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EPR, UV and IR Spectroscopic Methods for the Study of Crude

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ABSTRACT

This work for first time the systematizes research data obtained by EPR, UV, IR spectroscopy, and luminescence methods for light, medium, and heavy Azerbaijani crude oils. In light Surakhany oil (685 m depth), EPR analysis revealed asphaltene radicals and signals of metal oxides. The composition, kerosene, and diesel fractions of commercial oils from Sangachal-Deniz, Bulla-Deniz, Khara-Zira, and Alat-Deniz (ρ = 812.4 kg/m^3) were studied, where EPR spectra of Co, Mo, and Mn were detected. The composition, physicochemical properties, and paramagnetism of Zagly oil were examined together with aromatic hydrocarbons of groups I–IV. In group III, an EPR signal with ΔH = 7.5 mT and g = 2.4 was recorded. Groups I–III contain small amounts of benzene, naphthalene, phenanthrene, and anthracene derivatives. Kerosene fractions of heavy West Absheron oil (ρ = 912.5 kg/m^3) exhibited a similar EPR signal (ΔH = 7.5 mT, g = 2.4), also observed in group III aromatic hydrocarbons of Zaqly oil. In diesel fractions, an EPR signal with ΔH = 1.1 mT and g = 2.0023 was detected, corresponding to group II aromatic hydrocarbons. UV spectroscopy and luminescence measurements indicate that these radicals belong to substituted aromatic hydrocarbons. Balakhany oil (ρ = 919 kg/m^3) was investigated, with particular attention to aromatic hydrocarbons containing three or more condensed benzene rings responsible for visible luminescence. Absheron oil (ρ = 920 kg/m^3), its 50-degree fractions, and heavy petroleum residues were also studied. Residues from primary processing exhibit lower aromatic hydrocarbon content, improved environmental characteristics, and significant resource potential.

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1. Introduction

A significant contribution to the global oil production balance of the Absheron oil- and gas-bearing region of Azerbaijan comes from both onshore fields (Surakhany, Balakhany, Bibi-Heybat, Garadagh, Zagly, etc.) and offshore fields in the Caspian Sea (Neftyanye Kamni, Bahar, Chirag, Azeri, Shah-Deniz, Absheron, West-Absheron, etc.). This has generated considerable scientific interest in their study [1-3]. The development of the oil industry in the republic is largely determined by the level of technological solutions aimed at processing [4-15] and rational use of hydrocarbon raw material. With the rapid expansion of Azerbaijan's modern oil industry, the implementation of national oil-and-gas strategy, the growth of oil production, and the increase in exports to global markets, identifying new properties of Azerbaijani oils has become both scientifically and practically important. Crude oil, consisting mainly of hydrocarbons and heteroatomic compounds (sulfur-, nitrogen-, and oxygen-containing), also includes metal-containing [16-18] compounds, resin-asphaltene substances (RAS), and asphaltenes, which are responsible for the paramagnetism of oil [19]. This indicates the important role of unpaired electrons in petroleum systems and the need for a deep, systematic approach to understanding their lability under sunlight, which induces various photochemical processes—often radical-chain in nature. The presence of RAS, asphaltenes, and metals negatively affects refining performance and limits the application areas of petroleum products. Therefore, research aimed at a detailed study of the composition and properties of hydrocarbon raw materials is essential for developing effective technologies for extraction and processing.

2. Experimental Section

The paramagnetism of the oils was studied using a BRUKER BIO SPIN radiospectrometer (Germany) in the X-band (operating frequency 9.85 GHz). The high-frequency modulation amplitude (100 kHz) was varied within 150–650 mT. Field calibration was performed using an ultradispersed diamond (UDD) internal standard with g = 2.0036. EPR spectra were recorded at 25°C under minimal saturation conditions (microwave power 0.5 mW).

Electronic absorption spectra of several oil fractions were obtained using a Spekord UV-Vis spectrophotometer in the 200–800 nm range. IR spectroscopy was performed using a Fourier-transform spectrometer ALPHA (BRUKER, Germany). Electronic absorption spectra were also recorded on a JENNAY UV/Vis-680 spectrophotometer (190–1000 nm, 0.1 nm resolution) in diluted hexane solutions (10^{-2}).

3. Results and Discussion

In earlier studies, the objects of investigation were the oils of the Surakhany, Bibi-Heybat, and Balakhany fields of the Absheron Peninsula. The Surakhany field is one of the promising shallow oil deposits (658 m) of the peninsula. The yield of gasoline fractions (initial boiling point – 200°C) was 22.04 wt%.

The oil, consisting mainly of hydrocarbons and heteroatomic compounds (sulfur-, oxygen-, and nitrogen-containing), also contains metal-containing compounds and asphaltenes, whose presence negatively affects refining performance. The initial studies of this oil included determining its elemental composition using an Omega Handheld XRF Analyzer with adjustable voltage up to 40 kV and current up to 100 μA (with five filters). The microelement composition is presented in Table **1**.

According to the analysis, the content of microelements in the studied oil is low, except for Fe, Cu, and Ni. The hydrocarbon composition of native Surakhany oil was studied by liquid-adsorption chromatography. The results showed that the oil consists of paraffin–naphthenic hydrocarbons (75.42 wt%) and aromatic hydrocarbons (20.48 wt%). The content of resin-asphaltene substances (RAS) is 1.1 wt%. Using classical distillation by boiling intervals, a set of physicochemical parameters was determined (Table **2**).

The physicochemical analysis shows that Surakhany oil is sulfurous and paraffinic. EPR studies revealed the presence of asphaltene radicals ($R^{\cdot}_{\mathrm{asph}}$), as well as metal-containing compounds: iron oxides (ΔH = 168.1 mT, g = 2.24); copper oxide microcrystals (ΔH = 108.9 mT, g = 2.27), nickel oxides (ΔH = 117.4 mT, g = 2.21); significant amounts of $R\bullet_{\mathrm{asph}}$ (2×10^{16} spins/g) (ΔH = 0.88 mT, g = 2.0023) [20]. UV irradiation can significantly reduce the concentration of $R^{\bullet}_{asph}$. Earlier studies showed that RAS radicals have long-wavelength absorption up to 550 nm, enabling efficient use of solar energy [21]. Since RAS act as photosensitizers for the decomposition of organic compounds (paraffins, paraffin-naphthenes, alcohols), their excited states may play an important role in photochemical processes.

Table 1: Microelement content in Surakhany oil (well 1311) and its fractions.

Fraction (°C)	Cu	Ni	Fe	Zn	Nb	Mo	Pb	Bi
Crude oil	0.16	0.12	0.17	0.07	0.008	0.008	0.01	–
200–250	0.01	0.02	–	0.06	0.007	0.008	0.01	–
250–300	0.12	0.14	–	0.022	0.008	0.07	0.13	0.016
300–350	0.10	0.088	–	0.006	0.07	0.007	0.011	0.003
>350	0.10	0.075	0.028	0.006	0.007	0.006	0.016	–
400–450	0.088	0.10	–	0.006	0.007	0.007	0.009	0.004
450–500	0.12	0.082	0.024	0.006	0.007	0.007	0.015	0.004

Table 2: Physicochemical properties of Surakhany oil and its fractions.

Fraction (°C)	Molecular Weight	Density (g/cm³)	Viscosity at 40°C (mm²/s)	Refractive index n^{20}_{D}	RAS (wt%)	Pour Point (°C)	Acid Number (mg KOH/g)
Crude oil	202.64	0.8417	6.33	–	0.7809	–40	2.55
250–300	190.2	0.8319	3.30	1.4656	0.0379	–31	0.4782
300–350	201.8	0.8410	6.65	1.4710	–	–18	–
350–400	243.32	0.8679	18.64	1.4843	0.0851	0	0.3595
400–450	255.24	0.8743	26.90	1.4865	0.0878	+4	0.5257
450–500	280.16	0.8861	71.89	1.4932	0.1103	+12	0.5508

Commercial offshore oil (Sangachal-Deniz, Duvanny-Deniz, Khara-Zira, Bulla-Deniz, Alat-Deniz). This oil has density 719.5–828.4 kg/m³ (20°C); kinematic viscosity 0.63–4.12 mm²/s (20°C); pour point –65 to –21°C; high paraffin content (7.85 wt%); low resin content (4.25 wt%); low sulfur (0.3 wt%); low asphaltenes and coke (0.07 and 0.84 wt%). Distillation up to 350°C yielded 61.14 wt% fractions; residue was 38.86 wt% [22]. Physicochemical properties are shown in Table **3**.

Fractionation allowed detection of EPR spectra of rare-earth elements. In the kerosene and diesel fractions (140–320°C), EPR spectra were observed for: cobalt (ΔH = 5.12 mT, g = 4.76), molybdenum (g = 2.2); manganese Mn^{2+} (g = 2) [22].

3.1. Zagly Oil (Kuba Region)

This medium-heavy oil (density 847 kg/m³) is highly resinous, low-sulfur, low-paraffin, and contains: saturated hydrocarbons; heteroatomic compounds (N, S, O); metal-containing compounds; resin-asphaltene substances. Despite extensive studies of high-boiling fractions, primary processing products and heavy oil residues remain poorly studied. Heavy residues have advantages: lower aromatic hydrocarbon content; greater environmental safety; large resource potential [16-18].

Using the classical distillation method based on boiling temperature ranges, the following fractions were obtained: 120–230°C, 150–250°C, 150–280°C, 140–320°C, 140–350°C, and 180–350°C. These fractions are characterized by: Density: 719.5–828.4 kg/m³; Kinematic viscosity at 20°C: 0.63–4.12 mm²/s; Molecular weight: 105–189; Pour point: –65 to –21°C; Sulfur content: 0.3 wt%.

Table 3: Physicochemical properties of commercial offshore oil.

Fraction (°C)	Yield (%)	n^{20}_D	Molecular Weight	Density (kg/m³)	Viscosity (mm²/s)	Cloud Point	Crystallization	Pour Point
NBP–120	13.18	1.4072	105	719.5	0.63	–	–	–
NBP–200	30.37	1.4204	121	749.5	0.82	–	–	–
85–180	22.16	1.4217	124	754.5	0.84	–	–	–
120–230	21.77	1.4344	141	779.2	1.16	–	–60 (no crystals)	–65
150–250	18.35	1.4443	154	795.3	1.61	–	–51	–53
150–280	24.39	1.4484	162	804.5	1.99	–	–38	–40
140–320	36.00	1.4519	167	810.2	2.35	–25	–	–32
140–350	41.86	1.4550	173	816.2	2.73	–14	–	–29
180–350	33.11	1.4614	189	828.4	4.12	–9	–	–21

Fractionation enabled the identification of EPR spectra corresponding to rare-element ions. In the kerosene and diesel fractions (140–320°C), EPR spectra revealed: Cobalt (Co^{2+}): ΔH = 5.12 mT, g = 4.76; Trace amounts of molybdenum (Mo): g = 2.2; Medium and Heavy Crude Oils. Currently, worldwide interest has increased in the production and refining of medium and heavy crude oils with densities ranging from 847 to 920 kg/m³ and characterized by elevated viscosity. Among these is the Zagly crude oil from the Quba region of the Caspian area (ρ = 847 kg/m³) [25, 26]. This oil is: Highly resinous; Low in sulfur; Low in paraffin; A complex mixture of liquid hydrocarbons containing dissolved solid hydrocarbons, resin–asphaltene substances (RAS), and trace metals.

In addition to hydrocarbons, the oil contains heteroatomic compounds (nitrogen-, sulfur-, and oxygen-containing species), metal-containing compounds, and RAS, whose presence may adversely affect refining performance and product application.

Despite extensive studies of high-boiling fractions and secondary processing products—on the basis of which many important petroleum products have been developed—primary refining products and heavy petroleum residues (HPR) remain insufficiently investigated.

However, heavy petroleum residues obtained from primary refining offer significant advantages:

Lower aromatic hydrocarbon content; Reduced environmental impact; Substantial raw material potential.

3.2. Aromatic Hydrocarbons and Industrial Relevance

Modern industrial production is largely oriented toward obtaining aromatic hydrocarbons as feedstock for the synthesis of aromatic ketones, aldehydes, and acids.

Rational utilization of hydrocarbon resources is also associated with their potential application as: photosensitizers for decomposition of organic compounds; Inhibitors of photo- and thermo-oxidation; light stabilizers; luminescent materials.

Particular importance is attributed to aromatic hydrocarbons (AH) containing three or more condensed benzene rings, which exhibit luminescence [23, 24].

In products of primary oil refining, aromatic hydrocarbons consist predominantly of substituted derivatives, whereas in secondary processing products, both substituted and unsubstituted aromatic hydrocarbons are present.

3.3. Fractionation of Zagly Oil

After deasphalting and resin removal (GOST 11858-66), adsorption chromatography was performed using: adsorbent: ASK-grade silica gel; solvents: heptane, benzene, and alcohol–benzene mixture.

This procedure yielded Groups I–IV aromatic hydrocarbons of Zagly oil. Spectrometric analysis was conducted using an IRF-23 refractometer. The compositional analysis revealed: saturated (methane–naphthene) hydrocarbons: 40.5 wt%; aromatic hydrocarbons: 46.5 wt% resin–asphaltene substances: 13.2 wt%. Refractive indices: Group I AH: 1.5074; Group II AH: 1.5480; Group III AH: 1.5790.

Yields: methane–naphthene hydrocarbons: 40.5 wt%. Group I AH: 15.3 wt%; Group II AH: 4.5 wt%; Group III AH: 7.8 wt%.

The molecular weight increases progressively from methane–naphthene hydrocarbons to Group III AH, ranging from 859.7 to 972.2 [25, 26] (Table **4**).

Table 4: Physicochemical characteristics of isolated components of Zagly Crude Oil.

Product	Refractive Index (nD20)	Density (ρ, kg/m³)	Yield (wt%)
Methane–naphthene hydrocarbons	1.4685	859.7	40.5
Group I AH	1.5074	892.1	15.3
Group II AH	1.5480	932.1	4.5
Group III AH	1.5790	972.2	7.8
Group IV AH	1.6011	1043.6	18.7
Resins	–	1065.2	11.6
Asphaltenes	–	–	1.6

3.4. IR Spectroscopic Analysis

FTIR analysis (ALPHA Fourier spectrometer, BRUKER) of Zagly crude oil revealed that the IR spectrum of methane–naphthene hydrocarbons exhibits deformation vibration bands at 962, 973, and 1013 cm^{-1}, corresponding to C–H bonds of CH_2 groups in naphthenes.

Importantly, absorption bands characteristic of aromatic hydrocarbons are absent in this fraction [27-30]. In contrast, comparison of the IR spectra of Groups I–IV aromatic hydrocarbons (AH) with the methane–naphthene fraction shows that, in addition to the above-mentioned bands, the aromatic fractions display: Deformation vibrations at 700, 744, 813, and 868 cm^{-1}.

Stretching vibrations at 1604 cm^{-1}. These bands correspond to C–H and C=C bonds of substituted benzene rings, confirming the presence of substituted aromatic structures.

3.5. Luminescence Observations

Visual inspection of the aromatic hydrocarbon fractions under a PRK-4 mercury lamp (UV filter >200 nm), combined with structural-group analysis, demonstrated that Groups I–III AH contain small amounts of benzene, naphthalene, phenanthrene, and anthracene derivatives.

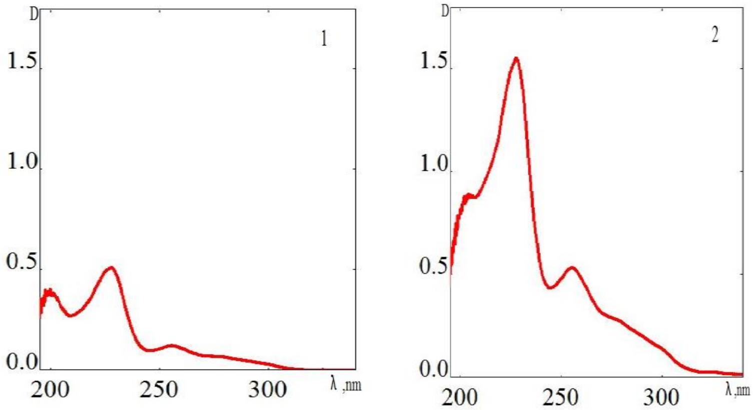
Groups I–II AH predominantly contain unsubstituted aromatic hydrocarbons. Group III AH contains both substituted and unsubstituted aromatic hydrocarbons.

All investigated crude oil fractions exhibited weak yellow luminescence under UV irradiation, characteristic of resin–asphaltene substances (RAS).

This behavior likely results from a relatively high concentration of asphaltene radicals ($R^{\bullet}_{asph}$), which effectively screen aromatic hydrocarbons and suppress their luminescence.

When the concentration of $R^{\bullet}_{asph}$ decreases, Groups II and III AH of Zagly oil exhibit blue–violet luminescence characteristic of substituted aromatic hydrocarbons, with absorption maxima corresponding to benzene: 200–215 nm; naphthalene: 220–230 nm; phenanthrene: 245–260 nm; anthracene: 272–290 nm.

Electronic absorption spectra of these fractions were recorded using a UV/Vis 680 spectrophotometer in diluted solutions (Fig. **1**).



1 – Group II AH (c = 0.01%); **2** – Group III AH (c = 0.01%).

Figure 1: Electronic absorption spectra of aromatic hydrocarbons of Groups II and III from Zagly crude oil.

3.6. EPR Investigation of Zagly Oil

The paramagnetic properties of Zagly crude oil and its Groups I–IV aromatic hydrocarbons were studied using EPR spectroscopy [25, 26].

Given that heavy crude oil inherently exhibits strong paramagnetism due to asphaltene radicals ($R^{\bullet}_{asph} \approx 10^{18}$ spins/g), which can mask the presence of aromatic hydrocarbon radicals at significantly lower concentrations, preliminary deasphalting and resin removal were performed.

EPR analysis of Group III AH revealed an intense signal with: Line width: $\Delta H = 7.5$ mT; g-factor: $g = 2.4$. This signal is attributed to substituted aromatic hydrocarbon radicals (Fig. **2**).

Similar aromatic radicals were detected in Groups II–III AH of Zagly oil, as well as in light fractions of West Absheron crude oil.

3.6. West Absheron Heavy Crude Oil

Due to depletion of conventional light and medium crude oil reserves, production of heavy, hard-to-recover oils with elevated viscosity has increased in recent years. Such oils cannot be transported under standard conditions without special treatment. West Absheron crude oil, produced from the Caspian Sea region of Azerbaijan, belongs to this category.

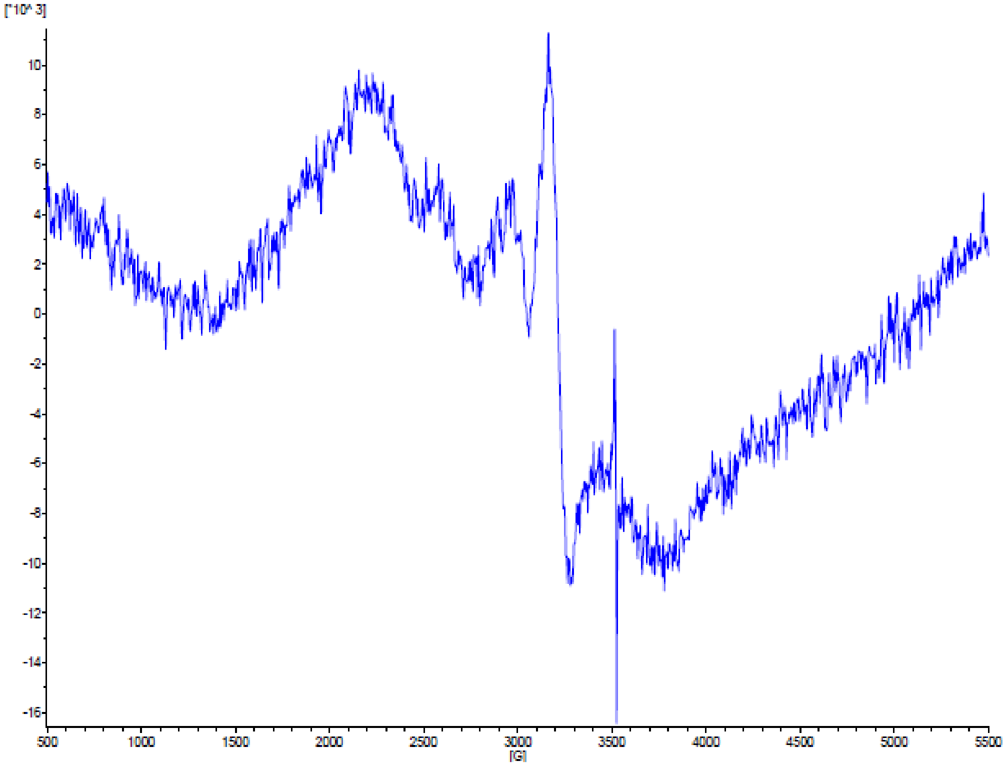


Figure 2: EPR spectrum of substituted aromatic hydrocarbon radical from Zagly crude oil.

Its physicochemical characteristics are: Density at 20°C: 912.5 kg/m^3; Molecular weight (0–200°C fraction): 136; Viscosity at 20°C: 91 mm^2/s; Pour point: –47°C; Solid paraffin: 1.16 wt%; Total sulfur: 0.38 wt% [31-36].

Modern industrial refining focuses on converting petroleum fractions into hydrocarbons that serve as feedstock for aromatic ketones, aldehydes, and acids.

With increasing production and processing of heavy oils such as West Absheron crude, comprehensive investigation of their group hydrocarbon composition and properties has become essential.

For this purpose, adsorption separation of native oil on KSM silica gel was applied, yielding: Methane–naphthene hydrocarbons; Individual groups of aromatic hydrocarbons; asphalt–resin compounds. The composition of West Absheron oil is: saturated (methane–naphthene) hydrocarbons: 42.40 wt%; aromatic hydrocarbons: 44.46 wt%; asphalt–resin compounds: 13.14 wt% [31, 32]. The structural-group composition of the isolated hydrocarbons was studied using IR [27-30] and UV spectroscopy.

After determination of physicochemical characteristics, West Absheron crude oil was subjected to atmospheric–vacuum distillation using an R/R Instrument (EDWARDS, USA), and the corresponding fractions were isolated (Table **5**).

3.7. IR Spectroscopic Analysis

The composition of the obtained fractions was studied by FTIR spectroscopy in the wavenumber range 600–4000 cm^{-1}. Band assignments were performed according to established methodologies. The IR spectra of West Absheron oil fractions exhibit: Deformation vibrations at 1376 and 1456 cm^{-1}; Stretching vibrations at 2852, 2920, and 2950 cm^{-1}.

These bands correspond to C–H bonds in CH_3 and CH_2 groups. Additional bands include: Deformation vibrations at 954 and 1031 cm^{-1} (C–H in naphthenic CH_2 groups); Deformation vibrations at 744, 812, and 873 cm^{-1}; Stretching vibration at 1603 cm^{-1}.

Table 5: Physicochemical characteristics of fractions of West Absheron crude oil.

Oil Fraction	Molecular Weight	Density at 20°C (kg/m³)	Pour Point (°C)	Refractive Index (nD20)	Kinematic Viscosity at 20°C (mm²/s)	Sulfur (wt%)	Yield (wt%)	Luminescence Color
IBP–200°C	136	773.1	—	1.4278	1.02	0.0455	8.84	Dark brown
85–180°C	135	771.9	—	1.4256	1.03	0.0502	5.46	Light yellow
120–230°C	167	810.0	Below –70	1.4440	1.63	0.0644	10.47	Same
150–280°C	198	838.1	Same	1.4605	2.78	0.0941	18.69	Same
140–320°C	218	851.5	Same	1.4688	3.65	0.1239	29.65	Same
140–350°C	232	860.6	Same	1.4740	4.95	0.1576	35.97	Violet–blue
180–350°C	241	866.1	Same	1.4780	6.06	0.1601	39.93	Same

The latter bands are characteristic of C–H and C=C bonds in benzene rings. Notably, in the fractions IBP–200°C, 85–180°C, and 120–230°C, absorption bands corresponding to aromatic C–C and C–H bonds are absent. The IR spectra of these three fractions are practically identical. Similarly, close spectral similarity is observed among the fractions 150–280°C, 140–320°C, 140–350°C, and 180–350°C.

From IR spectral analysis, the following structural parameters were determined: Aromaticity index (D1000/D726): 0.46–0.34; Branching index (D1380/D1465): nearly constant (0.59–0.56); Paraffinity index ((D720 + D1380)/D1604): increases from 6.03 to 10.53; Sulfur-related index (D1030/D1465): increases from 0.01 to 0.08.

In the aromatic triplet region (730–900 cm^{-1}), the intensity of absorption bands remains essentially unchanged. To confirm these observations, the structural-group composition of fractions 120–230°C, 140–350°C, and 180–350°C was further investigated, since these fractions exhibited an EPR signal corresponding to substituted aromatic hydrocarbon radicals [31-34].

3.8. UV–Vis Spectroscopy

Electronic absorption spectra were recorded using a UV/Vis 680 spectrophotometer (JENWAY) operating in the range 190–1100 nm with optical resolution of 0.1 nm. Hexane was used as the solvent. Spectra were measured in diluted solutions (10^{-2}).

The absorption spectra (Fig. **3**) demonstrate that the fractions 120–230°C, 140–350°C, and 180–350°C contain absorption bands characteristic of derivatives of benzene, naphthalene, phenanthrene, and anthracene.

The data presented in Table **6** indicate that all investigated fractions contain benzene derivatives, substituted naphthalenes, phenanthrenes, and anthracenes. Their concentration increases progressively with increasing boiling range. Anthracenes were not detected in the 120–230°C fraction.

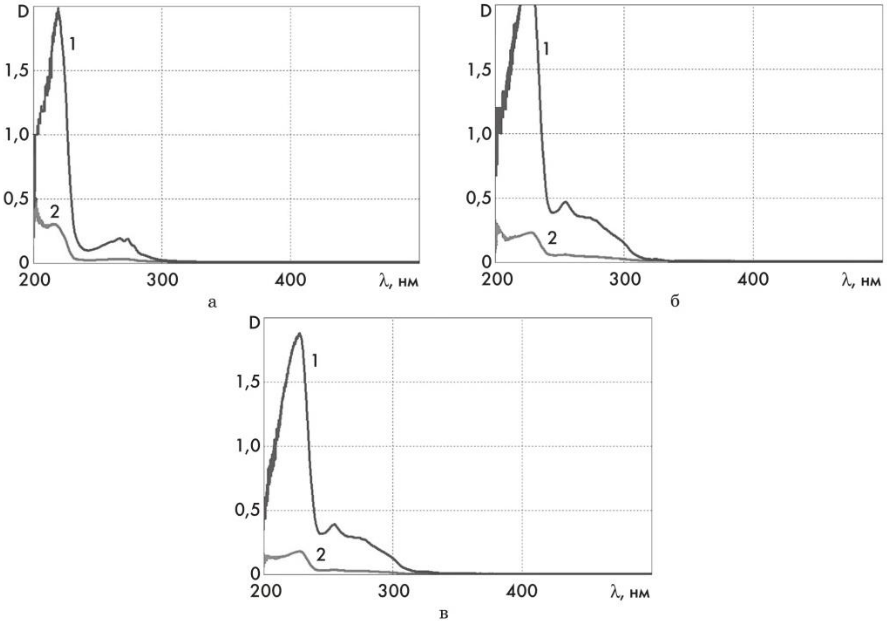
The data presented in Table **7** demonstrate that methane–naphthene hydrocarbons and Groups I–III aromatic hydrocarbons (AH) contain derivatives of benzene, substituted naphthalenes, phenanthrenes, and anthracenes. Their concentration increases progressively with fractionation [34].

3.9. Fractionation and Refractive Analysis

After deasphalting and resin removal (GOST 11858-66) using adsorption chromatography, Groups I–IV aromatic hydrocarbons were isolated and analyzed spectrometrically using an IRF-23 refractometer.

The oil contains: saturated methane–naphthene hydrocarbons: 42.40 wt%; refractive index: 1.4773; aromatic hydrocarbons (total): 44.56 wt%; resin–asphaltene substances (RAS): ~13 wt%; Distribution by groups: Group I AH (14.85 wt%): n_D^{20} = 1.5375; Group II AH (4.34 wt%): n_D^{20} = 1.5375; Group III AH (8.07 wt%): n_D^{20} = 1.5682; Group IV

AH (17.30 wt%): refractive index not reliably determined due to high concentration of asphaltene radicals. The molecular weight increases significantly from methane–naphthene hydrocarbons (254) to Group III AH (1305) [34].



a -fr. (120–230°C) (1 – C_1 = 0.06%; 2 – C_2 = 0.002%);
b -fr. (140–350°C) (1 – C_1 = 0.05%; 2 – C_2 = 0.03%); v -fr.(180–350°C) (1 – C_1 = 0.08%; 2 – C_2 = 0.002%)

Figure 3: Electronic absorption spectra of West Absheron oil fractions in heptane solutions.

Table 6: Content of aromatic hydrocarbons in fractions of West Absheron crude oil (wt%).

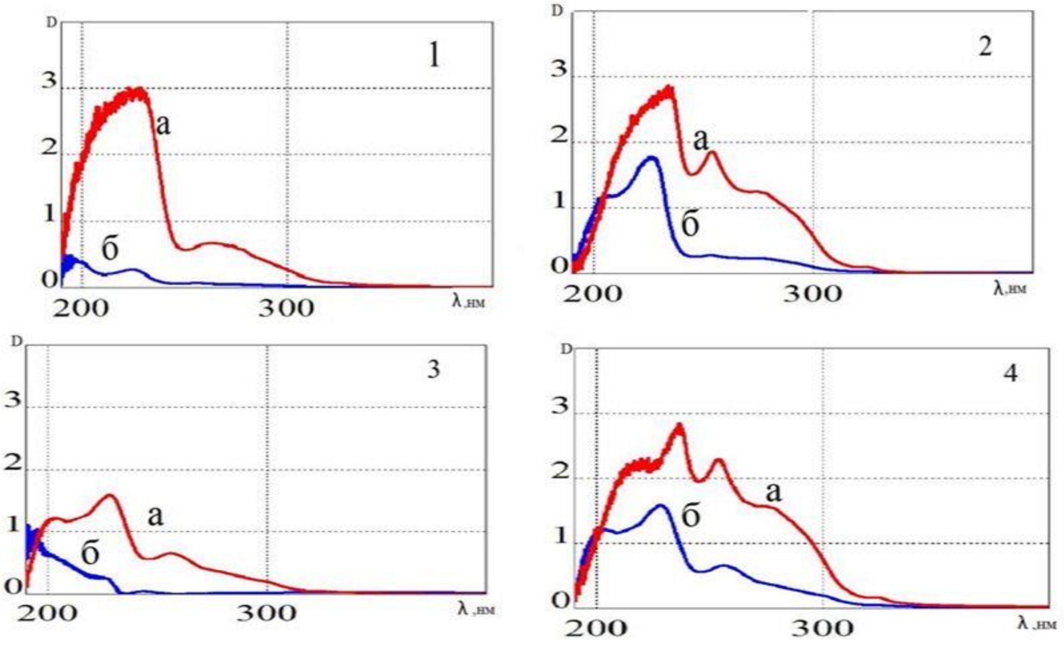
Fraction	Molecular Weight	Benzene Derivatives	Naphthalenes	Phenanthrenes	Anthracenes	Total
120–230°C (C_1=0.06%, C_2=0.002%)	167	5.35	7.82	0.03	—	13.2
140–350°C (C_1=0.05%, C_2=0.003%)	232	7.8	8.5	1.7	1.3	19.3
180–350°C (C_1=0.08%, C_2=0.002%, C_3=0.0002%)	241	8.5	9.3	3.3	1.4	22.5

Table 7: Content of aromatic hydrocarbons in West Absheron crude oil (wt%).

Hydrocarbon Fraction	Benzene Derivatives	Naphthalenes	Phenanthrenes	Anthracenes	Total
Methane–naphthene hydrocarbons	0.5	0.7	0.8	Trace	2.0
Group I AH	13.7	10.2	9.5	0.3	33.7
Group II AH	20.1	17.0	12.1	1.1	50.4
Group III AH	24.0	28.2	17.1	1.3	70.6

3.10. UV–Vis Spectroscopy of Aromatic Groups

In hexane solutions: Group I AH: (a) C_1 = 0.06%; (b) C_2 = 0.002%; Group II AH: (a) C_1 = 0.05%; (b) C_2 = 0.003%; Group III AH: (a) C_1 = 0.09%; (b) C_2 = 0.002%. The UV spectra (Fig. **4**) reveal absorption bands characteristic of: benzene derivatives: 200–215 nm; naphthalenes: 220–230 nm; phenanthrenes: 245–260 nm; anthracenes: 272–290 nm. The quantitative content of these compounds corresponds to the data presented in Table **7** [33-36].



1 – methane–naphthene hydrocarbons; **2** – Group I AH; **3** – Group II AH; **4** – Group III AH

Figure 4: Electronic absorption spectra of aromatic hydrocarbons in Groups I–IV of West Absheron crude oil.

3.11. Luminescence and EPR Behavior of Fractions

Visual observation under a PRK-4 mercury lamp (UVS-1 filter, >200 nm) showed: Fractions IBP–200°C, 85–180°C, and 120–230°C exhibit a slightly yellow luminescence. Their IR spectra lack absorption bands corresponding to aromatic C–C and C–H bonds of benzene rings. Fractions 140–320°C and 150–280°C show weak yellow luminescence typical of resinous compounds. The luminescence of these fractions is likely governed by higher-molecular-weight compounds excited via cascade energy transfer from lower-molecular-weight species [34].

3.12. EPR Observations

In fraction IBP–200°C, the concentration of asphaltene radicals ($R^{\bullet}_{asph}$) ($\Delta H = 0.88$ mT; $g = 2.0023$) decreases relative to the native oil. With further fractionation (starting from 85–180°C), a gradual reduction in $R^{\bullet}_{asph}$ concentration is observed. In fractions 120–230°C and 150–280°C, a distinct EPR line appears: $\Delta H = 7.5$ mT $g = 2.4$. Based on its linewidth, this signal cannot be attributed to metal-complex compounds. It corresponds to a substituted aromatic hydrocarbon radical, previously detected in Zagly crude oil [25]. Thus, these radicals can be confidently assigned to substituted aromatic hydrocarbons.

3.13. Correlation Between Radical Concentration and Luminescence

Fractions 120–230°C and 150–280°C exhibit light yellow luminescence characteristic of resin–asphaltene substances. This indicates that a sufficient concentration of $R^{\bullet}_{asph}$ remains to screen the aromatic hydrocarbons, suppressing their intrinsic luminescence.

When the concentration of $R^{\bullet}_{asph}$ decreases significantly, the higher-boiling fractions (140–350°C and 180–350°C) exhibit blue–violet luminescence characteristic of substituted phenanthrene and anthracene derivatives. IR spectroscopic analysis confirms that these fractions contain comparable concentrations of benzene aromatic rings. Structural-group analysis of fractions 120–230°C, 140–350°C, and 180–350°C further confirmed the presence of substituted aromatic hydrocarbon radicals, as evidenced by doublet EPR lines.

3.14. Balakhany Heavy Crude Oil

Another object of investigation was Balakhany heavy crude oil with the following physicochemical characteristics: Density at 20°C: 919 kg/m³; Molecular weight: 382; Viscosity (20–50°C): 135–25.8 mm²/s; Pour point: 37°C; Coke residue: 2.88%; Acid number: 2.5 mg KOH/g; Resins: 14%; Asphaltenes: 0.80%; Paraffin: 0.62%; Sulfur: 0.30%.

After deasphalting and resin removal (GOST 11858-66) by adsorption chromatography, Groups I–IV aromatic hydrocarbons were isolated and analyzed (IRF-23 refractometer): Group I AH: nD20 = 1.5220; Group II AH: nD20 = 1.5400; Group III AH: nD20 = 1.5770; Group IV AH: nD20 = 1.5808; Density range: 0.9342–1.0050 g/cm³; Molecular weight: 353–580 [35].

3.15. Luminescent Properties of Balakhany Aromatic Hydrocarbons

Upon excitation with unfiltered mercury lamp radiation (PRK-2) using UVS-1 filters (240–400 nm), the AH groups exhibit luminescence of varying color and intensity: Group I AH: no visible luminescence; Group II AH: weak blue luminescence; Groups III–IV AH: intense turquoise and yellow luminescence.

The presence of aromatic hydrocarbons indicates their potential role as photosensitizers of naphthene–paraffin hydrocarbons with radical formation. Particular practical importance is attributed to aromatic hydrocarbons containing three or more condensed benzene rings, which exhibit visible luminescence. In crude oil and petroleum products, aromatic hydrocarbons occur as both unsubstituted ("bare") and substituted derivatives [23, 24]. In primary refining products, aromatic hydrocarbons are predominantly substituted.

3.16. Absheron Heavy Crude Oil and High-Boiling Residues

The increasing heaviness of produced crude oils necessitates fundamentally new approaches to their rational utilization. Development of advanced technologies depends on detailed knowledge of composition, structure, and specific properties of petroleum components.

Absheron crude oil, produced from offshore depths of the Azerbaijani sector of the South Caspian Sea, is heavy (ρ = 920 kg/m³), highly resinous, low in sulfur, and low in paraffin.

According to GOST 2177-99: Yield up to 200°C: 4%; Yield up to 300°C: 26%. In this study, Absheron crude oil and its 50°C residual fractions were investigated. Distillation was performed up to 350°C (ASTM D_{2892}) and up to 500°C (ASTM D_{5236}).

Yields: 350–500°C: 27.62 wt%; Residue >350°C: 63.36 wt%; Residue >500°C: 35.86 wt%. The physicochemical characteristics of the Absheron oil fractions are presented in Table **8**.

Table 8: Physicochemical properties of Absheron oil fractions.

Fraction	Molecular Weight	Density, ρ20 (g/cm³)	Pour Point (°C)	Refractive Index nD20	Kinematic Viscosity (mm²/s)	Viscosity Index (40/100)	Acid Number (mg KOH/g)	Luminescence Color	Yield to Oil (%)
150–200°C	161	0.8033	–	1.4424	1.50 (20°C)	25.50	–	–	5.09
200–260°C	214	0.8498	–	1.4670	3.29 (20°C)	149.87	–	Slight violet	9.28
260–300°C	278	0.8850	–	1.4862	7.55 (20°C)	272.91	–	Violet	7.26
300–350°C	327	0.904	–62	1.4889	2.32 (100°C); 9.43 (40°C)	409.37	3.411	Bluish-violet	11.67
400–450°C	438	0.9322	–28	1.4950	47.09 (100°C); 84.43 (40°C)	–	0.31	Bluish-violet	16.00
450–500°C	506	0.9442	–12	1.5090	428.3 (40°C); 16.98 (90°C)	–	0.32	Turquoise	10.63
>500°C	–	0.9865	22	–	–	–	10.52	–	35.86

3.17. IR Spectroscopic Analysis of 50°C Fractions

The component composition of the 50°C fractions of Absheron oil was studied using Fourier-transform infrared (FTIR) spectroscopy in the range 600–4000 cm^{-1}. Owing to the use of Fourier transformation, IR spectrometry has become a widely accepted method for comprehensive investigations of crude oil composition from various deposits.

Most studies focus on identifying and quantifying hydrocarbon compounds, determining the presence of unbranched paraffinic structures, evaluating the degree of paraffin chain branching, assessing aromaticity, and related structural parameters.

From the obtained IR spectra, spectral coefficients were calculated based on the optical densities of absorption bands corresponding to hydrocarbons, resins, and asphaltenes. These data are presented in Table **9**.

Table 9: Content of hydrocarbons, resins, and asphaltenes in 50°C fractions of Absheron oil.

Product	C_1	C_2	C_3	C_4	A (%)
200–260°C	–	0.10	0.56	23.7	–
260–300°C	0.33	1.13	0.56	12.83	13.9
300–350°C	0.35	0.14	0.57	11.57	17.4
400–450°C	0.40	0.10	0.61	11.0	21.1
450–500°C	0.50	0.09	0.61	10.1	22.3
Resins	1.66	0.31	0.56	2.15	–
Asphaltenes	3.67	0.31	0.58	1.55	–

For comparative analysis of the fractions, the following spectral coefficients were used to characterize their structural-group composition: C_1 (aromaticity) = D1600 / D720; C_2 (oxidation) = D1710 / D1465; C_3 (branching) = D1380 / D1465; C_4 (paraffinity) = (D1380 + D720) / D1600. According to IR spectral data, the fractions show an increase in aromatic structures and a decrease in paraffinic structures with increasing boiling temperature. This trend is evidenced by the increase in the aromaticity index (A) and the decrease in the paraffinity coefficient (C_4).

IR spectra of resins and asphaltenes exhibit strong absorption bands of aromatic structures at 1600 cm^{-1} and in the aromatic triplet region (730–900 cm^{-1}). The increasing intensity of these bands is reflected in the higher aromaticity index of resins and asphaltenes (Table **9**).

An increase in absorption intensity at 1710, 1100–1200, 1030, and 400–500 cm^{-1} with increasing temperature likely indicates destructive oxidative processes accompanied by condensation of aromatic rings.

Thus, as follows from Table **9**, during fractionation the content of naphthenic, paraffinic, and aromatic hydrocarbons decreases, while the aromaticity coefficient increases. These results correlate well with EPR spectroscopy data: the concentration of asphaltene radicals in the residue >500°C increases from 4×10^{17} to 13×10^{17} spins/g [36].

The presence of unbranched paraffinic structures is confirmed by absorption bands at 720 and 1300 cm^{-1}. The degree of branching of paraffinic chains is evaluated from absorption bands at 1380 and 1465 cm^{-1} and characterized by the coefficient β, defined as the ratio of the intensities of characteristic absorption bands of CH_3 and CH_2 groups. The greater this ratio, the higher the degree of branching in paraffinic structures [36, 37].

For aromatic structures, the most informative absorption bands are located at 812–816 cm^{-1} and 1600 cm^{-1}. The higher the ratio of their intensities, the greater the degree of aromatization of the petroleum products.

UV–Vis spectroscopy (200–800 nm) performed using a "Spekord UV-Vis" spectrophotometer revealed the presence of phenanthrene, anthracene, and benzophenanthrene groups (295 nm).

The presence of polycyclic aromatic hydrocarbons (PAHs) in crude oil and its fractions indicates that they can act as photosensitizers in the decomposition of organic compounds such as alcohols and paraffins. Special irradiation is not required, since PAHs effectively absorb visible solar radiation.

The observed decrease in the EPR signal of asphaltene radicals during fractionation enabled the identification of EPR spectra corresponding to certain metal oxide ions. It was established that the EPR spectra of Absheron oil and its fractions mainly consist of: A single narrow signal characteristic of asphaltene radicals $R^{\bullet}_{ASFAL}$ (ΔH = 0.88 mT, g = 2.0023). A broad line attributed to: Iron oxide ions (ΔH = 168.1 mT, g = 2.24); Copper oxide microcrystals (ΔH = 108.9 mT, g = 2.27); Nickel chloride (ΔH = 117.4 mT, g = 2.2).

Due to the very low concentration of certain trace elements in Absheron oil, their EPR spectra fall below the sensitivity limit of the spectrometer and are not detected.

However, in fractions 150–200°C and 200–260°C an EPR signal (ΔH = 25 mT, g = 2.2) appears, becoming clearly distinguishable in the 260–300°C fraction. In heavier residual fractions (300–350°C, 400–450°C, 450–500°C), the concentration of asphaltene radicals increases, while the 25 mT EPR signal decreases in intensity but does not disappear.

According to IR spectral analysis, the 260–300°C fraction shows a decrease in both polyaromatic hydrocarbon concentration and total aromatic hydrocarbon content (Table **9**).

Commercial Absheron oil and its residual 50°C fractions were also investigated using a U.S. distillation apparatus up to 350°C ($ASTM_{2892}$) and up to 500°C ($ASTM_{5236}$). The yield of fractions from initial boiling point to 350°C and from 350–500°C was 36.46 wt% and 27.62 wt%, respectively. The yields of residues above 350°C and 500°C were 63.36 wt% and 35.86 wt%, respectively [38, 39].

Table 10: Physicochemical characteristics of Absheron crude oil and its residual fractions.

Sample Name	Yield to Oil (wt%)	Density at 20°C (kg/m³)	Pour Point (°C)	Coke (wt%)	Sulfur (wt%)	Conditional Viscosity at 30°C	80°C	100°C	Flash Point (°C, Closed Cup)	Flash Point (°C, Open Cup)
Commercial crude oil	–	919.4	–34	3.64	0.31	10.4	6	–	25	–
Residue >350°C (from crude oil)	63.36	959.0	±0	6.08	0.41	–	–	–	–	240
Residue >500°C (from crude oil)	35.86	986.5	22	10.52	0.54	–	–	–	–	316

3.18. EPR Spectroscopic Characteristics

The EPR spectra of the crude oil and its residual fractions consist of a single EPR signal with a linewidth: ΔH = 0.88 mT (for native commercial Absheron crude oil); ΔH = 0.64 mT (for fractions >350°C and >500°C). These signals are characteristic of asphaltene radicals. The calculated absolute concentrations and other parameters are presented in Table **10**.

The observed difference in EPR linewidths is attributed to structural variations. This is likely associated with the more compact structure of asphaltene radicals (molecules) subjected to thermal treatment (>350°C and >500°C), which are characterized by: Lower content of aliphatic substituents (K_1, K_4), Higher degree of condensed aromatic core development (K_1).

3.19. Practical Implications

The obtained spectroscopic results for Absheron oil and its residual fractions (>350°C and >500°C) indicate their potential rational use as binders in asphalt concrete for road construction.

However, as noted by other authors, preliminary investigation of the paramagnetic properties of mineral aggregates (stone and sand) is required. If the paramagnetism of the mineral material is comparable to that of the petroleum binder and the bitumen derived from it, good compatibility between materials is ensured [40-42]. Conversely, high paramagnetism of the mineral component may cause incompatibility effects, leading to premature aging of asphalt concrete.

3.20. General Considerations on Heavy Oil Residues

Despite the large number of studies devoted to high-boiling petroleum fractions and secondary processing products—on which many important petroleum products are based—primary processing products and heavy petroleum residues (HPR) remain insufficiently studied.

Heavy residues from primary processing offer several advantages: Lower content of aromatic hydrocarbons; Reduced environmental impact; Large raw material resources. Therefore, investigation of the 50°C fractions of Absheron oil and its residue (>500°C) is of significant importance.

4. Conclusion

Under the influence of ionizing radiation, excited molecules of polycyclic aromatic hydrocarbons (PAHs)—including naphthalene, phenanthrene, chrysene, benzophenanthrene, pyrene, and perylene groups (with a total content of approximately 40 wt%)—interact with organic compounds such as alcohols and paraffins [21, 43, 44].

These PAHs, known initiators of photochemical processes, act as photosensitizers. Through two-photon absorption, PAH molecules are transferred to a highly excited triplet state PAH* (T*).

Due to their relatively long triplet lifetimes (2.5–3 s), aromatic molecules can absorb an additional photon ($h\nu_2$), reaching a higher excited triplet level. The doubly photoactivated PAH molecule can then transfer energy to neighboring alcohol and paraffin molecules, leading to cleavage of valence bonds and formation of alkyl radicals ($R^{\cdot}$) and atomic hydrogen according to the scheme [43, 44]:

$$\text{PAH (S)} \rightarrow(h\nu_1)\ \text{PAH* (S*)} \rightarrow \text{PAH* (T)} \rightarrow(h\nu_2)\ \text{PAH* (T*)} + \text{R} \rightarrow \text{PAH (S)} + \text{PAH (S)} + \text{R}\bullet + \text{H} \tag{1}$$

where S, S* are the ground and excited singlet states, and T, T* are the triplet states of PAH molecules.

Atomic hydrogen reacts with hydrocarbons according to: $RH + H \rightarrow R^{\cdot} + H_2$ forming additional radicals and molecular hydrogen. The radicals $R^{\cdot}$ readily react with O_2, forming peroxide radicals.

4.1. Alternative Single-Quantum Mechanism

In petroleum systems containing resins and asphaltenes, photosensitized decomposition may also proceed via a single-quantum [21] mechanism using relatively low-energy photons (~2 eV):

$$R^{\cdot}\ \text{(resin/asphaltene)} + RH \rightarrow(h\nu)\ R^{*} + RH \rightarrow R^{*}H + R\bullet \rightarrow R\bullet + R^{\cdot} + H$$

Highly reactive and mobile atomic hydrogen migrates toward unpaired carbon electrons, promoting deactivation of the asphaltene molecular core and resulting in increased yield of light petroleum fractions. The presence of free radicals in resins and their photoexcited states under solar irradiation may significantly influence photochemical processes in petroleum systems. Long-wavelength optical irradiation activates otherwise stable radicals, facilitating cleavage of C–C and C–H bonds with formation of alkyl radicals and atomic hydrogen. Analysis of the obtained data suggests that, for the investigated light commercial crude oil, removal of resins and asphaltenes during fractionation may be beneficial and timely from the standpoint of photochemical stability and processing efficiency.

Conflict of Interest

The authors declare that there are no conflicts of interest regarding the publication of this paper.

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