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SUMMARY/ SAMMENDRAG

A condensate, an oil and an oil from a mud sample were analysed for correlation purposes. GC, GC-MS and δ^{13} C isotope analyses suggest that the three samples may have been generated from the same or similar types of source rocks. The condensate may have been generated from a more mature stage of the source rock.

KEY WORDS/STIKKORD 0il/0il Correlation	Haltenbanken
GC-MS	δ^{13} C isotopes

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1. JNTRODUCTION

Three samples from well 6407/1-3 (figure 1) were analysed for correlation purposes. Of the samples one was a condensate (B-2318), one an oil (B-2317) and the third one an oil from a mud sample (B-922).

The samples were submitted to gas chromatography (GC) and combined gas chromatography - mass spectrometry (GC-MS) in addition to $\delta^{13}C$ isotope analysis.

1.1 <u>Molecular ratios from terpane and sterane mass chromatograms</u> applied as maturity and source characteristic parameters.

Geochemical fossils or biological marker components are characteristic of the type of organic matter present at the time the sediments were deposited. The biological isomers of these components undergo changes due to increased maturity in particular, but also to a certain degree caused by migration and weathering processes.

1.1.1 Source characteristics parameters

In the m/z 191 mass chromatograms, representing terpanes, the hopanes and moretanes are the major components in most extracts and oils. Of the hopanes the C_{27} and C_{29} - C_{35} homologs are ubiquitous, while the C_{28} bisnorhopane is believed to be typical of certain types of source rocks. The amounts of tricyclic terpanes relative to the hopanes are also believed to a certain extent to be characteristic of the source rock. This is also the case for the component, probably gammacerane, sometimes seen to coelute with the 22S isomer of the C_{31} $17\alpha(H)$ -hopanes (H). In the sterane mass chromatograms, m/e 217 and m/e 218, the molecular weight distribution of the C_{27} - C_{29} regular steranes is believed to be representative of the original input of organic matter. The highest molecular weight compounds, the C_{29} steranes, represent organic matter of terrestrial origin, while the lower molecular weight analogs originate from more marine type environments.

1.1.2 Maturity parameters

The biological isomers of the hopanes, the $17\beta(H)$, $21\beta(H)$ -hopanes, undergo structural changes during the maturation process. The isomerisation

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reactions are thought to produced via the $17\beta(H)$, $21\alpha(H)$ -hopanes (moretanes) to the most stable $17\alpha(H)$, $21\beta(H)$ -hopanes. At equilibrium 100% of the $17\alpha(H)$ -hopanes are seen. The ratio $\alpha\beta/\alpha\beta+\beta\alpha$ is used to describe this reaction. In the extended hopanes ($\geq C_{31}$), the thermally stable S configurations at C-22 become increasingly more abundant as compared to the biologically preferred R configurations at increased maturity level. The equilibrium ratio is approximately 60% of the 22S configuration. Another ratio that is known to change with maturity is the Tm/Ts (Seifert et al., 1978) of the C₂₇ hopanes. The maturable $18\alpha(H)$ -trisnorneohopane (Tm) is reduced in intensity relative to the more stable $17\alpha(H)$ trisnorhopane (Ts), causing the Tm/Ts to decrease at increased maturity. This ratio is also believed to be source dependant, and this should be borne in mind when applying the ratio for maturity comparison. The amount of tricyclic terpanes is also to a certain extent seen to be maturity dependant.

Two isomerisation reactions taking place in the steranes are most commonly applied for maturity assignments from the m/z 217 mass chromatograms. The biologically preferred $14\alpha(H)$, $17\alpha(H)$ -isomers of the regular steranes is transformed to the thermally stable $14\alpha(H)$, $17\beta(H)$ -steranes, the %ßß approaching 75% at equilibrium. An equilibrium concentration of 50% is seen of the stable S configuration at C-20 as opposed to the 100% of the biological 20R epimer (Mackenzie et al., 1980). The abundance of rearranged steranes increases with increasing maturity.

One of the reactions taking place at an early stage of diagenesis is the aromatisation of steranes, leading to the formation of mono- and triaromatic analogs. This process is measured as the abundance of triaromatic relative to mono-aromatic compounds (% tri/tri + mono) in the m/z 231 and 253 mass chromatograms, respectively. In addition the degree of side chain cracking, as $%C_{20}/C_{26}$, 27 and $%C_{21}/C_{28,29}$ respectively, is applied. These cracking processes are also taking place during early diagenesis, and are used for maturity assignment together with the previously mentioned ratios.

1.1.3 Migration and weathering

The effect on the geochemical fossils of migration and weathering, is less apparent than the maturity induced changes. Migration is believed to cause an increase in the relative amounts of rearranged and $14\beta(H)$,



 $17\beta(H)$ regular steranes (Seifert and Moldowan, 1978, 1981). Severe biological alteration leads to the formation of desmethyl-hopanes (Seifert and Moldowan, 1979).

2. EXPERIMENTAL

2.1 Chromatographic Separation

The oils were topped under reduced pressure to constant weight prior to the chromatographic separation.

The topped oils were separated into saturated fraction, aromatic fraction and non hydrocarbon fraction using a MPLC system with hexane as eluant (Radke et al., Anal. Chem., 1980). The various fractions were evaporated on a Buchi Rotavapor and transferred to glass vials and dried in stream of nitrogen.

2.2 Gas Chromatographic Analysis

The saturated hydrocarbon fractions were each diluted with n-hexane and analysed on a HP 5730A gas chromatograph, fitted with a 25m OV-101 fused silica capillary column. Hydrogen (0.7ml/min) was used as carrier gas. The total aromatic fractions were after dilution with n-hexane, analysed on a Carlo Erba Fractovap Series 2150 GC fitted with a 20mm SE-54 fused silica column.

Injections on both systems were performed in the split mode (1:20). The temperature program applied was 80° C (2 min) to 260° C at 4° C/min.

In addition the whole oils were analysed on the HP 5730 A GC, applying a temperature program of the GC oven from -50° C (2 min.) to 280° C at 4° C/min.

The data processing for all the GC analyses was performed on a VG Multichrom System.

2.3 <u>Gas chromatography - mass spectrometry (GC-MS)</u>

GC-MS analyses were performed on a VG Micromass 70-70H GC-MS-DS system. The Varian Series 3700 GC was fitted with a fused silica DB-1 capillary column (30m x 0.3mm i.d.). Helium (0.7kg/cm^2) was used as carrier gas and the injections were performed in split mode $(1.5\mu$ l, split ratio 10:1). The GC oven was programmed from 120° C to 280° C at 4° C/min. after an initial isothermal period of 2 minutes for the saturated hydrocarbons,



while 70° C to 280° C at 5° C/min. was applied for the aromatic compounds.

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The saturated hydrocarbons were analysed in multiple ion mode (MJD) at a scan cycle time of approximately 2 secs. Full data collection was applied for the aromatic hydrocarbons at a scan time of 1 sec/decade. The mass spectrometer operated at 70eV electron energy and an ion source temperature of 200° C. Data acquisition was done by a VG data system.

Peak identification was performed applying knowledge of elution patterns in certain mass chromatograms. Calculation of peak ratios was done from peak height in the appropriate mass chromatograms.

3. RESULTS AND DISCUSSION

3.1 APJ gravity

The APJ gravities of the oils/condensate given in Table 1 show that sample B-922 (oil from mud) and B-2317 give similar values, while B-2318 is a much lighter condensate type sample.

3.2 GC of whole oils

All three samples seem to contain approximately the same distribution of hydrocarbons, the only difference being that the condensate (B-2318) contain hardly any n-alkanes above nC_{25} while the oil samples contain more of the high molecular weight n-alkanes. The relative distribution of n-alkanes, isoprenoids, cyclic and aromatic compounds is seen to be similar for all three samples.

3.3 Gross composition

The extraction data in Table 2 show some variations between the samples. These variations are believed mainly to be due to the experimental procedure rather than significant differences in the samples. The condensate (B-2318) seems to contain more non-hydrocarbons than the other two samples. This is probably so because the samples were topped at a too low temperature, i.e. the weights of EOM in the table are too high compared to what is left as saturated and aromatic hydrocarbons after the separation. Since the non-hydrocarbons are determined as the difference between EOM and the sum of hydrocarbons the non-hydrocarbons weights will be too high. The effect of this is most pronounced for the light condensate. The relatively low ratio of saturated to aromatic hydrocarbons and the high ARO/EOM in sample B-2317 is caused by a high weight of the aromatic fractions. Based on the similarities seen from the whole oil GC's it is believed that this high ARO value is due to some contamination of this sample.

3.4 GC-analysis of saturated hydrocarbons

A similar picture as from the whole oil GC's was seen from the saturated hydrocarbon GC's presented in figure 3. The main difference is the low abundance of high molecular weight hydrocarbons in the condensate. No



significant difference is seen in the isoprenoid/n-alkane ratios or the CPI (Table 3).

3.5 GC-analysis of aromatic hydrocarbons

In addition to the lower molecular weight naphthalene homologs showing higher relative abundance in the condensate (figure 4), the methylphenanthrene index (MPI) varies between the oils and the condensate. The higher MPI value (Table 4) for the condensate suggests higher maturity of this sample as compared to the two oils.

3.6 GC-MS analysis of saturated hydrocarbons

The three oil/condensate samples were analysed for their content of steranes and terpanes.

Only minor variations were seen in the three mass chromatograms that represent these compounds, m/z 191, 217 and 218 (Figure 5). From the molecular ratios presented in Table 5 a similar picture is seen. All the maturity ratios represent well mature hydrocarbons, and the finger-prints of the mass chromatograms suggest that the three samples probably are generated from the same or similar types of source rocks. The high relative abundance of tricyclic terpanes in sample B-2318 (DST 2) is probably due to lower absolute amounts of the hopanes (A-M), rather than a characteristic source difference.

3.7 GC-MS analysis of aromatic hydrocarbons

Mass chromatograms representing aromatic hydrocarbons are presented in Figure 6. The total ion chromatograms (TJC) resemble the ordinary gas chromatograms, and they show mainly alkylated naphthalene and phenan-threne homologs. Sample B-2318 (DST 2) seems to contain relatively less of the higher phenanthrene homologs as compared to the naphthalenes. But apart from this it is difficult to draw any firm conclusions from these complex chromatograms. The traces that represent the alkylated benzenes (m/z 92 and 106) show samples B-922 and B-2317 to be very similar, while B-2318 is seen to contain less of the higher molecular weight compounds. The distribution in the lower molecular weight range is, however, similar for all three samples. No significant variations are seen in any of the naphthalene, phenanthrene or dibenzothiophene chromatograms.

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The differences seen in the aromatic sterane traces (m/z 231 and 253) suggest that sample B-2318 is slightly different to the others. The higher relative intensities of the lower molecular weight steranes in this sample, is an effect of the general molecular weight distribution in this sample. Whether this is due to higher maturity when this oil was generated or is a fractionation effect during migration is difficult to say.

3.8 δ^{13} C isotope analysis

Saturated and aromatic hydrocarbon fractions in addition to aliquots of the whole oils were analysed for their carbon isotope distributions. The results given in Table 6 suggest that the three samples are very similar, and probably originate from the same or similar types of source rock.



From the analyses of these three oil samples we conclude that two of the samples are nearly identical, while the third sample (B-2318) shows a higher relative abundance of the lower molecular weight range compounds. The overall distributions in all three samples suggest they are all very similar, and originate from similar source rocks. The condensate seems to be of higher maturity than the two oils, and may thus have been generated from a more mature stage of the same rock as the one that have sourced the oils.



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JKU no.	Sample type	APJ gravity
B-922	oil from mud	31.0 ⁰
B-2317 B-2318	oil condensate	29.9 ⁰ 47.8 ⁰

Table 1. APJ gravities for oils/condensate.

TABLE : 2a.

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CONCENTRATION OF EOM AND CHROMATOGRAPHIC FRACTIONS

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COMPOSITION IN % OF MATERIAL EXTRACTED FROM THE ROCK

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TABLE 3.

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TABULATION OF DATA FROM THE BASCHROMATOGRAMS

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JKU no.	Sample type	MPI*
B-922	Oil from mud	0.65
B-2317	011	0.61
B-2318	Condensate	0.94

Table 4. Methyl phenanthrene indices (MPJ) for oils/condensate.

* MPJ = $\frac{1.5 (2-MP + 3MP)}{P + 1-MP + 9-MP}$

Table 5. Molecular ratios calculated from peak heights in terpane (m/z 191) and sterane (m/z 217) mass chromatograms.

	m/z 191			m/z 217			
JKU no.	Q/E ¹⁾	Tm/Ts ²⁾	αβ/αβ+βα ³)	%22S ⁴)	_{%ββ} 5)	%20S ⁶)	a/a+j ⁷⁾
B-922	0.17	0.6	0.96	62	80	63	0.88
B-2317	0.17	0.5	0.94	60	78	60	0.85
B-2318	0.28	0.5	0.94	62	78	58	0.88

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1) Relative abundance of tricyclic terpanes (Q/E in m/z 191).

2) B/A in m/z 191.

3) E/E+F in m/z 191.

4) % distribution between first and second eluting isomers of doublet J (m/z 191).

5) 2(r+s)/(q+t+2(r+s)) in m/z 217.

6) q/q+t in m/z 217.

7) Relative abundance of $C_{27}^{}$ rearranged steranes (a/a+j in m/z 217).

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JKU no.	Sample type	SAT	ARO	Whole oil
B-922	Oil from mud	-30.4	-29.1	-29.2
B-2317	011	-30.1	-29.1	-29.4
B-2318	Condensate	-30.5	-28.9	-29.2

Table 6. $\delta^{13}\text{C}$ isotope data for oil/condensate.





FIGURE 2

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WHOLE OJLS GC'S

C₅,C₆ etc. - n-alkanes CyC₆ - cyclohexane MeCyC₆ - methylcyclohexane



RAM DATA PLOT-CHANNEL 2 Data Scale Plot Box 1 of 1

Injection f: 1 Maximum signal (2) :5.943 .≩ Analysis :1703B922T Sample f Sample Name :B-922,T0TAL 01L,



Whole oil GC B – 922 Oil from mud



Whole oil GC B – 2317

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Analysis :707B2318T Sample f: 1 Injection f: 1
Sample Name :B-2318,1707,T.0IL,GH Maximum signal (%) :13.191 1 0f





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FJGURE 3

SATURATED HYDROCARBON GC'S

^C 15.20	etc.	-	n-alkanes
Pr		-	pristane
Ph		-	phytane

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FJGURE 4

AROMATIC HYDROCARBON GS'S

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Figure 5a.

Mass chromatograms representing terpanes (m/z 191)

Α	T _s , 18a(H)-trisnorneohopane	С ₂₇ Н ₄₆	(III)
В	T_m , 17 α (H)-trisnorhopane	C ₂₇ H ₄₆	(I,R=H)
С	17a(H)-norhopane	C ₂₉ H ₅₀	(I,R=C ₂ H ₅)
D	17β(H)-normoretane	C ₂₉ H ₅₀	(II,R=C ₂ H ₅)
Ε	17α(H)-hopane	C ₃₀ H ₅₂	$(I, R=C_3H_7)$
F	17β(H)-moretane	C ₃₀ H ₅₂	$(II, R=C_3H_7)$
G	$17\alpha(H)$ -homohopane (22S)	C ₃₁ H ₅₄	$(I,R=C_{4}H_{9})$
Η·	17α(H)-homohopane (22R)	C ₃₁ H ₅₄	$(I,R=C_{4}H_{9})$
	+ unknown triterpane (gammacerane?)		
I	17g(H)-homomoretane	C ₃₁ H ₅₄	(II,R=C ₄ H ₉)
J	$17\alpha(H)$ -bishomohopane (22S,22R)	C ₃₂ H ₅₆	$(I,R=C_5H_{11})$
κ	17α(H)-trishomohopane (22S,22R)	C ₃₃ H ₅₈	$(I, R=C_6H_{13})$
L	$17\alpha(H)$ -tetrakishomohopane (22S,22R)	C ₃₄ H ₆₀	$(I,R,=C_7H_{15})$
М	$17\alpha(H)$ -pentakishomohopane (22S,22R)	C ₃₅ H ₆₂	$(I,R=C_8H_{17})$
Z	bisnorhopane	C ₂₈ H ₄₈	• •
Х	unknown triterpane	C ₃₀ H ₅₂	
Ρ	tricyclic terpane	$C_{23}H_{42}$	(IV,R=C ₄ H ₉)
Q	tricyclic terpane	C ₂₄ H ₄₄	$(IV, R=C_5H_{11})$
R	tricyclic terpane (17R,17S)	C ₂₅ H ₄₆	$(IV, R=C_6H_{13})$
S	tetracyclic terpane	$C_{24}H_{42}$	(V)
T	tricyclic terpane (17R,17S)	C ₂₆ H ₄₈	$(IV, R=C_7H_{15})$











Figure 5b.

Mass	chromatograms	representing	steranes	(m/z	217	and	218)	

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$13\beta(H), 17\alpha(H)$ -diasterane (20S)	$C_{27}H_{48}$	(III,R=H)
13β(H),17α(H)-diasterane (20R)	$C_{27}H_{48}$	(III,R≈H)
13α(H),17β(H)-diasterane (20S)	C ₂₇ H ₄₈	(I∛,R=H)
13α(H),17β(H)-diasterane (20R)	C, 7H ₄₈	(IV,R=H)
13β(H),17α(H)-diasterane (20S)	C ₂₈ H ₅₀	(III,R=CH ₂)
13β(H),17α(H)-diasterane (20R)	C ₂₈ H ₅₀	(III,R=CH ₃)
$13\alpha(H), 17\beta(H)$ -diasterane (20S)	C ₂₈ H ₅₀	(IV,R=CH ₃)
+ 14a(H),17a(H)-sterane (20S)	$C_{27}H_{48}$	(I,R=H)
13β(H),17α(H)-diasterane (20S)	$C_{29}H_{52}$	(III,R=C ₂ H ₅)
+ 14α(H),17α(H)-sterane (20R)	C.,7H ₄₈	(II,R=H)
14β(H),17β(H)-sterane (20S)	$C_{27}H_{48}$	(II,R=H)
+ $13\alpha(H), 17\beta(H)$ -diasterane (20R)	C ₂₈ H ₅₀	(IV,R=CH ₃)
$14\alpha(H), 17\alpha(H)$ -sterane (20R)	$C_{27}H_{48}$	(I,R=H)
$13\beta(H), 17\alpha(H)$ -diasterane (20R)	C ₂₀ H ₅₂	(III,R=C ₂ H ₅)
$13\alpha(H), 17\beta(H)$ -diasterane (20S)	$C_{29}H_{52}$	(III,R=C ₂ H ₅)
$14\alpha(H), 17\alpha(H)$ -sterane (20S)	C ₂₈ H ₅₀	(I,R=CH ₃)
$13\alpha(H), 17\beta(H)$ -diasterane (20R)	C ₂₉ H ₅₂	(III,R=C ₂ H ₅)
+ 14β (Н),17β (Н)-sterane (20R)	$C_{28}^{H_{50}}$	(II,R=CH ₃)
14β(H),17β(H)-sterane (20S)	C ₂₈ H ₅₀	(II,R=CH ₃)
14u(H),17u(H)-sterane (20R)	C ₂₈ H ₅₀	(I,R=CH ₃)
14¤(H),17¤(H)-sterane (20S)	C ₂₉ H ₅₂	(I,R=C ₂ H ₅)
14β(H),17β(H)-sterane (20R)	C ₂₉ H ₅₂	(II,R=C ₂ H ₅)
+ unknown sterane		20
14ß(H),17ß(H)-sterane (20S)	C ₂₉ H ₅₂	(II,R=C ₂ H ₅)
14α(H),17β(H)-sterane (20R)	C29H52	(I,R=C ₂ H ₅)
5α(H)-sterane	C ₂₁ H ₃₆	(V,R=C,H ₅)
5a(H)-sterane	C22H38	(IV,R=C ₃ H ₇)
	13β(H),17α(H)-diasterane (20S) 13β(H),17α(H)-diasterane (20R) 13α(H),17β(H)-diasterane (20S) 13α(H),17α(H)-diasterane (20S) 13β(H),17α(H)-diasterane (20S) 13β(H),17α(H)-diasterane (20S) 13α(H),17β(H)-diasterane (20S) + 14α(H),17α(H)-sterane (20S) + 14α(H),17α(H)-sterane (20S) + 14α(H),17β(H)-sterane (20S) + 13α(H),17β(H)-diasterane (20R) 14β(H),17β(H)-diasterane (20R) 13β(H),17α(H)-diasterane (20R) 13β(H),17α(H)-diasterane (20R) 13β(H),17β(H)-diasterane (20S) 14α(H),17β(H)-diasterane (20S) 13α(H),17β(H)-diasterane (20S) 14α(H),17β(H)-diasterane (20S) 14α(H),17β(H)-diasterane (20R) + 14β(H),17β(H)-sterane (20S) 14β(H),17β(H)-sterane (20S) 14α(H),17β(H)-sterane (20S) 14β(H),17β(H)-sterane (20S) 14α(H),17β(H)-sterane (20S) 14β(H),17β(H)-sterane (20S) 14α(H),17β(H)-sterane (20S) 14α(H),17β(H)-	$13\beta(H), 17\alpha(H) - diasterane$ $(20S)$ $C_{27}H_{48}$ $13\alpha(H), 17\alpha(H) - diasterane$ $(20R)$ $C_{27}H_{48}$ $13\alpha(H), 17\beta(H) - diasterane$ $(20S)$ $C_{27}H_{48}$ $13\alpha(H), 17\beta(H) - diasterane$ $(20R)$ $C_{27}H_{48}$ $13\alpha(H), 17\alpha(H) - diasterane$ $(20S)$ $C_{28}H_{50}$ $13\beta(H), 17\alpha(H) - diasterane$ $(20S)$ $C_{28}H_{50}$ $13\alpha(H), 17\alpha(H) - diasterane$ $(20S)$ $C_{28}H_{50}$ $13\alpha(H), 17\alpha(H) - diasterane$ $(20S)$ $C_{27}H_{48}$ $13\beta(H), 17\alpha(H) - diasterane$ $(20S)$ $C_{27}H_{48}$ $13\beta(H), 17\alpha(H) - diasterane$ $(20S)$ $C_{27}H_{48}$ $13\beta(H), 17\alpha(H) - diasterane$ $(20R)$ $C_{27}H_{48}$ $14\alpha(H), 17\alpha(H) - sterane$ $(20R)$ $C_{27}H_{48}$ $14\alpha(H), 17\alpha(H) - sterane$ $(20R)$ $C_{29}H_{52}$ $14\alpha(H), 17\alpha(H) - sterane$ $(20R)$ $C_{29}H_{52}$ $13\alpha(H), 17\beta(H) - diasterane$ $(20S)$ $C_{29}H_{52}$ $14\alpha(H), 17\alpha(H) - sterane$ $(20S)$ $C_{29}H_{52}$ $14\alpha(H), 17\alpha(H) - sterane$ $(20S)$ $C_{28}H_{50}$ $13\alpha(H), 17\beta(H) - diasterane$ $(20R)$ $C_{28}H_{50}$ $14\alpha(H), 17\alpha(H) - sterane$ $(20S)$ $C_{29}H_{52}$ <t< td=""></t<>













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FIGURE 6

GC-MS CHROMATOGRAMS OF AROMATIC HYDROCARBONS

TIC		-	total ion chromatograms
m/z	92,106	-	alkylated monoaromatic hydrocarbons
m/z	142,156,170	-	alkylated naphthalenes
m/z	178,192,206	-	alkylated phenanthrenes
m/z	184,198,212	-	alkylated dibenzothiophenes
m/z	231	-	triaromatic steranes
m/z	253	-	monoaromatic steranes

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