

# Identification of the four rearranged hopane series in geological bodies and their geochemical significances

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**Abstract** Saturated fractions in a total of 23 oil samples and hydrocarbon source rocks from the Songliao, Tarim, and Ordos Basins have been analyzed by GC–MS and GC–MS–MS. According to the relative retention, mass spectral characteristics, and comparison with existing literature, a complete carbon number distribution ranging from C<sub>27</sub> to C<sub>35</sub> (without C<sub>28</sub>) in the 17 $\alpha$ (H)-diahopane series and early-eluting rearranged hopane series is identified. Compounds in the 18 $\alpha$ (H)-neohopane series (Ts and C<sub>29</sub>Ts) and 21-methyl-28-nor-hopane series (29Nsp and 30Nsp) are also noted. These four series of rearranged hopanes seem to occur in both brackish-saline lacustrine and coal measure environments. However, the coal measure and swamp environments being under an oxic condition, compared with brackish-saline lacustrines, are presumably more helpful to the formation of 30E. Diversity in the content and distribution patterns indicate that rearranged hopanes could serve as good indicators of organic facies, depositional environment and maturity in petroleum geology.

**Keywords** Rearranged hopanes · Early-eluting series · 21-methyl-28-nor-hopanes · Geochemical significances

## 1 Introduction

Rearranged hopanes refer to a class of biomarkers with carbon ring framework identical to that of regular hopanes, but with methane side chain carbon position being distinct from that of regular hopanes. Multiple homologues occur in hydrocarbon source rocks and crude oils. Currently, four series of rearranged hopanes have been reported. They are the 17 $\alpha$ (H)-diahopane series, the 18 $\alpha$ (H)-neohopane series, the early-eluting rearranged hopane series, and the 21-methyl-28-nor-hopane series (or 28-nor-spergulane series). 18 $\alpha$ (H)-22,29,30-trisnorneohopane (Ts), which has a rearranged methyl group at C-17, is one of the first hopanoid hydrocarbons to be rigorously characterized by X-ray (Whitehead 1974; Smith 1975). Moldowan et al. (1991) identified by nuclear magnetic resonance (NMR) spectroscopy techniques a member of the 18 $\alpha$ (H)-neohopane series (denoted “C<sub>29</sub>Ts”). They also determined (by X-ray crystallography) the structure of 17 $\alpha$ -15 $\alpha$ -methyl-27-norhopane (C<sub>30</sub> diahopane), a member of the 17 $\alpha$ (H)-diahopane series. Killips and Howell (1991) and Telnæs et al. (1992) noted the occurrence of a further series of unidentified rearranged hopanes in oils. Compounds in this pseudo homologous series are notable in eluting approximately two carbon numbers earlier than the regular hopanes. Farrimond and Telnæs (1996) reported this early-eluting rearranged hopane series, which appears to extend from C<sub>27</sub> to C<sub>35</sub> (without C<sub>28</sub> member). Then, the C<sub>30</sub> member (C<sub>30</sub> early-eluting rearranged hopane or 30E) of the series was synthesized by Nytoft (2007) in the laboratory. The C<sub>29</sub> 28-nor-spergulane (“X” in Huang et al. 2003), which is a member of the 21-methyl-28-nor-hopane series, was first found in some of the lacustrine oils from the Western Pearl River Basin offshore South China. Subsequently, Nytoft et al. (2006) indentified a new series of rearranged hopanes

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ranging from C<sub>29</sub> to at least C<sub>34</sub> as 28-nor-spergulanenes (or 21-methyl-28-nor-hopanes) using NMR spectroscopy. Most commonly, 29Nsp is always the dominant member of the series and seems to be particularly abundant in some oils from lacustrine source rocks in South East Asia.

In recent years, rearranged hopanes have received increasing attention as biological markers with applications for geochemical studies of petroleum source rocks and crude oils (Huang et al. 2003; Li et al. 2009; Zhang et al. 2009; Cheng et al. 2014). However, systematic identification of the four rearranged hopane series is not reported in the domestic and overseas literature. In this article, on the basis of previous studies, we identify the four series of rearranged hopanes. Their geochemical significances are also discussed, which provides the theoretical foundation for their applications in petroleum geology.

## 2 Experimental

### 2.1 Samples

Eight hydrocarbon source rocks were collected in China from the Songliao Basin (two mudstones), Tarim Basin (two coals and a carbonaceous mudstone), and Ordos Basin (three coals). A total of fifteen oil samples were chosen from the Songliao Basin.

### 2.2 GC–MS and GC–MS–MS

All source rocks were crushed into fine powder and extracted with a dichloromethane/methanol mixture (93:7 v/v) using a Soxhlet apparatus for 72 h. The asphaltene was removed from extracts and oils by precipitation in n-hexane. The deasphalted extracts and oils were fractionated into saturated, aromatic hydrocarbons and polar compounds using open alumina-silica gel (1:2) column chromatography. The solvents for column chromatography are petroleum ether, dichloromethane, and methanol, respectively. The saturated fractions of all the samples were analyzed using gas chromatography-mass spectrometry (GC–MS) and GC–MS–MS.

GC–MS was carried out with a HP 5973 mass spectrometer, coupled to a HP 6890 GC equipped with a HP-5MS fused silica capillary column (30 m × 0.25 mm i.d., film thicknesses 0.25 μm). The GC temperature was programmed to start at 50 °C for 1 min, increase to 100 °C at a rate of 20 °C/min, and from 100 to 310 °C at a rate of 3 °C/min with a final hold of 16 min. Helium was used as the carrier gas with a rate of 1.0 ml/min and the ionization source operated at 70 eV.

GC–MS–MS was performed using an Agilent 6890 N gas chromatograph connected to a Waters (Micromass)

Quattro Micro GC tandem quadrupole mass spectrometer. An HP-5MS, a Varian factorFour VF-5 ms column (30 m × 0.25 mm i.d., film thicknesses 0.25 μm) was used. The temperature program was 20 °C/min from 50 to 100 °C and 3 °C/min from 100 to 310 °C followed by 16 min at 310 °C.

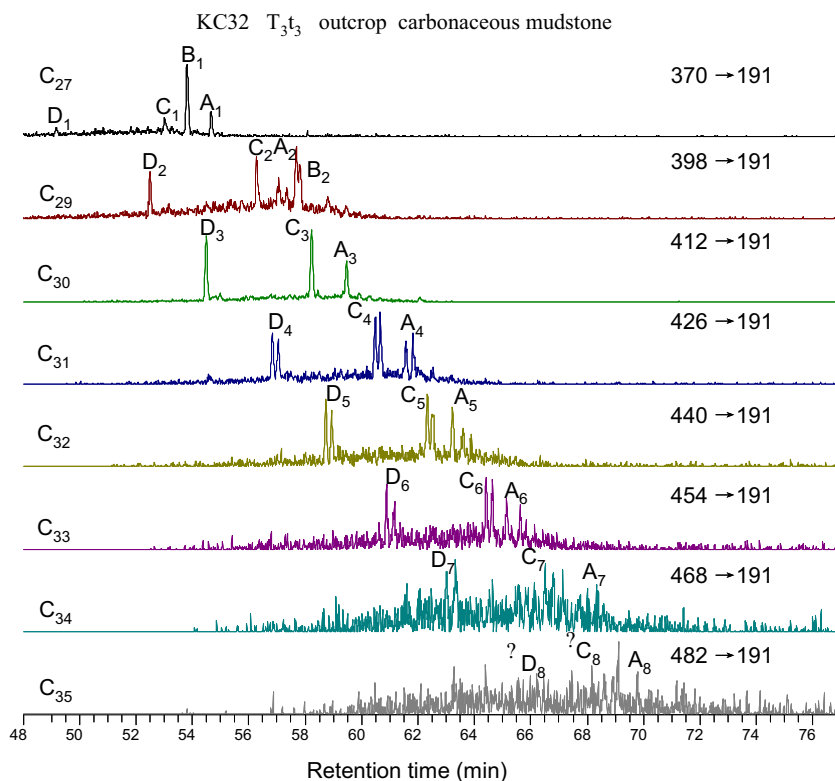
## 3 Results and discussion

### 3.1 Identification of 17α(H)-diahopanes and early-eluting rearranged hopanes

All samples were selected for GC–MS analysis. The pentacyclic triterpenes of oil samples and source rocks exhibit a complex composition. In order to determine the composition and carbon number distribution of these compounds, selected saturated hydrocarbon fractions were analyzed by GC–MS–MS. The use of GC–MS–MS parent ion analysis can reveal homologous series of compounds that are analytically commingled in simple GC–MS analyses. In addition to the ubiquitous regular hopanes, four series of rearranged hopanes can be distinguished: 17α(H)-diahopanes, 18α(H)-neohopanes, early-eluting rearranged hopanes, and 28-nor-spergulanenes. The 17α(H)-diahopane series and early-eluting rearranged hopane series are identified by their relative retention times in comparison with previous literature (Killops and Howell 1991; Moldowan et al. 1991; Telnæs et al. 1992).

As shown in Fig. 1, the 17α(H)-diahopane series and early-eluting rearranged hopane series are apparent from tandem mass spectrometry (MS–MS) data. The 17α(H)-diahopane series was first detected in crude oil from Prudhoe Bay in Alaska (Moldowan et al. 1991), ranging from C<sub>27</sub> to C<sub>35</sub> (although the C<sub>27</sub> member is not distinct), but with no C<sub>28</sub> member; the C<sub>31</sub>–C<sub>35</sub> members elute as pairs of 22S- and 22R-isomers in m/z 191 gas chromatograms. For the samples in this study, the series displays a complete carbon number range from C<sub>27</sub>, C<sub>29</sub>–C<sub>35</sub> (C<sub>1</sub>–C<sub>8</sub> in Fig. 1), with possible C<sub>35</sub> member in low abundance. Because of the lack of a distinct peak for the C<sub>28</sub> member in m/z 191 gas chromatograms, the m/z 384 → 191 transition was not monitored in GC–MS–MS analysis. Analogously, the early-eluting rearranged hopane series (D<sub>1</sub>–D<sub>8</sub> in Fig. 1) extends from C<sub>29</sub> to C<sub>35</sub> and a C<sub>27</sub> member is also apparent from the m/z 370 → 191 transition (retention time around 49.18 min). Doublets are resolved for members above C<sub>30</sub>. The C<sub>30</sub> member (30E) of the series is dominated and commonly high in continental oils of China. For instance, 30E is abundant in oils from the Yingmaili and Yaha regions of the Tarim Basin in western China (Zhu 1997; Zhu et al. 1997). Their carbon number distributions are comparable to those of the regular

**Fig. 1** Parent ion analysis of  $m/z$  191 daughter ions in the saturated fraction of hydrocarbon source rock (KC32, outcrop, carbonaceous mudstone) in the Tarim Basin. Note:  $A_1$ – $A_8 = C_{27}$ ,  $C_{29}$ – $C_{35}$  hopanes;  $B_1$ – $B_2 = Ts$ ,  $C_{29}Ts$ ;  $C_1$ – $C_8 = C_{27}$ ,  $C_{29}$ – $C_{35}$  diahopanes;  $D_1$ – $D_8 = C_{27}$ ,  $C_{29}$ – $C_{35}$  early-eluting rearranged hopanes



$17\alpha(H)$ -hopanes. The compounds of the  $17\alpha(H)$ -diahopane series and early-eluting rearranged hopane series are observed to have a linear relationship for GC retention time, indicating that their homologues differ only in the length of the side chain. Relative retention indices (based on hopane carbon numbers) of the various pseudohomologues are given in Table 1 and the series shown in Fig. 2.

Due to co-elutions and low concentrations, it is not possible to obtain mass spectra of all members of the  $17\alpha(H)$ -diahopanes and early-eluting rearranged hopanes. Mass spectra of the  $C_{30}$  diahopane and 30E are largely similar (Fig. 3a, b). They are all characterized by a  $m/z$  191 base peak, a  $m/z$  412 molecular ion and a pattern of peaks below  $m/z$  191 separated by 14 mass units. However, in comparison with the  $C_{30}$  diahopane, 30E has a small  $m/z$  287 fragment, which is considered to come from fragmentation through the B-ring. This ion ( $m/z$  287) is absent or insignificant in the spectra of the other compounds. The 30E elutes from the gas chromatograph ( $m/z$  191) behind Ts and just in front of Tm. The elutions of the other members of the series are exhibited in Fig. 1. It is clear that the early-eluting rearranged hopanes elute from the gas chromatograph approximately two carbon numbers earlier than the regular hopanes. Retention times of these four series of rearranged hopanes are provided in Table 1. The very elution of the early-eluting rearranged hopane series indicates that they are more volatile than the other hopane

and rearranged hopane series. These features should help in their identification.

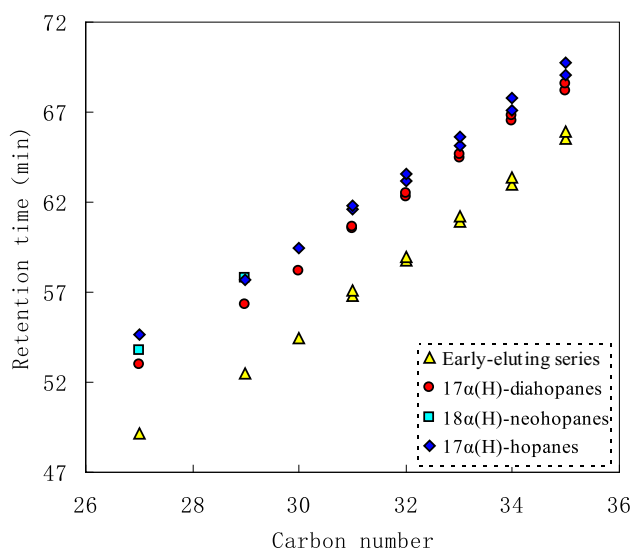
### 3.2 Identification of $18\alpha(H)$ -neohopanes and 28-norspergulanenes

According to relative retention time, we also identify the  $18\alpha(H)$ -neohopane series ( $B_1$ – $B_2$  in Fig. 1). It is clear from Figs. 1 and 2 that the carbon number distribution of the  $18\alpha(H)$ -neohopane series differs significantly from those of the  $17\alpha(H)$ -hopane and the other two rearranged hopane series. Compounds in the  $18\alpha(H)$ -neohopane series includes  $C_{27}$  and  $C_{29}$ – $C_{30}$  members; the  $C_{27}$  member (Ts) and  $C_{29}$  member ( $C_{29}Ts$ ) are widely recognized now, while the occurrence of higher homologues (above  $C_{31}$ ) is still unclear. This observation argues for at least partly different precursor biomolecules, the  $18\alpha(H)$ -neohopanes could be derived from diplopterol and diploptene or a  $C_{29}$  hopanoid (Moldowan et al. 1991; Farrimond and Telnæs 1996). Usually, Ts elutes well before Tm,  $C_{29}Ts$  is barely resolved from, and elutes after,  $C_{29}$  regular hopane. The  $C_{30}$  diahopane is small in comparison to the neighboring  $C_{29}Ts$ , but crudes and hydrocarbon source rocks in our study area show a higher  $m/z$  191 peak from the  $C_{30}$  diahopane than from  $C_{29}Ts$ . Using GC retention time, Farrimond and Telnæs (1996) tentatively identified the peak eluted after  $C_{30}$  regular hopane as the  $C_{30}$  member ( $C_{30}Ts$ ) of the

**Table 1** Hopane retention indices of selected carbon numbers of the three rearranged hopane series

Carbon number	Retention time (min)			
	Early-eluting series	17 $\alpha$ (H)-diahopane series	18 $\alpha$ (H)-neohopane series	17 $\alpha$ (H)-hopane series
27	49.18	53.01	53.81	54.66
29	52.49	56.27	57.82	57.69
30	54.50	58.22		59.47
31(22S)	56.84	60.49		61.57
31(22R)	57.05	60.67		61.82
32(22S)	58.73	62.33		63.22
32(22R)	58.94	62.50		63.59
33(22S)	60.89	64.42		65.15
33(22R)	61.17	64.64		65.62
34(22S)	63.01	66.51		67.13
34(22R)	63.33	66.80		67.75
35(22S)	65.57	68.17		69.10
35(22R)	65.97	68.58		69.79

C-22 stereochemistry is undetermined in the 17 $\alpha$ (H)-diahopane and early-eluting series



**Fig. 2** Retention indices (see Table 1) for different members of the hopane and rearranged hopane series, plotted versus carbon number

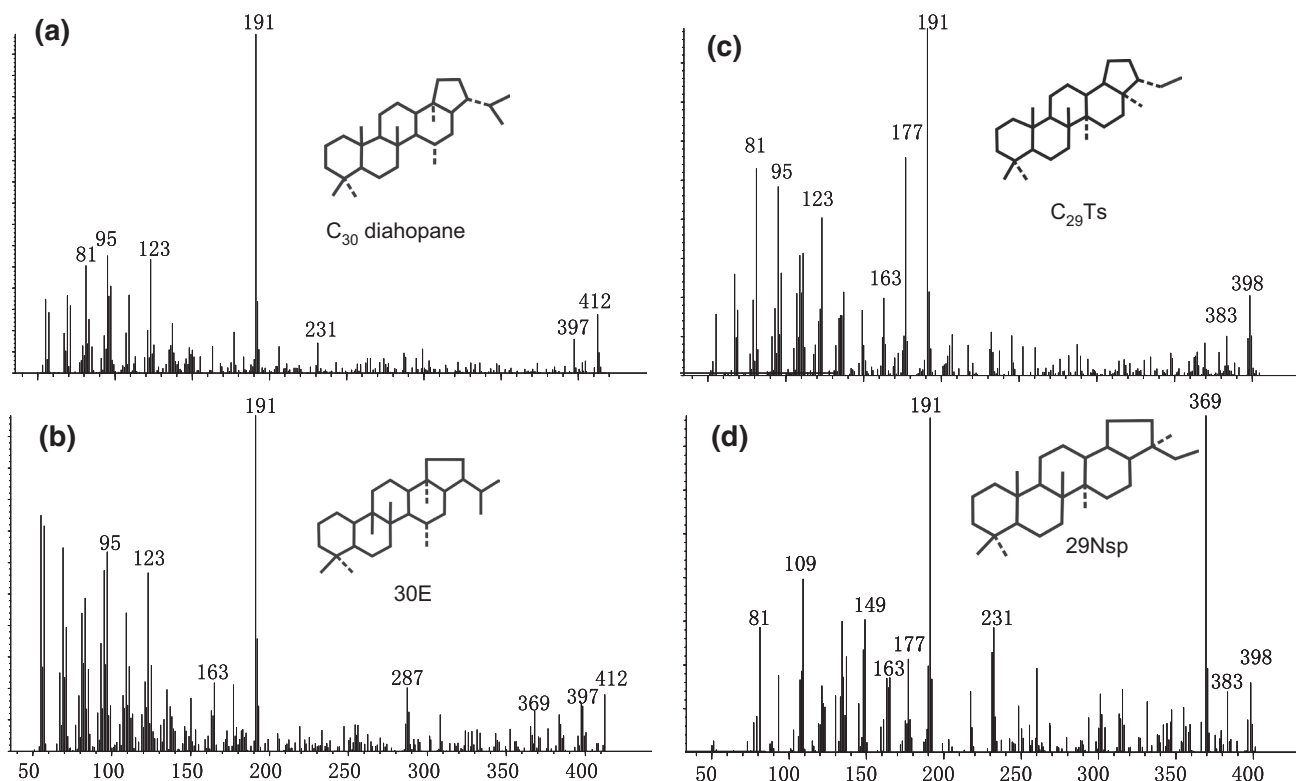
18 $\alpha$ (H)-neohopane series in  $m/z$  191 mass chromatogram. However, this member was not observed at the corresponding position in the  $m/z$  412  $\rightarrow$  191 transition in our data. The mass spectrum of  $C_{29}$ Ts displays a regular pattern of ions separated by 14 mass units below  $m/z$  191, a  $m/z$  191 base peak, a large  $m/z$  177 fragment, and a  $m/z$  398 molecular ion (Fig. 3c).

By combining with previous studies (Nytoft et al. 2006), two compounds in the 21-methyl-28-nor-hopane series (29Nsp and 30Nsp) are detected in individual oil samples and hydrocarbon source rocks (Fig. 4). The 21-methyl-28-nor-

hopane series is recognized later than the other three rearranged hopane series and their applications in petroleum are seldom reported. The carbon number distribution of the 21-methyl-28-nor-hopane series ranges from  $C_{27}$  to  $C_{34}$ ; the  $C_{29}$  member is always the dominant member of the series and can be detected in most crude oils or mature sediments using GC–MS. The 29Nsp elutes midway between  $C_{30}$  diahopane and 17 $\beta$ (H), 21 $\alpha$ (H)-30-norhopane ( $C_{29}$  moretane) in  $m/z$  191 mass chromatogram; the 30Nsp elutes after  $C_{30}$  diahopane (Nytoft et al. 2006; Huang et al. 2003). Similar to the  $C_{29}$  member of the 18 $\alpha$ (H)-neohopane series ( $C_{29}$ Ts), 29Nsp has a  $m/z$  191 base peak, a  $m/z$  398 molecular ion and a pattern of peaks of below  $m/z$  191 separated by 14 mass units typical of hopanes. The most notable feature of the mass spectrum for 29Nsp is the greater abundance of the  $m/z$  369 ion than recorded for  $C_{29}$ Ts (Fig. 3d). Because of the low content of 30Nsp, its mass spectrum was not obtained, and no members above  $C_{30}$  were detected.

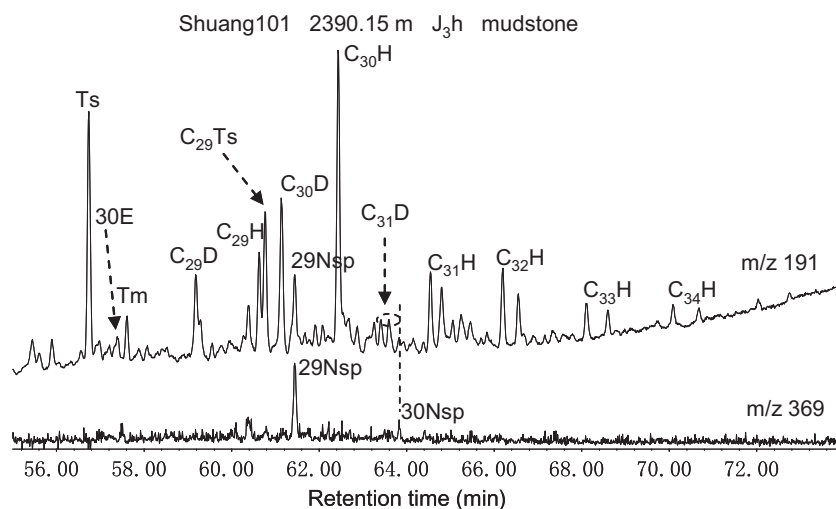
### 3.3 Geochemical significances of rearranged hopanes

Rearranged hopanes have been widely applied in maturity assessment of oils, oil-source rock correlation, petroleum study, etc. (Zhu et al. 2007; Li et al. 2009; Zhang 2013; Cheng et al. 2014). For instance, Horstad et al. (1990) measured  $C_{30}$  diahopane/( $C_{30}$  diahopane +  $C_{30}$  hopane) ratios, which increase with maturity in North Sea oils, and were useful to map maturity gradients in North Sea oil fields. Huang et al. (2003) distinguished the various petroleum source facies in the Western Pearl River Basin offshore South China according to the abundance of 29Nsp relative to bicadinanes



**Fig. 3** Mass spectra of (a) the  $C_{30}$  components of the  $17\alpha(H)$ -diahopane series and (b) early-eluting rearranged hopane series and (c) the  $C_{29}$  components of the  $18\alpha(H)$ -neohopane series and (d) 28-nor-spergulane series, all taken from the same full scan GC–MS analysis of oil sample (SW 103 well, 1907.8–1915.6 m) in the Songliao Basin

**Fig. 4**  $m/z$  191 and  $m/z$  369 mass chromatograms of a mudstone in the Songliao Basin with a high content of rearranged hopanes (Shuang 101 well, 2390.15 m, mudstone). *H* hopane; *D* diahopane; *Nsp* 28-nor-spergulane



W and T ( $m/z$  369). Li et al. (2009) differentiated two petroleum systems in the Huachang field of the Fushan Depression in terms of the relative concentration of diahopane and other maturity parameters. However, their genetic mechanisms and influencing factors are controversial.

Many scholars have explored the formation conditions and geochemical attributes of rearranged hopanes. The

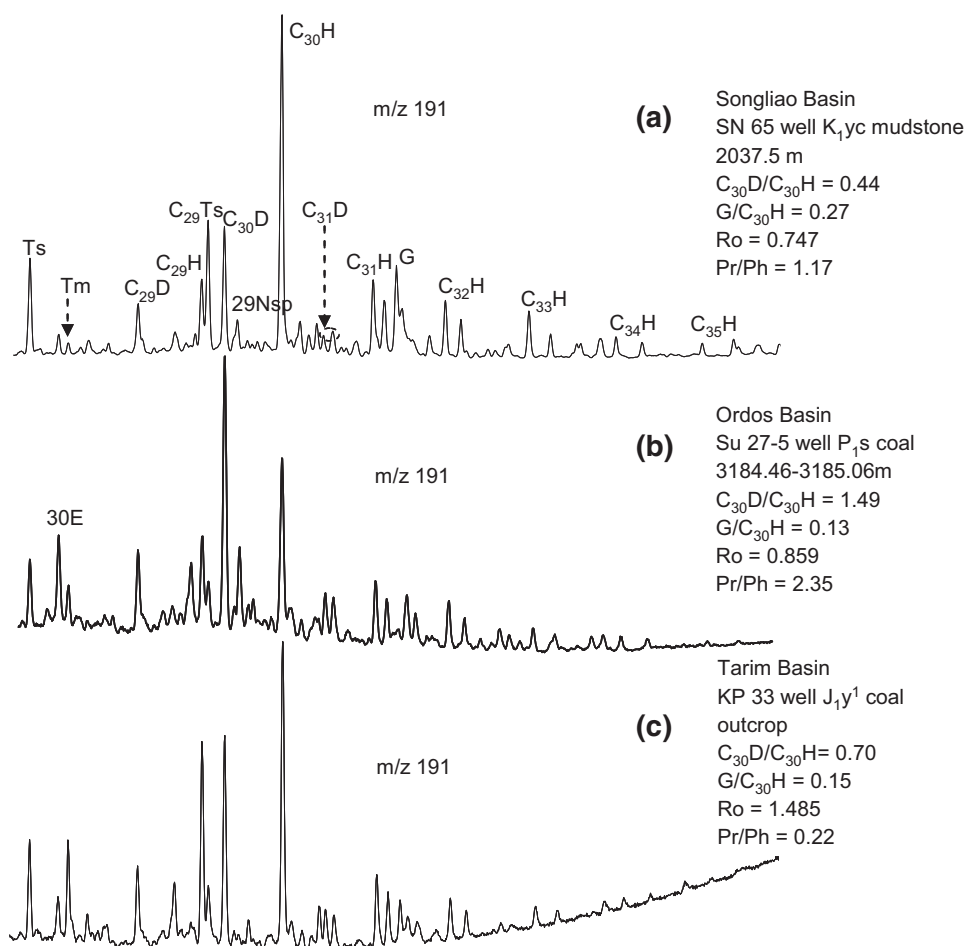
majority held that  $C_{30}$  diahopane may be formed by clay-mediated acidic catalysis under an oxic or suboxic environment (Philip and Gilbert 1986; Moldowan et al. 1991; Peters and Moldowan 1993; Farrimond and Telnæs 1996; Liu et al. 2014), whereas some scholars believed that clay catalysis under moderately alkaline conditions is helpful for the formation of  $C_{30}$  diahopane (Xiao et al. 2004). Zhu

et al. (2007) reported that source rocks and oils derived from marine or saline lacustrine environments, with a source dominated by aquatic organisms, contain low abundances of diahopanes and  $C_{29}$ Ts and almost lack the early-eluting rearranged hopanes. However, these compounds are rich in lacustrine sediments with a terrestrial higher plants input. Although the precursors of rearranged hopanes are not directly derived from terrestrial higher plants, a genetic connection between them seems apparent. Zhang et al. (2009) suggested that high to extremely high  $C_{30}$  diahopane values are indicative of sub-oxidizing environment of fresh-brackish water and shallow to semi-deep lake. Previous studies indicate that the Songliao Basin develops a brackish-saline lacustrine environment with a mixed input, whereas coal-measure source rocks develop well in sedimentary environments of the Tarim and Ordos Basins (Zhang and Zhu 1996; Zhang et al. 2013; Cheng et al. 2014). Overall, the conditions of the three basins contributed to the formation of rearranged hopanes.

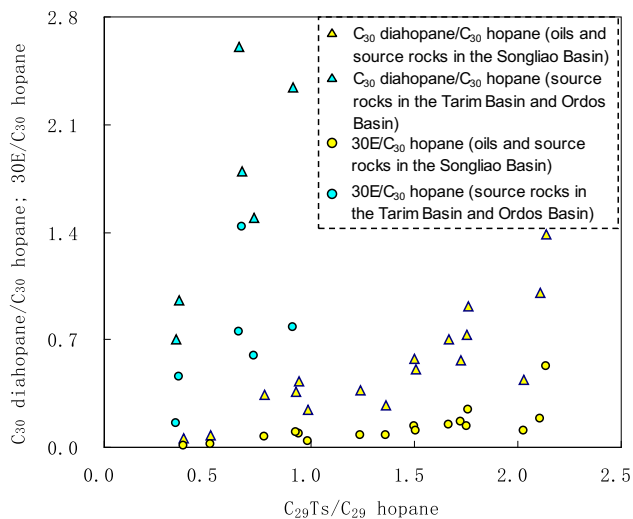
As shown in Fig. 5, both mudstones in the Songliao Basin and hydrocarbon source rocks in the Tarim and

Ordos Basins are found to contain variable contents of rearranged hopanes. Huang et al. (2003) and Nytoft et al. (2006) noted that 29Nsp is abundant in marine oils and lacustrine oils. We also found fairly abundant 29Nsp in coals of the Ordos Basin (Fig. 5b). The complexity of geological conditions and diverse study objects result in the differences in the influencing factors of rearranged hopanes; thus the concentrations of the other three series of rearranged hopanes differ across the three basins. Hydrocarbon source rocks in the Tarim and Ordos Basins commonly contain high abundances of  $C_{30}$  diahopane and 30E and low concentrations of  $C_{29}$ Ts. In contrast, the oils in the Songliao Basin are enriched in  $C_{29}$ Ts, and the amounts of  $C_{30}$  diahopane and 30E are relatively low (Fig. 6). In comparison with a brackish-saline lacustrine environment, the coal-measure and swamp environments under an oxic condition are more helpful to the formation of 30E. It is supported by Zhu et al. (2007) and Cheng et al. (2014), who found high contents of 30E in crude oils in the Jurassic reservoirs of central Sichuan Basin and the Lower Permian coal-measure source rocks in northeastern Ordos Basin,

**Fig. 5**  $m/z$  191 mass chromatograms of saturated fractions in representative source rocks from (a) SN 65 well in the Songliao Basin and (b) Su 27-5 well in the Ordos Basin and (c) KP 33 well in the Tarim Basin. *H* hopane; *D* diahopane; *G* gammacerane; *E* early-eluting rearranged hopane; *Nsp* 28-nor-spergulane; *Ro* vitrinite reflectance







**Fig. 6** The cross-plots of  $C_{30}$  diahopane/ $C_{30}$  hopane and  $30E/C_{30}$  hopane versus  $C_{29}Ts/C_{29}$  hopane

respectively. Moreover, abundant 30E has not been reported in a lacustrine environment. The relationship of  $C_{30}$  diahopane/ $C_{30}$  hopane and  $30E/C_{30}$  hopane with  $C_{29}Ts/C_{29}$  hopane are also noted. It is clear from Fig. 6 that  $C_{30}$  diahopane/ $C_{30}$  hopane and  $30E/C_{30}$  hopane ratios of oils in the Songliao Basin increase markedly slower than those of hydrocarbon source rocks in the Tarim and Ordos Basins with the increase of  $C_{29}Ts/C_{29}$  hopane ratios.

As mentioned above, different depositional environments exhibit diverse distributions of rearranged hopanes. It should be noted that rearranged hopanes in many oils and hydrocarbon source rocks from other Chinese sedimentary basins are much less abundant, even though their depositional setting, organic type and maturity level are comparable to those described above. This phenomenon is presumably related to a peculiar environment where a certain biological group highly enriched in precursors of these compounds thrived, or trace elements that cause a marked catalytic effect on the formation of the rearranged hopanes (Zhu et al. 2007). Although the precursors of these compounds and genetic mechanisms remain to be determined, the occurrence and distribution of these series of rearranged hopanes indicate that they may serve as good indicators of organic facies, depositional environment and maturity. The wide applications of geochemical parameters of rearranged hopanes in petroleum geology also confirm this (Huang et al. 2003; Li et al. 2009; Zhang 2013).

#### 4 Conclusions

Twenty-three oil samples and hydrocarbon source rocks from the Songliao, Tarim, and Ordos Basins have been analyzed by GC–MS and GC–MS–MS. Based on retention

time, mass spectral characteristics and comparison with other studies, four series of rearranged hopanes are systematically identified in oils and hydrocarbon source rocks in the Songliao, Tarim, and Ordos Basins: the  $17\alpha(H)$ -diahopane series ( $C_{27}$ ,  $C_{29}$ – $C_{35}$ ), the  $18\alpha(H)$ -neohopane series ( $C_{27}$ ,  $C_{29}$ – $C_{35}$ ), the early-eluting rearranged hopane series (Ts and  $C_{29}Ts$ ), and the 21-methyl-28-nor-hopane series ( $29Nsp$  and  $30Nsp$ ). These four series of rearranged hopanes develop well in both brackish-saline lacustrine environments and coal-measure environments. However, the coal-measure and swamp environments under an oxic condition are presumably more helpful to the formation of 30E. Even though their genetic mechanisms and influencing factors are controversial, the wide applications of geochemical parameters of rearranged hopanes in petroleum geology indicate that they could serve as good indicators of organic facies, depositional environment, and maturity.

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