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ORGANIC GEOCHEMISTRY AND BIOMARKER CHARACTERIZATION OF EOCENE COAL FROM THE BURGAS BASIN

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Abstract

This study presents new data on the organic geochemistry of sub-bituminous coals from two previously uninvestigated mine galleries (301 and 109) in the Burgas Basin (Bulgaria). The samples are characterized by low thermal maturity, as indicated by vitrinite reflectance, high volatile matter contents and biomarker compositions. Abundant hopanoids, moderate, uniform ash yields and high sulphur contents suggest deposition in a marine/brackish influenced low-lying mire with a stable water level.

n-Alkane distributions show a pronounced predominance of long-chain odd-numbered homologues, with maximum at *n*-C_{31}, indicating a significant contribution of leaf waxes from higher terrestrial plants and warm climate. Pristane/phytane ratios suggest deposition under slightly fluctuating redox conditions. Terpenoid assemblages indicate mixed vegetation input, with a substantial contribution from coniferous plants (particularly in gallery 301) and a very variable angiosperm proportions in gallery 109. Hopanoid distributions reflect low thermal maturity and enhanced microbial activity.

The obtained results extend existing knowledge by providing data from previously unstudied galleries in the Burgas Basin and allow for a more detailed characterization of origin and depositional environment of organic matter.

Key words: Bulgarian coals, biomarkers, organic geochemistry

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Introduction. Coal characteristics are controlled by multiple factors, including vegetation type, depositional environment and diagenetic processes. In addition to traditional petrographic and palaeobotanical approaches, biomarker analysis of extractable organic matter (EOM) has become a key tool for reconstructing the origin and evolution of coal organic matter. Biomarkers (chemofossils) provide valuable information on vegetation input, depositional conditions (Eh, pH), microbial activity and thermal maturity [1–5].

The Eocene Burgas Basin is an important coal-bearing basin in Bulgaria, where sub-bituminous coals were formed under paralic conditions influenced by marine and brackish environments. Previous studies have shown that the organic matter (OM) in these coals reflects a complex interplay between vegetation input, depositional setting and diagenetic processes [5–7].

This study provides new molecular and bulk organic geochemical data on sub-bituminous coals from mine galleries 301 and 109 in the Burgas Basin, representing previously uncharacterized sections of this coal-bearing system. While three samples from gallery 301 were previously reported [7], the present study expands the dataset by including new samples and integrating bulk geochemical parameters with detailed molecular data. This integrated approach allows for a more detailed characterization of vegetation input, depositional conditions, and diagenetic processes.

Geological settings and sampling. The Paleogene coal-bearing sediments in the Burgas Basin are part of the Ravnets Formation [8]. The basin includes two coal levels with up to four coal beds from the Eocene. The upper level comprises Seam A which contains the main economic resources of the Burgas deposit. The coals are subbituminous, with medium ash yield and high sulphur content. The present study is based on ten samples which were collected from coal seam A and originated from the face of "Black Sea" mine galleries 301 and 109.

Experimental. ***Proximate analysis, vitrinite reflectance, total organic carbon (TOC) and sulphur.*** The proximate analysis was carried out according to standard procedures: analytical moisture (W^a) following ISO 1015:1975 [9], ash yield according to ISO 602:2015 [10], and volatile matter (V^{daf}) according to ISO 20360:2020 [11].

Vitrinite reflectance (Ro, %) was measured using a Leica DM2500PM microscope equipped with a photometer, based on at least 50 measurements per sample. For microscopic analysis, samples were crushed to < 1 mm, mounted in epoxy resin, ground and polished.

Total organic carbon (TOC) and sulphur were determined using an Eltra Helios C/S analyser. Prior to TOC analysis, samples were treated with H_3PO_4 to remove carbonates. Measurements were performed in duplicate with an analytical error of less than 0.5%.

Gas chromatography–mass spectrometry (GC–MS). EOM was obtained from ~ 1.7 g of pulverized sample using an ASE 350 system with dichloro-

methane at 75 °C and 100 bar. The extracts were concentrated and asphaltenes were precipitated using *n*-hexane, followed by centrifugation. The resulting maltene fraction was separated into aliphatic, aromatic and polar (NSO) fractions by MPLC [2]. Biomarker compounds in the aliphatic and aromatic fractions were determined by GC–MS. Detailed procedure of GC–MS analysis and quantification of biomarkers was explained in the previous publication [7].

The TOC and sulphur, extraction, fractionation of EOM, as well as GC–MS analysis were carried out at the Montan University, Leoben, Austria. Proximate analysis and vitrinite reflectance measurements were performed at the Sofia University, Bulgaria.

Results and discussion. Three samples from gallery 301 (B310-2, B310-4, B310-5) were previously investigated [7], but are included here to allow direct comparison with newly obtained data.

Bulk geochemical characteristics. Bulk geochemical characteristics of the coal samples are listed in Table 1. Ash yields range from 10.41 to 16.17 wt.%, while total sulphur contents vary between 2.62 and 5.78 wt.%. TOC contents are relatively high, reflecting the organic-rich nature of the coals. These parameters are consistent with the low-rank (sub-bituminous) of the studied coals, as indicated by vitrinite reflectance (0.35–0.48%) and the relatively high volatile matter contents (51.67–59.81%). Elevated sulphur contents indicate deposition under marine/brackish-influenced conditions, typical for coastal peat-forming environments in the Burgas Basin [5]. Moderate, uniform ash yields and high sulphur contents suggest deposition under stable water column, which enabled good preservation of OM.

Extractable organic matter (EOM) fractional composition. Data on the yields and fractional compositions of extractable organic matter (EOM) are presented in Table 1. EOM yields range from 3.13 to 5.74 wt.% (57.54–104.31 mg/g TOC). The extracts are dominated by asphaltenes (53.07–66.12%) and polar (NSO) compounds (28.72–43.27%), whereas the aliphatic (1.42–4.26%) and aromatic (0.48–1.23%) fractions are minor. The predominance of asphaltenes and polar fraction indicates that the EOM is highly functionalized and composed of oxygen-rich, structurally complex constituents, typical for humic organic matter in low-rank coals. Similar extract compositions have been reported for previously studied coals from the Burgas Basin [5, 7].

Molecular composition of hydrocarbons. *n-Alkanes and isoprenoids.* *n*-Alkanes are the most abundant hydrocarbons in studied samples (Table 1). The *n*-alkane distributions are characterized by a pronounced dominance of long-chain odd-numbered homologues (n-C_{27-31}) (Table 2, Fig. 1, 2), with maximum at n-C_{31}. The carbon preference index (CPI) [12] values are high (5.58–10.10), indicating a strong odd-over-even predominance and supporting the low thermal maturity of the studied coals. The dominance of long-chain odd *n*-alkanes reflects a significant contribution of epicuticular waxes from higher terrestrial plants [1,3].

Table 1

Bulk organic geochemical data, yield and fractional composition of extractable organic matter of studied coals.
Note: Coal samples presented in the previous publication [7] are highlighted by grey shading

Gallery	Sample	Ash	W^a	V^{daf}	TOC	S_{total}	Ro	EOM	Aliphatic fraction	Aromatic fraction	Polar (NSO) fraction	Asphaltenes
		(wt%, db)		(wt%, daf)	(wt%, db)		(%)	%/mg/ gT OC	(wt% of EOM)			
301	B301-2	15.21	15.77	56.89	55.92	3.49	0.35	3.43/61.40	2.42	1.23	43.28	53.07
	B301-4	12.82	18.69	51.67	54.35	3.91	0.44	3.13/57.54	3.45	0.98	41.78	53.79
	B301-5	16.11	15.46	54.17	50.76	5.78	0.45	5.19/102.16	4.26	0.87	28.72	66.15
	B301-6	15.57	15.34	59.81	54.94	3.11	0.39	5.74/104.3	2.02	1.04	37.19	59.75
	B301-7	16.17	15.46	58.54	54.62	3.92	0.38	3.77/68.98	2.83	0.87	40.95	55.35
109	B109-2	12.90	17.77	51.80	57.92	4.49	0.48	3.83/66.09	2.21	0.92	39.39	57.48
	B109-4	15.66	17.60	53.68	61.30	3.25	0.36	5.29/86.29	1.69	0.84	33.55	63.92
	B109-5	12.96	17.33	54.99	51.37	2.62	0.35	3.85/74.97	1.54	0.60	3.5.54	62.32
	B109-7	12.82	15.07	55.69	61.17	3.87	0.41	4.36/71.36	1.42	0.95	36.98	60.65
	B109-8	10.41	18.07	52.28	52.30	2.69	0.45	4.43/84.66	2.23	0.48	37.21	60.08

db – Dry basis; daf – Dry and ash-free basis; W^a – Analytical moisture; V – Volatile matter; TOC – Total organic carbon; S_{total} – Total sulphur; Ro – Vitrinite reflectance; EOM – Extractable organic matter

Table 2

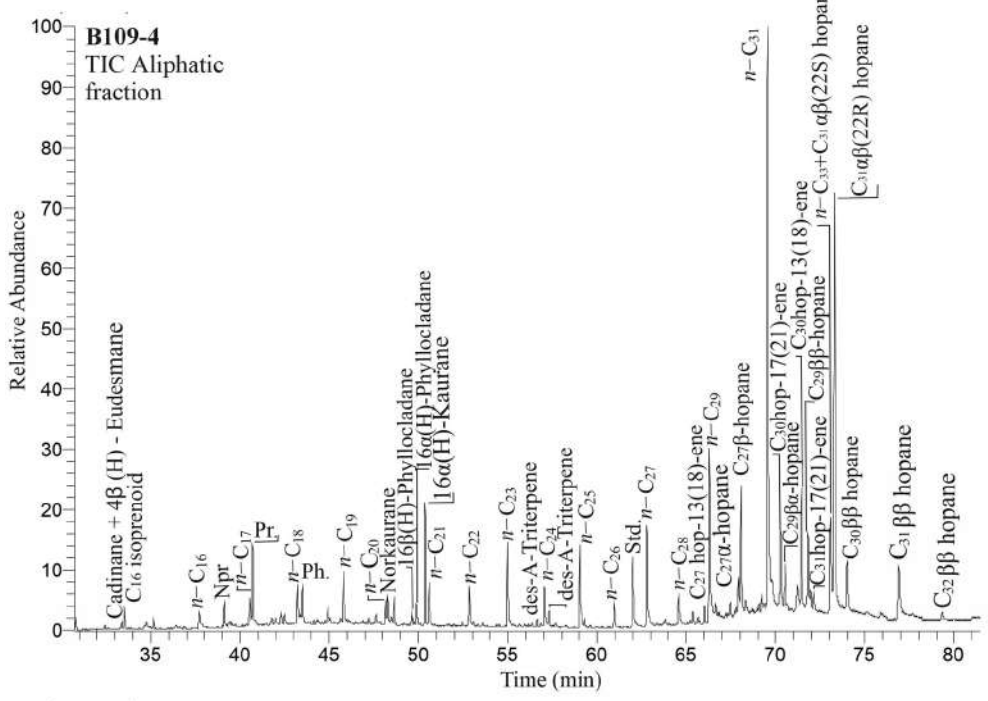
Concentrations of *n*-alkanes, proportions of *n*-C_{15-19}, *n*-C_{21-25} and *n*-C_{27-31} relative to sum of *n*-alkanes, CPI, pristane/phytane ratios, concentrations of sesquiterpenoids diterpenoids and angiosperm-derived triterpenoids, ratios of Di-/(Di- + Triterpenoids), concentrations of hopanes and C_{30} hop-17(21)-ene, and 22S/(22S+22R) C_{31} αβ hopane ratios of Burgas subbituminous coal.
Note: Coal samples presented in the previous publication [7] are highlighted by grey shading

Sample	B301-2	B301-4	B301-5	B301-6	B301-7	B109-2	B109-4	B109-5	B109-7	B109-8
n-Alkanes (μg/g TOC)	21.7	14.58	23.39	12.32	8.44	18.29	20.86	15.77	15.69	25.04
n-C_{15-19}/*n*-C_{15-33}	0.13	0.10	0.09	0.11	0.16	0.05	0.12	0.1	0.09	0.07
n-C_{21-25}/*n*-C_{15-33}	0.13	0.08	0.11	0.13	0.15	0.08	0.20	0.15	0.14	0.09
n-C_{27-31}/*n*-C_{15-33}	0.62	0.67	0.68	0.64	0.58	0.67	0.53	0.61	0.61	0.62
CPI*	8.56	10.10	8.45	7.50	7.27	6.97	6.61	6.44	5.93	5.58
(*n*-C_{31+33})/(*n*-C_{27+29})	1.78	2.09	1.79	1.49	1.77	2.25	2.05	1.39	1.45	1.88
AI**	0.68	0.70	0.67	0.65	0.70	0.69	0.71	0.64	0.65	0.68
Pr/Ph***	1.31	1.06	0.81	1.17	1.42	1.16	1.47	0.92	1.78	1.00
Sesquiterpenoids (μg/g TOC)	0.59	1.89	2.50	1.27	1.13	1.28	2.43	1.82	1.77	2.34
Diterpenoids (μg/g TOC)	5.30	0.55	2.11	5.64	3.68	1.05	8.32	0.83	0.36	0.73
Triterpenoids (μg/g TOC)	3.52	1.05	1.30	1.07	0.78	0.86	1.45	1.14	1.07	0.86
Di-/(Di-+Triterpenoids)	0.60	0.34	0.62	0.84	0.83	0.55	0.85	0.42	0.25	0.46
Hopanes (μg/g TOC)	13.95	7.85	6.75	9.48	6.69	2.64	13.31	6.29	5.65	3.57
C_{30} Hop-17(21)-ene (μg/g TOC)	0.63	0.35	0.39	0.42	0.31	0.19	0.47	0.25	0.16	0.09
22S/(22S+22R) C_{31} $\alpha\beta$ hopane	0.04	0.03	0.01	0.02	0.03	0.00	0.05	0.00	0.09	0.06

*CPI – carbon preference index, **AI – alkane index, ***Pr – pristane, Ph – phytane

$$CPI = 1/2 \times [\Sigma odd(n - C_{25} - n - C_{33})/\Sigma even(n - C_{24} - n - C_{32}) + \Sigma odd(n - C_{25} - n - C_{33})/\Sigma even(n - C_{26} - n - C_{34})]$$ [15]

$$AI = n - C_{31}/(n - C_{31} + n - C_{29})$$ [19]


B109-4
TIC Aliphatic
fraction
Relative Abundance
Time (min)
Cadinane + 4β (H) - Eudesmane
C16 isoprenoid
n−C16
Npr
n−C17
Pr.
n−C18
Ph.
n−C19
n−C20
Norkaurane
16β(H)-Phyllocladane
16α(H)-Phyllocladane
n−C21
16α(H)-Kaurane
n−C22
n−C23
des-A-Triterpene
n−C24
des-A-Triterpene
n−C25
n−C26
Std.
n−C27
n−C28
C27 hop-13(18)-ene
n−C29
C27α-hopane
C27β-hopane
n−C31
C30hop-17(21)-ene
C30hop-13(18)-ene
C29βα-hopane
n−C33+C31αβ(22S) hopane
C31hop-17(21)-ene
C29ββ-hopane
C31αβ(22R) hopane
C30ββ hopane
C31 ββ hopane
C32 ββ hopane

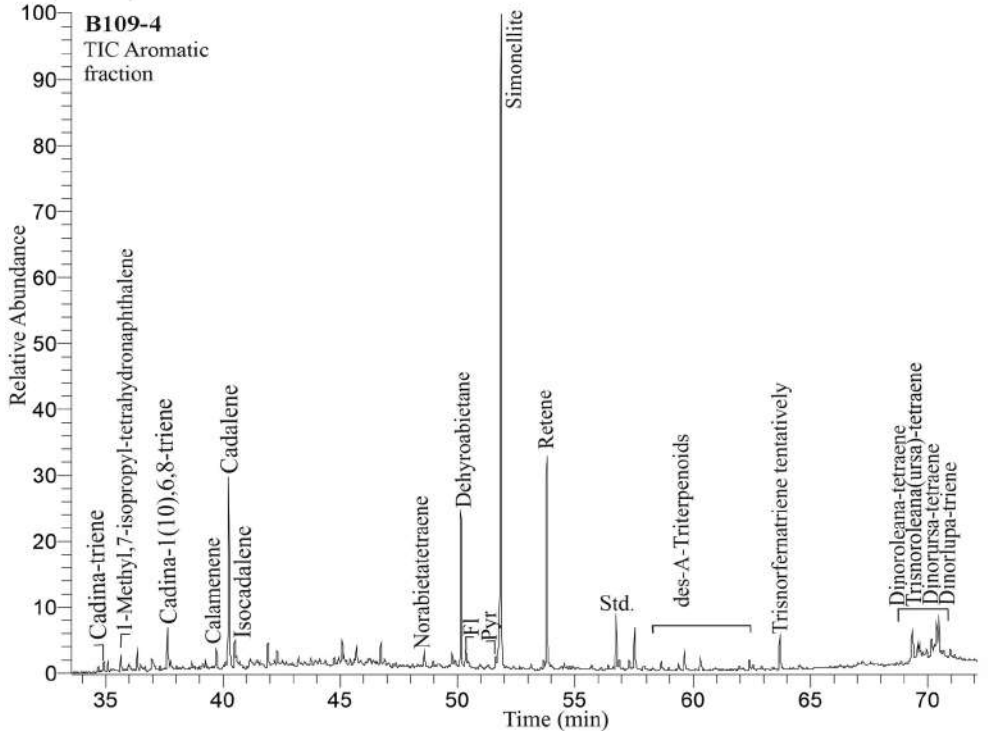

B109-4
TIC Aromatic
fraction
Relative Abundance
Time (min)
Cadina-triene
1-Methyl,7-isopropyl-tetrahydronaphthalene
Cadina-1(10),6,8-triene
Calamenene
Isocadalene
Cadalene
Norabietatetraene
Dehyroabietane
Fl
Pyr
Simonellite
Retene
Std.
des-A-Triterpenoids
Trisnorfernatriene tentatively
Dinoroleana-tetraene
Trisnoroleana(ursa)-tetraene
Dinorursa-tetraene
Dinorlupa-triene

Fig. 1. Total ion chromatograms (TICs) of the aliphatic and aromatic fraction of sample B109-4. Legend: Std. – internal standard (squalane for the aliphatic fraction and 1,1′-binaphthyl for the aromatic fraction), NPr – norpristane, Pr – pristane, Ph – phytane, ββ and αβ designate configurations at C-17 and C-21 in hopanes, (R) and (S) designate configuration at C-22 in hopanes, Fl – fluoranthene, Pyr – pyrene

←

Variations in the relative abundances of n-C_{27}, n-C_{29} and n-C_{31} suggest differences in vegetation input, with n-C_{27} and n-C_{29} generally associated with deciduous plant material, and n-C_{31} indicating a greater contribution from grasses or other herbaceous vegetation [13]. Otherwise, dominance of n-C_{31} was related to the warm climate plant waxes [14]. Notable prevalence of n-C_{31} and n-C_{33} over n-C_{27} and n-C_{29} [$(n\text{-}C_{31+33})/(n\text{-}C_{27+29})$ 1.39–2.25], associated with alkane index (AI > 0.5) [15] (Table 2) is consistent with warm climate during the Eocene [16, 17]. Short-chain homologues (n-C_{15-19}) and mid-chain (n-C_{21-25}) homologues have lower abundance (with relative proportion of up to 0.16 and 0.20, respectively, in the distribution of n-alkanes).

Pristane/phytane (Pr/Ph) ratios [18] vary from 0.81 to 1.78 (Table 2), indicating slightly variable redox conditions, from suboxic to weakly reducing, during peat accumulation. Limited variations of the Pr/Ph ratios are consistent with deposition in the low lying mire. However, in the immature coal, this parameter rather indicates predominantly terrestrial OM input than Eh settings in the peat-forming environment. This is documented by significant positive correlation between Pr/Ph (Table 2) and TOC (Table 1) ($r = 0.87$).

Sesquiterpenoids, diterpenoids and non-hopanoid triterpenoids. The sesquiterpenoid composition is characterized by compounds of the cadinane/cadinene/cadalene and eudesmane types (Fig. 1, 2, Table 2). These compounds are commonly associated with gymnosperms, particularly coniferous plants, and are typically derived from resinous tissues [4]. Diterpenoids are present in both aliphatic and aromatic fractions (Fig. 1, 2) of all samples, with locally higher concentrations in samples B301-6 and B109-4 (Table 2). In the aliphatic fractions, considerable amounts of tetracyclic diterpenoids, 16α(H)-kaurane, 16α(H)- and 16β(H)-phyllocladane are detected (Fig. 1, 2), with the prevalence of 16α(H)-phyllocladane in most samples. These compounds indicate a substantial contribution of conifer-derived resinous OM. In particular, 16α(H)-phyllocladane is associated with Araucariaceae, Cupressaceae and Taxodiaceae, whereas 16α(H)-kaurane is commonly linked to Araucariaceae [4]. Aromatic diterpenoids such as dehydroabietane, simonellite and retene are also present, indicating contributions from abietane-type precursors. Simonellite predominates among the aromatic diterpenoids (Fig.Fig. 1, 2). Diterpenoids with abietane skeleton are commonly linked to Pinaceae, although these compounds may also derive from ferruginol, which is widespread in Cupressaceae and Podocarpaceae [4]. The Podocarpaceae were distinctive, but generally an uncommon part of the Northern Hemisphere

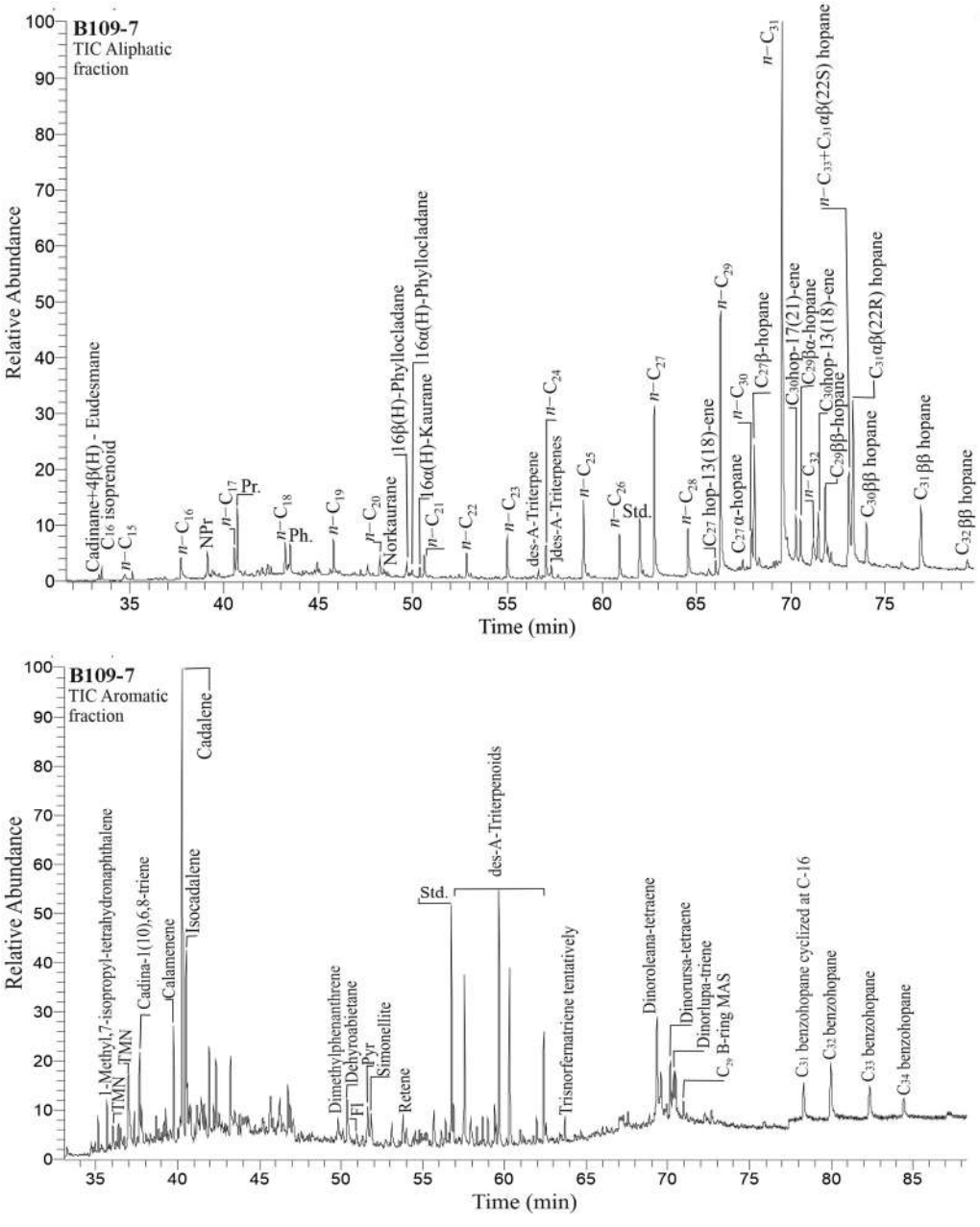


Fig. 2. Total ion chromatograms (TICs) of the aliphatic and aromatic fraction of sample B109-7. For peak assignments see legend of Fig. 1

flora during the Eocene, particularly in Europe [19]. Therefore, overall, the diterpenoid assemblage indicates a dominant input of families Araucariaceae, Cupressaceae and Taxodiaceae to the precursor OM.

Non-hopanoid triterpenoids (aliphatic and aromatic derivatives, including ring-A-degraded and pentacyclic structures) of the oleanane, ursane and lupane types, indicative for angiosperms, occur in all samples in considerable amounts (Fig. 1, 2, Table 2). This is consistent with palaeobotanical data [6] and the previous studies of the Burgas Basin coals [5, 7]. The Di-/(Di- + Triterpenoids) ratio [3, 5] ranges from 0.25 to 0.85 (Table 2), indicating substantial variability in the relative contributions of gymnosperm and angiosperm vegetation during peat formation. Coals from gallery 301 are mostly characterized by larger contribution of gymnosperms, whereas samples from gallery 109 showed highly variable proportions of coniferous and angiosperm vegetation.

Hopanoids. The hopanoid distributions are characterized by the presence of C_{27} to C_{32} hopanes, with an absence of C_{28} homologues. The most abundant compound is C_{31} 17α(H),21β(H),22(R) hopane followed by C_{27} 17β(H),21β(H) hopane. C_{30} Hop-17(21)-ene and C_{30} hop-13(18)-ene are detected in all samples, but in lower amounts than hopanes (Fig. 1, 2). The presence of $\beta\beta$-hopanes, unsaturated hopenes, and the S/(S+R) ratio of C_{31} $\alpha\beta$ hopanes (<0.1; Table 2) reveal low thermal maturity of the Burgas coals.

The overall hopanoid assemblage indicates a significant contribution of bacterial biomass during peat formation. In particular, the prevalence of C_{31} hopanes supports the activity of hopanoid-producing bacteria [20], which is consistent with the stable water column and non-acidic pH conditions due to the marine/brackish influence [5]. Neutral to slightly alkaline conditions in the paloemire are also documented by the presence of small foraminifers and ostracods [8], which carbonate shells cannot be preserved in the acidic environment. The C_{30} hop-17(21)-ene (Fig. 1, 2; Table 2) may originate directly from bacteria or can be formed by diagenetic isomerisation of diploptene (C_{30} hop-22(29)-ene). The latter is dehydration product of diplopterol, found in ferns, mosses, lichens, fungi, as well as in hopanoid-producing bacteria [20, 21]. Since the C_{30} hop-13(18)-ene was also detected in studied samples (Fig. 1, 2) the possible origin from ferns cannot be excluded [22]. However, given the marine/brackish influence on the peat environment, it seems reasonable to assume that C30 hop-17(21)-ene originates, at least in part, from sulphate-reducing bacteria [23].

Conclusions. The studied coals from galleries 301 and 109 of the Burgas Basin are low-rank (sub-bituminous), as documented by vitrinite reflectance, high volatile matter contents and biomarker compositions. Peatification processes in the marine/brackish influenced low-lying mire. The stable water level enabled OM preservation. The prevalence of *n*-alkanes in hydrocarbon fractions with the predominance of *n*-C_{31} homologue indicates a dominant input of plant waxes from the higher-plants, which thrived under warm climate conditions. Terpenoid assemblages reveal mixed vegetation input, with a substantial contribution from coniferous plants (Araucariaceae, Cupressaceae, Taxodiaceae) and a variable angiosperm component. The latter often dominated in gallery 109. Hopanoid abun-

dances and distributions reflect significant microbial input and neutral to slightly alkaline conditions. Overall, the data indicate peat formation in a dynamic coastal neutral-slightly alkaline environment, characterized by mixed vegetation (thrived in humid, warm climate), slightly variable redox conditions and active diagenetic microbial processes.

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