NOTES

High molecular weight (C_{35}^{+}) *n*-alkanes of Neogene heavily biodegraded oil in the Qianmiqiao region, North China

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Abstract **With wax content of 1.62%, heavy oil has been produced from the sandstone reservoirs of Neogene Guantao Formation ($\mathrm{Ng1}^{\mathrm{III}}$). In the GC and GC-MS RIC profiles of its aliphatic fraction, *n*-alkanes are totally lost, which shows the result of heavy biodegradedation. However, the remaining trace C_{13}—C_{36} *n*-alkanes can be still seen from its *m/z* 85 mass chromatogram. In addition, a complete series of C_{35}—C_{73} high molecular weight (HMW) *n*-alkanes was detected by high-temperature gas chromatography (HTGC). The HMW *n*-alkane series shows a normal distribution pattern, a major peak at nC_{43}, obvious odd-carbon-number predominance, CPI_{37-55} and OEP_{45-49} values up to 1.17 and 1.16—1.20 respectively. The present study not only has conformed the strong resistibility of HMW *n*-alkanes to biodegradation in crude oils as concluded by previous researchers, but also has provided some significant information on source input and maturity for the heavily biodegraded oil in the Qianmiqiao region.**

Keywords: biodegradation, heavy oil, high-temperature gas chromatography (HTGC), high molecular weight (HMW) wax fraction, HMW *n*-alkane.

In organic geochemical literature, it was traditionally said that *n*-alkanes are removed preferentially to other components during crude oil biodegradation[1,2]. In way of *in vitro* simulation experiments of crude oil biodegradation, however, Setti et al.[3] and Heath et al.[4] have successively discovered that due to the steric hindrance of large paraffin molecules to the bacterial enzymes, the biodegradation rate for $>C_{28}$ *n*-alkanes decreases along with increasing molecular chain-length, there should be a "cut-off" for the biodegradation of *n*-alkanes somewhere between nC_{40} and nC_{45} under their experimental conditions[4]. Both the experiments have made much headway on the research of HMW *n*-alkane biodegradation[3,4], but no heavily biodegraded oil in the reservoir is concerned.

Since 1986, the development of high-temperature gas chromatography (HTGC) and its pretreatment technique has extended the carbon-number range of compounds analyzed and identified, hence this makes it possible to detect C_{40}—C_{100} high molecular weight (HMW) wax fraction[6,7]. Hsieh et al. proposed that some heavily biodegraded oil contains trace amount of HMW hydrocarbons, which are not clearly visible in gas chromatograms of the whole oil, but become evident upon isolation of the HMW wax fraction. The HTGC of HMW hydrocarbons isolated is mainly composed of alkylcyclohexanes, no $>nC_{40}$ alkanes are available[8]. Moreover, so far there has been no detailed report on the HMW *n*-alkanes of heavily biodegraded oil in a real reservoir.

In the present study, the HMW *n*-alkanes of heavily biodegraded oil from a Neogene sandstone reservoir of well Ban-14-1 in the Qianmiqiao region are reported in detail.

1 Geological setting

Located in the north of the Huanghua Depression Bohai Gulf Basin, North China, the Qianmiqiao region is tectonically a structural zone of fault-blocks within the NE-SW trending Ordovician burial-hill zone, which is sandwiched by the Dazhangtuo and the Gang-8 Well Faults. The Ordovician carbonates are directly overlaid with Mesozoic and Tertiary sediments, and these sediments are thin at the top of burial-hills and thick at the flank parts as drape strata. The superface of Ordovician carbonates appears as three main burial-hill highs (fig. 1).

The well Ban-14-1 is situated at the lower position of a middle high in the burial-hill zone (fig. 1). Within the depth interval of 1753—1901 m, altogether 3 shallow reservoirs were found respectively in the sand section Nos. Ⅰ and Ⅲ of the Neogene Guantou Formation ($\mathrm{Ng1}^{\mathrm{I}}$ and $\mathrm{Ng1}^{\mathrm{III}}$) as well as in the sand layer of the Eogene Dongying Formation (Ed). By means of the drill stem test (DST), their oil productive capacity was totally up to 208.2 m^3/d. Therefore, the discovery of these shallow reservoirs in well Ban-14-1 has revealed a new target zone for oil exploration in this area.

As a typical shallow reservoir, the depth of $\mathrm{Ng1}^{\mathrm{III}}$ reservoir ranges from 1804 m to 1806.1 m in well Ban-14-1. The crude oil produced is characterized by high density (0.9704 $g \cdot cm^{-3}$) and viscosity (317.1 $mPa \cdot s$) as well as low solidifying point (−18℃) and wax content (1.62%) as well as abundant resin plus asphaltene (34.9%), thus it is attributed to a typical heavy oil.

2 Experimental

(i) Sample pretreatment. Two portions of the heavy oil were taken from the $\mathrm{Ng1}^{\mathrm{III}}$ reservoir in well Ban-14-1. By means of column chromatography, aliphatic and aromatic fractions were separated from one portion and preserved for the further gas chromatography (GC)

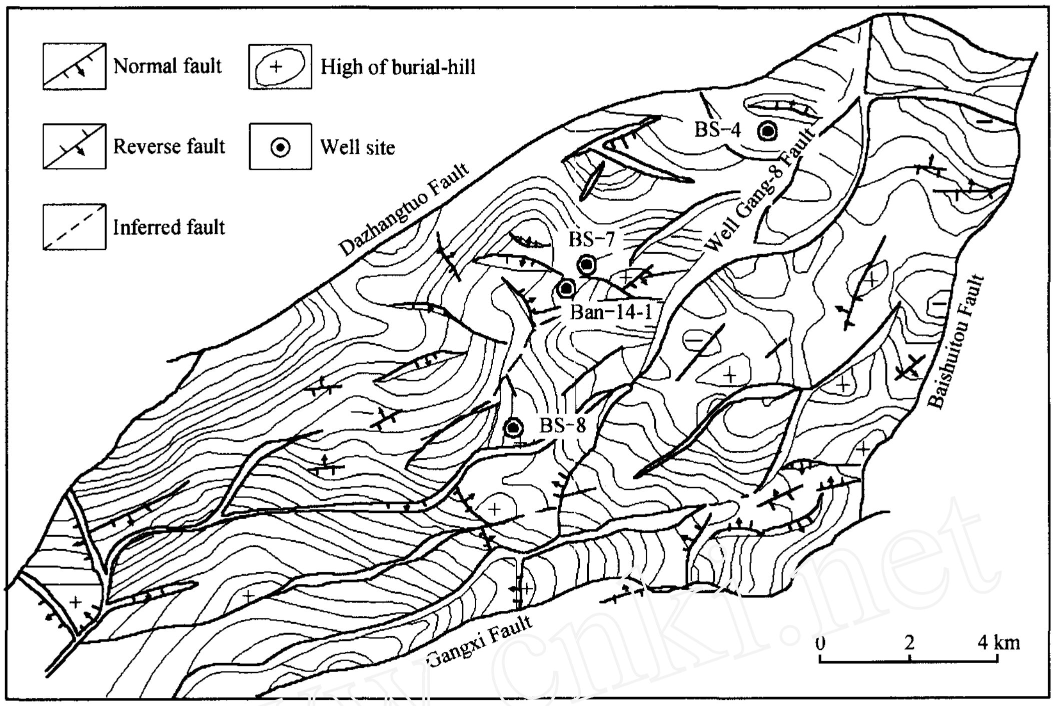

Fig. 1. Tectonic framework of the Qianmiqiao burial-hill and well sites of Ban-14-1.

and/or GC/MS analyses. Owing to the low wax content, another portion had to be concentrated for the separation of HMW wax fraction. Based on the practical wax content, 372 g heavy oil had been dissolved in 50 mL tetrahydrofuran solvent and the solution was saved overnight for concentration and precipitation of the HMW wax fraction, and then, the precipitate was filtered and collected for HTGC analysis of HMW *n*-alkanes.

(ii) GC and HTGC

(1) GC analysis. The aliphatic fraction was analyzed on a Shimadzu Modal GC-17A gas chromatograph equipped with a fused silica capillary column coated with SE-54 (25 m×0.25 mm, i.d.). N_2 was used as carrier gas with a split ratio of 1∶100. The temperatures of both vaporizer and detector were set at 300℃. The temperature was programmed from 100℃ (held for 1 min) to 300℃ (for 20 min) at 4℃/min.

(2) HTGC analysis. The precipitate of HMW wax fraction was dissolved in the hexane∶carbon bisulfide (*v/v* 1∶1) solvent. The squalane (chromatographic grade) was used as an internal standard for identification and quantification. The HTGC was run on the same instrument as GC, but a Ultra ALLOY-1 high temperature capillary column (5 m×0.25 mm, i.d.×0.5 μm) and He_2 carrier gas were used. Both vaporizer and detector temperatures were set at 405℃. The temperature program was from 100℃ (held for 2 min) to 410℃ (for 5 min) at 15℃ /min.

(iii) Gas chromatography-mass spectrometry (GC-MS). The GC-MS analyses of aliphatic and aromatic fractions of the heavy oil were performed on a Finnigan Modal 710 quadrupole instrument fitted with a DB-5 fused silica capillary column (30 m×0.32 mm, i.d.). He_2 carrier gas and EI (70 eV) mode were used. Both vaporizer and detector temperatures were set at 300℃. The temperature programs were respectively from 100℃ (held for 1 min) to 220℃ at 4℃/min, then from 220℃ to 300℃ (for 5 min) at 2℃/min for the aliphatic fraction, and from 100℃ (held for 1 min) to 300℃ (for 5 min) at 3℃/min for the aromatic fraction.

3 Results

(i) Bitumen composition analysis. Compared with common crude oils, the bitumen composition of the $\mathrm{Ng}^{\mathrm{III}}$ heavy oil in well Ban-14-1 makes a feature of very low aliphatic (42.6%), higher aromatic (31.4%), relatively high resin (19.0%), relatively low asphaltene (7.1%) contents, and thus, the heavy oil appears as a low aliphatics/aromatics ratio (0.8—1.34) and a high resin/asphaltene ratio (1.8—4.0), which show the characteristics of some immature oil[9] or biodegraded oil.

(ii) GC and GC-MS analyses. In the GC and GC-MS RIC profiles of the aliphatic fraction of $\mathrm{Ng\,1}^{\mathrm{III}}$ heavy oil, almost all *n*-alkanes and most isoprenoid hydrocarbons are lost, the GC baselines appear as a remarkable hump, implying a large quantity of unresolved complex matter (UCM). According to the criteria of biodegradation ranks proposed by Peters and Moldowan[10], the Ng 1^{III} heavy oil shows typical GC or RIC features of heavily biodegraded oil (Rank No. 6; fig. 2(a)).

By comparison with the GC and RIC profiles, however, mass chromatogram shows evidently higher sensitivity and detecting level. As far as the Ng 1^{III} heavy oil is concerned, even though both the series of *n*-alkanes and isoprenoid hydrocarbons are undetectable in GC and RIC profiles (fig. 2(a)), a remaining trace series of C_{13}—C_{36} *n*-alkanes can still be detected in the *m/z* 85 mass chromatogram, but no $>C_{36}$ HMW hydrocarbons are visible in routine GC and GC-MS analyses (fig. 2(b)).

Moreover, according to GC-MS analysis, both the series of C_{32}—C_{34} benzohopanes and C_{27}—C_{29} ring-C monoaromatic sterenes are detected from the aromatic fraction of the Ng1^{III} heavy oil in well Ban-14-1$^{1)}$ indicating the immaturity of the heavy oil[11, 12].

In addition, each maturity parameter of triterpanes in the aliphatic fractions shows very similar values among three Ng and Ed shallow reservoirs in well Ban-14-1, i.e. the C_{31} αβ-hopane 22S/(22S+22R) ratios range from 0.56—0.58 with differences of less than 0.02, and the Ts/Tm ratios also show very close values with differences of less than 0.06 (table 1). In this case, therefore, the variation of both the parameters is consistent with each other and the triterpane epimers in both parameters seem to be free of biodegradation.

Utterly different from the triterpane parameters, however, the sterane maturity parameters, C_{29} ααα-sterane 20S/20S+20R) and C_{29} sterane ααα/(ααα+αββ), show a larger variation, the differences for each parameter can be up to 0.12 (table 1). Most possibly, sterane epimers in these parameters were biodegraded to different extent,

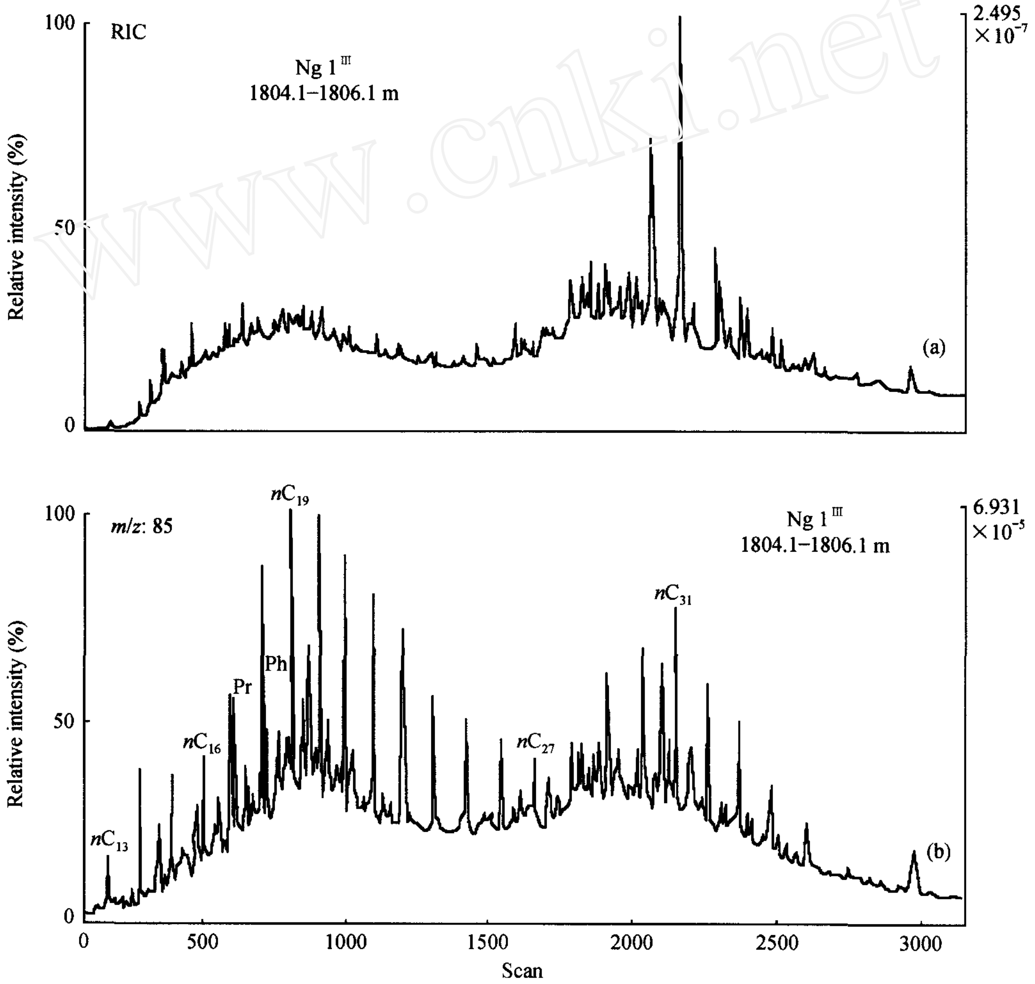

Fig. 2. RIC (a) and *m/z* 85 mass chromatogram (b) of the aliphatic fraction from the Ng1^{III} heavy oil in well Ban-14-1. Pr, Pristane; Ph, phytane; IP_i, isoprenoid hydrocarbons; nC_i, normal alkanes; i, carbon number.

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1) Lu Hong, Study on Hydrocarbon Sources of Oil Pool in Qianmiqiao Bured Hill Region, Huanghua Depresson (Ph. D. Thesis), University of Petroleum (Beijing), China, 2000.

Table 1 Maturity parameters of the low-waxy heavy oil in the shallow reservoir

Well No.	Depth/m	Position	$C_{31}\ \frac{22S}{22S+22R}$ *1	$\frac{Ts}{Tm}$	$C_{29}\ \frac{\alpha\beta\beta}{\alpha\alpha\alpha+\alpha\beta\beta}$ *2	$C_{29}\ \frac{20S}{20S+20R}$ *3
Ban-14-1	1753.8—1757.8	Ng1$^{\text{I}}$	0.58	1.00	0.36	0.45
Ban-14-1	1804.1—1806.1	Ng1$^{\text{III}}$	0.57	0.96	0.44	0.44
Ban-14-1	1894.1—1901.9	Ed	0.56	0.94	0.48	0.57

*1 C_{31} αβ-hopane 22S/(22S+22R); *2 C_{29} sterane αββ/(ααα+αββ); *3 C_{29} ααα-sterane 20S/(20S+20R).

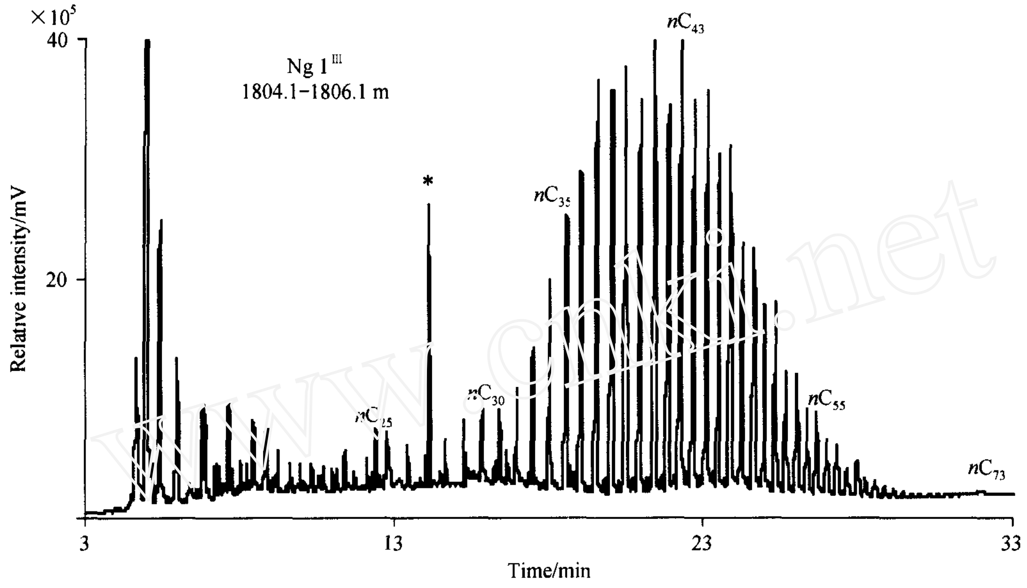

Fig. 3. HTGC of the HMW wax fraction in the heavy oil from the Ng1$^{\text{III}}$ reservoirs in well Ban-14-1. * Internal standard squalane peak.

which makes all the sterane maturity parameters invalid.

(iii) HTGC. Using HTGC technique, a successive n-alkane series has been detected from the HMW wax fraction of the Ng1$^{\text{III}}$ heavy oil in well Ban-14-1, its carbon-number range is extended to nC_{14}—nC_{73} and the abundance of C_{35}^{-} n-alkanes is remarkably reduced due to biodegradation, but the HMW C_{35}—C_{55} n-alkanes still keep high abundance without any indication of biodegradation, displaying a normal distributional pattern with a major peak at nC_{43} as well as an obvious odd carbon-number preference with CPI_{37-55} and OEP_{45-49} values of 1.17 and 1.16—1.20 respectively (fig. 3).

4 Discussion

(ⅰ) Origin of the HMW n-alkanes in the Ng1$^{\text{III}}$ heavy oil. As for the origin of HMW hydrocarbon compounds, two possible genetic mechanisms were summarized by Philp et al.: one mechanism is that the HMW hydrocarbons are derived from the corresponding natural precursors; another possibility is that these compounds result from the di- and trimerization of lower molecular weight precursors[7].

Tulloch (1976) had indicated that n-alkanes are probably present in all plants, some plants (such as many members of the Crassulaceae) contain 40%—60% hydrocarbons; the major alkanes contain an odd number of carbons ranging from about C_{21} to C_{37}, even an unusual hydrocarbon isolated from the grass *Lepyochloa digitata* apparently has 62 carbons[13]. Killops et al. (2000) reported the HMW n-alkane distribution detected by HTGC from a New Zealand immature coal, the odd-over-even predominance of its n-alkanes is most remarkable within the rang of nC_{51}—nC_{57} and showing a CPI_{51-57} value up to 1.79 at the corresponding maturity of 0.5%R_o; but the odd-over-even predominance is reduced with increasing maturity; while the CPI_{51-57} value is only 1.09 in a New Zealand coal-sourced mature waxy oil sample. The authors further proposed that the C_{40}^{+} n-alkanes may be liberated by thermal decarboxylation of esters from cutan/cutin sources within kerogen[14].

Del Rio et al. reported a series of HTGC results of hydrocarbons isolated from oils, ozocerite, microcrystaline waxes, Fischer-Tropsch synthetic wax and wax deposits collected from the drill stem pipe, the maximum of carbon numbers for their HMW *n*-alkanes detected is as high as $n\mathrm{C}_{75}$ with major peaks ranging from $n\mathrm{C}_{38}$ to $n\mathrm{C}_{42}$ but without any odd-over-even predominance, based on which, it was proposed that these HMW hydrocarbons were derived from the low molecular weight precursors by way of di- and trimerizations[15].

In comparison with the above-mentioned HTGC results in literature, the HMW wax fraction of Ng1$^{\mathrm{III}}$ heavy oil in well Ban-14-1 has high similarities with those in the immature coal reported by Killops et al.[14]. The development of $\mathrm{C}_{31}-\mathrm{C}_{73}$ *n*-alkanes in the heavily biodegraded Ng1$^{\mathrm{III}}$ oil shows the resistibility of HMW hydrocarbons to biodegradation[3,4], whereas the evident odd-over-even predominance of HMW *n*-alkanes is also indicative of the immaturity of the heavy oil[14,16] as well as the source input of higher plant waxes[13,16]. As for the above genetic mechanisms under discussion, therefore, the contribution of higher plant waxes for the origin of the HMW *n*-alkanes in the Ng1$^{\mathrm{III}}$ heavy oil seems to be more convincing.

(ii) Significance of the odd-over-even predominance in HMW *n*-alkanes. In the recent literature, Hsieh et al. and Killops et al. have approached the significance of the odd-over-even predominance in HMW hydrocarbons[8,14]. Owing to the long-chain alkylcyclohexanes as main component and for lack of $\geqslant\mathrm{C}_{40}$ *n*-alkanes in oil samples analyzed, Hsieh et al. only calculated and proposed $\mathrm{CPI}^{\mathrm{a}}$ as a parameter of sedimentary environments instead of a maturity parameter, i.e. $\mathrm{CPI}^{\mathrm{a}}_{42-46} << 1$ is indicative for non-marine oils, while $\mathrm{CPI}^{\mathrm{a}}_{42-46} > 1$ for marine oils[8].

In New Zealand coal studied by Killops et al., however, CPI_{51-57} values appear as a general tendency of variation synchronous with CPI_{25-31} values. In an immature coal (R_o 0.5%), the *n*-alkane series shows a remarkable odd-over-even predominance with CPI_{25-31} and CPI_{51-57} values as high as 1.32 and 1.79 respectively. As the maturity of coal increases, both the CPI_{25-31} and CPI_{51-57} values decrease to 1.14 and 1.32 respectively at the maturity level of 1.00%R_o[14]. Therefore, the odd-over-even predominance parameter CPI can yet be regarded as an effective maturity indicator in HMW *n*-alkanes.

As for the HMW wax fraction of heavily biodegraded Ng1$^{\mathrm{III}}$ oil in well Ban-14-1, *n*-alkanes are the major component with evident odd-over-even predominance, and alkylcyclohexanes are undeveloped. Since monoaromatic sterenes and benzohopanes, as immaturity indicators, are also detected in the Ng1$^{\mathrm{III}}$ heavy oil1), the CPI_{37-55} value of 1.17 and OEP_{45-49} of 1.16 or 1.20 in the HMW *n*-alkanes should be the indicators of immaturity for the heavy oil.

5 Conclusion

A shallow oil reservoir has been found from the sand section No. III of the Neogene Guantou Formation (Ng1$^{\mathrm{III}}$) in well Ban-14-1 within the Qianmiqiao region. As immature biodegraded oil, the reservoired Ng1$^{\mathrm{III}}$ heavy oil is characterized by high density and viscosity as well as low solidifying point and wax content in its physical properties.

Almost alkanes are totally lost in the Ng1$^{\mathrm{III}}$ heavy oil produced from well Ban-14-1, but both remaining trace series of *n*-alkanes and isoprenoid hydrocarbons can still be detected from *m/z* 85 mass chromatogram. Sterane epimers are, to different extent, under the influence of biodegradation, which makes sterane maturity parameters invalid. Nevertheless, triterpane maturity parameters of the same heavy oil among three adjoining Ng and Ed shallow reservoirs are still consistent, these triterpane epimers seem not to be biodegraded. Therefore, the Ng1$^{\mathrm{III}}$ heavy oil has reached the level of heavy biodegradation (Rank No. 6).

Even though the heavily biodegraded Ng1$^{\mathrm{III}}$ oil has low wax content (1.62%), it still contains considerably abundant $\mathrm{C}_{35}-\mathrm{C}_{73}$ HMW *n*-alkanes. These HMW *n*-alkanes also keep an obvious odd-number-carbon predominance with CPI_{37-55} and OEP_{45-49} values of 1.17 and 1.16—1.20 respectively. The study of HMW *n*-alkanes of the Ng1$^{\mathrm{III}}$ oil in well Ban-14-1 not only conforms the strong resistibility of HMW *n*-alkanes to biodegradation in crude oils as concluded by Setti et al. (1993) and Heath et al. (1997), but also provides some important geochemical information on source input, maturity and even oil-source correlation for the heavily biodegraded oil. In addition, this result can also be used for reference in other cases of biodegraded oils.

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Surveying of the deformed terraces and crust shortening rate in the northwestern Tarim Basin: Comment

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The paper of Shen et al., entitled "Surveying of the deformed terraces and crust shortening rate in the northwest Tarim Basin", was published in Chinese Science Bulletin (Vol. 46, No. 12)[1]. Shen et al. found the deformation of Late Pleistocene to Holocene terraces of the Boguzi River across the Artushi Anticline in the northwest Tarim Basin close to the Pamir, and made level survey and differential GPS measurement, which is of great importance to geodynamics for research on the coupling of Tianshan Mountains uplifting and Tarim Basin depression. But their understanding to the deformation mechanics of terraces and the calculation methods of crustal shortening are open to discussion. Therefore, we discuss it with Shen Jun et al.

1 Formula for calculating the crustal shortening

Shen Jun et al. gave the calculating formula for crustal shortening:

$$\Delta L \approx \tan\theta \times H' \qquad (1)$$

and pointed out that θ was dip of the main thrust located in the core of the anticline . But H' was not explained, and we speculate that H' is the magnitude of warping up of terraces in table 1. While Shen Jun et al. did not give the deduction process of the formula, and just said that it was "a simple equation"[1]. The question is that eq. (1) is not right for the function between crustal shortening (ΔL), dip of thrust (θ) and magnitude of warping up of terrace (H'). We infer that the relation between ΔL, θ and H' in Shen et al.'s paper should be as shown in fig. 1. If so, the equation should be

$$\Delta L \approx \cot\theta \times H'. \qquad (2)$$

So, the relation shown in eq. (1) is absolutely impossible.

2 Calculation of crustal shortening rate

Shen et al. gave the magnitudes of all parameters for calculating the crustal shortening rate in table 1[1]. The question in table 1 is that the crustal shortening is not what is calculated by eq. (1). According to eq. (1) and 40 $\pm 2°$ of θ[1], crustal shortening should be smaller than the magnitude of warping up of every terrace respectively, and corresponding warping up rate and crustal shortening