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Petroleum generation timing and source in the Northern Longmen Shan Thrust Belt, Southwest China: Implications for multiple oil generation episodes and sources

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Petroleum generation timing and source in the Northern Longmen Shan Thrust Belt, Southwest China: Implications for multiple oil generation episodes and sources

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Abstract

The temporal evolution of hydrocarbons (~500 million barrels oil) and its relationship to the orogenic events of the Longmen Shan Thrust Belt have been extensively debated. The hydrocarbons occur as solid bitumen, as dykes and/or coatings within/along faults/fractures, and as present day oil seeps. Here utilizing organic geochemistry, we demonstrate that all the bitumen exhibit similar organo-gechemical characteristics, and were sourced from the Late Neoproterozoic–Early Cambrian Doushantuo and Qiongzhusi formations. In contrast, the organic geochemistry of the present day oil seeps are distinct from that of the bitumen, and suggest that the source is the Permain Dalong Formation.

Bitumen rhenium-osmium data indicate that the Late Neoproterozoic–Early Cambrian Doushantuo and Qiongzhusi formations underwent two temporally distinct oil generation events; initially during the Early Ordovician (ca.486 Ma) prior to the Caledonian Orogeny, and secondly during the Jurassic (ca.165 Ma) coinciding with the Indosinian-Yanshan orogenies. In contrast, the rhenium-osmium data of the present day oil seeps are too similar to yield a meaningful age, although the source is considered to have underwent hydrocarbon maturation between the Triassic and Jurassic. The temporal hydrocarbon evolution in the the Longmen Shan Thrust Belt also provides implication for the hydrocarbon evolution and future exploration of the adjacent petroliferous Sichuan Basin.

1. Introduction

Source rock burial and maturation history, coupled with hydrocarbon generation and subsequent migration are key factors of a petroleum system, which are often temporally associated with regional tectonic events (Bordenave and Hegre, 2005; Moretti et al., 1996; Urien et al., 1995; Yahi et al., 2001). For example, (1) in the Berkine (Ghadames) Basin, eastern Algeria, hydrocarbon maturation of the Silurian, Llandoverian – Wenlockian souce rock and associated oil generation directly relates to the timing of the Cretaceous Austrian Orogeny (Yahi et al., 2001), and (2) in the foreland of the Sub Andean Zone in Bolivia, three stages of tectonic accretion are suggested to have controlled three phases of sedimentation and oil generation during the Cenozoic (Moretti et al., 1996; Urien et al., 1995).

The key to understanding the direct relationship between tectonism and the evolution of a petroleum system are the accurate estimates for the timing of the related tectonism and that of the hydrocarbon generation, expulsion and accumulation. Recent successes in determining age constraints and the relationship between tectonism and petroleum evolution has been achieved through the application of both radiometric (e.g., Re-Os, Ar-Ar, Apatite Fission Track (AFT)) and indirect techniques (e.g., basin/tectonic models) (Boles et al., 2004; Fall et al., 2015; Ge et al., 2016).

The Sichuan Basin in the South China Block records multiple tectonic events (e.g., Ordovician-Devonian Caledonian, Late Triassic Indosinian, Late Jurassic Yanshan, and the Cenozoic Himalaya orogenies) (Chen and Wilson, 1996; Dai et al., 2009; Harrowfield and Wilson, 2005; Jin et al., 2010; Sun, 2011; Yan et al., 2011). The majority of the hydrocarbon reserves of the Sichuan Basin are distributed close to its border regions (e.g., Longmen Shan Thrust belt, Micang Shan Uplift, Daba Shan Orogenies) (Li et al., 2015; Li et al., 2001; Liu et al., 2011; Ma et al., 2010) (Fig. 1B). The basin has current reserve estimates of ~30 Bbbl (billions of barrels) of oil and ~180 Tcf (Trillion cubic feet) of gas (Zhang and Zhu, 2006; Zou et al., 2014a). Key examples are the giant Puguang Gas field that is located adjacent to the Daba Shan orogenic belt in the northeast Sichuan Basin which possesses ~12 proven original in-place Tcf gas (Ma et al., 2007b), the great Yuanba gas field (~2 Tcf proven gas) that lies near the Micang Shan Uplift in the northern Sichuan Basin (Liu et al., 2011), and several gas fields possessing ~1 Tcf gas (e.g., Dayi, Majing and Pingluoba) that occur near the Longmen Shan Thrust belt (Liu et al., 2011) (Fig. 1B). In addition to the gas fields in the Longmen Shan Thrust belt, hydrocarbons are present as bitumen. The total bitumen accumulation, which is recoverable is estimated to yield a reserve in excess of 500 million barrels (Mbbl) of oil (Liu et al., 2003).

The Longmen Shan Thrust belt is located between the Songpan-Ganze Terrane and the Sichuan Foreland Basin, and marks the western margin of the Sichuan Basin (Fig. 1A). The belt is tectonically complex due to multiple orogenic events from the Palaeozoic to present (Caledonian, Indosinian–Yanshan, Himalaya) (Jin et al., 2010; Yan et al., 2003). Abundant hydrocarbons predominantly occur in Neoproterozoic to Permian strata and are typically spatially associated with thrust faults and associated fracture systems (Fig. 1C) (Dai et al., 2009; Huang and Wang, 2008; Liu et al., 2003). To date, the origin, age and the evolution of the hydrocarbons is debated. For example, either organic-rich strata of the Late Neoproterozoic-Early Cambrian (Dai et al., 2009; Liu et al., 2009; Tian, 2009; Wei et al., 2008; Xie et al., 2003) or Permian (Liu et al., 2003; Rao et al., 2008; Wang et al., 1997) are considered to be the main source rocks. Additionally, basin burial history and fluid inclusion analyses propose multiple hydrocarbon generation and migration events, e.g., between the Ordovician and Silurian (Wang and Li, 1999; Wei et al., 2008) and during the Late Triassic (Liu et al., 2009), as well as during the Cenozoic (Liu et al., 2003; Rao et al., 2008).

As a petroleum component, bitumen records significant information regarding petroleum evolution, including hydrocarbon generation, migration, accumulation and alteration (Hwang et al., 1998; Parnell and Swainbank, 1990; Selby et al., 2005; Summons et al., 2008; Zhu et al., 2001). Recently, Re-Os isotope dating of oil and bitumen has shown good potential for determining the absolute timing of hydrocarbon generation (Cumming et al., 2014; Finlay et al., 2011; Ge et al., 2016; Georgiev et al., 2016; Lillis and Selby, 2013; Selby and Creaser, 2005; Selby et al., 2005; Selby et al., 2007). In this study, we present new Re-Os data and organic geochemistry of bitumen and present day oil seeps from the Northern Longmen Shan Thrust belt. The data are discussed with the previous hydrocarbon evolution knowledge, as well as U-Pb, Ar-Ar and Apatite Fission Track (AFT) dates that constrain the timings of tectonism to understand the petroleum evolution and its relationship with tectonism. Our data provide not only an improved understanding of the petroleum evolution within the Longmen Shan Thrust belt, but also provides implications for the potential utility of Re-Os hydrocarbon chronometer to help constrain the absolute timing of both hydrocarbon generation and associated tectonism in petroleum systems worldwide.

2. Geological Setting

The NE-SW striking Longmen Shan Thrust belt is ~500 km long and ~50 km wide. The belt is bordered by the Micang Shan uplift to the north, the Kangdian paleo uplift to the South, the Songpan-Garze Belt to the west, and the Sichuan Basin to the east (Burchfiel et al., 1995; Dirks et al., 1994; Jin et al., 2010) (Fig. 1C). Longitudinally, the Longmen Shan Thrust belt is divided into three sub-structural belts by four major faults: the Maoxian-Wenchuan, Beichuan-Yingxiu, Anxian-Dujiangyan and the Guangyuan-Dayi faults (Fig. 1C). Further, the belt can also be separated geographically into three areas: the northern, middle and southern segments (Fig. 1C) (Chen and Wilson, 1996; Deng et al., 2012; Jin et al., 2010; Li et al., 2008; Liu et al., 2016; Wang et al., 2015; Yan et al., 2011).

The Longmen Shan Thrust belt has experienced a complex tectonic evolution since the Early Palaeozoic (Chen and Wilson, 1996; Dai, 2011; Jin et al., 2010; Yan et al., 2011). The initial tectonic events were associated with the Palaeozoic Caledonian Orogeny caused by the closure of the Tethys ocean, with thrusting causing numerous unconformities between the Early Palaeozoic strata (Jin et al., 2010). Following the Caledonian Orogeny (Early Devonian), the Longmen Shan belt changed into a passive continental margin throughout the Devonian and Permian that was associated with extensional tectonism (Jia et al., 2006; Li et al., 2012; Tian, 2009; Zhou et al., 2013). The most severe deformation recorded in the Longmen Shan thrust belt relates to the Late Triassic to Early Cretaceous NW to WNW directed under-thrusting of the South China block beneath the North China block (Chen and Wilson, 1996; Dai et al., 2009; Jin et al., 2010; Liu et al., 2005; Yan et al., 2011). These tectonic events resulted in structural unconfomities within the Triassic and between the Upper Triassic and Lower Jurassic strata (Tian, 2009), numerous faults (e.g., Beichuan-Yingxiu Faults, Anxian-Dujiangyan Faults and the high angle reverse fault in this study (Fig. 3B)) (Arne et al., 1997; Chen et al., 1995; Wilson et al., 2006), intensive folding of the Jurassic strata and led to the uplift and erosion of Cretaceous strata (Li et al., 2008). The absolute timing of tectonism is constrained by Sensitive High Resolution Ion Microprobe analysis (SHRIMP) U–Th–Pb monazite, conventional U-Pb titanite, Sm–Nd garnet, and Rb–Sr muscovite and biotite ages on metamorphic rocks from the Danba Domal Metamorphic

Terrane ~100 km northwest of the southern sector of the Longmen Shan Thrust Belt. The available geochronology yield three age groups (ca. 200, ca. 160 and ca. 120 Ma) (Huang et al., 2003; Jin et al., 2010; Jin et al., 2008; Yan et al., 2011). Further age constraints for the timing of tectonism are given by $^{40}Ar/^{39}Ar$ garnet and zircon fission track dates (ca. 110 - 130 Ma) from the middle district of the Longmen Shan Thrust Belt (Liu et al., 2001), and $^{40}Ar/^{39}Ar$ muscovite and sericite dates (ca. 237 - 183 Ma) from the basement complex, detachment fault zone and ductile deformation zone from the northern part of the Longmen Shan Thrust Belt (Yan et al., 2011). The most recent tectonism recorded by the Longmen Shan Thrust Belt occurred during the Cenozoic as a result of the India-Asia continental collision (Dai, 2011; Li et al., 2008; Yan et al., 2011). This event further reactived previous existing thrust faults and caused exhumation along the belt (Harrowfield and Wilson, 2005; Lei et al., 2012; Yan et al., 2011). Low-temperature thermochronology, such as apatite fission track (AFT) and (U-Th)/He methods, indicate a series of uplift events since the Late Cretaceous (Arne et al., 1997; Deng et al., 2012; Lei et al., 2012; Yan et al., 2011). Additionally, present day activity along the Longmen Shan Thrust Belt is evidenced by the 2008 Wenchuan earthquake (7.9 M_w – epicentre in Wenchuan City) and the 2012 Ya'an earthquake (6.6 M_w – epicentre in Ya'an City) (Feng et al., 2014).

The northern segment of the Longmen Shan Thrust Belt, located to the north of Anxian County, extends for ~200 km (Fig. 1C). This segment of the belt contains several major thrust sheets and a blind frontal thrust zone, with most of the folds and thrust sheets emplaced towards the southeast (Jia et al., 2006; Jin et al., 2010; Jin et al., 2009a). Precambrian to Quaternary strata are present in the northern Longmen Shan Thrust Belt (Jia et al., 2006; Jin et al., 2009a). The Precambrian to Cambrian units mainly consist of organic-rich black shales and siltstones with a total thickness of ~200 m (Rao et al., 2008; Wang et al., 2005; Xie et al., 2003). The Ordovician to Silurian units are largely absent due to uplift and erosion during the Caledonian Event (ca. 450 – 400 Ma) in the Yangtze Block. Devonian and/or Carboniferous strata, which mainly consist of dolomite and limestones, uncomformably overlie the older units and possess a thickness ~50 – 250 m. The Permian strata, which have a total thickness ~270 - 470 m, consist of limestone and black shales (Rao et al., 2008; Wang et al., 2005; Xie et al., 2003). The Early-Mid Triassic strata include ~750 m of limestones interbedded with sandstones or shales. The Late Triassic units of ~400 m thickness comprise interbedded sandstones and mudstones (Zhou et al., 2013). The overlying Jurassic and Cretaceous fluviolacustrine sediments comprise mudstones, sandstones and siltstones with a total thickness of up to 4500 m (Fig. 2).

The Upper Neoproterozoic to Lower Cambrian shales and middle Permian black mudstones are considered as the potential source rocks (Xie et al., 2003; Zhou et al., 2013) to several petroleum systems (e.g., Ningqiang, Tanjingshan and Kuangshanliang) in the northern Longmen Shan Thrust Belt (Chen et al., 1994; Huang and Wang, 2008; Tissot and Welte, 1984). Reservoir units include the Upper Cambrian, Lower Devonian, Lower and Upper Permian, Lower Triassic and Upper Jurassic carbonates, sandstones and/or siltstones (Chen and Wilson, 1996; Li et al., 1999; Worley and Wilson, 1996; Zhou et al., 2013). Devonian marine mudstones, Triassic gypsum, and Jurassic to Cretaceous mudstones act as seals to the several petroleum systems (Zhou et al., 2013) (Fig. 2).

The Kuangshanliang petroleum system is characterized by the largest and most complete anticline in the frontal thrust zone of the northern Longmen Shan Thrust Belt

(Fig. 3A) (Chen et al., 2005). The anticline comprises Cambrian strata in the core, surrounded by Ordovician to Triassic units (Fig. 3A). Bitumen and present day oil seeps in the Kuangshanliang anticline occur in the 565 m thick marine clastics of the Lower Cambrian Changjianggou Formation (Fig. 3A, B) as dykes or along faults and/or fractures that trend NW-SE. The calculated total hydrocarbon reserves in the Kuangshanliang anticline are ~70 Mbbl of oil (Tian, 2009).

3. Samples

Bitumen and oil seep samples were collected between 2010 and 2011 from field outcrops of bitumen dykes, fault planes, fault zones, fractures and seeps, in the Cambrian Changjianggou Formation (Figs. 3, 4). The densely forested nature of the area has resulted in there being no known outcrops of the bitumen dykes. The bitumen dykes were only accessible through old mine adits. Samples were obtained through three old mine adits approximately 8 km north of Shangsi, Jiange County (Fig. 3A). Access to these adits has been prohibited since 2012 for health and safety reasons. The dykes are ~0.5 - 10 m wide and occur over a known strike (NW) distance of ~50 m. The dykes typically run parallel to faults that show a dextral motion and dip ~35º towards the southwest. The contacts between the bitumen dykes and the country rock are sharp, although bitumen also impregnates the wallrocks up to ~10 cm from the edge of the dyke.

Two dykes (Dykes 1 and 2) are located within ~500 m of each other. Dyke 1 was accessed from the mine adit Shang Kuang Dong (upper bitumen hole). The dyke strikes NW over a distance of at least 100 m, and averages a width of 70 - 100 cm. The eastern contact with the Cambrian Changjianggou Formation sandstone is sharp, but the western contact is brecciated (Fig. 4A). The breccia zone is ~10 cm wide and contains 1 - 5 mm clasts of both the country rock (sandstone) and bitumen. Inward from the brecciated area, the western edge of Dyke 1 is intensively fractured over 10-20 cm. Three bitumen samples (11SKD-3d, 11SKD-4d and 11SKD-5d) were taken, ~2 m apart, from the centre of the dyke, which represents the least fractured part of Dyke 1 (Fig. 4B). A fourth sample (SKD-1f) was taken from a cross-cutting bitumen-filled fracture (Fig. 4B). Dyke 2 was accessed from mine adit Xia Kuang Dong (lower bitumen hole). Two bitumen samples (XKD-1d; XKD-2d) were collected from the center of this dyke, ~5 m apart (Fig. 4C). Dyke 2 is narrower than Dyke 1 (20 - 50 cm), and strikes NW for at least 100 m. Both the western and eastern contacts with the host rock are sharp, with the adjacent country rocks being impregnated with bitumen up to 10 cm from the contact. In contrast to Dyke 1, the eastern margin of Dyke 2 is part of a low-angle reverse fault (Fig. 4C). The fault plane strikes NE and dips 34° to the NNW, with transport direction towards the SE. These fault characteristics are consistent with the overall pattern and geometry of thrusting in the northern Longmen Shan Thrust belt (Chen et al., 2005; Tian, 2009). The fault plane and associated gouge is confined to a 5 cm thick, clay-rich siltstone interbed of the Cambrian Changjianggou Formation sandstone. The gouge has a sharp upper contact and diffuse basal contact. The bitumen impregnated into the sandstone country rock indicates a SE-ward fault motion syn-post emplacement of the bitumen Dyke 2 (Fig. 4D).

Seven additional bitumen samples (GY1d-6d and HSCD-1d) were collected from a third Dyke (Dyke 3) from the Huoshicun mine hole, ~2 km to the south of Dykes 1 and 2. Samples in Dyke 3 were collected along the centre of the main bitumen dyke body (Fig. 4E). Dyke 3 has a similar strike orientation to that of Dykes 1 and 2 (NW), and possesses a similar width to Dyke 2 (30 – 50 cm). Both the eastern and western dyke contacts are sharp, with little to no bitumen impregnated into the sandstone, and with no evidence of post-emplacement fracturing or faulting observed.

In the Northern Longmen Shan Thrust Belt, dextral strike-slip faults striking NE and dipping 30-40º NW are well developed (Burchfiel et al., 1995; Chen et al., 2005; Yan et al., 2011). In this study, the bitumen samples were also collected from two separate faults (Fig. 4F,G). Samples LXB-1f and LXB-2f were collected from the same fault, which strikes NE and dips 75º NW (Fig. 4F). The samples were taken ~2 m apart. Sample 11LXB-1f was collected from part of a slickenline striking NE with a dip of 45º NNW (Fig. 4G).

At the Huoshicun mine hole, oil was observed seeping through the Changjianggou Formation sandstone, three oil samples (Oil-3, Oil-5, Oil-7) were collected over a distance of 1 m (Fig. 4H).

4. Analytical Protocols

The Gas Chromatography (GC) and Gas Chromatography-Mass Spectrometry (GC-MS) analyses on nine bitumen samples (GY-1, 3, 5, 11SKD-4, 11LXB-1, HSCD-1, SKD-1, XKD-1 and LXB-1) and two present day oil seep samples (Oil-3 and Oil-5) were conducted in Wuxi Institute of Petroleum Geology, Sinopec, China and Weatherford Laboratories, USA, following the analytical procedure of Hackley et al., 2013 . The bitumen samples were first cleaned with distilled water, to ensure there were no weathered contaminants on the surface, and then crushed to 100 mesh size using an agate pestle and mortar. Approximately 100 g of bitumen was put into a Soxhlet extractor for 72 hours to obtain the chloroform extract (asphalt). The asphalt was then precipitated using n-hexane. For the oil samples, ~30 mg of crude oil sample was dissolved in 50 mL of *n*-hexane and left for 12 h at room temperature. The solution was then filtered, with all the filtrates collected and evaporated under nitrogen gas to 0.5 mL. A chromatographic column (30 cm × 10 mm in diameter) was prepared using a mixed stationary phase of activated silica gel and alumina at a ratio of 3:2 by referring to relevant literature (e.g., Yang et al., 2009). The concentrated sample was transferred to the chromatographic column for further separation. The saturated hydrocarbon fraction was eluted with *n*-hexane (25 mL). The fractions were then carefully concentrated under nitrogen flow to 0.5 mL for GC-MS analysis. The GC-MS system consisted of an Agilent 7890 GC, and an Agilent 5975C mass spectrometer. A DB-5MS column 50 m × 0.25 mm × 0.25 μm was used. High purity helium (99.9995%) was used as a carrier gas at a flow rate of 1.0 mL/min. The injector temperature was 300°C. The injection volume was 1.0 L. All injections were done with a 7683B series autosampler. The oven temperature was programmed from 50°C (1 min hold) to 100°C at 10°C /min, and then to 310°C (20 min hold) at 2°C /min. The mass spectrometer was operated in the electron impact mode (70 eV). The temperature of ion source and transfer-line were set at 230°C and 300°C, respectively. The scanned mass range was from 50 to 550 u. The temperature of the Quadrupole was held at 150°C.

For Re-Os analysis, approximately ~0.2 - 1.0 g bitumen was separated from the 16 samples. All samples were isolated without metal contact and handpicked. Samples were crushed to ~1 mm grains using an agate pestle and mortar. For the oil seeps, the asphaltene fraction was analyzed as Re and Os are predominantly contained within the asphaltene fraction of oil (Selby et al., 2007). The asphaltenes were precipitated from the oil using 40 times volume of *n*-heptane (~1 g oil with 40 ml solvent) at room temperature for at least 8 hrs. The asphaltene abundance of the present day oil seeps are between 9.48 and 13.54 % (Table 1). The Re and Os isotopic compositions, and abundances of the bitumen and asphaltene from the oil were analysed at the Laboratory for Source Rock and Sulfide Geochronology and Geochemistry (a member of the Durham Geochemistry Centre) at Durham University following published analytical procedures (e.g. Selby et al., 2005; Selby et al., 2007). Approximately 100 - 200 mg of bitumen or asphaltene were dissolved and equilibrated with a known amount of ^{185}Re and ^{190}Os spike solution by inverse *aqua-regia* (3 ml HCl and 6 ml HNO_3) in a Carius tube for 24 hours at 220˚C. Osmium was isolated and purified from the inverse *aqua-regia* by $CHCl_3$ solvent extraction at room temperature and micro-distillation. The Re was isolated using HCl-HNO_3 based anion chromatography. The purified Re and Os were loaded on Ni and Pt filaments and analyzed using Negative Ion Thermal Ionization Mass Spectrometry (N-TIMS). Measured Re and Os ratios were corrected for mass fractionation using ^{185}Re/^{187}Re = 0.59738 (Gramlich et al., 1973) and ^{192}Os/^{188}Os = 3.08261, spike and blank contributions. All data were blank corrected based on the total procedural blanks values of Re (1.6 ± 0.025 pg) and Os (0.05 ± 0.004 pg), with an average ^{187}Os/^{188}Os ratio of ~0.22 ± 0.06 (n = 4). All uncertainties include the propagated uncertainty in the standard, spike calibrations, mass spectrometry measurements, and blanks. In-house Re (Restd) and Os (AB2) solutions were analyzed as a monitor of reproducibility of isotope measurements. The analyses presented in this study were conducted prior to using DROsS as our in-house control solution (Nowell et al., 2008). The ^{187}Os/^{188}Os values of the Os standard solution AB2 during this study were 0.1611 ± 0.0066, with the ^{185}Re/^{187}Re values of the Re standard solution being 0.5984 ± 0.0002. These values are in agreement with those previously published for

AB2 and Restd (Cumming et al., 2014; Finlay et al., 2011, 2012; Lillis and Selby, 2013; Rooney, 2011). The $^{185}Re/^{187}Re$ ratios for samples of this study were corrected for the measured difference of the $^{185}Re/^{187}Re$ value for Restd and the $^{185}Re/^{187}Re$ value of 0.59738 ± 0.00039 (Gramlich et al., 1973). The Re–Os data of this study are regressed using the program *Isoplot* V. 4.15 (Ludwig, 2003) with ^{187}Re decay constant of $1.666\times10^{-11}a^{-1}$ (Smoliar et al., 1996). The input data contains $^{187}Re/^{188}Os$ and $^{187}Os/^{188}Os$ ratios with their total 2σ uncertainty and associated error correlation, Rho.

5. Results

This study presents results of organic geochemistry for nine bitumen and two present day oil seeps, and Re-Os data for the sixteen bitumen samples and three present day oil seeps. The detailed results of the organic geochemistry and Re-Os analysis are presented below.

5.1 GC-MS results

Nine samples from the three bitumen dykes and bitumen from faults/fractures were selected for detailed organic geochemistry analysis (Table 1). Component analysis show that the asphaltene fraction occupies more than 98 % of the total bitumen sample (Table 1). The saturate fraction gas chromatograms (SFGCs) of the analyzed bitumen samples are dominated by humps of unresolved compounds (UCM) with some discrete peaks superimposed (Fig. 5). The UCM exhibited by the SFGCs indicates that the samples have been extensively biodegraded with compounds, such as *n*–alkanes and acyclic isoprenoids, removed by microbial action. This is also supported by the presence of Nor-25-hopane (Wenger and Isaksen, 2002) (Fig. 5). Gas chromatograms show that the

Carbon Preference Index (CPI) value based on the formula (NC23+NC25+NC25)+ (NC25+NC27+NC29)/(2*(NC24+NC26+NC28)) ranges from 0.35 to 2.59, with majority of samples possessing CPI values between ~0.91 and 1.21 (except LXB-1f and HSCD-1d). The calculated Pr/Ph ratio for samples 11SKD-4d (Dyke 1), XKD-1d (Dyke 2) and HSCD-1d (Dyke 3) range form 0.47 to 0.95. The bitumen samples show peak values for tricyclic terpanes (C_{19} to C_{30}) at C_{21} or C_{23} (Fig. 5). The C_{23}/C_{21} tricyclic terpane values range from 0.89 to 1.67, with an average of 1.35. The C_{24} tertracyclic/C_{26} tricyclic terpane ratios range from 1.00 to 2.61, with only two samples (11SKD-4 and SKD-1) possessing ratios >2.0 (Table 1). The hopanes (C_{27} to C_{35}) exhibit peaks at C_{29} or C_{30} (Fig. 5). The abundance of the C_{31} to C_{35} hopanes decrease with increasing carbon number. In addition to the presence of C_{30} diahopane, Ts (18α(H)-trisnorhopane), and Tm (17α(H)-trisnorhopane), and gammacerane are also detected (Fig. 5). The Ts/(Ts+Tm) values range from 0.20 to 0.38, with an average of 0.31, DH_{30}/H_{30} values range from 0.02 to 0.08, with an average of 0.05, and C_{32} hopane S/(S+R) values range from 0.54 to 0.62, with an average of 0.58. The bitumen samples yield a gammacerane index of 0.12 to 0.22, with an average of 0.16. The ratio of Nor-25-hopane/hopane range from 0.07 to 0.19, with the exception of sample SKD-1f, which has a ratio of 0.07, all other samples possess a similar ratio (0.15).

Sterane compounds including C_{21} pregnane, C_{22} sterane, diasterane and C_{27}-C_{29} sterane were also detected (Fig. 5). The ratio of S_{21}/S_{22} range from 2.36 to 2.58, with an average of 2.44. C_{27}, C_{28}, C_{29} steranes of all the bitumen display a similar V-shape distribution which occupy ~30.2, 16.0 and 53.8 %, respectively, with C_{29} sterane exhibiting the highest abundance. The ratio of C29ααα20S/(20S+20R) and C29ββ/(ββ+αα) vary from 0.46 to 0.52 and 0.49 to 0.57, respectively, which yield a similar Ro value (~0.9). The organic geochemistry of the bitumen samples analyzed in this study suggest that all the bitumen has middle to high maturity, and was sourced from similar organic matter derived from marine algae deposited in an anoxic environment (De Grande et al., 1993; Didyk, 1978; Peters et al., 2005; Seifert and Moldowan, 1986).

However, the organic analysis of two of the present day oil seep samples (Oil-3 and Oil-5) show very different organo-geochemical features as compared to the bitumen. This observation is critical given that these samples are taken in close spatial (~20 m) proxmity to the bitumen samples GY-6 and HSCD-1, which suggests that the organo-geochemical characteristics of the bitumen have not been appreciably affected by the present oil seeps. The SFGCs display UCM and suggest severe biodegradation of the present day oil seeps (Fig. 5). Given the lack of the majority of the *n*-alkanes, the data related to CPI or Pr/Ph values can not be calculated. Terpanes and hopanes, except for the Ts/(Ts+Tm) ratio (0.31 vs 0.36), of the oil are similar to the bitumen. Further, all of the other parameters, for example gammacerane/hopane (3.91-7.59), C_{24} tertracyclic/C_{26} tricyclics (0.51-0.52) and DH_{30}/H_{30} (3.10-5.54) values are distinctly different in comparison to the bitumen samples (Table 1). Only C_{21} and C_{22} steranes in the oil were dectected, which yield a S_{21}/S_{22} ratio of 0.21 and 0.20, respectively, which are also different as compared to the bitumen. The limited organic data for the present day oil seep samples suggests that the organic fraction of the oil is derived from a source rock deposited in a hypersaline, suboxic and clay-rich environment (Peters and Moldowan, 1993; Zumberge, 1987).

5.2 Re-Os results

The Re and Os abundances of all the bitumen samples range between 283.1 and 547.9

ppb, and 4.06 and 15.3 ppb, respectively (Table 2). These values are significantly elevated from those of the average continental crust (Esser and Turekian, 1993), but similar to previously reported bitumen samples, and the organic-rich sedimentary rocks (Cohen et al., 1999; Esser and Turekian, 1993; Georgiev et al., 2016; Ravizza and Turekian, 1992; Rooney et al., 2010; Selby and Creaser, 2005; Xu et al., 2009; Xu et al., 2014). The $^{187}Re/^{188}Os$ values of the bitumen range from 229.5 to 595.1 and exhibit a radiogenic $^{187}Os/^{188}Os$ composition of 2.79 to 3.56 (Table 2). Repeat analyses of a single bitumen sample (11SKD-4d-rpt) yield highly reproducible (<1%) Re (~512.2 ± 1.8 vs 518.8 ± 1.3 ppb) and Os (14478.3 ± 46.3 vs 14605.3 ± 75.8 ppt) concentrations, and $^{187}Re/^{188}Os$ (230.7 ± 0.9 vs 231.5 ± 1.0) and $^{187}Os/^{188}Os$ (2.84 ± 0.01 vs 2.83 ± 0.01) values (Table 2). Similar reproducibility has been shown by previous studies (Lillis and Selby, 2013; Selby et al., 2005).

Collectively the bitumen Re-Os isotope data from the three dykes, fault and fracture surfaces does not show any linear relationship, and shows a large range in isotope compositions (Fig. 6A). Herein we discuss the Re-Os data of each bitumen occurrence separately. The three samples from Dyke 1 have extremely similar Re-Os isotope compositions, and as a result do not yield a meaningful Re-Os date (674 ± 490 Ma). Bitumen from a fracture (SKD-1f) that postdates Dyke 1 exhibits broadly similar $^{187}Os/^{188}Os$, but higher $^{187}Re/^{188}Os$ values than that of the dyke (Table 2). The two bitumen samples from Dyke 2 show distinctly different Re-Os isotope compositions in comparison to Dyke 1, specifically with respect to the $^{187}Re/^{188}Os$ values. Although, dates derived from only two samples may not be completely reliable geologically, the Re-Os data from the two Dyke 2 bitumen samples may suggest that bitumen formation occurred during the Early Jurassic (181 ± 41 Ma). Bitumen from Dyke 3 exhibit the largest variation in Re-Os isotope space, with the compositions specifically different to Dyke 1 (with the exception of GY-5d) and Dyke 2. The Re-Os data of Dyke 3 yield a Model 3 (which assumes that the scatter in the degree of fit of the data is a combination of the assigned uncertainties, plus a normally distributed variation in the $^{187}Os/^{188}Os$ values (Ludwig, 2003)) date of 503 ± 140 Ma (Mean Squared Weighted Deviation, MSWD = 90), with an initial $^{187}Os/^{188}Os$ value of 0.91 ± 0.71 (Fig. 6B). The Re-Os date and its uncertainty is largely controlled by sample GY-5d that controls the lower anchor of the best-fit line of the data, and HSCD-1 and GY-1d which plot above the best-fit line, respectively. Using the Re-Os date of 503 Ma to calculate the initial $^{187}Os/^{188}Os$ (Os_i) values, shows that samples HSCD-1 and GY-1d possess Os_i values (~1.0) that are slightly more radiogenic than the samples GY-2d, 3d, 4d, 5d, and 6d (0.85 - 0.89), and we consider that the samples HSCD-1 and GY-1d are the principal controls on the Re-Os date and uncertainty (Table 2). Considering only the samples (GY-2d, 3d, 4d, 5d, and 6d) that possess similar Os_i values, the Re-Os data yield a more precise Re-Os date of 483 ± 27 Ma, with an Os_i value of 0.97 ± 0.13.

The Re-Os isotope data of bitumen sampled from faults and fractures show no linear trends. Sample 11LXB-1f possesses similar $^{187}Re/^{188}Os$ and $^{187}Os/^{188}Os$ values to those of Dyke 3, specifically GY-3d and 4d. In contrast, the Re-Os data of SKD-1f, LXB-1f and 2f are similar to that for Dyke 2. Combined, the bitumen Re-Os data of Dyke 2 and from the fractures represented by samples SKD-1f, LXB-1f and 2f define a broadly positive correlation between the $^{187}Re/^{188}Os$ and $^{187}Os/^{188}Os$ compositions, and yield a Re-Os date of 158 ± 76 Ma, with an Os_i value of 1.85 ± 0.61 (MSWD = 79) (Fig. 6E). The uncertainty in this date is because samples (LXB-1f, SKD-1f, LXB-2f, XKD-2d) though very close to the linear regression, still deviate from the line of best-fit. The Os_i

values calculated at 158 Ma show that samples LXB-1f and SKD-1f possess less radiogenic Os_i values (1.80 and 1.75) in comparison to XKD-1d, XKD-2d and LXB-2f (1.87 to 1.91) (Table 2). Treated separately the Re-Os data for samples XKD-1d, XKD-2d and LXB-2f, and samples LXB-1f and SKD-1f record a Mid Jurassic age (162 ± 14 Ma, with an Os_i value of 1.87 ± 0.12, and 172.6 ± 8.1 Ma, with an Os_i value of 1.66 ± 0.06, respectively) (Fig. 6F).

The present day oil seep samples possess very different Re-Os systematics as compared to all the bitumen samples. In contrast to the bitumen samples the asphaltene fractions of the oil seep samples possess much lower Re and Os abundances (Re of 7.7 to 9.6 ppb, Os of 90.3 to 127.2 ppt) (Table 2). The $^{187}Re/^{188}Os$ and $^{187}Os/^{188}Os$ values of the oils are very similar, 496.3 to 579.3 and 2.89 to 2.93, respectively (Table 2). As the three oil seep samples possess very similar Re and Os isotope compositions, no meaningful Re-Os date can be determined.

6. Discussion

6.1 Bitumen and Oil Geochemistry and source tracing

In this study, the biomarker analysis shows that all the bitumen from the dykes, fractures and faults possess similar organo-geochemical characteristics, distinct from the present day oil seeps. The bitumen molecular compostition (*n*-alkanes, terpanes and steranes) are interpreted to suggest that the bitumen organic matter derived from a marine source deposited in an anoxic setting, are mature (Ro: ~0.8 - 1.0) and biodegraded (Pr/Ph ratio = 0.47-0.95; Gammacerane/H30 = ~0.16; C_{23}/C_{21} tricyclic = ~1.35; C_{24} tertracyclic / C_{26} tricyclics = ~1.41; Ts/(Ts+Tm) = ~0.31; diahopane/hopane = ~0.05 and H_{32} S/(R+S) homohopane = ~0.58; the 25-nor-hopane/hopane = ~0.15)

(Table 1) (Peters and Moldowan, 1993; Wenger and Isaksen, 2002; Zumberge, 1987). Further, the sterane chromatogram (m/z = 217), pregnane/homopregnane ratio (~2.44), as well as the V-shape C_{27}-C_{29} sterane distribution, with C_{29} being the largest component, implies that all the bitumen in the study area is derived from the same source rock (e.g. Peters and Moldowan, 1993; Wu et al., 2012). The C29 ααα S/(S+R) and C29 ββ/(ββ+αα) ratios (0.49 and 0.53, respectively) also indicate that the bitumen was generated during peak oil generation (Georgiev et al., 2016; Peters and Moldowan, 1993).

In comparison to the bitumen samples the present day oil samples are severely biodegraded and are slightly less mature (Ts/Ts+Tm ratio = 0.36) (Table 1; Fig. 5). The remaining biomarker parameters (gammacerane/hopane = 7.59 and 3.91; C_{24} tertracyclic/C_{26} tricyclics = 0.52; diahopane/hopane = 5.54 and 3.10; pregnane/ homopregnane = 0.21) are supportive of the organo-geochemical signature of the present day oil seeps being derived from the organic matter deposited in a sub-oxic marine-continental sedimentary environment (Peters and Moldowan, 1993; Zumberge, 1987). The Late Neoproterozoic to Early Cambrian Doushantuo and Qiongzhusi formations and the Upper Permian Dalong Formation are considered to be the principal source rocks in the Kuangshanliang area (Huang et al., 2011; Lin et al., 2011; Liu et al., 2009; Sun et al., 2009; Wei et al., 2008). Previous work shows that the geochemical parameters of both C_{23} tricyclics/C_{24} tertracyclic and pregnane/homopregnane are higher (>2.5 and ~2.0) for the Late Neoproterozoic-Early Cambrian formations than for the Upper Permian Dalong Formation (<1.6 and ~1.0) (Wu et al., 2012). In this work, the C_{23} tricyclics/ C_{24} tertracyclic and pregnane/homopregnane ratio of the bitumen samples are respectively ~2.42 and ~2.44, however, for the present day oil seep samples, these organic parameter ratios are only ~0.25 and 0.20 (the ca. 5 times lower ratio compared with Permian mudstone may be caused by the biodegradation of the present day oil seeps). Given that hydocarbons possess similar biomarker characterisitcs to that of its source unit (Cole et al., 1987; Pusey, 1973; Wu et al., 2012; Zhang et al., 2000), the results of this study suggest that the bitumen and oil seeps are sourced from different units, with the bitumen being sourced predominantly from the shales of the Late Neoproterozoic Doushantuo and Early Cambrian Qiongzhusi formations, and the present day oil seeps from the mudstones of the Permian Dalong Formation. The identification of the Doushantuo and Qiongzhusi formations being the source of the bitumen in the Kuangshanliang area further supports the oil-source correlation based on the similar $\delta^{13}C$ values for bitumen and the Doushantuo and Qiongzhusi formations (bitumen = -35.71 ‰ to -27‰ (Wu et al., 2012; Zhou et al., 2013); Precambrian-Cambrian = -30.3‰ to -35.4‰; (Wu et al., 2012; Zhou et al., 2013)), in contrast to the oil and Dalong Formation (oil = -25.9‰ to -27.7‰ (Wu et al., 2012); Permian Dalong Formation = -25.9‰ to -27.7‰ (Liang, 2007; Zhou et al., 2013)).

6.2 Multiple phases of petroleum generation

Previous studies suggest that oil generation in the North Longmen Shan Thrust Belt and the adjacent Sichuan Basin is a result of the hydrocarbon maturation of the Late Neoproterozoic - Early Cambrian shales (e.g., Late Neoproterozoic Doushantuo and Early Cambrian Qiongzhusi formations) during the Middle Ordovician (Zhou et al., 2013; Zou et al., 2014b). The only Re-Os dataset that provides a robust estimation of oil generation during the Ordovician is the bitumen from Dyke 3. As discussed above, all the Re-Os bitumen from Dyke 3 yield a Model 3 date of 503 ± 140 Ma (Fig. 6B).

However, considering only the Re-Os bitumen data of Dyke 3 that possess similar Os_i values (GY-2d, 3d, 4d, 5d, and 6d; Os_i = 0.85 - 0.89) calculated at 503 Ma, a Re-Os date of 483 ± 10 Ma is determined. The absolute reason why the samples HSCD-1 and GY-1d possess slightly elevated Os_i values (~1.0) in comparison to the majority of the bitumen from Dyke 3 is not known. But the reasoning could be related slight post-depositional disturbance to the Re-Os systematics and/or continuous hydrocarbon generation. Both determined Re-Os ages are in agreement within uncertainty, with the more precise age determined, by the bulk of the sample set, providing an age that agrees well with modeling for the timing of burial (~2500 m and ~100 ºC) and source rock maturation of the Doushantuo and Qiongzhusi formations in the Northern Longmen Shan Thrust Belt and adjacent Sichuan Basin (Liu et al., 2009; Yuan et al., 2012; Zhou et al., 2013).

In contrast to Dyke 3, the bitumen Re-Os data from Dyke 1 yield no meaningful age because of the limited spread in $^{187}Re/^{188}Os$ and $^{187}Os/^{188}Os$ values. However, the bitumen Re-Os isotope compositions of Dyke 1 are similar to that of sample GY-5d from Dyke 3 (Table 2). Calculated at the age of Dyke 3 (483 Ma), the Re-Os bitumen data of Dyke 1 yield Os_i values (0.96-0.99) which is similar to the range of that determined for Dyke 3 (0.96 - 0.98; except GY-1 and HSCD-1) (Table 2). Based on the similarity of the GC-MS (e.g., m/z 191 and 217; Fig. 5) and Re-Os data of bitumen from the Dyke 1 and Dyke 3, we consider the bitumen to be of the same oil generation episode. Together, the Re-Os data from both Dyke 3 (except GY-1 and HSCD-1) and Dyke 1 yield a Model 3 date of 486 ± 15 Ma (Fig. 6D).

In comparison to the bitumen from Dyke 1 and 3, the Re-Os characteristics of the five bitumen samples from Dyke 2 and faults/fractures have very different Re-Os systematics. Calculated at 486 Ma the Re-Os data of Dyke 2 and faults/fractures yield negative $^{187}Os/^{188}Os$ values (-0.05 to -1.04; Table 2). Furthermore, relative to a ca.486 Ma reference isochron, the five samples of Dyke 2 and faults/fractures have higher $^{187}Re/^{188}Os$ ratios for a given $^{187}Os/^{188}Os$ (Fig. 6E). This suggests that these bitumen samples are either of a different generation age to that of Dyke 1 and 3, or the Re-Os bitumen systematics of the Dyke 2 and faults/fractures have been disturbed.

The Re-Os data of the five bitumen samples from Dyke 2 (XKD-1d, XKD-2d) and fault/fractures (SKD-1f, LXB-1f, LXB-2f) together yield a Model 3 Re-Os date of 158 ± 76 Ma ($^{187}Os/^{188}Os$ = 1.85 ± 0.61, MSWD = 79) (Fig. 6E). For the Re-Os isotope compositions (e.g., $^{187}Re/^{188}Os$ and $^{187}Os/^{188}Os$) to yield a statistically meaningful isochron date, the samples comprising the dataset must have formed contemporaneously, must possess the same initial ($^{187}Os/^{188}Os$) isotope ratio, and the isotope systematics must not have been affected post formation (Cohen et al., 1999; Kendall et al., 2009; Selby et al., 2007). For these five bitumen samples, the degree of scatter about the best-fit line of the data, as given by the MSWD, is 76. The high MSWD indicates that one of the criteria for developing a statistically meaningful isochron has not been met. Although post-depositional effects, diffrerent sample localities, and contemporaneity between the sample set may affect the Re-Os data, the positive correlation of the Re-Os data may indicate that the major reason for the scatter could be a result of variable initial $^{187}Os/^{188}Os$ values. Using the Re-Os date derived by the isochron (158 Ma), initial $^{187}Os/^{188}Os$ values (Os_i) yield two populations for the sample set: (1) three samples with Os_i values of ~1.89 (samples XKD-1d, XKD-2d, LXB-2f); and (2) two samples with Os_i values of ~1.77 (samples SKD-1f; LXB-1f) (Table 2). Considering the sample set as two distinct populations, the three bitumen samples (XKD-1d, XKD-2d, LXB-2f) yield a Model 1 Re-Os date of 162 ± 14 Ma (Os_i = 1.87 ± 0.12; MSWD = 0.95; Fig. 6F). Although only two samples, the Re-Os data for bitumen samples SKD-1f and LXB-1f define Re-Os date of 172.7 ± 8.1 Ma (Fig. 6F). Both the Re-Os dates are within uncertainty and suggest that these five bitumen formed broadly contemporaneously during the Middle Jurassic at 162 - 173 Ma.

The $^{187}Os/^{188}Os$ composition of an hydrocarbon at its time of generation is inherited from its source (Finlay et al., 2011; Lillis and Selby, 2013; Selby and Creaser, 2005; Selby et al., 2005; Selby et al., 2007). The difference in the initial $^{187}Os/^{188}Os$ compositions of the two temporally distinct bitumen samples (~0.95 vs ~1.85; Fig. 6; Table 2) could indicate the bitumen could have been derived from different source rocks. However, the organic geochemistry for all the bitumen samples are indicative of the source rock being the Late Neoproterozoic to Early Cambrian Doushantuo and Qiongzhusi formations. As such, the more radiogenic initial $^{187}Os/^{188}Os$ compositions of bitumen formed during the Jurassic (~1.85) is the result of the greater duration of radioactive ingrowth of ^{187}Os from the decay of ^{187}Re in the source rock since its deposition. Although no Re-Os data was obtained for the potential source rock samples in this study, Re-Os data of the Late Neoproterozoic – Early Cambrian shales from the South China Block (Yangtze Gorges area (Kendall et al., 2009) and Zunyi, Guizhou province (Jiang et al., 2007)) yield Os_i values at ca. 485 Ma and ca. 165 Ma of 0.89 - 0.98 and 1.54 - 2.01, respectively. The bitumen Os_i values at ca. 486 Ma and ca. 165 Ma in this study all fall into this range, which further supports the bitumen are derived from the same source, but during two separate phases of oil generation.

The Longmen Shan Thrust Belt records a series of complex tectonic events since the Palaeozoic (Chen and Wilson, 1996; Dai, 2011; Jin et al., 2010; Yan et al., 2011). Burial history models (Zhou et al., 2013; Zou et al., 2014b) coupled with the Re-Os dates of the Dykes 1 and 3 suggest that oil generation of the Late Neoproterozoic Doushantuo and Early Cambrian Qiongzhusi formations in the Northern Longmen Shan Thrust Belt and the adjacent Sichuan Basin occurred during the Middle Ordovician. Oil generation ceased during the Caledonian Orogeny (~450 – 400 Ma) due to more than 2000 m of uplift and denudation (Wang et al., 1989; Wang et al., 2007; Zhuang, 1985; Zou et al., 2014b). The maturation history of the Early Cambrian Qiongzhusi Formation based on five different wells across the southwest Sichuan Basin indicates that the shales did not enter the oil window between the Late Devonian and Carboniferous (Liu et al., 2009). However, since Triassic, the Northern Longmen Shan Thrust Belt has been affected by the Indosinian–Yanshan orogenies following the collison between the North and South China blocks (Liu et al., 2005). Compressional tectonics in the Longmen Shan Thrust Belt and Sichuan Basin continued into the Late Jurassic from the paleogeography model (Jin et al., 2009b; Liu et al., 1996); apatite fission track date of 162 ± 23 Ma (Arne et al., 1997); and post-tectonic granitoid magmatism at ~160 Ma (Jin et al., 2008) (Fig. 7). The sheared bitumen accummulations observed in the sandstone country rock (Fig. 4D) also suggests that the bitumen emplacement of Dyke 2 may be syn-tectonic. The Re-Os dates (ca.162 - 172 Ma) of the five bitumen samples from Dyke 2 and fault/fractures coincide with the timing of the tectonism in the Longmen Shan Thrust Belt during the Jurassic. The organic geochemistry of the Dyke 2 and fault/fracture bitumen suggest that it is sourced from the Late Neoproterozoic – Early Cambrian Doushantuo and Cambrian Qiongzhusi formations. These units were buried to more than 5000 m and re-entered the oil window (Ro ~1.2 %) during the Middle Jurassic (Liu et al., 2009; Zou et al., 2014b). The hydrocarbon generation intensity of the Cambrian source rock units from the

Yangtze block (China) indicates that the Triassic and Middle Jurassic were the two peak oil generation intervals (Liang, 2007). Further, analysis on the Zi 1 and Gaoke 1 Wells from the center of the Sichuan Basin found that the Lower Cambrian shales achieved peak oil generation during the Middle Jurassic (ca. 175 – 161 Ma) (Liu et al., 2009; Zhang et al., 2005). Fluid inclusion homogenization temperatures (~120 ºC) and basin modeling in the Weiyuan gas field in the southwest Sichuan Basin also indicate that oil generation and migration occurred during the Triassic and Juriassic (ca. 200 – 170 Ma) (Fig. 7) (Ma et al., 2007a; Tang et al., 2004; Zou et al., 2014b).

Integrating previous research work and the organic geochemistry, and Re-Os isotope analysis results of this study, we propose that the hydrocarbon evolution in Kuangshangliang area happened as follows:

(1) During the Early Palaeozoic, the Late Neoproterozoic Doushantuo and Early Cambrian Qiongzhusi formations were buried to a depth of more than 2500 m and entering the oil window (Ro ~0.80), and leading to the first phase of oil generation. Following this oil generation event, the Caledonian Orogeny (~450 - 400 Ma) resulted in ~2000 m of uplift and thus halted hydrocarbon maturation of the Late Neoproterozoic and Early Cambrian source rocks (Fig. 7A, B).

(2) As a result of the collison following the Indosinian-Yanshan orogenies during the Triassic and Jurassic, the Late Neoproterozoic Doushantuo and Early Cambrian Qiongzhusi formations were buried to a depth of more than 5000 m (Ro ~1.2) (Fig. 7C), leading to the second phase of oil generation from these formations.

(3) Although no meaningful Re-Os age can be obtained from the present day oil seeps, the organic geochemisty data generated in this study along with the previous research work indicates that this oil may have been generated during the Mesozoic from a

Permian source, i.e. the Dalong Formation (Fig. 7C).

(4) Since the Cretaceous, continued tectonics, due to the collision between the Indian and Asian plates, has caused the rapid uplift and denudation of the entire Longmen Shan Thrust Belt (Dai, 2011; Yan et al., 2011). This erosion effect has exhumed the majority of the traps and reservoirs within the the petroleum systems (Fig. 7D).

7. Implications and Conclusions

Combining the bitumen and present day oil seep organic geochemisty and Re-Os isotope geochronology from Kuangshanliang area we provide quantitative constraints on the petroleum evolution within the northern Longmen Shan Thrust Belt and adjacent basins that record a similar temporal tectonic evolution. The organic geochemisty of all the bitumen in the Kuangshanliang area from both dykes and fault/fractures possess similar characteristic and suggest they are sourced from shales of the Late Neoproterozoic to Early Cambrian Doushantuo and Qiongzhusi formations. In contrast, the few organic geochemistry of the present day oil seeps indicate the oil seeps possess distinct characteristics in comparison to the bitumen (e.g., lower tT24/TR26 (~0.5) value, higher GAM/H_{30} (3.9-7.6), DH_{30}/H_{30} (3.1-5.5) ratio and $\delta^{13}C$ (-25.9 ‰ to -27.7 ‰) and are suggestive of being derived from the Permian Dalong Formation.

The Re-Os isotope analysis showed that the Kuangshanliang area bitumen which has been derived from liquid hydrocarbon has two distinct episodes of generation. The Re-Os data for bitumen from Dyke 1 and 3 yield a date of ca. 486 Ma. This Latest Cambiran to Earliest Ordovician age agrees well with previous understanding that the Late Neoproterozoic – Early Cambrian shales of the Doushantuo and Qiongzhusi formations in the Longmen Shan Thrust Belt and adjacent Sichuan Basin first entered into the oil window during the Ordovician based on basin burial modeling, and source rock maturation history (Liu et al., 2009; Yuan et al., 2012; Zhou et al., 2013). In contrast, the Re-Os data of bitumen from Dyke 2 and the fault/fractures yield a Middle Jurassic age (ca. 172 - 162 Ma). This Middle Jurassic age is coincident with the timing of the Indosinian-Yanshan orogenies (Arne et al., 1997; Jin et al., 2009b; Liu et al., 1996; Yan et al., 2003), which lead to the second phase of oil generation from the shales of the Late Neoproterozoic Doushantuo and Early Cambrian Qiongzhusi formations (Liu et al., 2009; Zou et al., 2014b). Additionally, the timing is in agreement with the basin modelling and the homogenization temperatures (~120 ºC) of fluid inclusions in dolomite and quartz from the adjacent Sichuan Basin showing oil generation and migration occurred between the Triassic and Juraissic (Ma et al., 2007a; Tang et al., 2004; Zou et al., 2014b).

This research shows that Re-Os isotope analyses of bitumen have the potential to record multiple oil generation episodes in complex tectonic settings. In addition to the Longmen Shan Thrust Belt and the adjacent Sichuan Basin, multiple hydrocarbon generation phases related to tectonism are also reported in the Maracaibo Basin of Venezuela (Eocene and Miocene to present two continuous oil genertion episodes) (Lugo and Mann, 1995; Talukdar et al., 1986) and the Tarim Basin of Northwest China (two phases of oil generation during the Late Silurian and Late Permian) (Xin et al., 2011). Thus, hydrocarbon (bitumen and oil) Re-Os chronology could aid in quantitatively constraining the petroleum evolution in basins worldwide, which may enhance our understanding of both the temporal and spatial evolution of a hydrocarbon system.

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Figure Captions

Fig. 1. A) Regional map of the Longmen Shan Thrust Belt and the adjacent Sichuan Basin and Songpan-Garze Belt in the SE and NW, respectively. The shaded area is expanded in Figure 1B and 1C; B) Simplified map of the Sichuan Basin showing the distribution of gas fields with different orogenic belts. Substantially modified after Li et

al., 2015; Li et al., 2001; Ma et al., 2010; C) Structural map of the Longmen Shan Thrust Belt showing the bitumen outcrop distribution, and the location of the Cambrian cored Kuangshanliang anticline (our study area). Substantially modified after Tian, 2009.

Fig. 2. Stratigraphy, hydrocarbon system and tectonic events in the North Longmen Shan area. Substantially modified after Chen and Wilson, 1996; Wu et al., 2012.

Fig. 3. A) Simplified geological map of the Kuangshanliang anticline; B) Detailed geology feature in the Kuangshanliang area and locations of the bitumen and present day oil seep samples.

Fig. 4. Bitumen and oil sample locations and field relationships. A) Bitumen Dyke 1 showing the relationship between the dyke and the country rock. A') detailed image of the breccia zone shown in A. B) Bitumen sample (11SKD-3d, 11SKD-4d, 11SKD-5d, SKD-1f) locations in Dyke 1 and related fault/fractures. C) Bitumen sample (XKD-1d, XKD-2d) locations in Dyke 2. D) Syn / post thrust fault in Dyke 2. E) Bitumen sample (HSCD-1d and GY1d-6d) locations in Dyke 3. F) Bitumen samples (LXB-1f, LXB-2f) from a fault zone. G) Bitumen sample (11LXB-1f) from a fault plane. H) Present day oil seep samples (Oil-3, Oil-5, Oil-7) occurring in the Early Cambrian Qiongzhusi Formation.

Fig. 5. Total Iron Chromatogram (TIC), m/z 191 and m/z 217 mass chromatograms of the bitumen (11SKD-4d, XKD-1d, GY-3d, HSCD-1d, LXB-1f) and the present day oil seeps (Oil-5) in the Kuangshangliang area.

Fig. 6. A) Traditional $^{187}Re/^{188}Os$ vs $^{187}Os/^{188}Os$ plot showing all the Re-Os data for bitumen from the dykes and faults/fractures, as well as the present day oil seeps in the Kuangshanliang anticline (Bold for Dyke 1 bitumen; Underline for Dyke 2 bitumen; Italic for Dyke 3 bitumen; Regular font for fault/fracture bitumen and Bold Italic for the present day oil seeps). B) The Re-Os isotope data of Dyke 3 bitumen. C) The Re-Os isotope data of bitumen from Dyke 1 and 3 and fault bitumen sample, 11LXB-1f. D) The Re-Os isotope data of Dyke 1 and 3 bitumen (without HSCD-1 and GY-1). E. The Re-Os isotope data of all Dyke 2 and fault/fracture bitumen. F) The Re-Os isotope data of Dyke 2 and fault/fracture bitumen based on Os_i values groups (~1.82 and ~1.89). Data-point ellipses are shown with 2-sigma absolute uncertainty. Data labels are sample numbers listed in Table 2.

Fig. 7. The relationship between petroleum generation and tectonism. Shown is a comparison of the Re-Os ages with source rocks (Xie et al., 2003; Zhou et al., 2013), published basin model and fluid inclusion results (Ma et al., 2007a; Tang et al., 2004) and muscovite $^{40}Ar/^{39}Ar$ ages (Li et al., 1999; Yan et al., 2011), Jurassic zircon fission track ages (Arne et al., 1997) in Longmen Shan Thrust Belt. The schematic cartoon model shows the hydrocarbon evolution in the Kuangshanliang anticline, Northern Longmen Shan Thrust Belt. A) First phase of oil generation during the Ordovician, before the Caledonian Orogeny. B) Oil generation ceased due to uplift caused by the

Caledonian Orogeny (ca. 450 - 400 Ma). C) Second phase of oil generation during the Middle Jurassic Indosinian-Yanshan orogenies. D) Present condition of the bitumen and present day oil seeps after Cenozoic Himalayan Orogeny.

Figure 1

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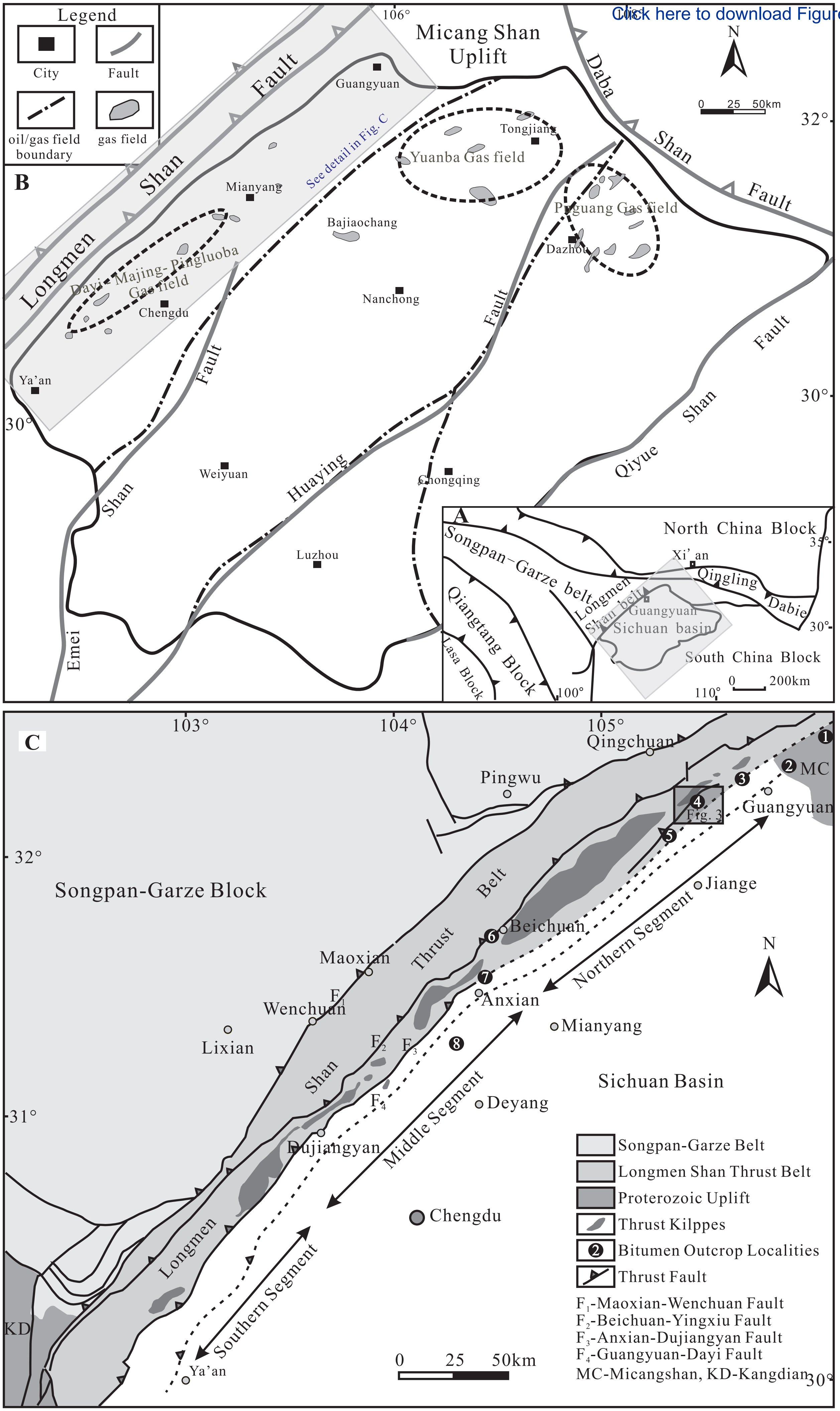

Figure 2

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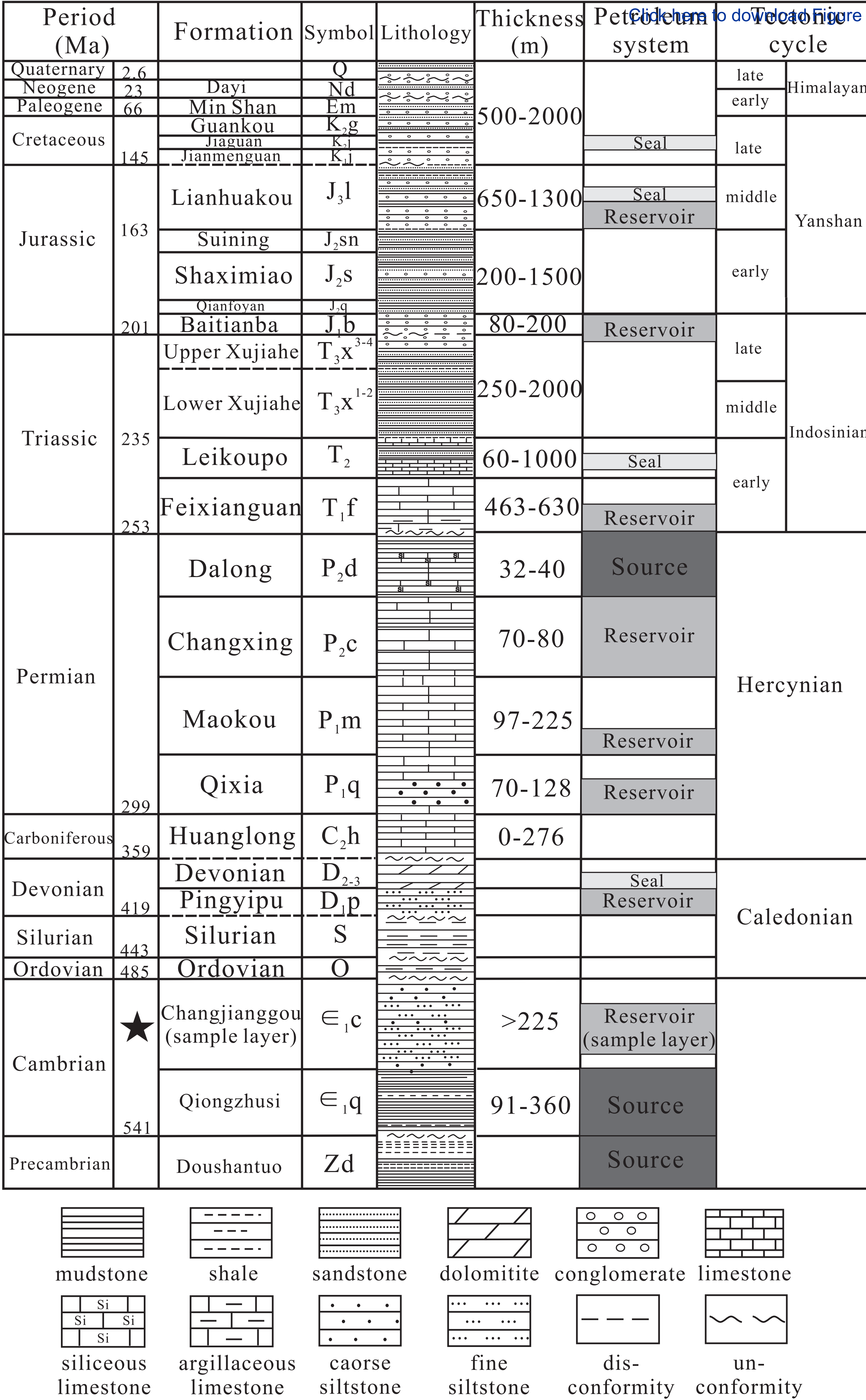

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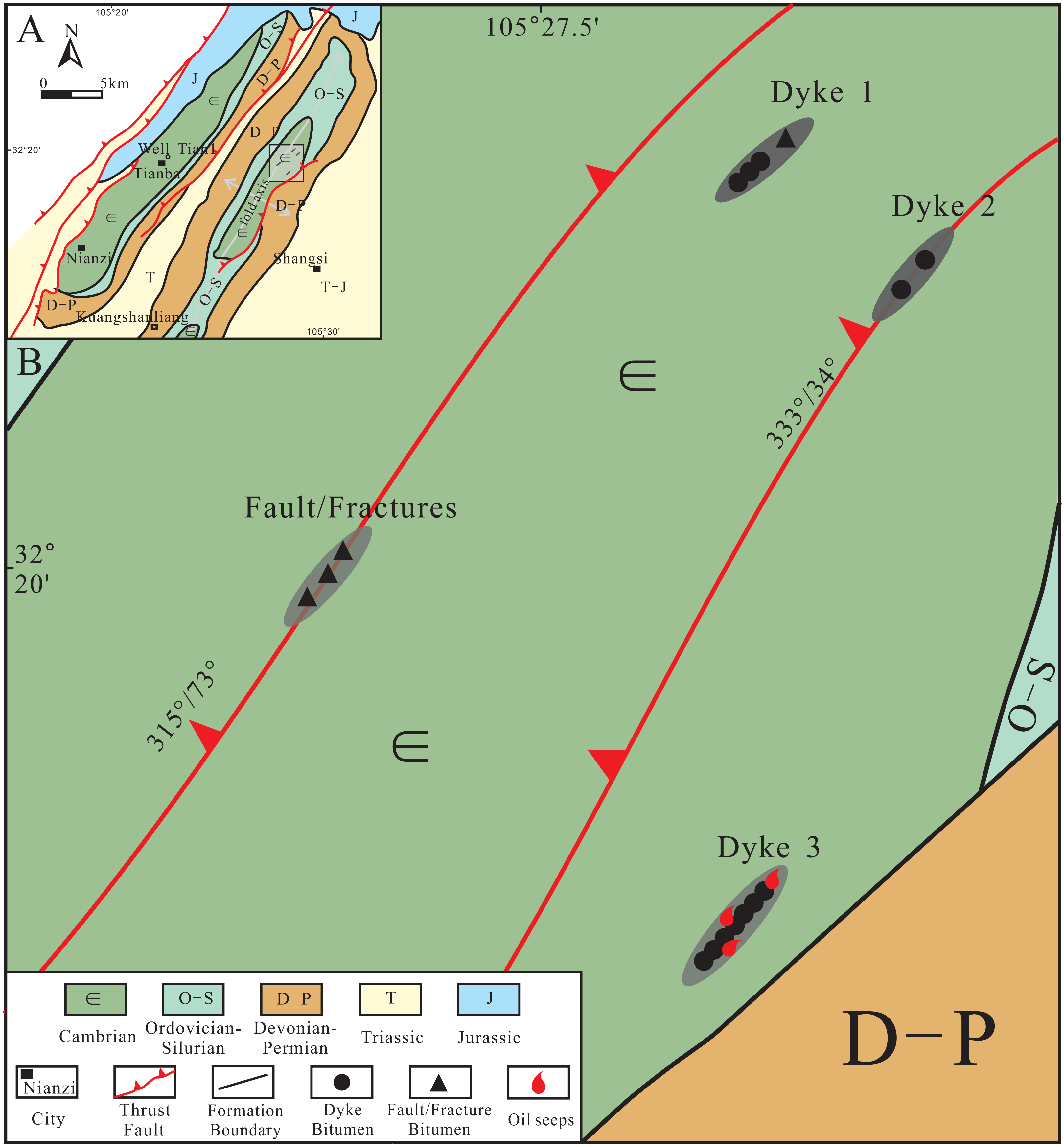

Figure 4

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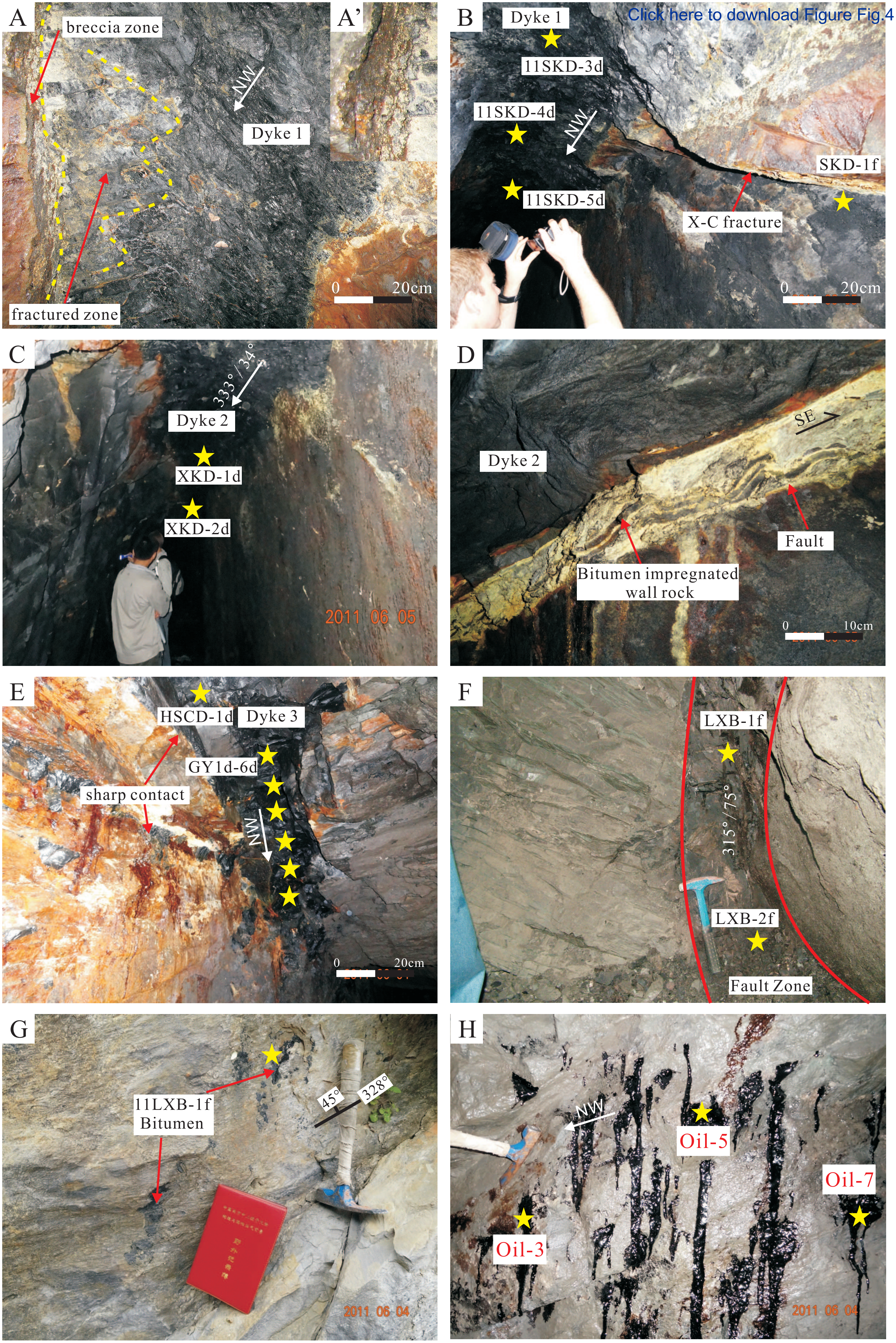

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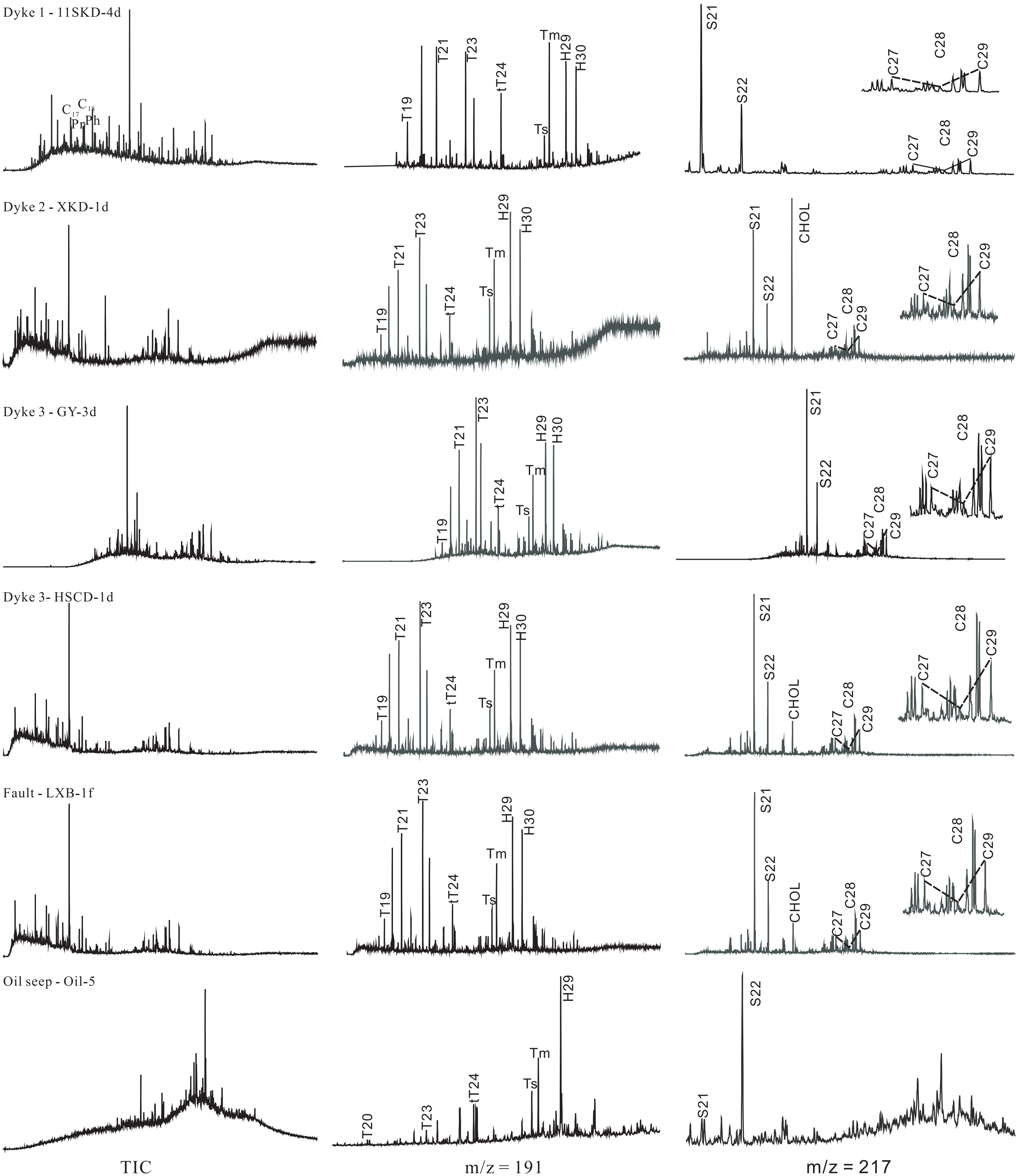

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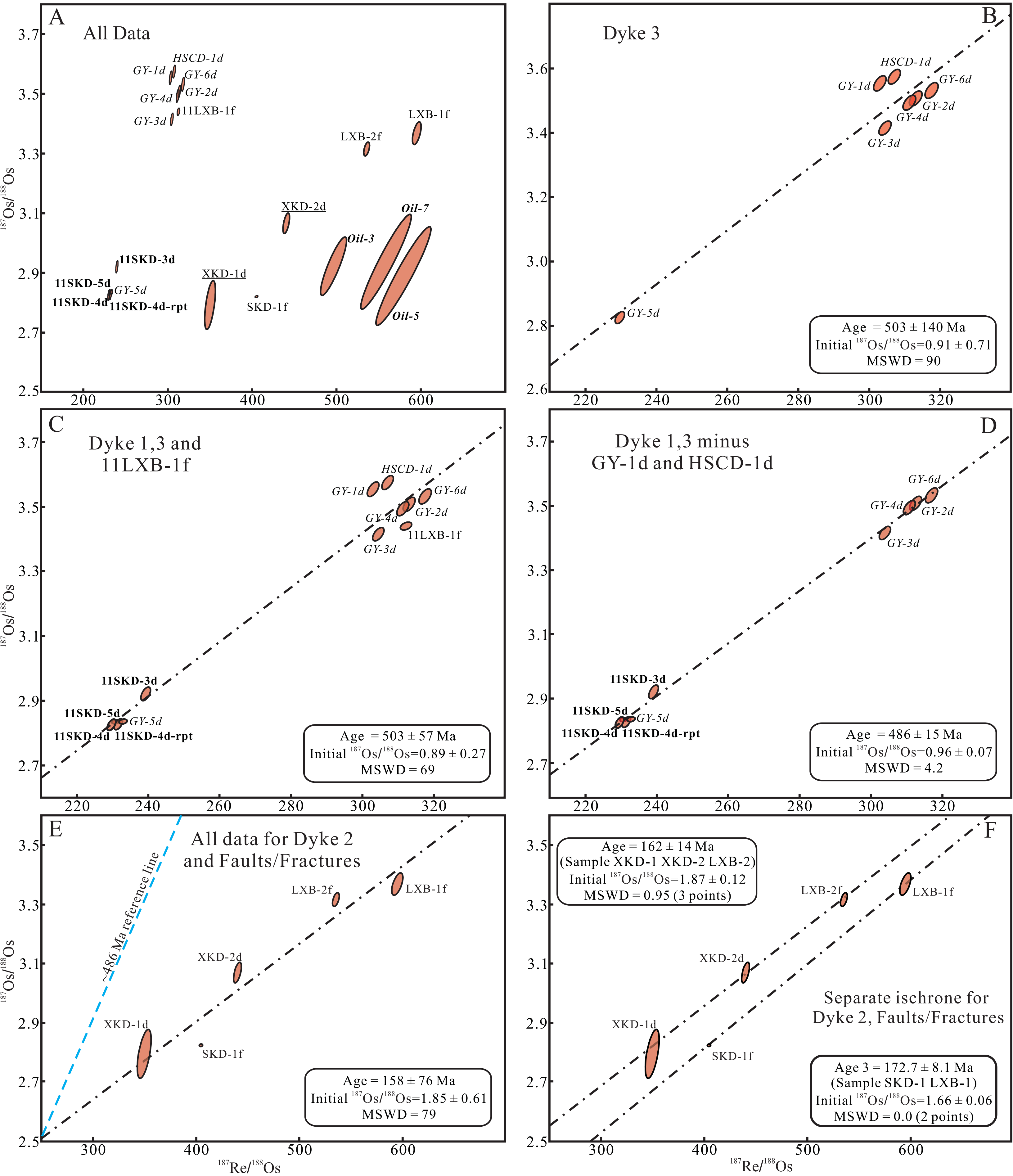

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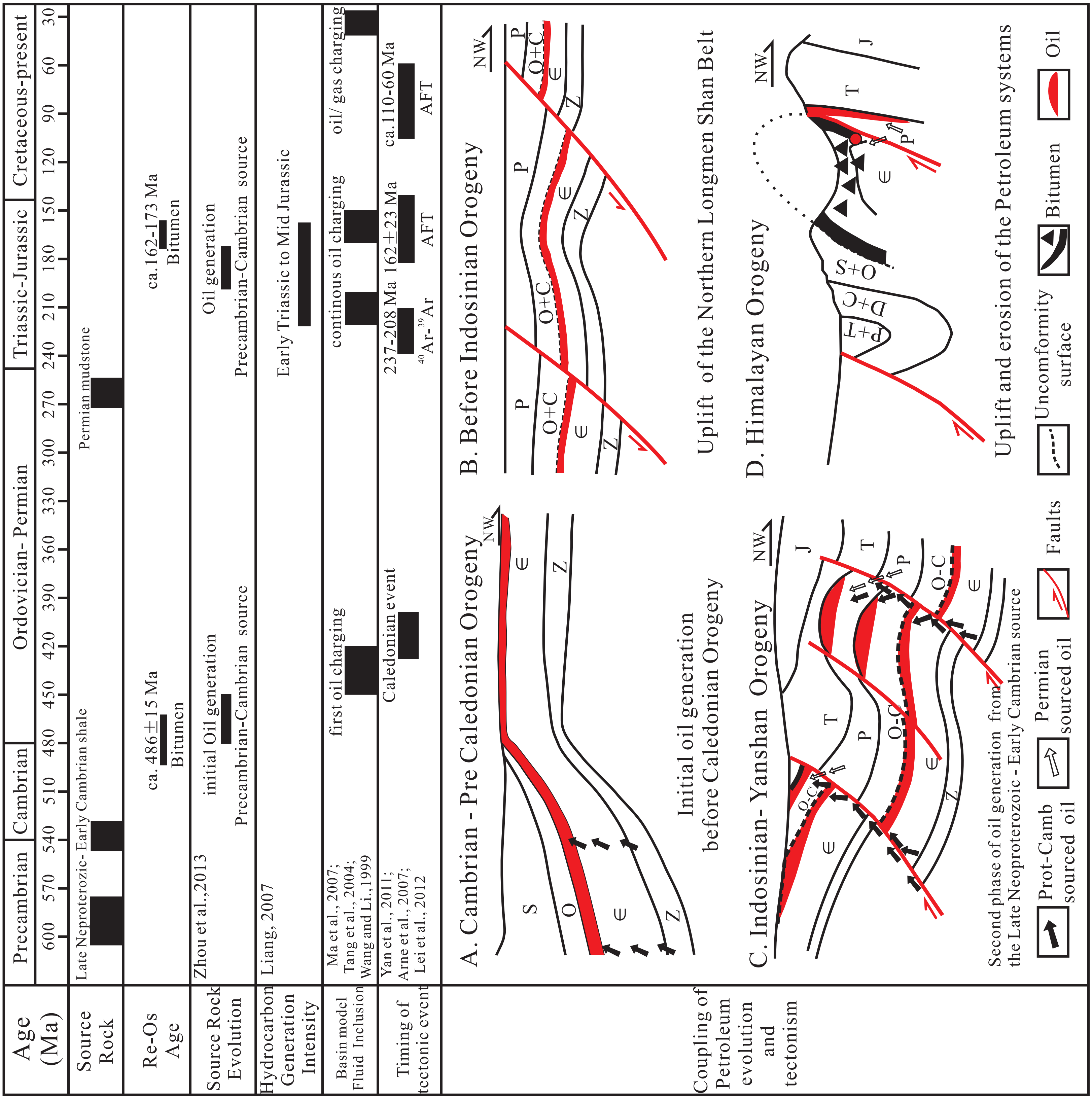

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Table 1. The biomarker characteristics of the bitumen and present day oil seeps from the Kuangshangliang area, Northern Longmen Shan Thrust Belt

Sample name	ASPH (%)	CPI	Pr/ C17	Ph/ C18	Pr/Ph	GAM /H30	Ts/ Ts+Tm	TR23/ TR21	TR23 / tT24	tT24/ TR26	DH30 /H30	H32 S/(R+S)	NOR25H /H30	S21/ S22	C27R (%)	C28R (%)	C29R (%)	C29S /(S+R)	C29ββ/ (ββ+αα)
Dyke 1																			
11SKD-4d	/	/	0.96	1.71	0.72	0.14	0.2	0.90	1.50	2.32	0.06	0.6	0.16	2.55	34.5	19.5	46	0.5	0.49
Dyke 2																			
XKD-1d	98	1.05	0.4	1.05	0.47	0.22	0.38	1.45	2.41	1.21	0.08	0.57	0.14	2.58	24.8	15.7	59.5	0.51	0.51
Dyke 3																			
GY-1d	/	1.04	/	/	/	0.18	0.32	1.67	2.85	1.03	0.05	0.54	0.16	2.41	30.1	15.2	54.7	0.52	0.53
GY-3d	/	1.16	/	/	/	0.19	0.32	1.57	2.88	1	0.05	0.56	0.16	2.42	27.7	14.4	57.9	0.5	0.53
GY-5d	/	0.91	/	/	/	0.13	0.31	1.31	2.20	1.43	0.05	0.58	0.17	2.5	30.4	16.2	53.4	0.52	0.55
HSCD-1d	99	2.59	0.7	0.91	0.95	0.15	0.35	1.49	3.01	1.06	0.05	0.62	0.13	2.45	32.2	16.3	51.5	0.47	0.57
Fault and fractures																			
SKD-1f	98	0.94	/	/	/	0.12	0.24	1.00	1.28	2.61	0.02	0.6	0.07	2.46	29	15.2	55.7	0.46	0.5
11LXB-1	99	1.21	/	/	/	0.17	0.32	1.38	2.82	1.01	0.05	0.6	0.17	2.26	30.8	16	53.2	0.47	0.55
LXB-1f	98	0.35	/	/	/	0.18	0.33	1.38	2.83	1.00	0.06	0.58	0.19	2.32	31.90	14.80	53.40	0.46	0.55
Oil																			
Oil-3	13.54	/	/	/	/	7.59	0.37	/	/	0.51	5.54	/	/	0.21	/	/	/	/	/
Oil-5	9.48	/	/	/	/	3.91	0.36	/	0.26	0.52	3.1	/	/	0.2	/	/	/	/	/

Table 2. Re-Os elemental and isotopic data of the bitumen and present day oil seeps from the Kuangshanliang area, Northern Longmen Shan Thrust Belt.

Sample	latitude	longitude	Re (ppb)	Re blk %	Os (ppt)	Os blk %	$^{187}Re/^{188}Os$	$^{187}Os/^{188}Os$	rho	Osi_{503}	Osi_{486}	Osi_{483}	Osi_{158}
Dyke 1													
11SKD-3d	32°20'27"	105°27'47"	403.5(1.0)	0.004	11094.0(64.7)	0.007	239.1(1.1)	2.92(0.02)	0.560	/	/	0.99	/
11SKD-4d	32°20'25"	105°27'48"	512.2(1.8)	0.003	14478.3(46.3)	0.006	230.7(0.9)	2.84(0.01)	0.260	/	/	0.97	/
11SKD-4d-rpt	32°20'25"	105°27'48"	518.8(1.3)	0.010	14605.3(75.8)	0.046	231.5(1.0)	2.83(0.01)	0.567	/	/	0.96	/
11SKD-5d	32°20'26"	105°27'47"	547.9(1.9)	0.009	15347.3(50.3)	0.042	232.8(0.9)	2.84(0.01)	0.238	/	/	0.96	/
Dyke 2													
XKD-1d	32°20'26"	105°27'49"	332.9(1.1)	0.007	6182.4(80.9)	0.107	349.7(5.2)	2.79(0.07)	0.591	/	-0.05	/	1.87
XKD-2d	32°20'25"	105°27'49"	334(1.2)	0.007	5058.7(38.4)	0.133	440.2(3.2)	3.07(0.03)	0.576	/	-0.51	/	1.91
Dyke 3													
GY-1d	32°19'21"	105°27'47"	305.6(0.8)	0.008	7033.5(40.5)	0.012	302.9(1.3)	3.56(0.02)	0.581	1.01	/	1.11	/
GY-2d	32°19'20"	105°27'45"	320.3(0.8)	0.007	7105.0(40.4)	0.012	313.1(1.4)	3.51(0.02)	0.582	0.87	/	0.98	/
GY-3d	32°19'22"	105°27'47"	303.7(0.8)	0.008	6869.0(38.1)	0.012	304.4(1.3)	3.42(0.02)	0.577	0.85	/	0.96	/
GY-4d	32°19'21"	105°27'46"	293.5(0.7)	0.008	6538.1(37.2)	0.013	311.3(1.4)	3.49(0.02)	0.577	0.88	/	0.98	/
GY-5d	32°19'22"	105°27'47"	524.8(1.3)	0.005	14895.1(79.1)	0.005	229.5(1.0)	2.83(0.01)	0.562	0.89	/	0.97	/
GY-6d	32°19'20"	105°27'48"	329.3(0.8)	0.007	7216.8(42.4)	0.012	317.6(1.4)	3.53(0.02)	0.567	0.86	/	0.97	/
HSCD-1d	32°19'21"	105°27'47"	284.1(0.7)	0.006	6464.5(36.3)	0.013	307.0(1.3)	3.58(0.02)	0.581	0.99	/	1.10	/
Fault and Fracture													
11LXB-1f	32°20'07"	105°27'20"	311.1(1.1)	0.005	6875.7(29.5)	0.013	312.2(1.3)	3.44(0.01)	0.408	/	/	/	/
SKD-1f	32°20'26"	105°27'47"	525.9(1.8)	0.004	8460.4(20.0)	0.074	404.8(1.4)	2.82(<0.01)	0.468	/	-0.47	/	1.75
LXB-1f	32°20'06"	105°27'19"	352.2(1.2)	0.005	4058.2(31.6)	0.413	595.1(4.2)	3.37(0.03)	0.582	/	-1.47	/	1.80
LXB-2f	32°20'06"	105°27'20"	334.4(1.1)	0.007	4259.3(25.6)	0.409	535.6(2.8)	3.32(0.02)	0.505	/	-1.04	/	1.90
Oil													
Oil-3	32°19'20"	105°27'48"	9.6(0.1)	0.185	127.2(1.9)	3.803	496.3(12.6)	2.92(0.08)	0.88	/	/	/	/
Oil-5	32°19'21"	105°27'47"	8.1(0.1)	0.312	91.7(2.0)	3.632	579.3(26.6)	2.89(0.14)	0.948	/	/	/	/
Oil-7	32°19'20"	105°27'46"	7.7(0.1)	0.308	90.3(1.9)	3.460	558.6(24.5)	2.94(0.13)	0.947	/	/	/	/

Note: asphaltene fraction of the oil were fisrt precipitated using 40 times volume of *n*-heptane (~1 g oil with 40 ml solvent) at room temperature for at least 8 hrs and Re-Os analyses are conduct on the asphaltenes.

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Dataset
appendix Re-Os data table 20170227.xlsx

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