



Rhenium–osmium (Re–Os) geochronology of crude oil from lacustrine source rocks of the Hailar Basin, NE China

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Received: 3 July 2020
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Abstract

Re–Os radiometric dating of crude oil can be used to constrain the timing of hydrocarbon generation, migration or charge. This approach has been successfully applied to marine petroleum systems; however, this study reports on its application to lacustrine-sourced natural crude oils. Oil samples from multiple wells producing from the Cretaceous Nantun Formation in the Wuerxun-Beier depression of the Hailar Basin in NE China were analysed. Subsets of the Re–Os data are compatible with a Cretaceous hydrocarbon generation event (131.1 ± 8.4 Ma) occurring within 10 Myr of deposition of the Nantun Formation source rocks. In addition, two younger age trends of 54 ± 12 Ma and 1.28 ± 0.69 Ma can be regressed from the Re–Os data, which may reflect the timing of subsequent hydrocarbon generation events. The Re–Os geochronometer, when combined with complementary age dating techniques, can provide direct temporal constraints on the evolution of petroleum system in a terrestrial basin.

Keywords Re–Os dating · Lacustrine crude oils · Hailar basin · Petroleum system evolution

1 Introduction

Oil and natural gas are expected to meet over 50% of the world's energy needs by 2040 (ExxonMobil 2019). Providing temporal constraints on the evolution of a petroleum system within a sedimentary basin is of fundamental importance to exploration targeting by industry. The rhenium–osmium (Re–Os) geochronometer is a promising absolute dating technique for organic-rich rocks and their

associated hydrocarbon derivatives (see reviews in Cohen 2004; Stein and Hannah 2015; Li et al. 2017; Liu and Selby 2018). Both Re and Os are redox-sensitive and organophilic, and can be enriched in hydrocarbon source rocks, tar sands, crude oil, and bitumen. Based on the naturally occurring radiometric decay of ^{187}Re to ^{187}Os , Re–Os geochronology of such organic-rich samples has the potential to constrain the timing of hydrocarbon maturation, migration or charge in petroleum systems (Selby and Creaser 2005a). Moreover, the initial $^{187}\text{Os}/^{188}\text{Os}$ composition of liquid hydrocarbons can potentially fingerprint their source rock provenance (Finlay et al. 2011).

Previous studies have successfully applied Re–Os geochronology to petroleum systems in marine-dominated basin environments (Ravizza and Turekian 1989; Cohen et al. 1999; Selby and Creaser 2005a, b; Rooney et al. 2010; Finlay et al. 2011; Lillis and Selby 2013; Zhu et al. 2013; Bertoni et al. 2014; Ge et al. 2016, 2018; Georgiev et al. 2019). However, lacustrine sedimentary basins—which are important sources of hydrocarbons in China—can be more challenging (Li et al. 2017) because sediments of subaerial provenance are likely to have low initial Re and Os concentrations (Creaser et al. 2008; Baioumy et al. 2011). Here, we report the first successful

Edited by Jie Hao

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Re–Os radiometric dating of lacustrine-sourced natural crude oils from the Wuerxun-Beier depression of the Hailar Basin in NE China, providing direct Re–Os dating constraints on a petroleum system operating within a terrestrial basin. By combining these data with previous basin histories and apatite fission-track analysis, we not only investigate the temporal evolution of the Wuerxun-Beier petroleum system, but also demonstrate the potential application of the Re–Os chronometer in better understanding other terrestrial petroliferous basins worldwide.

2 Geological setting

Covering an area of more than 70,000 km², the Hailar Basin is located in Hulun Buir of the Inner Mongolia Autonomous Region and is one of the most important petroliferous continental basins in NE China (Fig. 1). The Wuerxun-Beier depression, with an area of about 5,220 km², is a large second-order tectonic unit in the central fault belt of the Hailar Basin (Zhang and Peng 2002; Wu et al. 2006; Cui and Ren 2011). The Wuerxun-Beier petroleum system has received considerable attention due to its huge resource potential with total proved reserves of oil and gas exceeding 200 million

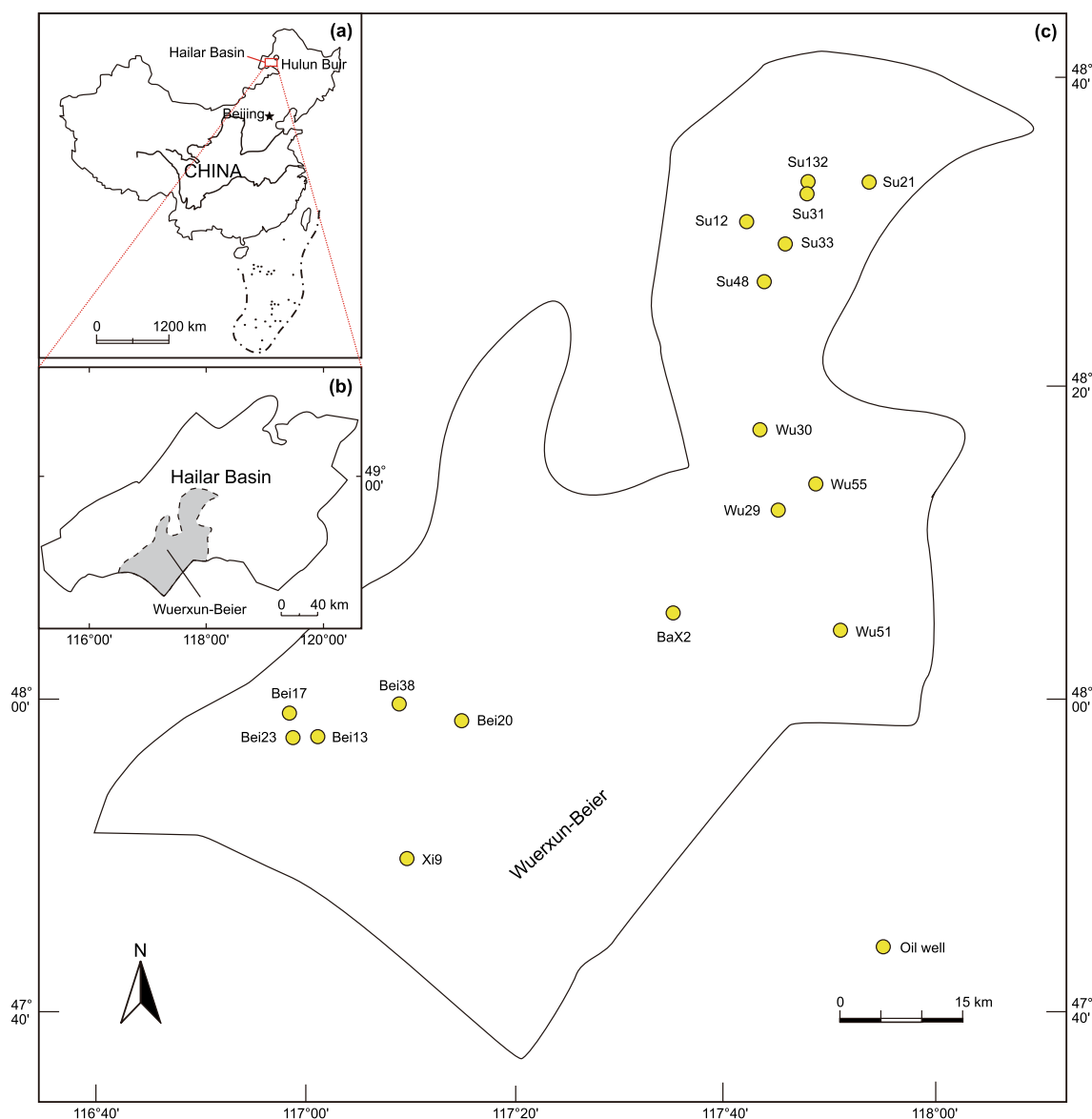


Fig. 1 **a** Schematic map shows the location of the Hailar Basin. **b** Location of the Wuerxun-Beier depression in the Hailar Basin (Modified after Gong 2012). **c** Locations of oil wells used for sampling in this study

tons (Ping et al. 2013), making it a major target of hydrocarbon exploration in the basin.

Six main sedimentary sequences are recognised in the Wuerxun-Beier depression resting on the Paleozoic

metamorphic basement (Fig. 2), from bottom to top the Lower Cretaceous Tamulangou, Tongbomiao, Nantun, Damoguaihe and Yimin Formations, as well as the Upper Cretaceous Qingyuangang Formation (Ping et al. 2013;

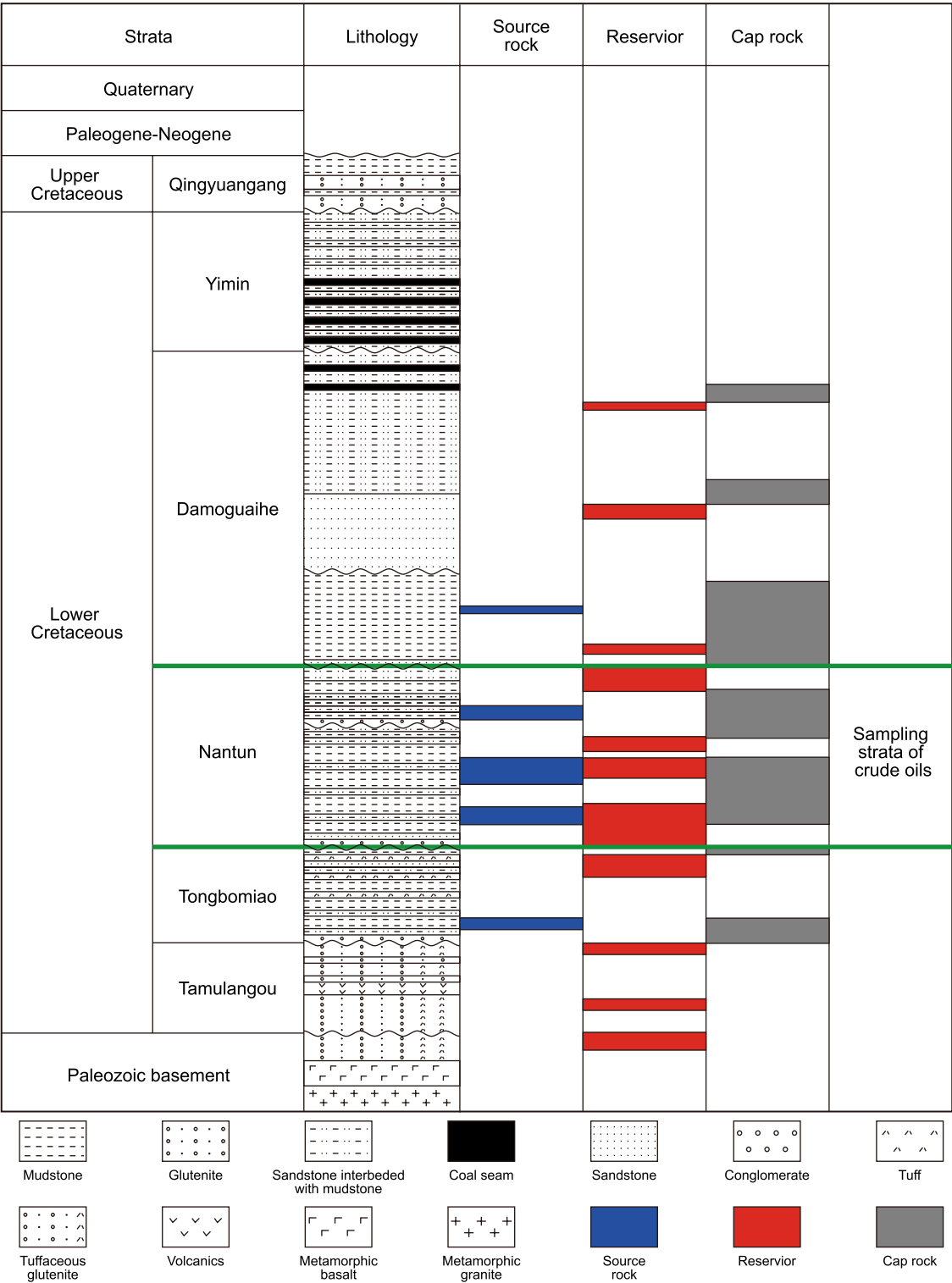


Fig. 2 Simplified stratigraphic column of the Wuerxun-Beier depression in the Hailar Basin

Wang et al. 2018). Previous studies observed possible source rocks (including organic-rich dark mudstones, coal mudstones, and coal seams, etc.) in the Tongbomiaio (K_1t) Formation, Nantun (K_1n) Formation, and Damoguaihe (K_1d) Formation (Fig. 2). The K_1t Formation source rocks mainly contain thermally mature to postmature Type II kerogen, and the K_1n Formation source rocks have both mature Type I and mature Type II kerogens. In contrast, the K_1d Formation source rocks contain immature Type III kerogen.

Recent investigations of oil-oil and oil-source rock correlations by chemometric methods suggest that the highest contributing source rocks for the Wuerxun-Beier petroleum system is the K_1n Formation, with the K_1t and K_1d Formations providing only a limited contribution (Wang et al. 2018). The K_1n Formation source rocks are lacustrine sediments ranging from deepwater mudstones to marshy deltaic sediments (Lu et al. 2012) with interbedded sandstones and mudstones (Fig. 2). Furthermore, because the petroliferous horizons in the Wuerxun-Beier depression occur mainly within the K_1n Formation, it has been argued that it functions as a stratigraphically self-contained (source, reservoir and seal unit) petroleum system in the Hailar Basin (Fig. 2) (Ping et al. 2013).

3 Samples and methods

Crude oil samples were recovered from 17 producing petroleum wells from the K_1n Formation in the Hailar Basin, providing a broad geographic distribution of Nantun oils from the Wuerxun-Beier petroleum system (Fig. 1). Because the asphaltene fraction (the heavy organic fraction) is the main carrier of Re (> 90%) and Os (> 83%) within typical crude oil samples (Selby et al. 2007; Liu and Selby 2018),

we separated asphaltene from the Wuerxun-Beier Nantun crudes to determine their $^{187}\text{Re}/^{188}\text{Os}$ and $^{187}\text{Os}/^{188}\text{Os}$ ratios.

The Re–Os isotopic analyses were carried out at the John de Laeter Centre (JdLC) at Curtin University in Perth, Western Australia. The asphaltene contents were separated from the Wuerxun-Beier Nantun crudes using a modified protocol from Selby et al. (2007). For each sample, 1 g of crude oil was weighed into a 50 mL centrifuge tube. For separation of the asphaltene component, 40 mL of *n*-heptane was added to each centrifuge tube. To thoroughly mix the oil and *n*-heptane, the tubes were first shaken by hand for 60 s and then placed on a Vari-Mix™ Platform Rocker at 200 rpm (revolutions per minute) for 24 h (Fig. 3a). The following day the samples were transferred into a refrigerator set to 5–6 °C and allowed to sit overnight. The samples were then removed from the refrigerator and centrifuged at 2500 rpm for 5 min, after which, in most samples, the asphaltene can be observed at the base of the centrifuge tube. Once each sample has been centrifuged, the *n*-heptane (and thus the maltene fraction) were decanted into a waste beaker (Fig. 3b). After being weighed, double-distilled chloroform (CCl_4) was then pipetted into each 50 mL centrifuge tube to transfer the asphaltene separate into Carius tubes for digestion. If a sample did not contain enough asphaltene after separation (about 120 to 200 mg of asphaltene was targeted for each Re–Os analyses), the process was repeated on another 1 g of crude oil and the asphaltene aliquots were mixed and weighed before being transferred to Carius tubes.

The Carius tubes were laid flat onto a sloped hotplate at 70 °C to evaporate all CCl_4 before digestion. After a Re and Os tracer was added to each sample, the samples were dissolved in inverse *aqua-regia* (3:1 HNO_3/HCl) at 240 °C within sealed Carius tubes (Fig. 4a) for at least 48 h (Selby and Creaser 2001). The solvent extraction method

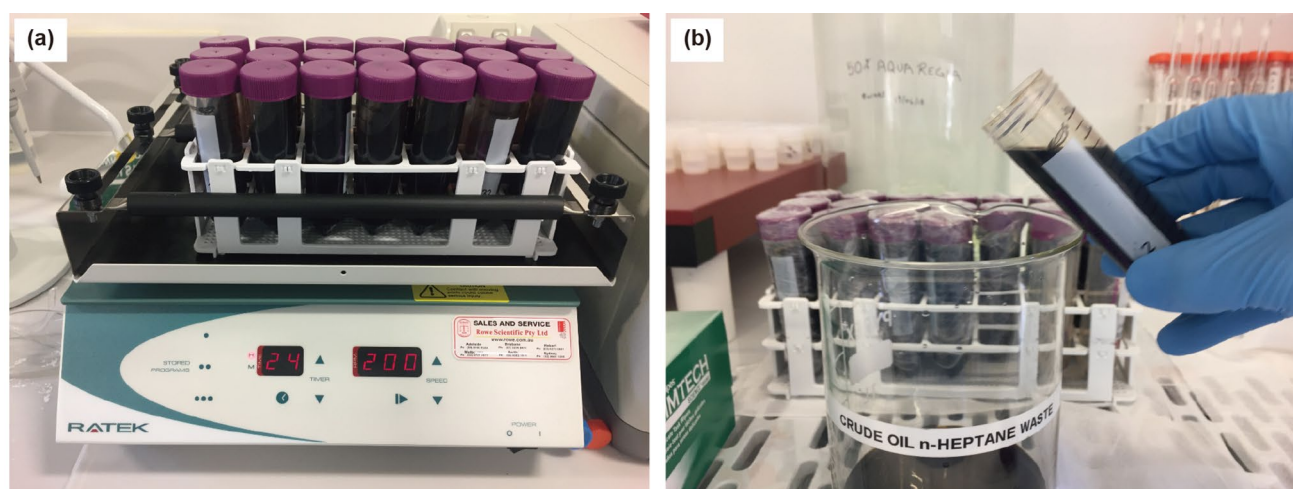


Fig. 3 Asphaltene separation for the Wuerxun-Beier Nantun oils in the Hailar Basin. **a** 1 g of oil sample in 40 mL of *n*-Heptane mixing overnight. **b** Decanting of *n*-Heptane and maltene leaving asphaltene separate in the bottom of the centrifuge tube

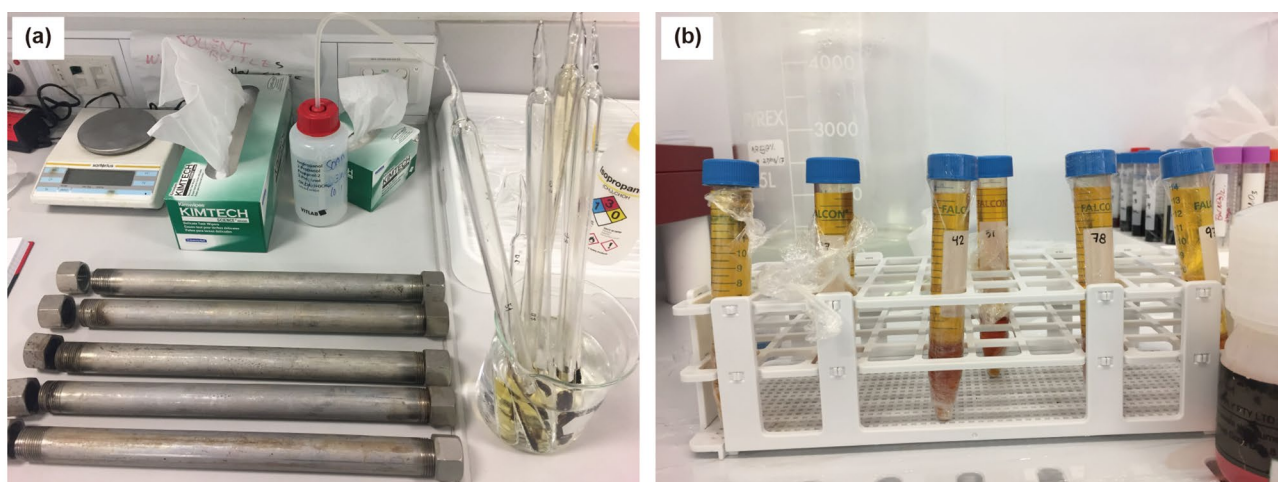


Fig. 4 Sample digestion and solvent extraction for the Wuerxun-Beier Nantun oils in the Hailar Basin. **a** Preparing sealed Carius tubes and protective metal jackets for digestion. **b** Oil samples in centrifuge tubes ready for centrifuge and Os solvent extraction. Reverse *aqua regia* (upper part) can be observed not mixing with the chloroform (lower part)

was utilised to extract the Os from the inverse *aqua-regia* (Fig. 4b) and a micro-distillation protocol was used to purify the Os for analyses (Brick et al. 1997; Selby and Creaser 2001). The Re was processed separately by taking the remaining inverse *aqua-regia* solution through an anion chromatography procedure.

The Os isotopic compositions were determined by the N-TIMS method (Negative-Thermal Ionization Mass Spectrometry) on a ThermoFisher Triton TIMS. The Re isotopic compositions were analysed using the ID-ICP-MS method (Isotope Dilution Inductively Coupled Plasma Mass Spectrometry) on a ThermoFisher ELEMENT XR ICP-MS. The average blanks during this study were 3.4 picogram for Re, and 0.02 picogram for Os. For the present analyses, the measured Re and Os concentrations of homogenised asphaltene isolated from NIST Research Material 8505 (RM 8505) crude oil were 16.128–16.699 parts per billion (ppb, 10^{-9}) and 162.31–166.88 parts per trillion (ppt, 10^{-12}), with $^{187}\text{Re}/^{188}\text{Os}$ and $^{187}\text{Os}/^{188}\text{Os}$ ratios of 571.6–573.2 and 1.612–1.640, respectively (Table 1), consistent with previously published data by Liu and Selby (2018). The Re–Os mass fractions and isotopic compositions of asphaltene fractions from the terrestrial crude oils in the Hailar Basin are presented in Table 2.

4 Results and discussion

4.1 Challenge of low Re–Os concentrations and mitigation procedures

The asphaltene fractions of the Wuerxun-Beier Nantun oils contain 0.047–5.308 ppb Re and 1.07–13.97 ppt Os (Table 2), values that are significantly lower than asphaltene fractions separated from crude oils in USA (Lillis and Selby 2013; Liu et al. 2019), UK (Finlay et al. 2011), Venezuela (Liu and Selby 2018), and the Persian Gulf (Liu et al. 2019), with tens to hundreds of ppb Re and several hundreds to thousands of ppt Os. Such low Re and Os concentrations may lead to higher analytical uncertainties, due to higher blank contributions (relative to the sample concentrations), as well as lower absolute precision of measured isotopic ratios for both Re and Os, induced by their low signal intensities at the limit of technique. To mitigate the influence of such challenges, we carried out thorough distillation of relevant chemicals (e.g. triple-distilled H_2O_2 -purified concentrated nitric acid) and used new clean Teflon beakers in order to reduce blank levels, and created a specific spike for these low-level samples to minimise the weight error and optimize spike/sample ratio.

Table 1 Re–Os mass fractions and isotopic compositions of homogenised asphaltene from NIST Research Material 8505 (RM 8505) crude oil

Sample	Re, ppb	2 σ	Os, ppt	2 σ	$^{187}\text{Re}/^{188}\text{Os}$	2 σ	$^{187}\text{Os}/^{188}\text{Os}$	2 σ
RM NIST 8505-1	16.128	0.405	162.31	0.58	571.6	15.0	1.612	0.017
RM NIST 8505-2	16.699	0.181	166.88	0.47	573.2	7.1	1.640	0.013

ppb parts per billion, 10^{-9} ; ppt parts per trillion, 10^{-12}

Table 2 Re–Os isotopic compositions of asphaltene fractions from the terrestrial crude oils in the Hailar Basin

Sample	Well	Depth, m	Formation	Re, ppb	2 σ	Os, ppt	2 σ	$^{187}\text{Re}/^{188}\text{Os}$	2 σ	$^{187}\text{Os}/^{188}\text{Os}$	2 σ	Rho
6	BaX2	1835–1939.8	Nantun	0.451	0.007	3.33	0.03	720.6	19.6	0.922	0.028	0.617
67	Bei13	1583–1593	Nantun	0.097	0.002	3.37	0.02	147.7	4.0	0.660	0.018	0.286
26	Bei17	1438–1453	Nantun	5.308	0.103	3.97	0.12	6614.6	485.0	0.337	0.076	0.301
25	Bei20	1887–1919	Nantun	0.598	0.004	3.15	0.07	920.9	50.0	0.162	0.033	0.260
27	Bei23	1456.4–1493	Nantun	0.280	0.004	13.97	0.31	100.7	5.4	0.465	0.036	0.660
104	Bei38	2299–2304	Nantun	0.107	0.002	2.54	0.02	211.4	6.1	0.415	0.013	0.370
10R	Su12	1309–1356.8	Nantun	0.083	0.010	2.45	0.07	172.6	23.4	0.594	0.053	0.377
3	Su132	1850–1856	Nantun	0.275	0.004	1.94	0.03	868.9	24.9	2.242	0.077	0.618
95	Su132	1876.8–1878	Nantun	2.497	0.052	12.68	0.09	1077.0	27.1	1.161	0.041	0.222
88	Su21	1188.8–1193	Nantun	0.054	0.001	7.67	0.19	34.3	2.1	0.197	0.036	0.279
77R	Su31	1744–1755	Nantun	0.117	0.022	2.29	0.07	277.4	56.4	1.099	0.095	0.315
52	Su33	1719.4–1799.4	Nantun	0.064	0.002	1.07	0.02	299.4	13.2	0.465	0.028	0.453
90	Su48	1848.4–1856	Nantun	0.105	0.003	6.51	0.08	80.3	3.0	0.377	0.019	0.423
16	Wu29	2493–2572	Nantun	0.259	0.002	3.38	0.12	387.1	33.7	0.487	0.061	0.691
17	Wu30	1671–1675	Nantun	0.710	0.011	2.20	0.07	2846.7	124.4	6.469	0.321	0.763
21	Wu51	1471–1474	Nantun	0.047	0.001	1.07	0.01	221.8	8.1	0.462	0.016	0.428
42R	Wu55	1526.6–1529	Nantun	0.492	0.015	1.12	0.02	2126.3	125.9	0.197	0.029	0.287
51	Xi9	2620–2624	Nantun	0.497	0.004	4.76	0.36	503.0	87.1	0.131	0.089	0.253

ppb parts per billion, 10^{-9} ; ppt parts per trillion, 10^{-12} ; Rho, associated error correlation at 2σ (Ludwig 1980)

4.2 Re–Os geochronology of the Hailar oils

The samples have variable $^{187}\text{Re}/^{188}\text{Os}$ ratios ranging from 34.3 to 6614.6, and exhibit $^{187}\text{Os}/^{188}\text{Os}$ ratios ranging from relatively non-radiogenic (0.131) to radiogenic (6.469) (Table 2). Considering that the source wells range from 10 to 100 km apart, it is unlikely that the Wuerxun-Beier crude samples represent a single population despite them all being sourced from the K_1n Formation (Lu et al. 2012; Ping et al. 2013; Wang et al. 2018). The fact that no single linear trend is evident in the Re–Os results (Fig. 5) confirms the lack of a single population, with the regression of all Nantun oils yielding only a nominal Model 3 (indicating excess, “geologic” scatter) Re–Os age of 13 ± 28 Ma (2σ), with significant uncertainty (215%) and a considerable mean square weighted deviation (MSWD) of 456.

Three separate trends, however, were observed in the results (Fig. 5), and each subgroup was regressed individually to test for possible age significance (Table 3 and Fig. 6). Population A trend consists of seven oils which yielded an Early Cretaceous age of 131.1 ± 8.4 Ma, with an initial $^{187}\text{Os}/^{188}\text{Os}$ value of 0.29 ± 0.12 (Fig. 6b). Population B consists of six oils yielding a Tertiary age of 54 ± 12 Ma, and an initial $^{187}\text{Os}/^{188}\text{Os}$ value of 0.22 ± 0.11 (Fig. 6c). Finally, Population C represents five relatively non-radiogenic oils yielding a Pleistocene age of 1.28 ± 0.69 Ma and an initial $^{187}\text{Os}/^{188}\text{Os}$ value of 0.16 ± 0.02 (Fig. 6d).

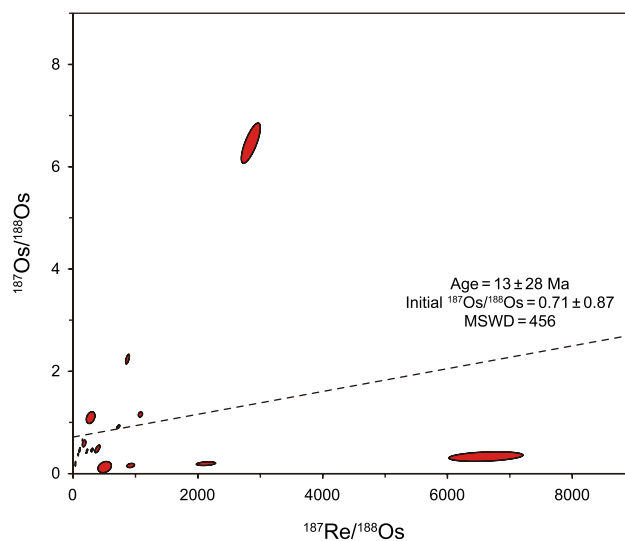


Fig. 5 $^{187}\text{Re}/^{188}\text{Os}$ versus $^{187}\text{Os}/^{188}\text{Os}$ plot showing all of the Re–Os data for the Wuerxun-Beier Nantun oils in the Hailar Basin. The nominal Model 3 (indicating excess, “geologic” scatter) Re–Os age was regressed using the ISOPLOT program (ver. 4.15) (Ludwig 2012). Data point uncertainties are 2 sigma (2σ). MSWD, mean square weighted deviation

4.3 Linking Re–Os ages to petroleum system evolution

The Early Cretaceous Re–Os isochron age of 131.1 ± 8.4 Ma for the Wuerxun-Beier Nantun oils (Table 3 and Fig. 6b) is

Table 3 Re–Os geochronology results of terrestrial crude oils in the Hailar Basin

Population No	<i>n</i>	Age (Ma)	2σ	MSWD	Model	Os _i	2σ	Samples	Description
A	7	131.1	8.4	26	3	0.29	0.12	3, 10R, 17, 27, 67, 77R, 90	Early Cretaceous hydrocarbon generation
B	6	54	12	15	3	0.22	0.11	6, 16, 21, 52, 95, 104	Cenozoic multiple hydrocarbon generation or expulsion
C	5	1.28	0.69	2.4	1	0.161	0.024	25, 26, 42R, 51, 88	Cenozoic multiple hydrocarbon generation or expulsion

n number of samples, *MSWD* mean square weighted deviation
Model number is from Ludwig (2012); Os_i, initial ¹⁸⁷Os/¹⁸⁸Os value

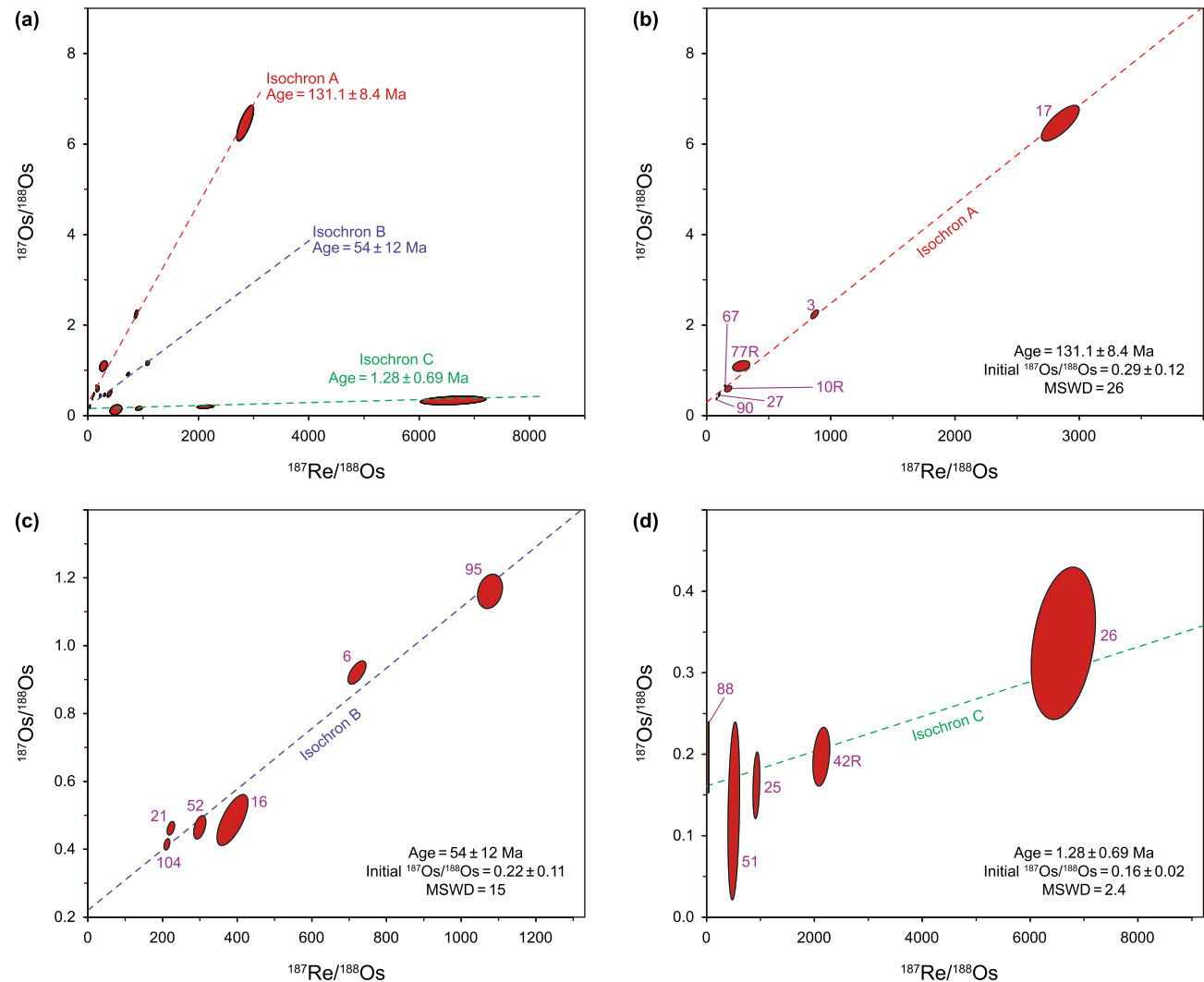


Fig. 6 Re–Os isochron diagrams for the Wuerxun-Beier Nantun oils in the Hailar Basin, showing the calculated regression ages. Regressions were calculated and plotted with the ISOPLOT program (ver. 4.15) (Ludwig 2012). Data point uncertainties are 2 sigma (2σ). The data labels in italics indicate the oil sample numbers listed on Tables 2 and 3. MSWD, mean square weighted deviation. **a** Re–Os isotope data of all the Wuerxun-Beier Nantun oils. **b** Re–Os isochron for Population A. **c** Re–Os isochron for Population B. **d** Re–Os isochron for Population C

equivalent to the chronostratigraphic age of the K_1n Formation (131–136 Ma; He et al. 2013), and suggests that these hydrocarbon samples may have been generated within 10 million years of deposition indicating a period of rapid sedimentation and burial. The Re–Os age is concordant with independently derived thermal models of hydrocarbon generation based on apatite fission-track dating (Cui and Ren 2011).

Regressions of the remaining oils yield two Cenozoic Re–Os isochron ages of 54 ± 12 Ma (Population B) and 1.28 ± 0.69 Ma (Population C) (Table 3 and Fig. 6c, d) implying that the Wuerxun-Beier Nantun oils do not represent a single population despite them all being sourced from the K_1n Formation (see Sect. 2). Apatite fission-track dating (Cui and Ren 2011) has demonstrated that multiple re-heating events have occurred since the Tertiary, which if correct, provides corroboration that the Cenozoic Re–Os ages can be interpreted as oil maturation-expulsion events linked to a later period of basin evolution. Structural geology and stress field analysis studies (Liu et al 2012) also provide evidence for Cenozoic disturbances of the Hailar Basin, yet it is unknown whether these events are significant enough to generate significant volumes of compartmentalised hydrocarbons.

As illustrated by this study in the Hailar Basin, Re–Os radiometric dating of lacustrine-sourced natural crude oils can be extremely challenging. This is due not only to higher analytical uncertainties for the low to ultra-low Re and Os concentrations in the asphaltene fractions of some oil samples (Table 2), but also be further hampered by multiple tectonic and hydrocarbon generation events in many basins (Cui and Ren 2011; Liu et al 2012). However, hydrocarbon Re–Os geochronology can aid in quantitatively constraining the temporal evolution of petroleum system in a terrestrial petroliferous basin, especially when the results can be cross-checked and augmented by additional dating methods (e.g. apatite fission-track, fluid inclusions analysis, basin modelling, in situ U–Pb dating of carbonates, and in situ Rb–Sr and (U–Th)/He dating of siliciclastic sediments, etc.) and organic geochemistry (e.g. chemometric methods, biomarkers, clumped isotope, and site-specific stable isotope technologies, etc.).

5 Conclusions

This study presents new Re–Os geochronology for lacustrine crude oils from the Wuerxun-Beier depression of Hailar Basin in NE China. Corroborated with previous basin modelling and apatite fission-track analysis, the Re–Os dating of these oils is interpreted to provide an absolute age of 131.1 ± 8.4 Ma of an Early Cretaceous hydrocarbon generation event in the Wuerxun-Beier lacustrine petroleum

system. Further work is necessary to validate the geological significance of the younger Re–Os isochron ages of 54 ± 12 Ma and 1.28 ± 0.69 Ma. This case study represents the first successful application of the Re–Os geochronometer to quantitatively constrain the absolute timing of petroleum generation in a terrestrial basin.

Acknowledgements This research was funded by Daqing Oilfield Co., Ltd. project entitled “Geochronology and oil-source rock correlations in complex reservoirs (No. DQYT-1201002-2018-JS-150)”, and PetroChina Co., Ltd. project (No. 2016E-0202). The authors would like to appreciate Dr. Chunhua Shi for his assistance with sampling.

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