

Apatite fission-track and Re-Os geochronology of the Xuefeng uplift, China:
Temporal implications for dry gas associated hydrocarbon systems

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Analytical protocols

Apatite Fission Track analysis was conducted at Apatite to Zircon, Inc., following the protocols of (Donelick et al., 2005). In brief, the apatite grains were isolated using standard mineral separation techniques (crushing, magnetism, heavy liquids and hand picking under a binocular microscope) from the sandstone samples. The apatite grains were then mounted in resin, polished, and etched by a eutectic melt of NaOH + KOH (at ~220°C) for 12-72 hours to expose the fission tracks. The polished and etched grains were then cleaned in reagent-grade 48% HF for 15 minutes at 23°C. The amount and the density of the fission track lengths were measured at 1562.5x dry magnification using unpolarized transmitted light and a Nikon Optiphot 2 microscope. The concentration of U in the apatite was determined by instruments of New Wave YAG 213 nm laser ablation (LA) system in line with a Finnigan Element 2 magnetic sector, inductively coupled plasma, mass spectrometer (ICP-MS). For detailed information about the LA-ICP-MS settings and data acquisition parameters see (Shen et al., 2012).

Rhenium and Os analysis was conducted at the Laboratory for Source Rock and Sulfide Geochronology and Geochemistry (a member of the Durham Geochemistry Centre) at Durham University following the analytical protocols of ((Selby, 2007; Selby and Creaser, 2003; Selby et al., 2007)).

For Re-Os analysis, approximately ~0.2-1.0 g bitumen was separated from the limestone rocks without metal contact and crushed to ~1 mm grains using an agate pestle and mortar. Using the Carius tube technique (Shirey and Walker, 1995). Approximately 100~200 mg of bitumen 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 HNO₃) in a Carius tube for 24 hours at 220°C. Osmium was isolated and purified from the inverse *aqua-regia* by CHCl₃ solvent extraction at room temperature and micro-distillation. The Re was isolated using HCl-HNO₃-

based anion chromatography. The purified Re and Os were loaded to Ni and Pt filaments and analyzed using negative ion thermal ionization mass spectrometry (NTIMS, (Creaser et al., 1991; Völkening et al., 1991)) on a ThermoElectron (TRITON) mass spectrometer. Re was measured using Faraday collectors and Os in peak hopping mode using a secondary electron multiplier (SEM).

Total procedural blanks for Re and Os are <3 and <1 pg, respectively, with an average $^{187}\text{Os}/^{188}\text{Os}$ ratio of ~0.25, and all the data were blank corrected (n=3). All uncertainties include the propagated uncertainty in the standard, spike calibrations, mass spectrometry measurements, and blanks. In-house Re (Restd) and Os (DROsS) solutions were analyzed as a monitor of reproducibility of isotope measurements. The $^{187}\text{Os}/^{188}\text{Os}$ values of the Os standard solution DROsS during this study were 0.1611 ± 0.0066 , with the $^{185}\text{Re}/^{187}\text{Re}$ values of the Re standard solution being 0.5984 ± 0.0002 . These values are in agreement with those previously published for DROsS and Restd (Cumming et al., 2014; Finlay et al., 2011, 2012; Lillis and Selby, 2013; Rooney, 2011). The Re–Os data of this study are regressed using the program *Isoplot* V. 4.15 (Ludwig, 2003) with ^{187}Re decay constant of $1.666 \times 10^{-11} \text{ a}^{-1}$ (Smoliar et al., 1996). The input data contains $^{187}\text{Re}/^{188}\text{Os}$ and $^{187}\text{Os}/^{188}\text{Os}$ ratios with their total 2σ uncertainty and associated error correlation, Rho.

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Table DR1. Results of the Apatite fission track analysis from the Western margin of Xuefeng Uplift

Sample name	Location		Formation	Elevation (m)	Lithology	nGrains	P-age (Ma)	95%-Cl (Ma)	95%+Cl (Ma)	nlength	Length±1σ (μm)	Dpar (μm)
	Latitude	Longitude										
3-18	27°57'03"	110°07'11"	T ₃	166	sandstone, T ₃	36	70.98	5.56	6.02	204	12.66±2.21	2.28
3-67	28°28'24"	108°55'42"	S ₂	408	sandstone, S ₂	36	97.67	6.62	7.09	205	12.91±1.69	2.56
3-97	28°37'48"	108°35'28"	T ₂	441	sandstone, T ₂	35	155.79	12.4	13.46	202	12.87±1.67	2.56
2-3	26°26'36"	106°42'24"	J ₁	1090	sandstone, J ₁	38	149.60	8.59	9.11	206	13.20±2.08	2.54
2-33	26°34'37"	107°19'02"	O ₁	1140	sandstone, O ₁	38	123.43	7.29	7.75	200	13.71±1.43	2.52

Footnote: T₃ = Late Triassic Xiaojiangkou formation, S₂ = Middle Silurian Shiniulan formation, T₂ = Middle Triassic Guangling formation, J₁ = Early Jurassic Ziliujing Formation, O₁ = Early Ordovician Dawan formation

Table DR2. Re-Os isotopic data for Type A and B bitumen from the Majiang and Wanshan reservoirs of the Xuefeng Uplift.

Sample name	Location		Re (ppb)	±	Os (ppt)	±	¹⁸⁷ Re/ ¹⁸⁸ Os	±	¹⁸⁷ Os/ ¹⁸⁸ Os	±	rho	^a IOs (430)	
	Latitude	Longitude											
Type A bitumen													
WL-B01	26°40'48"	107°49'09"	4.32	0.03	206.4	1.1	121.2	1.7	1.66	0.02	0.824	0.79	
WL-B02	26°40'48"	107°49'09"	1.67	0.03	84.4	1.0	113.6	3.7	1.62	0.05	0.847	0.80	
WL-B04	26°40'49"	107°49'09"	2.73	0.03	131.2	1.1	120.4	2.5	1.66	0.03	0.845	0.79	
WL-B05	26°40'49"	107°49'09"	2.67	0.03	134.9	1.1	113.6	2.3	1.60	0.03	0.833	0.79	
WL-B06	26°40'48"	107°49'10"	1.52	0.03	76.5	1.0	114.2	4.1	1.62	0.05	0.848	0.80	
Type B bitumen												^b IOs (70)	^c IOs (80)
MJ1-2	26°14'02"	107°45'05"	8.19	0.05	98.5	1.6	497.0	13.8	1.97	0.08	0.689	1.39	1.31
MJ-S1-B	26°29'25"	107°34'42"	6.14	0.04	217.7	3.6	163.4	4.9	1.68	0.07	0.711	1.49	1.46
MJ-S2-B	26°29'25"	107°34'43"	9.43	0.11	340.3	3.8	157.9	3.3	1.53	0.04	0.608	1.34	1.31
MJ-S4-B	26°29'26"	107°34'42"	15.28	0.28	423.7	4.7	206.3	5.2	1.56	0.04	0.489	1.32	1.29
HBZ-S1-B	26°14'43"	107°33'53"	4.86	0.04	314.8	6.7	88.4	3.7	1.56	0.09	0.697	1.46	1.45
HBZ-S4-B	26°14'44"	107°33'53"	7.93	0.05	323.8	4.2	140.4	3.1	1.58	0.05	0.700	1.42	1.39
HBZ-S6-B	26°14'43"	107°33'54"	5.36	0.04	354.0	5.6	86.8	2.5	1.58	0.06	0.687	1.47	1.46
HBZ-S7-B	26°14'44"	107°33'54"	8.44	0.05	498.1	7.8	97.3	2.7	1.58	0.06	0.697	1.47	1.45
XR-S1-B	26°19'42"	107°46'45"	6.56	0.04	255.5	4.2	149.0	4.3	1.70	0.07	0.706	1.52	1.50
WS-S3-B	27°31'29"	109°13'41"	6.43	0.05	135.7	2.4	280.2	9.2	1.86	0.08	0.749	1.54	1.49
WS-S4-B	27°31'28"	109°13'41"	7.44	0.12	131.8	2.0	333.4	10.6	1.86	0.06	0.682	1.47	1.41
WS-S4-B6	27°31'29"	109°13'42"	2.51	0.04	40.0	0.9	373.9	16.5	1.95	0.11	0.662	1.52	1.45

All uncertainties shown are 2 σ . Rho is the association error correlation (Ludwig, 1980). ^aIOs calculated at 430 Ma, ^bIOs calculated at 70 Ma, ^cIOs calculated at 80 Ma.