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Geochemical Study of Oils and Oil Source Rock from the Eastern Drava and Slavonija-Srijem Depressions, Pannonian Basin, Croatia

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Ključne riječi: biomarkeri, geokemijski parametri, nafta, naftnomatična stijena, organska geokemija.

Abstract

A Middle Miocene oil source rock has been identified by geochemical logging of exploration wells in the Eastern Drava depression (EDD) and in the Slavonija-Srijem depression (SSD) at the south-east margin of the Pannonian Basin, Croatia. The source rock contains Type II to II/III kerogen, and reaches early maturity stage at depths of about 2400 m. The Mesozoic sequences were found either to be absent or to be poor in organic matter within the analysed wells.

According to C₂₇-C₂₈-C₂₉ sterane and C₂₇-C₂₈-C₂₉ monoaromatic steroid distributions, gammacerane indices, C₃₅ homohopane indices and presence of C₃₀ steranes, the EDD and SSD oils were generated from the same marine source rock. The distribution of homohopanes and high Ni/(Ni + V) porphyrin ratios indicates that the related source rock was deposited under suboxic conditions. The oils appear to have been generated from the source rock during the early to middle stages of maturity. The oils are waxy, with pour points up to 30°C and sulphur contents not exceeding 0.7%.

Sažetak

Temeljem geokemijskih podataka za istražne bušotine identificirana je srednjomiocenska naftnomatična stijena u Istočnodravskoj i Slavonsko-srijemskoj potolini, na jugoistočnom rubu Panonskog bazena, Hrvatska. Matična stijena sadrži kerogen tipa II. do II./III. Stadij rane zrelosti matična stijena doseže otprilike na dubini zaliježanja od 2.400 m. U ispitivanim bušotinama za mezozojske je slojeve utvrđeno da su siromašni organskom tvari ili da slojevi nedostaju.

Prema raspodjeli C₂₇-C₂₈-C₂₉ sterana i C₂₇-C₂₈-C₂₉ monoaromatskih steroida, te gammaceranskih indeksa, C₃₅ homohopanskih indeksa i prisutnosti C₃₀ sterana, EDD i SSD nafte su otpuštene od iste marinske matične stijene. Raspodjela homohopana i visoki porfirinski omjeri Ni/(Ni + V) ukazuju da je odnosna matična stijena istaložena u suboksičnim uvjetima. Nafte su otpuštene od matične stijene u stadiju njezine rane do srednje zrelosti. To su visokoparafinske nafte sa stišištem do 30°C, a sadržaj sumpora ne prelazi 0.7%.

1. INTRODUCTION

The Eastern Drava (EDD) and Slavonija-Srijem (SSD) depressions share the common geological history of the south Pannonian Basin (ROYDEN & HORVÁTH, 1988). This area has been studied by HERNITZ (1983) and PANDŽIĆ (1986). The Basement, which is mostly composed of a metamorphic complex intruded by Hercynian granites (PAMIĆ, 1986), is overlain by the Palaeozoic phyllites, quartzites, tuffs, extrusives and clastics.

From Permo-Triassic to Late Triassic a subsiding carbonate platform was widespread, generating limestones and dolomites. Pelagic sedimentation in the Jurassic mainly produced muddy limestones.

Cretaceous lithofacies include both shelf and deep sea clastics. In the Tethyan region, related to ocean anoxic events, "Scaglia" facies were generated in the Upper Cretaceous. Compressional tectonics, which had started during the Jurassic, initiated uplift and altered patterns of sedimentation. Therefore remarkable unconformities occur along the Basin margins in the Creta-

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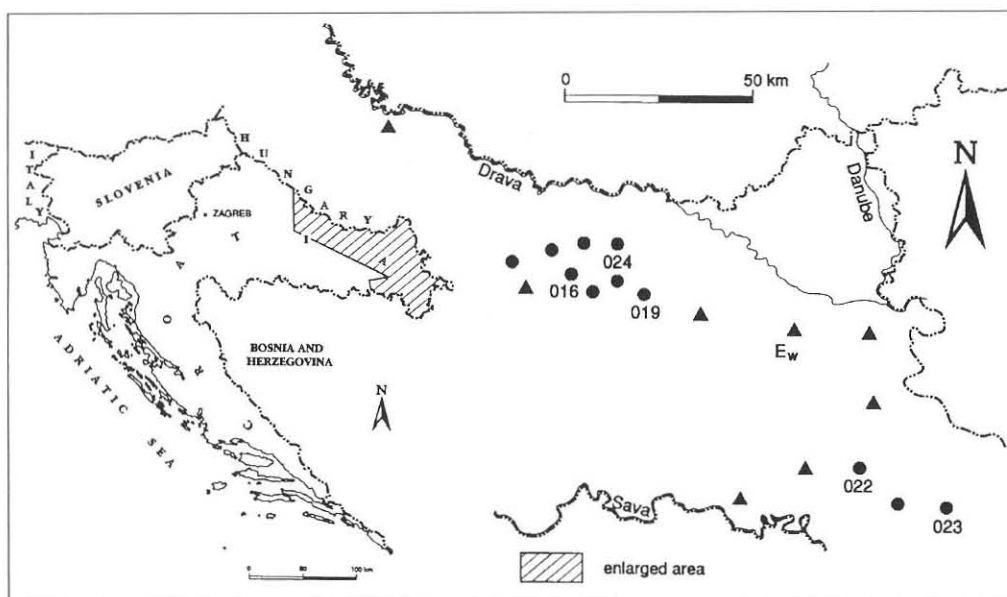


Fig. 1 Sample location map. Legend: ● - oil wells; ▲ - exploration wells (only the wells subjected to further study are additionally labeled).

ceous. Above the unconformities Cretaceous flysch deposits are found which consist of reworked pelagic material, terrigenous clays and turbidites.

Shallow marine carbonate sedimentation continued in the Palaeogene only in a small area and was followed by a period of extensive erosion of the Mesozoic strata.

During the Neogene, when compressional tectonics turned to an extensional regime, rifting and subsidence

took place, associated with taphrogenic folding and volcanism. During the Mid-Late Miocene, rapid sedimentation accumulated up to 2 km thick sequences in the depocenters. Frequent vertical and lateral facies changes are characteristic of these deposits, mainly due to differences in the geological structures of older surrounding strata which supplied material to the Miocene depressions. The rapid sedimentation lasted until the

SAMPLE	O2	O16	O19	O7	O24	O8	O2	O1	O3	O22	O23
PHYSICAL PROPERTIES											
gravity, kg/l	0.874	0.871	0.860	0.843	0.877	0.859	0.899	0.951	0.882	0.897	0.870
API, at 15°C	29	31	35	35	31	33	27	35	28	26	30
pour point, °C	27	26	30	24	27	30	30	24	21	-9	19
ISOTOPE DEPLETION											
$\delta^{13}\text{C}$, ppt	-25.6	-25.5	-26.0	-26.4	-25.8	-26.5	-25.9	-26.1	-24.6	-24.8	-25.3
δD , ppt	-158	-160	-148	-138	-141	-152	-143	-141	-154	-154	-152
ELEMENTAL COMPOSITION											
H/C	1.8	1.7	1.8	1.9	1.9	1.9	1.8	1.8	1.7	1.6	1.8
S, %	0.7	0.7	0.4	0.2	0.3	0.5	0.6	0.2	0.3	0.4	0.4
Ni/(Ni+V)	0.9	0.8	*	*	1.0	0.9	1.0	0.9	1.0	1.0	1.0
GROUP COMPOSITION											
SAT, %	68.0	67.9	72.4	70.7	67.7	65.3	0.53	0.74	60.7	41.8	59.5
ARO, %	16.0	17.1	15.0	19.6	24.6	22.2	28.1	17.0	23.7	35.9	24.5
NSO, %	12.5	11.0	8.6	7.5	6.3	10.0	14.5	5.1	13.8	19.3	14.0
ASPH, %	3.5	4.0	4.0	2.2	1.4	2.5	4.4	3.9	1.8	3.0	2.0
GC PARAMETERS											
CPI	0.94	0.96	0.98	0.97	0.98	0.99	0.99	0.99	1.00	**	0.97
Pr/Phyt	0.89	0.78	0.87	0.99	0.98	0.82	1.00	1.05	1.14	0.92	0.96
Pr/nC ₁₇	0.87	0.68	0.67	0.54	0.66	0.64	0.95	0.63	1.40	5.68	0.77

Table 1 Bulk properties of the EDD and SSD oils. Legend: * = no porphyrins; ** = sample biodegraded.

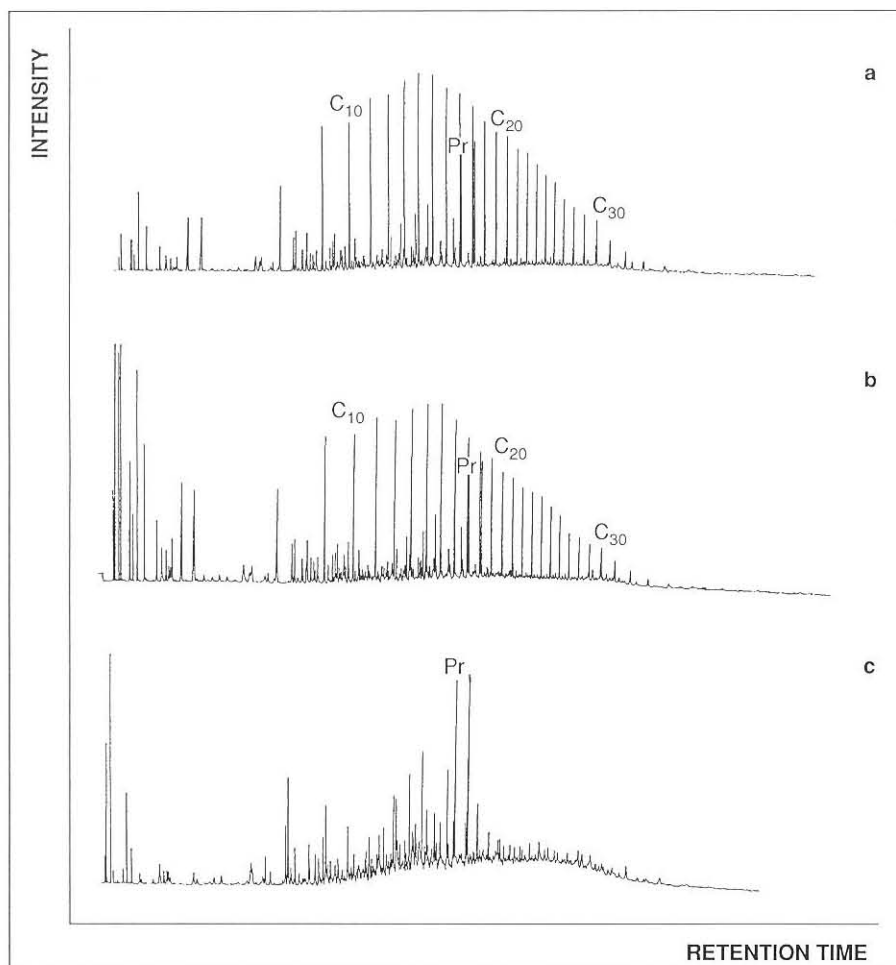


Fig. 2 Examples of the whole oil gas chromatograms a) O19: unimodal n-alkane distribution - the range of the intensive peaks belongs to n-alkanes; b) O23: bimodal/trimodal n-alkane distribution - the range of the intensive peaks belongs to n-alkanes; c) O22: biodegraded oil sample - intensive peaks belong to i-alkanes.

Pliocene when deposits from coarse- to fine-grained clastics and pelites were deposited.

Along the EDD and SSD the recent temperature gradients are comparatively high (4-6°C/100m) probably as a consequence of a relatively thin crust and the rifting characteristics of the Pannonian Basin which is well known for high heat flow (e.g., DÖVENYI & HORVÁTH, 1988).

Commercial oils are found in both the EDD and SSD. They are trapped in porous and/or fractured reservoirs, mainly in the Miocene Moslavačka Gora formation and in the Mesozoic strata, as well as in fractured basement. The related source rock has been studied recently (ALAJBEG et al., 1990; BARIĆ et al., 1992; VULAMA, 1994; HERNITZ et al., 1995; BARIĆ et al., 1995). This paper contributes detailed geochemical data as further evidence of this source rock.

2. EXPERIMENTAL DATA

Oils O5, O16, O19, O7, O24, O8, O2 and O1 from the EDD as well as oils O3, O22 and O23 from the SSD (Fig. 1) were studied for their physical properties: gravity by ASTM D 1289 and pour point by ASTM D 97; elemental analysis: C, H, S, V and Ni; GC data:

whole oil gas chromatogram, carbon preference index (CPI) (BRAY & EVANS, 1961), pristane-to-phytane ratio (Pr/Phyt) and pristane-to-normal heptadecane ratio (Pr/nC₁₇); group composition (Table 1): saturates (SAT), aromatics (ARO), polars (POL) and asphaltenes (ASPH) (PETERS & MOLDOWAN, 1993, p. 54), as well as carbon and hydrogen isotope depletion (SCHOELL, 1984).

The oils O16, O19 and O24 from the EDD and the O22 and O23 oils from the SSD, which were found to express some differences in bulk properties, were selected for biomarker study; triterpanes, steranes, and monoaromatic and triaromatic steroids (SEIFERT & MOLDOWAN, 1986; PETERS & MOLDOWAN, 1993, p. 151-202 and p. 227-250). Cuttings from eight exploration wells (Fig. 1) were examined for source rock identification. This included total organic carbon (Fig. 8), free hydrocarbons yield (S₁), kerogen pyrolyzate yield (S₂), hydrogen index (HI=100 S₂/TOC) and the temperature of the S₂-peak maximum (Tmax). The organic matter in Miocene strata from the exploration well E_w (Fig. 1) was studied microscopically for vitrinite reflectance determination (R_o). Pyrolysis in an off-line tube reactor (ALAJBEG & ŠTIPAK, 1985) and gas chromatography of oil asphaltene pyrolysates was also performed.

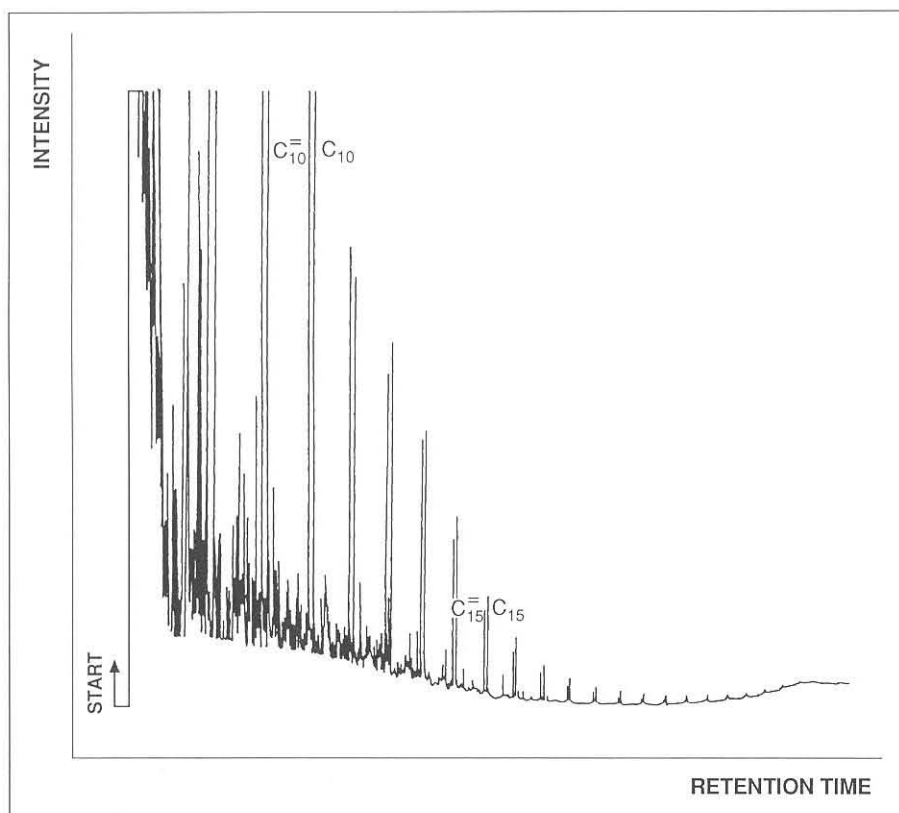


Fig. 3 GC chromatogram of asphaltene pyrolysis products for O19; the intensive peak doublets belong to 1-alkenes and n-alkanes, e.g. $C_{10}^=$ is related to 1-decene and C_{10} to n-decane.

3. RESULTS AND DISCUSSION

The study of the genetic relationships among the oils relied on their bulk properties and biomarkers.

The EDD and SSD oils are found to be rather waxy as reflected in pour points of up to 30°C (Table 1). The exception is the pour point -9°C of O22 which was discovered in a relatively shallow trap (depth <1000 m, trap temperature 65°C) where it underwent biodegradation. For this oil the biodegradation is confirmed by the high predominance of pristane over n-heptadecane ($Pr/nC_{17}=5.68$), a comparatively low H/C (1.6) and saturates yield (41.8%) as well as in the low intensity of normal alkanes as shown in whole oil gas chromatogram (Fig. 2c). The other oils show an intensive n-alkanes range, sometimes exceeding n- $C_{35}H_{72}$, arranged in a (roughly) unimodal distribution (e.g. O19, in Fig. 2a, by biomarkers found to be comparatively the most mature one) and in a non-unimodal (bimodal/trimodal) distribution (e.g. O23, in Fig. 2b, found to be of the comparatively lowest maturity level). Almost all the oils show a slight predominance of even n-alkanes (CPI slightly less than one - Table 1).

The aliphatic structure of the studied oils is also reflected in the products of asphaltene pyrolysis (e.g. O19), which are dominated by the series of doublets of n-alkanes and the related 1-alkenes (Fig. 3) as well as a H/C atomic ratio up to 1.9 (Table 1).

The waxy character and the type of n-alkane distribution, may indicate that organic matter of algal (or reworked algal) origin served as the predominating oil precursor, which was possibly deposited in carbonate-

rich sediments (PETERS & MOLDOWAN, 1993). In those oils where porphyrins are detected, Ni porphyrins are found to predominate strongly over V=0 porphyrins, suggesting a suboxic to oxic depositional environment of the source rock which released the studied oils (e.g., MOLDOWAN et al., 1986). This is also reflected by the low to moderate sulphur content (0.2% to 0.7%) in oils.

The SSD oils, in comparison to the EDD oils, are found to be isotopically heavier (Table 1) and lower in pour points and H/C ratios.

3.1. BIOMARKER RESULTS - - OIL/OIL CORRELATION

For biomarker analysis, 3 EDD samples were chosen: O19 as the example which reaches the high pour point (30°C), and high API gravity (33°) and has no measurable porphyrins; O24 as the example which contains no V=0 porphyrins [$Ni/(Ni+V)=1$] and reaches high H/C ratio (1.9); O16 which is characterized by the lowest H/C ratio (1.7) among the studied EDD oils and comparatively high sulphur (0.7%). Furthermore 2 SSD oils were taken: the biodegraded O22, and O23 which was found to be similar in physical properties to O3.

The biomarker source parameters for oil-to-oil correlation of the five select oils from EDD and SSD indicate close correlation among the oils. The distributions of C_{27} - C_{28} - C_{29} steranes were measured using metastable reaction monitoring (MRM) GC MS. The steranes measured include the sum of isomers $\alpha\alpha\alpha 20S$, $\alpha\alpha\alpha 20R$, $\alpha\beta\beta 20S$, $\alpha\beta\beta 20R$, and the measured distributions clus-

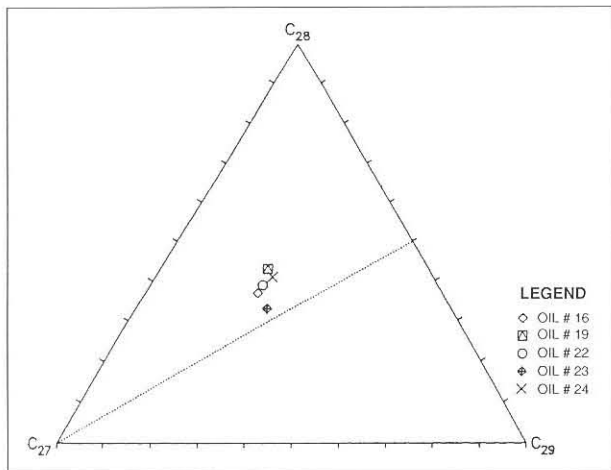


Fig. 4 Sterane distribution (m/z 217) for select EDD and SSD oils; C_{27} = cholestanes; C_{28} = ergostanes; C_{29} = stigmastanes.

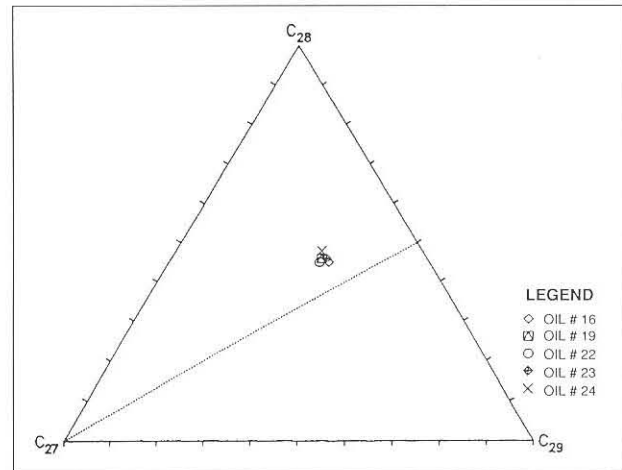


Fig. 5 MAS distribution (m/z 253) for select EDD and SSD oils; C_{27} = cholestane type MA steroids; C_{28} = ergostane type MA steroids; C_{29} = stigmastane type MA steroids.

ter in a group indicating the similarity of oil for sterol precursors (Fig. 4, Table 2). Sterane distributions reflect the algal (eukaryotic) input to the source (MACKENZIE, 1984).

The distributions of C_{27} - C_{28} - C_{29} C-ring monoaromatic steroids (MAS), including the sum of isomers $5\alpha 20S$ $5\alpha 20R$ $5\beta 20S$ $5\beta 20R$ dia-20R, dia-20S, cluster in a very tight group independently confirming the sterane results (Fig. 5). C-ring monoaromatic steroids are thought to be derived from sterols with a higher degree of unsaturation than is necessary for the formation of steranes with, perhaps, a double bond in the side-chain (e.g. MOLDOWAN & FAGO, 1986; RIOLO et al., 1986), reflecting precursors other than the steranes. The

steranes and MAS together indicate that the three EDD oils and two SSD oils examined were very probably generated from the same source.

Homohopane concentrations in the different oils follow a similar pattern (Fig. 6), decreasing with increasing C-number. This observation together with a significant preference for the C_{32} homolog, suggest a mildly suboxic environment of deposition for the source rock (MOLDOWAN et al., 1992). 24-n-Propylcholestane was detected by (MRM) GC MS (Fig. 7) indicating marine algal input typically associated with a marine depositional environment (MOLDOWAN et al., 1990). Low gammacerane indices [$100 \times \text{gammacerane}/17\alpha$, $21\beta(\text{H})$ -hopane] suggest that the source rocks were

SOURCE PARAMETERS								
SAMPLE	STERANES			MA STEROIDS			A	B
	a	b	c	a	b	c		
O16	0.39	0.37	0.24	0.21	0.45	0.34	0.72	8.7
O24	0.35	0.41	0.29	0.21	0.48	0.31	0.74	6.7
O19	0.34	0.43	0.23	0.23	0.46	0.32	0.93	8.1
O22	0.37	0.39	0.24	0.23	0.45	0.32	1.08	7.4
OO23	0.39	0.33	0.28	0.21	0.46	0.33	0.95	10.2
MATURITY PARAMETERS								
SAMPLE	Ts/(Ts+Tm)	C_{32} .HOMOHOPANE	STIGMASTANE 22S/(22S+22R)	$TAS_{28}/(TAS_{28} + MAS_{29})$ $20S/(20S+20R)$				
O16	0.35	0.58	0.46	0.88				
O24	0.38	0.56	0.45	0.82				
O19	0.41	0.57	0.51	0.94				
O22	0.42	0.56	0.43	0.89				
O23	0.33	0.54	0.29	0.85				

Table 2 Biomarker parameters for the EDD and SSD oils. Legend: A = gammacerane index; B = C_{35} homohopane index; a = $C_{27}/\Sigma C_{27}-C_{29}$; b = $C_{28}/\Sigma C_{27}-C_{29}$; c = $C_{29}/\Sigma C_{27}-C_{29}$.

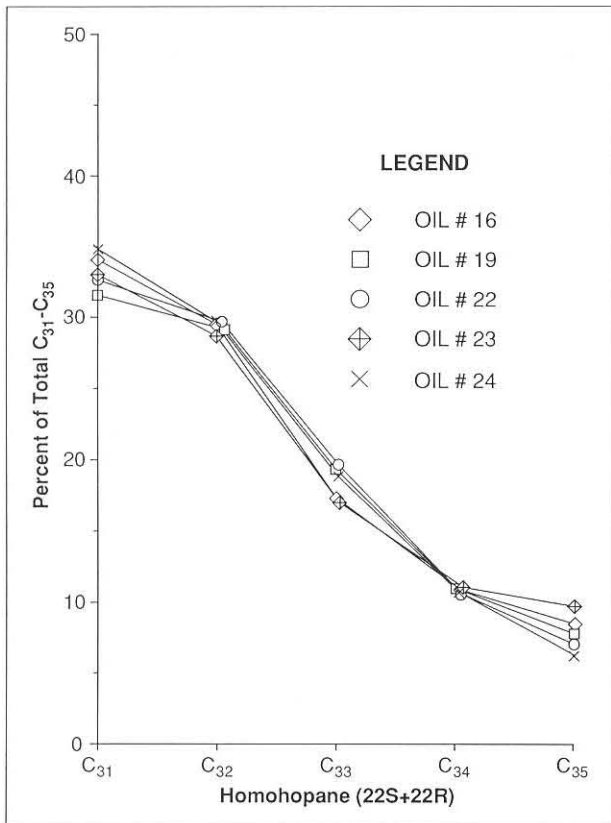


Fig. 6 Homohopane distribution for select EDD and SSD oils.

deposited at normal or reduced, rather than elevated marine salinities.

Predominating Ni porphyrins also show that oxygen was available during early diagenesis (LEWAN, 1984; MOLDOVAN et al., 1986; BARWISE, 1990). Oleanane as the possible indicator of terrestrial plant input to the source (after angiosperms appearance in Cretaceous) was not found in significant concentrations

in any of these oils. Low rearranged to normal sterane ratios $[13\beta, 17\alpha(H), 20S + 20R] / [14\alpha, 17\alpha(H), 20S + 20R + 14\beta, 17\beta(H), 20S + 20R]$, point to a carbonate mineral matrix for the source rock (MOLDOVAN & FAGO, 1986; MELLO et al., 1988; PETERS & MOLDOVAN, 1993).

Biomarker maturity parameters (Table 2), which reflect thermal transformations of biomarkers, including $T_s / (T_s + T_m)$, the isomer ratios of $22R / (22R + 22S)$ and $17\beta, 21\alpha(H) / 17\alpha, 21\beta(H)$ configurations in hopanes (i.e., moretanes/hopanes) and $14\alpha, 17\alpha(H) / [14\alpha, 17\alpha(H) + 14\beta, 17\beta(H)]$ and the $20R / (20R + 20S)$ configuration in C_{29} -steranes as well as steroid aromatization were used. The $22S / (22S + 22R)$ homohopane ratios between 0.55 and 0.60 suggest full epimerization and that the oils are more mature than the initial generation stage (SEIFERT & MOLDOVAN, 1986). However, the sterane isomerization parameters are less than fully equilibrated suggesting that generation occurred before the peak of the oil window (PETERS & MOLDOVAN, 1993). Regarding the biomarker parameters the maturity level of the oils studied for biomarkers can be ranked in the following order: $O23 < O22, O16$ and $O24 < O19$, where O23 was generated in the late early oil window and O19 was generated near peak. High temperature gradients improved the maturity rank of the EDD and SSD oils.

3.2. SOURCE ROCK STUDY

In order to identify the oil source rock, cuttings from eight wells (Fig. 1) were studied. The organic matter from the exploration well E_w underwent additional study as the representative sample. In E_w the Miocene strata between 2,400 m and 2,680 m are found to be a source rock, with average TOC values ranging from 0.5% to 1%. Hydrogen indices range up to 475 in

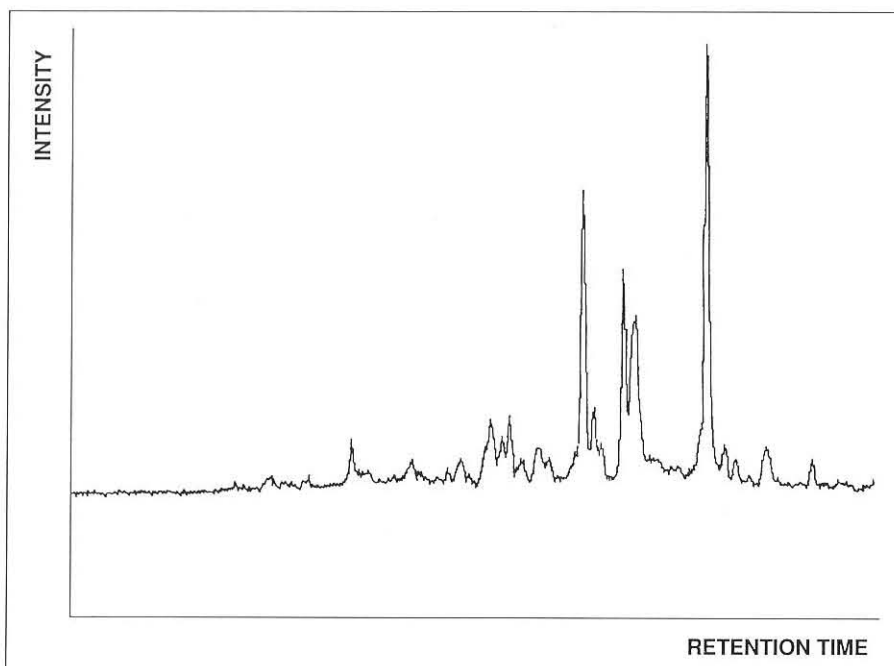


Fig. 7 Mass fragmentogram m/z 414 \rightarrow 217 from metastable GC MS analysis for an EDD oil showing a distribution of 24-n-propylcholestanes.

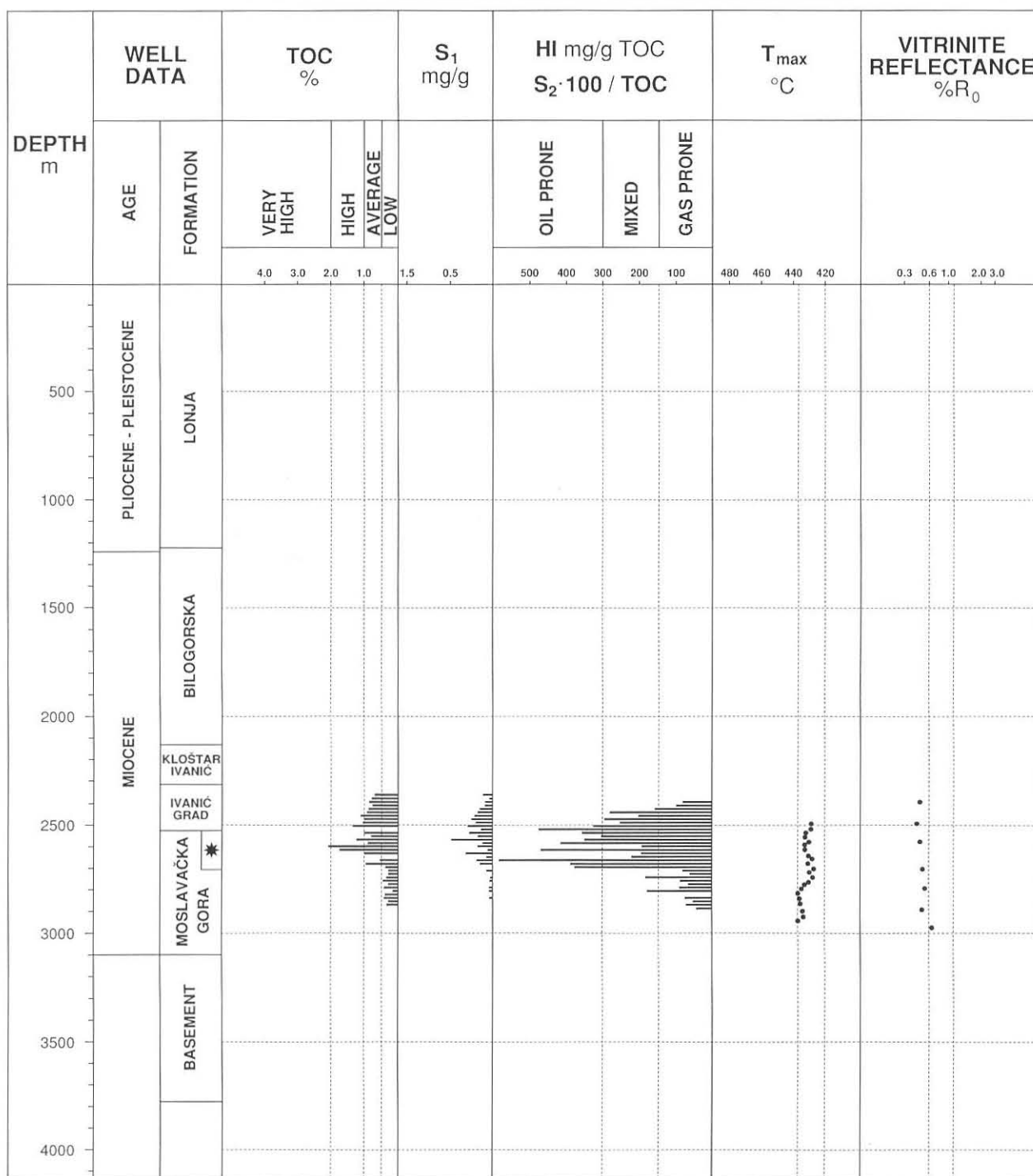


Fig. 8 Geochemical log of the exploration well E_w, Star - the Križevci Formation.

the deeper part of the source rock (Fig. 8; Križevci član in Moslavačka Gora formation) and reflect the predominating oil-prone character of the kerogen (Type II). In overlying strata, organic matter turned to mixed oil/gas prone (Type II/III) and then to gas prone (Type III) in the Ivanić Grad formation.

Regarding the fact that the sediments of this interval are predominantly represented by clastics, the change might reflect the upward increasing input of non-oxidized terrestrial macerals. However, some clasts of these clastic sediments were reworked from older formations and they may contain various types of organic

matter of different diagenetic and thermal history. The non-uniformity of the organic facies was reflected in three recognizable vitrinite populations: autochthonous vitrinites, the values of which increase with depth (Table 3) in accordance with the geothermal gradient; vitrinites with reflectance values indicating they might have been earlier diagenetically (primarily) partly oxidized or secondarily reworked; and vitrinites with reflectance values which most probably represent grains from older, reworked formations.

The Miocene source rock is also found in other exploration wells. Mesozoic strata are irregularly dis-

DEPTH (m)	POPULATIONS		
	1st	2nd	3rd
2380	0.522	0.965	2.021
2480	0.509	0.955	1.868
2580	0.538	1.039	2.119
2680	0.557	1.018	1.909
2780	0.572	1.048	2.016
2880	0.566	1.057	2.025
2970	0.609	1.075	2.012

Table 3 Vitrinite reflectances for organic matter in Miocene strata in the well E_w.

tributed in the EDD and in SSD, and they are poor in organic matter.

4. CONCLUSION

Geochemical evidence suggests that EDD and SSD oils have been generated from the same source rock, deposited in a carbonate mineral matrix under marine suboxic conditions. The oils were generated at the early to middle maturity stage of the source. Differences in oil properties should be ascribed to different migration and trapping histories or different maturity levels at various locations.

A Miocene source rock is identified containing autochthonous and allochthonous organic matter, predominantly Type II/III. Mesozoic strata are found to be absent and with no recent oil generating potential. Thus, the Miocene appears to be the only consistent source throughout the EDD and SSD that can account for the commercial oil in this basin. Because of the modest prolificity of this source, a rather efficient trapping system is postulated, including short migration distances and good cap rocks, in order to account for the known oil accumulations. These conditions are consistent with the geology of the EDD and SSD.

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