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# The final closure time of the eastern segment of the Paleo-Asian Ocean: Insights from geochronology and geochemistry of Permian-Triassic sedimentary sequence in Wangqing, Jilin Province, China

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#### Abstract

The Central Asian Orogenic Belt is the world's largest accretionary orogenic belt, associated with the closure of the Paleo-Asian Ocean (PAO). However, the final closure timing of the eastern PAO remains contentious. The Permian-Triassic sedimentary sequences in the Wangqing area along the Changchun-Yanji suture zone offer important clues into this final closure. New data on petrology, whole-rock geochemistry, zircon U-Pb geochronology and zircon Hf isotopes of sedimentary rocks from the Miaoling Formation and Kedao Group in Wangqing area provide new insights into the final closure of the eastern end of the PAO. The maximum deposition ages of the Miaoling Formation and Kedao Group have been constrained to the Late Permian (ca. 253 Ma) and early Middle Triassic (ca. 243 Ma), respectively. These sedimentary rocks exhibit similar geochemical characteristics, showing low textural and compositional maturities, implying short sediment transport, with all detrital zircons suggesting their origins from felsic igneous rocks. The  $\varepsilon_{Hf}(t)$  values of the Miaoling Formation range from -6.09 to 12.43 and from -2.20 to 7.59 for the Kedao Group, implying these rocks originated from NE China. Considering our new data along with previously published data, we propose that a reduced remnant ocean remained along the Changchun-Yanji suture zone in the early Middle Triassic (ca. 243 Ma), suggesting the final closure of the eastern PAO likely occurred between the latest Middle Triassic and early Late Triassic.

#### Highlights:

1. Miaoling Formation and Kedao Group were deposited around 253 Ma and 243 Ma, respectively.

2. The eastern segment of the Paleo-Asian Ocean closed in a scissor-like manner along the Changchun-Yanji suture zone.

3. Remnants of the Paleo-Asian Ocean still existed in the early Middle Triassic.

# 1. Introduction

The Central Asian Orogenic Belt (CAOB), the largest accretionary orogenic belt on Earth, is situated between the Siberian Craton to the north, and the North China and Tarim Cratons to the south (Sengör et al. 1993; Windley et al. 2007; Wilde, 2015; Xiao et al. 2015; Xu et al. 2015; Chen et al. 2022; Li, H.D. et al. 2022; Fig. 1a) and reflects a complex tectonic evolution closely connected to the closure of the Paleo-Asian Ocean (PAO) (Khain et al. 2002; Windley et al. 2007; Xiao et al. 2015; Zhang et al. 2019; Wu et al. 2007; Pei et al. 2016). Northeastern China (NE China), also called the Xing'an-Mongolian Orogenic Belt (XMOB; Sun et al. 2004; Xu et al. 2014), is located in the eastern segment of the CAOB and has witnessed the amalgamation of NE China massifs during Paleozoic and early Mesozoic, which from west to east are Erguna, Xing'an, Songnen and Jiamusi-Khanka massifs or terranes, all of which are separated by major faults or suture belts (Wu et al. 2007, 2011; Zhou et al. 2011a, b, 2015; Zhou & Wilde, 2013; Cao et al. 2012, 2013; Santosh & Somerville, 2013; Sun et al. 2013; Xu et al. 2014; Mi et al. 2017; Liu et al. 2017; Li, 2006; Tang et al. 2013; Fig. 1a). This amalgamation of NE China (XMOB) and the North China Craton (NCC) along the Solonker-Xar Moron-Changchun-Yanji Suture (SXCYS) is commonly recognized as the final closure of the PAO, supported by evidence from magmatic rocks, structural features and palaeontological data (Sun et al. 2004; Jia et al. 2004; Wu et al. 2007; Cao et al. 2013; Zhou et al. 2017; Wang et al. 2015a, b; Li et al. 2017; Yang et al. 2017; Gu et al. 2018; Du et al. 2019). Nonetheless, there are still no definite conclusions about the final



**Figure 1.** Tectonic sketch map of the Central Asian Orogenic Belt (a; Zhou & Wilde, 2013) and NE China (b; Liu et al. 2017).

closure time of the PAO, as different scholars present varied perspectives. The closing process of the PAO during the Permain-Triassic has been inferred through the study of intrusive rocks exposed in the NCC and igneous rocks in NE China (Cao et al. 2013; Guan et al. 2022; Han et al. 2020, 2021; Song et al. 2018; Wang et al. 2015b; Wu et al. 2007; Yuan et al. 2016; Yu et al. 2022). Some scholars, using high-quality paleomagnetic data and geological evidence, suggest the closure around 250 Ma (Zhao et al. 2013; Ren et al. 2023). Conversely, others, focusing on sedimentary rocks, argue differently (Du et al. 2019, 2021; Han et al. 2019; Liu J et al. 2017; Shi et al. 2020; Sun et al. 2022; Wang et al. 2015a). Most studies agree that the final suturing occurred during the Permian-Triassic (Cao et al. 2013; Du et al. 2019, 2021; Guan et al. 2022; Han et al. 2019, 2020, 2021; Li, 2006; Li & Zhao, 2007; Liu J et al. 2017, 2020; Shi et al. 2020; Song et al. 2018; Sun et al. 2022; Wang et al. 2015a, b; Wu et al. 2007; Yuan et al. 2016), while some propose it occurred before the Permian (Zhang et al. 2008; Shi et al. 2010), or even before the Late Devonian (Xu & Chen, 1997; Zhao et al. 2013; Xu et al. 2015; Zhu & Ren, 2017), or between the Late Devonian and Early Carboniferous (Tang, 1989; Hong et al. 1995). Others suggest that closure spanned from the Permian to Triassic, encompassing late Early Permian (Yu et al. 2022; Feng et al. 2010; Liu et al. 2010), Middle to Late Permian(Sengör et al. 1993; Chen et al. 2000, 2009; Jian et al. 2010; Lin et al. 2013), Late Permian (Li, 2006; Wu et al. 2011), Late Permian to Early Triassic (Sengör et al. 1993; Li, 1998, 2006; Xiao et al. 2003; Sun et al. 2004; Zhang et al. 2004; Wu et al. 2007, 2011; Xu et al. 2009; Peng et al. 2012; Cao et al. 2013; Eizenhöfer et al. 2014; Li et al. 2014; Wilde, 2015; Han et al. 2015; Guo et al. 2016; Wu & Li, 2022), Late Permian to Middle Triassic (Jia et al. 2004; Wang F et al. 2015; Xiao et al. 2015; Liu et al. 2017; Guan et al. 2023) and Middle-Late Triassic (Peng et al. 2012; Zhou & Wilde, 2013). This controversy largely arises from insufficient time constraints and sedimentological data for the transition from subduction to collision in the eastern segment of CAOB (Zhang et al. 2023).

This uncertainty about the provenance of Late Permian to Triassic sedimentary rocks in this area and the absence of constraints from related orogenic events further exacerbate the differing viewpoints. The Wangqing area in eastern Jilin Province, adjacent to the Changchun-Yanji suture zone, plays a key area in elucidating the final closure of the eastern end of PAO (Fig. 2). This study focuses on petrology, geochronology and geochemistry investigations conducted on sandstone samples from the Middle Permian Miaoling Formation and Upper Triassic Kedao Group in the Wangqing area, aiming to enhance the understanding of the evolution and closure history of the eastern PAO from a perspective of provenance analysis.

#### 2. Geological setting and sample descriptions

# 2. a. Geological setting

The SXCYS is bordered by the eastern CAOB to the north and the NCC to the south. NE China is located in the east segment of the CAOB, also known as the XMOB, which consists of numerous micro-blocks, including the Erguna Block, Xing'an Block, Songnen-Zhanggaungcai Range and Jiamusi-Khanka massifs or terranes (Ge et al. 2005; Huang et al. 2006; Li, S.K. et al. 2020). The NCC, the oldest and largest known craton in China, is divided into the Western Block and the Eastern Block, which amalgamated along the Trans-North China Orogen around 1.85 Ga (Zhao et al. 2012). Jilin Province features a series of discontinuous Permian-Triassic sedimentary sequences along the Changhun-Yanji suture, witnessing the amalgamation of NE China blocks and the NCC, preserved within the extensive "granite ocean" (Cao et al. 2013). The study area in the Wangqing area in the east of Jilin Province, adjacent to the southeastern XMOB and the northern NCC (Fig. 1a), is pivotal in reconstructing the final closure process of the PAO and the collision between the NCC and NE China.

The tectonic evolution history of NE China is complex, featuring Phanerozoic granitoids and Paleozoic to Mesozoic sediments, such as the Middle Permian Miaoling Formation and the Triassic Kedao Group. Intrusions associated with the PAO tectonic domain are primarily I-type granites and mafic to ultramafic rocks, while those related to the Paleo-Pacific tectonic Paleo-Asian Ocean: geochronology of Permian-Triassic sedimentary sequence



Figure 2. Detailed geological map of the Wangqing area showing the stratigraphic distribution and sampling locations.

domain consist mainly of A-type and I-type granites, with fewer mafic intrusions. Different formations often exhibit distinct lithological characteristics and different fossil assemblages. Previous studies on the Miaoling Formation have focused on volcanic interlayers (Lu et al. 2022), while the definition of the Kedao Group remains unclear (Yu, 2017). Therefore, for our study, we select the Miaoling Formation and the Kedao Group in the Wangqing area, Jilin Province, as our sampling locations are closer to the Changchun-Yanji suture zone, providing better representation.

Sun (1988) defined the Miaoling Formation as marine siliceous pyroclastic rocks intercalated with intermediate-felsic lavas, siltstone and limestone lenses. It exhibits a conformable contact with the upper Hongtaiping Formation and an angular unconformable contact with the lower Kedao Group. Its lower part is predominantly composed of grey and grey-green feldspathic quartz sandstone, greywacke and siltstone, intercalated with thin lenses of limestone; the upper part is mainly characterized by sandstone, siltstone and shale, interbedded with thick lenses of limestone.

Yin et al. (2011) concluded that the Kedao Group consists of Permian marine strata, divided into the Shanguqi and the Tanqian formations, which are mainly composed of fluvio-lacustrine deposits. Its upper part is in parallel unconformity with the Daxinggou Group, and the lower part is in angular unconformity with the Miaoling Formation. This group consists mainly of conglomerate, tuffaceous siltstone, arkose and shale with intercalated allochthonous limestone blocks (Shen et al. 2019; see Fig. 3 for more details).

# 2. b. Sample descriptions

Five samples were collected in the study area (Fig. 2), including mudstone 23JL34, grey-black mudstone 23JL36 and graywacke 23JL38 from the Miaoling Formation and grey-black siltstone 23JL30, blueish-grey mudstone 23JL33 from the Kedao Group.

#### 2. b.1. Miaoling formation

Samples 23JL34 (43°34′57.35″N, 129°34′0.75″E) and 23JL36 (43°25′36.77″N, 129°32′16.37″E) are both mudstones with over 90% argillaceous content; the primary difference is their colour under microscope, with 23JL34 appearing yellow-brown (Fig. 4a and d) and 23JL36 grey-black (Fig. 4b and e).

Sample 23JL38 (43°3'12.36"N, 129°51'48.77"E) is a graywacke from Shixian town, characterized by low maturity, with 50% matrix and 50% clastic fragments, featuring 70% quartz, 5% polycrystalline quartz and 25% feldspar, with poorly sorted, subrounded grains and aligned mineral fragments (Fig. 4c and f).

#### 2. b.2. Kedao group

The Kedao Group is dominantly exposed in Miaoling, Kedao, Xiangrenping, Houdidong, Wangqing and Sidonggou in the Yanbian area, with an exposed area of approximately 189 km<sup>2</sup> (RGSR, 2007). Predominantly composed of conglomerate and tuffaceous siltstone, these strata contain few fossils and exhibit contested age estimates (BGMRJP, 1997). While some studies suggest a Permian origin (BGMRJP, 1997), recent evidence indicates an Early-Middle Triassic age, supported by Triassic fossils (Zhou, 2009) and geochronologic data from the Yanji area.

Sample 23JL30 (43°27′53.65″N, 129°43′24.31″E) is a greyblack siltstone from the eastern Miaoling Quarry, characterized by low maturity and composed of 35% matrix and 65% clastic fragments. It comprises 80% quartz and 20% feldspar, featuring a clastic texture with poorly sorted, sub-rounded grains and a clear alignment of mineral fragments (Fig. 5a and c).

Sample 23JL33 (43°34′0.38″N, 129°36′3.54″E) is a blueish-grey mudstone in the southwest of Tianqiaoling town, containing over 90% argillaceous materials and presenting challenges for mineral identification (Fig. 5b and d).

#### 3. Analytical methods

#### 3. a. LA-ICP-MS zircon U-Pb dating

Selected zircon grains (Fig. 6) were mounted into an epoxy resin disc and polished to about half-sections to expose grain interiors. Cathodoluminescence (CL) images coupled with transmitted and reflected light micrographs were obtained to examine the internal structures. Zircon U-Pb isotopic and trace element analyses were performed using an ASI<sup>TM</sup> RESOlution-LR Series 193 nm excimer laser ablation instrument (LA) coupled with Thermo Scientific<sup>TM</sup> iCAP<sup>TM</sup> RQ series inductively coupled plasma mass spectrometry (ICP-MC) at Key Laboratory of Orogen and Crust Evolution, Peking University and Hebei Key Laboratory of Strategic Critical Mineral Resources, Hebei GEO University. The laser spot diameter is 29 $\mu$ m, the frequency is 6 Hz and the energy density is 3 J/cm<sup>2</sup>. High-purity argon gas is used as the carrier gas and high-purity He and N<sub>2</sub> gas are used to increase sensitivity. The background of <sup>204</sup>Pb and <sup>202</sup>Hg is usually less than 100 cps. Calibration for the zircon U/Pb ratios and trace elements was carried out by using the standard zircon 91500 (1062 Ma; Wiedenbeck et al. 1995) and glass standard NIST 610, respectively. Zircon standard Plésovice (337 Ma; Sláma et al. 2008) and GJ-1 are also used to supervise the deviation of age measurement/calculation. Those external standards were analyzed once per five unknown samples. The contents of trace elements in all zircons were also calculated using 91Zr as an internal standard. Isotopic ratios and element concentrations of zircons were calculated using GLITTER (ver. 4.4.2, Macquarie University) and iolite ver. 4.3.0. The common lead was corrected

| System   | Formation                                  |  | Section   | Description   |
|--|--|--|---|---|
| Jurassic   | Manhe Fm.<br>J <sub>2</sub> mh             |  | $\begin{array}{c} \lor \land \lor \land \lor \land \lor \land \lor \land \lor \land \lor \\ \land \lor \land \lor \land \lor$ | Intermediate volcanic rock assemblage and pyroclastic rock  |
| Permain  | Daxinggou Group<br>T <sub>3</sub> D        | Tianqiaoling Fm.<br>T <sub>3</sub> tq                |   | Rhyolite, dacite  |
|  |  | Malugou Fm.<br>T <sub>3</sub> m                      |   | Sandstone, conglomerate sandstone, siltstone,<br>with fossilized plants such as <i>Neocalamites carrerei</i><br><i>Cladophlebis, Cycadoca rpidim</i> , and <i>Padozamites</i> . |
|  |  | Tuopangou Fm.<br>T <sub>3</sub> t                    | · · · · · · · · · · · · · · · · · · ·   | Andesite, dacite, and intermediate-acid volcanic rocks.   |
|  | Kedao Group<br>T <sub>3</sub> K            | Tanqian Fm.<br>T <sub>3</sub> ta<br>23JL30<br>23JL33 |   | Upper part: Feldspathic coarse sandstone, siltstone.<br>Lower part: Siltstone, mudstone and fossils.  |
|  |  | Shanguqi Fm.<br>T <sub>3</sub> ŝ                     |   | Upper part: Feldspathic coarse sandstone, siltstone.<br>Lower part: Conglomerate and sandstone.   |
| Triassic   | Miaoling Fm.   23JL34 ∖     P₂m   23JL36 / |  |   | Sandstone, limestone, siltstone and animal fossils.   |
|  | Hongtaiping Fm.<br>P <sub>.</sub> h        |  | Y A Y A Y A Y A<br>A Y A Y A Y A Y A<br>Y A Y A   | Andesite, limestone, pyroclastic rocks and intermediate-acid volcanic rocks.  |
|  |  |  |   |   |
| v v andesite • sandstone - Mudstone □ Limestone C Carbonaceous 6 Fossil ★ Samples   X X rhyolite •• siltstone v▲v Andesitic volcaniclastic rock •• Conglomerate rock |  |  |   |   |

**Figure 3.** Stratigraphic columns of the study area with sampling locations.



Figure 4. Field photographs (a-c) and photomicrographs (d-f) of analyzed samples from the Miaoling Formation.

using LA-ICP-MS Common Lead Correction (ver. 3.15), following the method of Anderson (2002). Concordia diagrams and weighted average plots were obtained using Isoplot/Ex (3.0) (Ludwig, 2003). Probability density distribution plots were produced using AgeDisplay (Sircombe, 2004). Uncertainties on individual analyses are reported at the  $2\sigma$  and mean ages for pooled U/Pb analyses are calculated at a 95% confidence interval.

All the errors are within  $2\sigma$  interval, and the isotopic ages plotted in the diagrams for the relative probability of the ages of the samples. The <sup>207</sup>Pb/<sup>206</sup>Pb age is used when the age is over 1000 Ma,

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**Figure 5.** Field photographs (a-b) and micrographs (c-d) of analyzed samples from the Kedao Group.



**Figure 6.** Cathodoluminescence (CL) images of representative detrital zircons from all dated samples. Circles mark dating spots (red for U-Pb isotopic tests, yellow for Hf isotopic tests.). Below zircons refer to the U-Pb ages, above are dating numbers.

while the <sup>206</sup>Pb/<sup>238</sup>U age is used when the age is younger than 1000 Ma due to the imprecise measurement of <sup>207</sup>Pb in young grains (Sircombe, 1999). The ages with discordance degree over 10% were excluded when Mapping.

### 3. b. Major and trace element determinations

The siltstone samples used for whole-rock analysis were crushed to ~200 mesh in an agate mill after the removal of altered surfaces. Major and trace elements compositions were determined by using an X-ray fluorescence spectrometer and ICP-MS (Agilent 7700x), respectively, at the Key Laboratory of Mineral Resources Evaluation in Northeast Asia, Ministry of Natural Resources, Jilin University, Changchun, China, after the sample powders had been dissolved in Teflon bombs. The analytical results for the BHVO-1 (basalt), BCR-2 (basalt) and AGV-1 (andesite) standards

yielded values of analytical precision that were better than 5% for major elements and 10% for trace elements (Rudnick et al. 2004).

# 3. c. Hf isotope analysis

Zircon Hf isotope ratios were measured using a Neptune (Plus) MC-ICP-MS (ThermoFisher Scientific<sup>TM</sup>, USA) equipped with an ASI<sup>TM</sup> RESOlution-LR Series 193 nm excimer laser ablation instrument, which is hosted at the Hebei Key Laboratory of Strategic Critical Mineral Resources, Hebei GEO University, China. These analyses were performed with a laser beam diameter of ca. 43  $\mu$ m and 5 Hz repetition rate, yielding a signal intensity of ~1.2 v at <sup>179</sup>Hf during the analysis of the standard zircon GJ-1. The ablation time is 40 s, yielding pits of 30–40  $\mu$ m deep. Masses 172, 173, 175, 176, 177, 178, 179 and 180 were simultaneously measured in static-collection mode. Standard zircons GJ-1 were



Figure 7. U-Pb concordia diagrams of detrital zircons from the Miaoling Formation; ellipses represent 2σ uncertainties (blue ellipses represent the group of the youngest concordant ages).

used as external standards and were analyzed twice before and after every 10 analyses. The resulting data were normalized to  $^{179}\mathrm{Hf}/^{177}\mathrm{Hf}=0.7325$  using an exponential correction for mass bias. The interference of  $^{176}\mathrm{Lu}$  on  $^{176}\mathrm{Hf}$  was corrected by measuring the intensity of the interference-free  $^{175}\mathrm{Lu}$  isotope and using the recommended  $^{176}\mathrm{Lu}/^{175}\mathrm{Lu}$  ratio of 0.02655. The mean  $^{173}\mathrm{Yb}/^{172}\mathrm{Yb}$  ratio on the zircon sample itself was measured to calculate the mass fractionation factor  $\beta_{\mathrm{Yb}}$ , and the signal intensity of  $^{176}\mathrm{Yb}$  was calculated based on the signal intensity of  $^{173}\mathrm{Yb}$  and the calculated  $\beta_{\mathrm{Yb}}$ , and then used to correct the interference of  $^{176}\mathrm{Hf}$ .

# 4. Analytical results

# 4. a. Zircon U-Pb dating

#### 4. a.1. Miaoling formation

A total of 142 concordant U-Pb dating analyses from three samples in this study were obtained, and the results are given in Supplementary Table 1. All dated zircons, ranging in size from  $30-220 \mu$ m, are euhedral to subhedral with some corrosion marks (Fig. 6). Their high Th/U ratios of 0.02-2.17 (over 98% are above 0.1) and clear oscillatory zones (Fig. 6) indicate a magmatic origin (Wu & Zheng, 2004).

In sample 23JL34, 42 zircon grains yield concordant ages ranging from 248  $\pm$  9 Ma to 915  $\pm$  26 Ma (Fig. 7a; Supplementary Table 1), grouped as 248–284 Ma (42 grains, peaking at 254 Ma), 316–500 Ma (12 grains, peaking at 410 Ma) and 638–915 Ma (6 grains) (Fig. 7b). The youngest ages yield the <sup>206</sup>Pb/<sup>238</sup>U weight mean age of 252  $\pm$  3.7 Ma (Fig. 7c; Mean Square Weighted Deviation (MSWD) = 0.086, n = 18), with a mean Th/U ratio of 0.64.

In sample 23JL36, 50 concordant zircons show concordant ages from 247  $\pm$  15 Ma to 428  $\pm$  10 Ma (Fig. 7d; Supplementary Table 1), predominantly in the age group of 247–276 Ma (47 grains, peaking at 258 Ma) (Fig. 7e). The youngest zircons yield the <sup>206</sup>Pb/<sup>238</sup>U age of 253  $\pm$  2.9 Ma (Fig. 7f; MSWD = 0.22, n = 28), with a mean Th/U ratio of 0.53.

For sample 23JL38, 50 zircon grains yield concordant ages from 249  $\pm$  6 Ma to 1420  $\pm$  19 Ma (Fig. 7g; Supplementary Table 1), which can be subdivided into groups of 249–303 Ma (25 grains, peaking at 256 Ma), 463–553 Ma (10 grains, peaking at 503 Ma) and 623–1420 Ma (15 grains) (Fig. 7h). The youngest zircons yield the <sup>206</sup>Pb/<sup>238</sup>U weight mean age of 254  $\pm$  3.1 Ma (Fig. 7i; MSWD = 0.30, n = 13), with a mean Th/U ratio of 0.57.



Figure 8. U-Pb concordia diagrams for detrital zircons from the Kedao Group; ellipses represent 2<sup>o</sup> uncertainties (blue ellipses represent the group of the youngest concordant ages).

#### 4. a.2. Kedao group

A total of 89 concordant U-Pb dating analyses from two samples in this study were obtained, and the results are given in Supplementary Table 1. All dated zircons, ranging in size from 30 to 200  $\mu$ m, are euhedral to subhedral with some corrosion marks (Fig. 6). They exhibit high Th/U ratios of 0.08–2.13 (over 98% are above 0.1) and clear oscillatory zones (Fig. 6), indicating a magmatic origin (Wu & Zheng, 2004).

For sample 23JL30, 57 zircons yield concordant ages ranging from 238±8 Ma to 1178±34 Ma (Fig. 8a; Supplementary Table 1), falling into groups of 238–271 Ma (29 grains, peaking at 246 Ma), 303–363 Ma (6 grains, peaking at 337 Ma), 405–532 Ma (11 grains) and 707–1178 Ma (11 grains) (Fig. 8b). The youngest concordant zircon yields the  $^{206}$ Pb/ $^{238}$ U age of 245 ± 2.7 Ma (Fig. 8c; MSWD = 0.31, n = 18), with a mean Th/U ratio of 0.52.

For sample 23JL33, 32 concordant zircons yield a broad age spectrum ranging from 237  $\pm$  6 Ma to 1801  $\pm$  19 Ma (Fig. 8d; Supplementary Table 1), which can be divided into groups of 237–310 Ma (22 grains, peaking at 242 Ma), 445–708 Ma (9 grains, peaking at 460 Ma and 518 Ma) and 1801 Ma (Fig. 8e). The youngest zircons with a mean Th/U ratio of 0.66 yield the <sup>206</sup>Pb/<sup>238</sup>U age of 242±4.8 Ma (Fig. 8f; MSWD = 0.48, n = 7).

# 4. b. Major and trace elements

# 4. b.1. Major elements

Whole-rock major and trace elemental analyses were carried out on the fifteen sandstones and the data are provided in Supplementary Table 2. Samples from the Miaoling Formation have higher contents of <sup>T</sup>Fe<sub>2</sub>O<sub>3</sub>, CaO, MgO, Na<sub>2</sub>O, Ti<sub>2</sub>O, P<sub>2</sub>O<sub>5</sub> and MnO, but lower contents of SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub> and K<sub>2</sub>O than those from the Kedao Group. The SiO<sub>2</sub>/Al<sub>2</sub>O<sub>3</sub> ratios are relatively low (3.13– 5.68; average 3.92), classifying the samples as immature rocks (Peittijohn et al. 1972; Herron, 1988). In the lithological



Figure 9. Geochemical classification diagrams of the Miaoling Formations and Kedao Group (after Pettijohn et al. 1972 and Herron, 1988).

discrimination (Fig. 9), all samples from both formations are plotted in the greywacke area, consistent with our microscopic observations (Figs. 4 and 5).

#### 4. b.2. Trace elements

The chondrite-normalized Rare Earth Elements (REEs) distribution patterns of all samples are similar to those of the average upper continental crust (Rudnick & Gao, 2014), featuring enriched Light Rare Earth Elements (LREEs) ((La/Yb)<sub>N</sub> = 1.79–7.13; averagely 4.29), flat Heavy Rare Earth Elements (HREEs) ((Gd/Yb)<sub>N</sub> = 0.95–2.42; averagely 1.45) and slight to moderate negative Eu anomalies (Fig. 10a), with no Ce anomaly. The



Figure 10. Chondrite-normalized REE patterns (left) and Primitive Mantle trace element diagrams (right) for the studied sandstones. The normalizing values for REE and trace elements are from McDonough & Sun (1995) and Boynton (1984), respectively. Data for the average upper continental crust are from Rudnick & Gao (2014).



Figure 11. Hf isotopic compositions of detrital zircons from Miaoling Formation (yellow) and Kedao Group (blue) in the study area (Yang et al. 2006).

enrichment of LREEs and depletion in HREEs are obvious for all samples, reflected in their similar right-dipping curves. The mantle-normalized multi-element variation diagram of trace elements shows a consistent trend (Fig. 10b), indicating depletion of high-field strength elements (HFSE) Nb, Ta and of large-ion lithophile elements Sr, while showing slight enrichment in Ba, Th, La, Ce, Nd, and Sm in HFSE and Zr, K in large-ion lithophile elements, although the enrichment of La and Ce is not conspicuous.

## 4. c. Hf Isotope results

We conducted Hf isotope analysis on representative Paleozoic to Mesozoic zircon grains from sedimentary rocks of the Miaoling Formation and Kedao Group (Supplementary Table 3 and Fig. 11).

The Hf isotopic compositions of 28 detrital zircons from the Miaoling Formation (23JL34, 23JL36 and 23JL38) were analyzed. Paleozoic zircons (495–254 Ma) exhibit  $^{176}$ Hf/ $^{177}$ Hf ratios ranging from 0.2823145 to 0.2829695, with  $\varepsilon_{\rm Hf}(t)$  values varying between -6.09 and 12.43, and the  $T_{\rm DM2}$  are 1830–493 Ma. Mesozoic zircons (251–247 Ma) exhibit  $^{176}$ Hf/ $^{177}$ Hf ratios ranging from 0.282512 to 0.282947, with  $\varepsilon_{\rm Hf}(t)$  values varying

between -3.90 and 11.5, and the  $\rm T_{DM2}$  ranging from 1272 to 488 Ma (Supplementary Table 3).

The Hf isotopic compositions of 12 detrital zircons from the Kedao Group (23JL30 and 23JL33) were analyzed. Paleozoic zircons (438–252 Ma) exhibit <sup>176</sup>Hf/<sup>177</sup>Hf ratios ranging from 0.2824738 to 0.282837, with  $\varepsilon_{\rm Hf}(t)$  values varying between -2.20 and 7.59, and the  $T_{\rm DM2}$  of 1499–794 Ma. Mesozoic zircons (250–239 Ma) exhibit <sup>176</sup>Hf/<sup>177</sup>Hf ratios ranging from 0.2825632 to 0.282849, with  $\varepsilon_{\rm Hf}(t)$  values varying between -2.07 and 7.66, and the  $T_{\rm DM2}$  of 1407–784 Ma (Supplementary Table 3).

# 5. Discussion

# 5. a. The sedimentary ages of the miaoling formation and kedao group

The deposition time of a sedimentary strata is much younger than its sediments, allowing detrital zircons within a formation to help constrain the maximum deposition ages (Fedo, 2003). This method relies on the assumption that the U-Pb system of analyzed zircons remained undisturbed by post-depositional tectonic-metamorphic or hydrothermal events (Zeh & Gerdes, 2012). In this study, combined with age-diagnostic fossils and intruded dykes, the mean age of the youngest overlapping zircon grains at  $2\sigma$  uncertainty (Dickinson & Gehrels, 2009) was used to estimate the maximum deposition ages of the Miaoling Formation and Kedao Group in Wangqing area.

#### 5. a.1. Miaoling formation

Extensive research has been conducted on the Miaoling Formation; however, its deposition time remains a point of contention. Gathering all data from these three samples (23JL34, 23JL36 and 23JL38), the maximum depositional age of the Miaoling Formation was determined to be late Permian by the 57 youngest concordant zircons having a weighted mean  $^{206}$ Pb/ $^{238}$ U age of 253 ± 1.9 Ma (Fig. 14d; MSWD = 0.19, n = 57). Among all 142 concordant zircons, only 21 are older than 600 Ma, with the oldest three grains yielding the  $^{207}$ Pb/ $^{206}$ Pb age of 1005 ± 36 Ma, 1209 ± 24 Ma and 1420 ± 19 Ma (Fig. 14c). Phanerozoic ages peak at 256 Ma (Fig. 14c). This late Permian maximum depositional age is obviously younger than the previously published youngest cluster of zircons in sandstones with the age of ca. 265 Ma (Lu et al. 2022).



Figure 12. A-CN-K weathering diagram of major elements (after Nesbitt & Young, 1984) in sandstones from the Wangqing area. The solid arrow represents the ideal weathering trend line of each igneous rock, according to data from Condie (1993). A: Al<sub>2</sub>O<sub>3</sub>, CN: CaO<sup>+</sup>+Na<sub>2</sub>O, K: K<sub>2</sub>O.

#### 5. a.2. Kedao group

Divergent views exist regarding the sedimentary ages of the Kedao Group, ranging from the Late Permian to the Late Triassic (Zhou, 2009; Yu, 2017; Zhou et al. 2017; Du et al. 2019). Combining all the data from two samples together, the 20 youngest concordant zircons of the Kedao Group yielded a weighted mean  $^{206}$ Pb/ $^{238}$ U age of 243 ± 2.7 Ma (MSWD = 0.24, n = 20) (Fig. 14b), implying the maximum depositional age of Middle Triassic (ca. 243 Ma). Precambrian zircons constitute a significant proportion in all analyses (15.7%), with 14 grains older than 600 Ma, while the Phanerozoic ages with peaks of 245 Ma and 468 Ma (Fig. 14a).

# 5. b. Provenance analysis of Miaoling Formation and Kedao Group in the study area

The chemical index of alteration (CIA) is useful for quantitatively assessing weathering intensity (Fedo et al. 1995; Nesbitt & Young, 1984). The samples show an average CIA value of 68.60, reflecting weak initial chemical weathering (Fig. 12a; Fedo et al. 1995). In the A-CN-K diagram (Fig. 12b), the average Chemical Index of Weathering is 77.85, about 10% higher than the CIA. The weathering trend deviates slightly to the right (Fig. 12a), indicating a potassium metasomatism influence during diagenesis, which indicates a nearby source and a short transportation distance. The intersection of the weathering trend line and the plagioclase-Kfeldspar line indicates a higher plagioclase content than K-feldspar in parent rocks (Fedo et al. 1995), indicating a provenance related to felsic igneous. Analyzed detrital zircons, in this study, show a positive correlation between U and Y concentrations, suggesting origins from intermediate-acidic igneous rocks such as granite, syenite and pegmatite. The trace element analysis of zircons also indicates that the parent rocks were primarily felsic igneous, although some points suggest minor mixture of mafic rocks (Fig. 13a, b, c, and d). Overall, these characteristics indicate that the REE contents of the sandstones approximate the global average for continental upper crust, with their provenance related to felsic rocks in the upper crust.

Both the sandstones from Miaoling Formation and Kedao Group exhibit characteristics of greywackes (Figs. 4, 5, and 9), with low compositional and textural maturities indicating a nearby provenance. These samples contain a high proportion of argillaceous materials and various sizes of mineral clasts. The CL images of euhedral detrital zircons further support a relatively near source. Possible provenance areas for these formations include the NCC to the south and the NE China massifs (Zhangguangcai Range and Jiamusi-Khanka block) to the north (Fig. 1a). Detrital zircons carry geochronological information about their source rocks and have high diagenesis stability (Wu & Zheng, 2004), providing them a valuable for provenance analysis of sandstones at plate margins or orogenic belts.

The age distributions of these two formations exhibit the same characteristics, as evidenced by variations in the proportion of each age group and the distribution of peak ages (Fig. 14). The Miaoling Formation is characterized by the overwhelming majority of Phanerozoic zircons (81.3%), with 118 out of 145 grains. A peak age of ca. 256 Ma can be perfectly related to the Permian granitic magmatism (256-252 Ma; Yu et al. 2013) and magmatic activities linked to the westward subduction of an ancient oceanic plate in the southern Jiamusi-Khanka block (272-257 Ma; Long et al. 2019). A peak age of ca. 504 Ma is consistent with the ~500 Ma late Pan-African crystal basement in NE China massifs (Xiong et al. 2020; Hua et al. 2019; Li, Y. et al. 2017; Wen et al. 2017; Wang et al. 2012; Wu et al. 2011; Yang et al. 2014; Zhou et al. 2011b, 2015, 2018), representing typical differences from the NCC. In addition, a minor peak at 920 Ma in the Neoproterozoic is consistent with the magmatic activities in NE China, implying a strong affinity. Both the Kedao Group and Miaoling Formation exhibit similar magmatic activity peaks at 245 Ma, 468 Ma and 876 Ma, while zircon grains older than 1000 Ma are rare in both formations, lacking distinct peaks, which highlights a clear deviation from the zircon curve of the NCC (Fig. 14).

Vermeesch (2013) proposes multidimensional scaling (MDS) as a valuable statistical tool for geological data analysis. The solid line signifies the strongest correlation, while the dashed line represents the second strongest correlation. Through MDS analysis, both the Miaoling Formation and Kedao Group have a higher affinity with NE China (Fig. 15). Zircons from these formations exhibit similar Hf isotopic compositions consistent



Figure 13. The fields of zircon compositions used as discriminants for different rock types (Belousova et al. 2002). (a) Zircon Y versus U, (b) Zircon Nb versus Ta, (c) Zircon Y versus Yb/Sm, (d) Zircon Y versus Nb/Ta, (e) Zircon Nb/Hf versus Th/U (Yang et al. 2012) and (f) Zircon Hf/Th versus Th/Nb (Yang et al. 2012).

with those from the CAOB, in line with the Hf isotope results of the magmatic rocks in the study area (Fig. 11; Shi et al. 2022; Lu et al. 2022; Shi et al. 2020), indicating a certain level of data reliability. The  $T_{DM2}$  did not provide evidence for an age of 2.5 billion years. Based on various evidence, we can confidently infer that the samples from both the Miaoling Formation and Kedao Group originated from NE China (Zhangguangcai Range and Jiamusi-Khanka block). Additionally, combined with the findings from other studies, we can boldly infer that the Permian to Triassic stratigraphic source areas in the eastern part of Jilin Province are more closely related to NE China (Sun et al. 2023; Du et al. 2019,

2021; Zhang et al. 2019), further supported by Hf isotope results from igneous rocks exposed in the surrounding area (Cao et al. 2013).

# 5. c. Tectonic setting

The oceanic subduction-continental collision system has become a research hotspot in recent years (Dai et al. 2018; Li et al. 2018a, b, 2019; Yu et al. 2019a, b, c; Zhang et al. 2019). The amalgamation of the NE China (XMOB) and the NCC along the SXCYS is widely believed to record the subduction and



Figure 14. a-b. Age probability histograms of detrital zircons with concordant ages and the youngest weight mean age from the Kedao Group; c-d. Age probability histograms of detrital zircons with concordant ages and the youngest weight mean ages of the Miaoling Formation; e. The data originate from NE China (Zhangguangcai Range and Jiamusi-Khanka block; data from Du et al. 2016; Meng et al. 2011; Wang et al. 2012, 2015; Yu et al. 2013; Luan et al. 2017; Li, Y. et al. 2017; Long et al. 2019; Xue et al. 2023; Hua et al. 2019; Li, H.D. et al. 2022; Meng et al. 2017; Mou et al. 2023; Pu et al. 2015; Wen et al. 2017; Xiong et al. 2020; Zhang et al. 2021; Zhao et al. 2021; Zhao et al. 2023; Zhou et al. 2013). f. The data originate from North China Craton; data from Liu et al. 2020, 2021; Chen et al. 2017, 2020; Fu et al. 2018; Li, G.S. et al. 2022; Liu et al. 2018; Pei et al. 2014; Peng et al. 2020; Peng & Wang, 2018; Shao et al. 2014; Yang et al. 2022; Zhang et al. 2015; Zhang et al. 2022; Zhang et al. 2013).

closure of the PAO (Sun et al. 2023), as indicated by the evidence from magmatic rocks, structural features and palaeontological data (Sun et al. 2004; Jia et al. 2004; Wu et al. 2007; Cao et al. 2013; Wang et al. 2015a, b; Li et al. 2017; Yang et al. 2017; Zhou et al.

2017; Gu et al. 2018; Du et al. 2019). In contrast to previous studies, we take a perspective of provenance analysis by concentrating on the sedimentary strata through petrology, geochronology and geochemistry in the Wangqing area along the



**Figure 15.** Multidimensional scaling (MDS) analysis results of Miaoling Formation, Kedao Group and surrounding potential source areas (NE China and North China Craton). a. After the standard K-S test. b. After the standard Kuiper test. (Vermeesch, 2013).

suture zone between the NCC and NE China (Fig. 1b). The Permian Miaoling Formation major consists of polycyclic sandstone, siltstone, silty mudstone and is the product of an arc-basin series. The Tanqian Formation, representing a muddy construction beneath the Carbonate Compensation Depth line of the abyssal plain, and the Shanguqi Formation, reflecting continental slope turbidites, together constitute the Kedao Group, which belongs to a deep-water slope-basinal sedimentary system (RGSR, 2007). The samples analyzed in this paper align well with previously established conditions. The Miaoling Formation and Kedao Group show distinct characteristics in various tectonic diagrams based on both major and trace elements (Figs. 9 and 10), with all geochemical data indicating their orogenic origin (Fig. 12e and f).

Zircon transport processes mainly influence the age distribution of detrital zircons in the strata, with peak age compositions effectively reflecting the tectonic environment of the sedimentary basin (Cawood et al. 2012). The convergent plate boundary is characterized by a large proportion of zircon ages close to the deposition age of the sediment, whereas sediments in collisional, extensional and intracratonic settings contain higher proportions of older ages reflecting the history of the underlying basement (Cawood et al. 2012). The majority of detrital zircons from the Miaoling Formation (70%) and Kedao Group (64%) have crystallization ages (CA) close to the depositional ages (DA) (CA-DA < 100 Ma), similar to the sedimentary facies of a convergent environment (Fig.16).

Verma & Armstrong-Altrin (2013) developed new discriminant-function-based major-element diagrams for classifying siliciclastic sediments from island or continental arc, continental rift and collision settings. The results of all sandstones indicate that the Wangqing area is still within the continental island arc (Fig. 17), reaffirming previous conclusions and indicating ongoing subduction of the PAO in this area during the Middle Triassic.

Recent studies on PAO evolution have documented considerable sedimentary evidence. Research on the southern part of the Great Xing'an Range and the Songliao Basin in NE China indicates that the PAO closed on the western side of the Changchun-Yanji suture zone in the Late Permian (He et al. 2023; Zhang et al. 2023). However, an alternative argument proposes a delayed closure of the eastern segment between the Middle Triassic and the Late Triassic (Zhang et al. 2019; Du et al, 2019). Igneous rocks in this region provide additional supporting evidence (Cao et al. 2013; Sun et al. 2023). Considering our new data with previously published data, Sun (2023) suggests that the volcanic interlayers of the Yangjiagou Formation formed in a syn-collisional setting,



**Figure 16.** Summary plot of the general fields for convergent (A: red field), collisional (B: blue field), and extensional basins (C: greenfield). From the variations observed between the different fields, a model that predicts the tectonic setting of sedimentary packages of unknown origin is proposed based on differences between the crystallization and depositional ages (CA-DA) of the zircons.

implying a remnant ocean basin remained along the Changchun-Yanji suture zone during the Early Triassic. Furthermore, simulations of the crustal thickness in the central Jilin Province show that the crust gradually thickened from 280 Ma to 245 Ma, with the subduction of oceanic crust contributing to the continuous thickening of the continental crust (Guan et al. 2023). On a broader scale, Liang et al. (2019) summarize thermochronological data suggesting that significant strike-slip movement likely occurred in the Late Triassic, attributed to the eastward extrusion of the XMOB and far-field forces related to Late Triassic convergence following the final closure of the PAO.

The Miaoling Formation and the Kedao Group mainly originated from continental island arcs of NE China, including the Zhangguangcai Range and Jiamusi-Khanka block. During their sedimentation, a reduced remnant ocean basin remained in the Wangqing area, indicating that the PAO has not yet completed its closure until the early Middle Triassic (ca. 243 Ma; Fig. 18). Combining previous research suggests the final closure of the easternmost segment of the PAO likely occurred between the latest Middle Triassic and early Late Triassic in a scissor-like manner.



Figure 17. New discriminant-function multidimensional diagram for high-silica (a) and low-silica (b) clastic sediments from three tectonic settings (arc, continental rift and collision) (Verma & Armstrong-Altrin, 2013).



**Figure 18.** A sketch of the tectonic evolutionary pattern of the Paleo-Asian Ocean during 253–243Ma.

#### 6. Conclusions

This study investigated the geochronology and geochemistry of the sandstones of the Miaoling Formation and the Kedao Group in the Wangqing area of NE China to determine their provenance and tectonic setting.

1. New LA-ICP-MS zircon U-Pb dating results show that the sedimentary rocks from the Miaoling Formation were deposited around 253 Ma, while the maximum depositional age of the Kedao Group is early Middle Triassic (ca. 243 Ma).

2. The Miaoling Formation and the Kedao Group are dominantly composed of Phanerozoic sediments originating from the NE China, suggesting that their parent rocks were likely felsic igneous rocks from an orogenic tectonic setting.

3. The provenance of these sedimentary strata has remained relatively consistent, implying that around 243 Ma, a small remnant ocean basin of PAO still existed in this area, and the continental blocks on the north and south sides had not yet completely converged. The final closure of the easternmost segment of the PAO likely occurred between the latest Middle Triassic and early Late Triassic in a scissor-like manner.

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