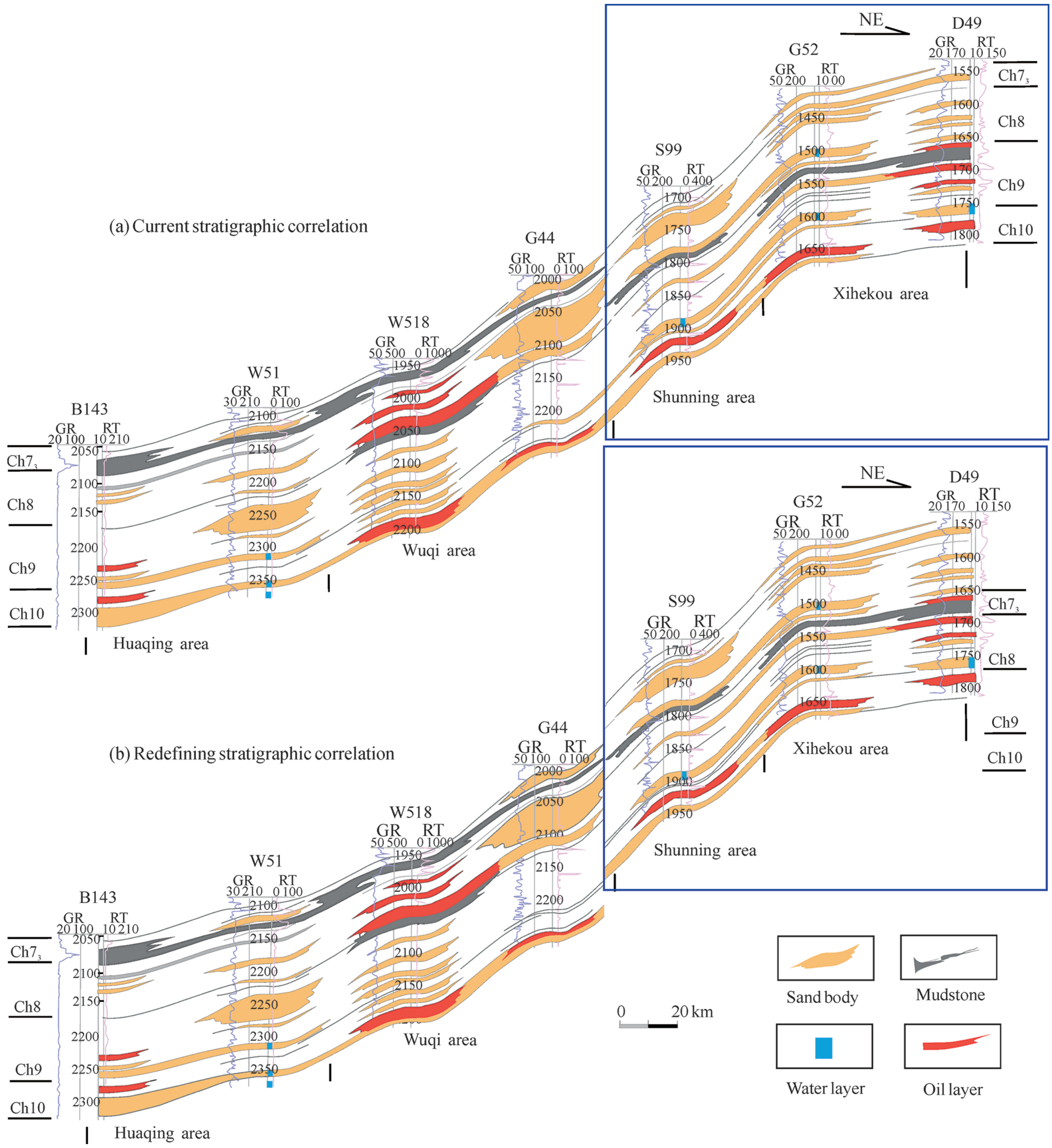

Fig. 4. Before and after stratigraphic redetermination in the Huachi–Wuqi–Shunning–Xihekou area, Ordos Basin (see Fig. 2 for locations of the wells).

important role with regards to scientific research based on the recovery of lower organisms. According to the new chronological data, the formation age of the Chang 7 source rock is redefined as the early Middle Triassic and it will be recognized as the major continental source rock for the earliest large-scale reservoir formation subsequent to the end-Permian mass extinction (252 Ma). The age data obtained in this study will provide an important time scale for the study of paleoclimate and the biological evolution of continental strata.

6 Conclusions

Using high-precision zircon ID-TIMS U-Pb dating, two important insights have been obtained for the the Chang 9 shale in northern Shaanxi and the Chang 7 shale in Tongchuan, southeast Shaanxi.

(1) The formation time of the Chang 9 shale in northern Shaanxi is consistent with that of the Chang 7 oil shale in the Tongchuan region. It is considered that the Chang 9 shale in northern Shaanxi is not in fact really Chang 9 oil shale, but Chang 7 oil shale. The area of the main source

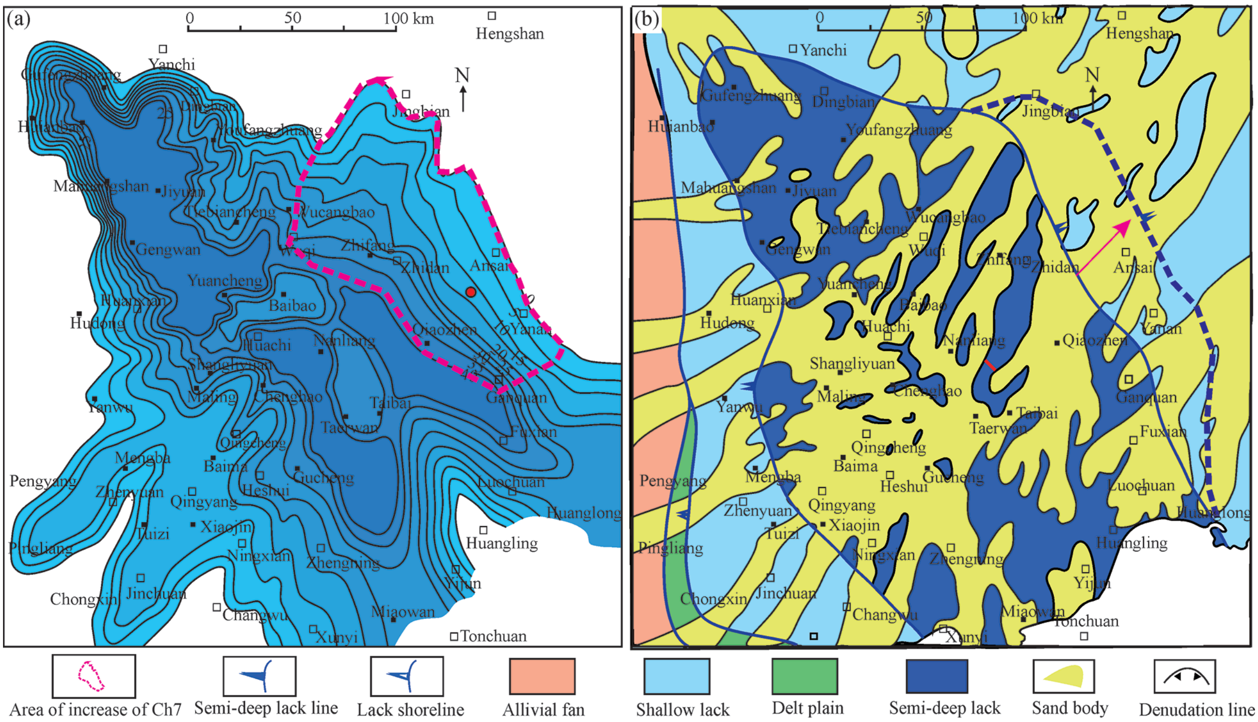

Fig. 5. The distribution range of the revised Chang 7 source rocks and the revised Chang 7 sedimentary facies.

rock for Chang 7 is thus expanded to the northeast, along with the oil and gas exploration prospects associated with it. This will guide exploration for the real Chang 9 source rock at greater depths.

(2) Both the Chang 7 source rock in the Tongchuan region and Chang 9 in northern Shaanxi belong to the Middle Triassic Ladinian stage, which will now be regarded as the earliest known continental source rock with large scale in the world after the end-Permian mass extinction (252 Ma). The age data will provide an important time scale for the study of paleo-climate and biological evolution in the continental strata.

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