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Compositional analysis of oil C36+ using gas chromatography

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The educational-methodical manual for undergraduate and graduate students of the full-time form of training direction 21.03.01 and 21.04.01 Petroleum Engineering in the profile "Development of oil and gas fields". The training of specialists in vocational education institutions should meet the requirements of employers. An important component is to gain skills in determining the component composition of oil for subsequent numerical simulation of the composition of the reservoir fluid and familiarization of students with the gas chromatography.

List of Contents

Terms and definitions *

Carrier gas – a gaseous or vaporized substance that flows through a sorbent layer to transport the substances to be identified

Gas chromatography – a chromatography in which the mobile phase is in a gas or vapor state

Capillary column – a gas chromatography column in which the walls of the column and a liquid or solid applied to the column walls act as a stationary phase

Internal standard method – a method for calculating concentrations of components of the sample to be determined by adding a specific amount of a known compound (internal standard) to the sample and determining its concentration as a well-resolved peak on a chromatogram

Non-volatile residue – the unrecorded components of a C36+ oil sample

Chromatogram zero line – the section of a chromatogram representing the differential detector signal recording during the exit of pure carrier gas from the column

Chromatographic peak – the section of the chromatogram accompanying the release of a sample component from the column

Flame ionization detector (FID) – an ionization gas chromatography detector in which the ionization source is a flame and the saturation current is measured

Peak area – is the area bounded by the contour of the peak and its base

The corrected area of the sample chromatogram – is the difference between the areas of the sample and blank chromatograms

Theoretical total chromatogram area – the area of the chromatogram that would be obtained by eluting the whole sample from the column

Chromatogram – the graphical representation of the signal from a gas chromatograph detector as a function of time

Peak width – is the length of the baseline segment bounded by two tangents drawn through the inflection point of each side of the chromatographic peak

* Terms and definitions are given according to [1,2].

Symbols

ν – the molal quantity

φ – the mole fraction

$\mathbf{S_{C_n}}$ – the peak area of n-alkane with number of carbon atoms n

$\mathbf{m_{C_n}}$ – the mass of n-alkane sample with the number of carbon atoms n

$\mathbf{m_{CS_2}}$ – the mass of carbon disulfide

A – the sum of the areas of all the peaks in a chromatogram of an oil sample with a standard internal

AIS – the area on chromatogram of oil sample with internal standard iso-C_{16} with the peak of standard internal

B – the sum of the areas of all peaks on a chromatogram of an oil sample without an internal standard to the C_{35} component

BIS – the area on a chromatogram of an oil sample without internal standard corresponding to the position of the internal standard peak

$\mathbf{K_{mass.i}}$ – the mass sensitivity factor of the i-component of the mixture

$\mathbf{K_{mass.i.rel}}$ – the relative mass sensitivity factor of the i-component for n-hexane

$\mathbf{K_{mass.n\text{-}hexane}}$ – the absolute coefficient of mass sensitivity of n-hexane

Mr – the molar mass of a component

$\mathbf{m_o}$ – the mass of oil sample

$\mathbf{m_{st}}$ – the mass of internal standard

$\mathbf{S_i}$ – the peak area of the i-component calculated on a chromatogram without internal standard

$\mathbf{S_{i\text{-}st}}$ – the peak area of the component of oil, calculated on a chromatogram of oil with standard internal

$\mathbf{S_T}$ – total theoretical area of a chromatogram

$\mathbf{X_{Cn}}$ – the mass fraction of n-alkanes in the mixture

$\mathbf{X_i}$ – the mass fraction of oil components ignoring non-volatile residue

ω – the mass fraction of oil components including non-volatile residue

$\mathbf{X_{st}}$ – the mass fraction of an internal standard in the mixture

z – the ratio of the sum of the areas of hydrocarbon peaks on chromatograms obtained when analyzing oil with and without adding the internal standard

1. Summary of test method

The oil sample is analyzed on a gas chromatograph with a flame ionization detector (FID) in the column thermostat temperature programming mode to determine the component composition. The fraction of fixed residuals is determined by performing additional analysis of the sample with the addition of an internal standard. This method proposes using isohexadecane (hereinafter referred to as iso-C16) as the internal standard, ACROS, quality level 98%.

2. Measurement conditions

– ambient temperature (20±5) °C;

– relative humidity from 30% to 80%;

– atmospheric pressure from 80.0 to 106.7 kPa (630 to 800 mm Hg);

– AC voltage ($220 \pm \frac{22}{33}$) V;

– AC frequency (50±1) Hz;

– no mechanical influences, external electric and magnetic fields affecting the operation of the apparatus;

– absence of aggressive gases and vapors.

3. Measuring instruments, auxiliary devices, materials and reagents

Measuring instruments and accessories

1) Gas chromatograph including:

– control unit;

– a FID with a detection limit of $2 \cdot 10^{-12}$g/s (by carbon) and capable of operating continuously at the maximum temperature of the column to be used (the detector must be connected to the column so that there are no cold spots between them);

– a sample injection system that is isothermal at the maximum temperature of the column or temperature-programmable heating, or that introduces the sample directly into the column;

– a thermostat that provides programmable regulation of temperature rise rate and maintains the set temperature with an error not exceeding 0.1°C over the whole operating temperature range;

– a cooling system for the column thermostat (for samples with an initial boiling point below 90°C);

– software to acquire process and store chromatographic information.

The software package used shall provide:

– conversion of a continuously integrated detector signal into areas of narrow sections of a chromatogram no wider than 0.1 s;

– subtract the area of the narrow section of the zero line from the area of the corresponding section of the chromatogram of the sample being analyzed.

2) Capillary chromatographic columns:

– DB-1 - 30 m length, 0.53 mm inner diameter, 0.25 μm film thickness liquid stationary phase (polydimethylsiloxane);

– TG-BOND Q - 30 m length, 0.32 mm inner diameter, 10 μm film thickness liquid stationary phase (divinylbenzene).

3) Laboratory balance (according to GOST R 53228) of I accuracy class (maximum load - 210 g, minimum load - 0,01 g, discontinuity of counting d = 0,1 mg, calibration range e = 1 mg).

4) Micro-syringe made by SGE, Hamilton, or analogue with a capacity of 10 μL.

5) Gas-tight syringe made by Agilent, Hamilton, or similar types of glass and PTFE piston, 1 ml capacity.

6) Medical syringes according to GOST ISO 8537-1 with a capacity of 1 ml.

7) 20 ml glass vials with screw-on caps with two-layer septa (silicone/PTFE).

8) 2 ml glass vials with screw-on caps with double layer septa (silicone/PTFE).

Materials and reagents

- helium gaseous purified grade A with a helium content of at least 99.99%;
- hydrogen generator with at least 99.99% purity;
- air (compressor) of grade 0 (hydrocarbon-free) according to GOST 17433;
- n-pentane, analytical reagent quality;
- n-hexane, analytical reagent quality;
- n-heptane, analytical reagent quality;
- n-octane, analytical reagent quality;
- n-nonane, analytical reagent quality;
- n-decane, analytical reagent quality;
- n-decane, analytical reagent quality;
- n-undecane, analytical reagent quality;
- n-dodecane, analytical reagent quality;
- iso-hexadecane, analytical reagent quality;
- n-hexadecane, analytical reagent quality;
- eicosan, tech. quality (≤97%);
- tetracosan, tech. quality (≤97%);
- dotriacontan, tech. quality (≤97%);
- hexatriacontane, tech. quality (≤97%);
- certified mixture of C1-C5 hydrocarbon gases (GSO №10540-2014);
- carbon disulfide with a basic substance content of not less than 99%.

4. Preparation for measurement

Switch on the compressor for the air supply to the FID, check the operating air pressure. Open the helium cylinder and check the working pressure. Turn on the hydrogen generator and wait until it is in operation. Start-up the gas chromatograph. Check for leaks and set up according to chromatograph operating manual.

Determine operating method and record zero line (Table 1).

Table 1

Measurement conditions

Parameter Value	Value	
	For composition C5-C36+	For composition C1-C5
Capillary column	DB-1	TG-BOND Q
Column length, m	30	30
Column diameter (inside diameter), mm	0.53	0.32
Liquid stationary phase of capillary column	Polydimethylsiloxane	Divinylbenzene
Liquid stationary phase film thickness, μm	0,25	10
Detector	FID	FID
Carrier gas flow rate (helium), cm^3/min	10	10
Volume flow rate of propellant gas through detector, cm^3/min	20	20
Hydrogen flow rate, cm^3/min	30	30
Air flow rate, cm^3/min	300	300
Start temperature of column thermostat (hold time 1 to 2 min), °C	35	50

Column temperature controller heating rate, °C/min	15-20	10
End temperature of column (holding time 6 min), °C	340-350	260
Evaporator temperature, °C	320-340	280
Detector temperature, °C	340-350	260
Sample dilution (reference material) with carbon disulphide	1:1	-
Sample volume injected, μL	0,2	250
Sample flow divider (Split ratio)	1:40	1:30

5. Conduct of measurements

5.1. Procedure of measurements

Start a temperature program of column heating oven after entering a calibration mixture of analyzed sample into the chromatograph or during a blank test. Measurement conditions (table 1) and their duration during a blank experiment, chromatograph calibration, and sample analysis should be the same.

5.2. Blank experiment

After the chromatograph reaches the operating regime, record the zero line under the conditions given in Table 1. A blank test should be conducted to account for the effect of an increase in the baseline near the maximum column temperature. Factors affecting the stability of the baseline: the chromatography column affect, accuracy of detector temperature control, degree of stability of carrier and gas flows feeding the detector, leaks, instrumental drift, etc.

A blank test allows obtaining additional information about the operation of the equipment and controlling the residual content in the system of the components of the previous sample. A blank test should be performed before the analysis of the calibration mixture and samples, but it is also recommended to carry it out at the end of a series of analyzes.

Before performing calculations, subtract the chromatogram of the blank experiment from the chromatogram of the sample or the calibration mixture.

5.3. Chromatograph calibration

Use an accurately weighed mixture to determine mass sensitivity factors of n-alkanes from C_5 to C_{36}, dissolved in carbon disulfide. It is allowed to use a calibration mixture ASTM® D2887.

Calibration mixture of individual n-alkanes from C5 to C36 and iso-C16 in carbon disulfide with a mass fraction of each component from 0,01% to 0,1% and do it as follow. Alternately, add from 30 mg to 80 mg of solid n-alkanes (C20, C24, C32, C36) in the vial (size 20 cm^3), which has a screw cap with a hole and it should be equipped with a septum. Weigh the vial before and after adding each component and write the results in grams accurate to 0.0001 g. Then, add carbon disulfide (10 to 15 cm^3) by

pipette; the vial is leakless closed and weighed. Next, inject 130-280 mg of liquid n-alkanes (C_5, C_6, C_7, C_8, C_9, C_{10}, C_{11}, C_{12}, C_{16}) and iso-C_{16} in the vial by syringe through the septum in the cap. By the difference between the results of two sequence weighing operations, it is necessary to calculate the mass of each n-alkane and its mass fraction in the mixture X_{Cn} (%) using the formula (1):

$$X_{Cn}=\frac{m_{C_n}}{\sum m_{C_n}+m_{CS_2}}*100, \quad (1)$$

where

m_{C_n} – sample weight of n-alkane with the number of carbon atom n, g;

m_{CS_2} – mass of carbon disulfide, g.

If it is necessary to analyze the aromatic compounds (toluene, ethylbenzene, ortho-/ meta-/ paraxylol) it is required to add them to the calibration mixture.

After preparing the calibration mixture, switch the chromatograph to the operating mode (table 1) and put the sample (0.2 μL) properly into the chromatograph using a micro-syringe.

Using the chromatogram of a mixture of n-alkanes, determine the values of their retention time which is equal to the registration time of maximum peak of individual n-alkane.

The obtained values of retention time, mass components and peak area are written in the table 2, and account for mass sensitivity coefficients $K_{mass.i}$ and relative mass sensitivity factors for components $K_{mass.i\ rel}$ on n-hexane using formula (2):

$$K_{mass.i}=\frac{m_{C_n}}{S_{C_n}}, \quad (2)$$

where

S_{C_n} – peak area of n-alkane with number of carbon atoms n.

Relative mass sensitivity factors for components (n-hexane) accounted by formula (3):

$$К_{mass.i.rel}=\frac{К_{mass.i}}{К_{mass.\ n-hexane}}, \quad (3)$$

where

$К_{mass.n\text{-}hexane}$ – the absolute coefficient of mass sensitivity of n-hexane.

Table 2

An example of filling in data based on the results of chromatographic measurements of a calibration mixture of n-alkanes

Component	Retention time, min	Peak area S_{C_n}, units	Mass m_{C_n}, g	Relative mass sensitivity factors $К_{.mass.i.rel.}$ on n-hexane*
C_5				
C_6				1,000
C_7				
…				
C_{36}				

* *$К_{mass.i.rel}$ should be 0,98 -1,05. The result should be considered acceptable if there is some gradual deterioration in the convergence of the sensitivity coefficients of the component C_5 and heavier components after n-C_{20}.*

Additionally, we use a certified mixture of C1-C5 hydrocarbon gases to determine the retention time of lower-boiling oil components for calibration. We use a capillary column TG-BOND Q according to Table 1 for light hydrocarbons. To calculate the content of light hydrocarbons C1-C5, we use the values of the relative mass sensitivity coefficients given in Table 3.

Table 3

Relative mass sensitivity factors (on n-hexane) (Carrier gas – helium)

Component	Relative mass sensitivity factors $K_{mass.i.rel.}$ on n-hexane
C_1	1,114
C_2	1,047
C_3	1,023
iso-C_4, n-C_4	1,012
iso-C_5, n-C_5	1,005
C_6	1,000

6. Preparation of oil sample

6.1. Sample preparation with internal standard

Weigh a vial (2 ml) cap and septum, add 1 ml of oil sample, close and weigh again. To add an internal standard, add 8 mg of iso-C16 through the septum using a syringe is needed. Then, the vial should be weighed and write all results accurately up to the fourth decimal place. Determine the mass of the oil sample and the internal standard by the difference in the mass of the vial before and after their addition. Contents of the vial intensely mix for 20 minutes until the sample is completely dissolved.

Samples with a low viscosity at room temperature can be added into the chromatograph without carbon disulfide. Samples of viscous or paraffinic oil products should be diluted with carbon disulfide; the degree of dilution and the volume of the sample solution added into the chromatograph should be according to Table 1.

6.2. Sample preparation without internal standard

Add 1 ml of oil sample in the vial (2 ml) using a syringe. Dilute the mixture with an equal volume of carbon disulfide. Then, close the vial tightly by a cap with a septum. Stir intensely for 10 minutes until the sample is completely dissolved.

7. Analysis of the oil sample

Add an oil sample (0,2 μL) with an internal standard into the chromatograph evaporator using a micro-syringe and perform the analysis under the same conditions, which were done with the blank experiment and the study of the calibration mixture (Table 1). Then, with a micro-syringe, inject an oil sample without an internal standard into the chromatograph evaporator and carry out the analysis under the same conditions. It is allowed to analyze an oil sample with and without an internal standard in any sequence. Typical chromatograms of oil samples with and without an internal standard are shown in Figures 1 and 2.

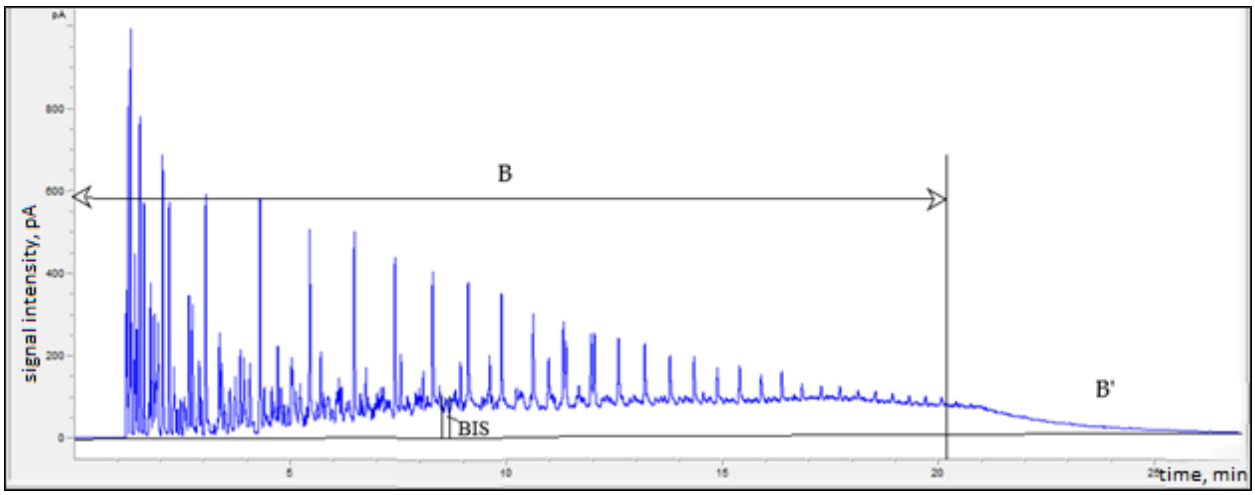

Figure 1 – Chromatograms of oil samples without an internal standard

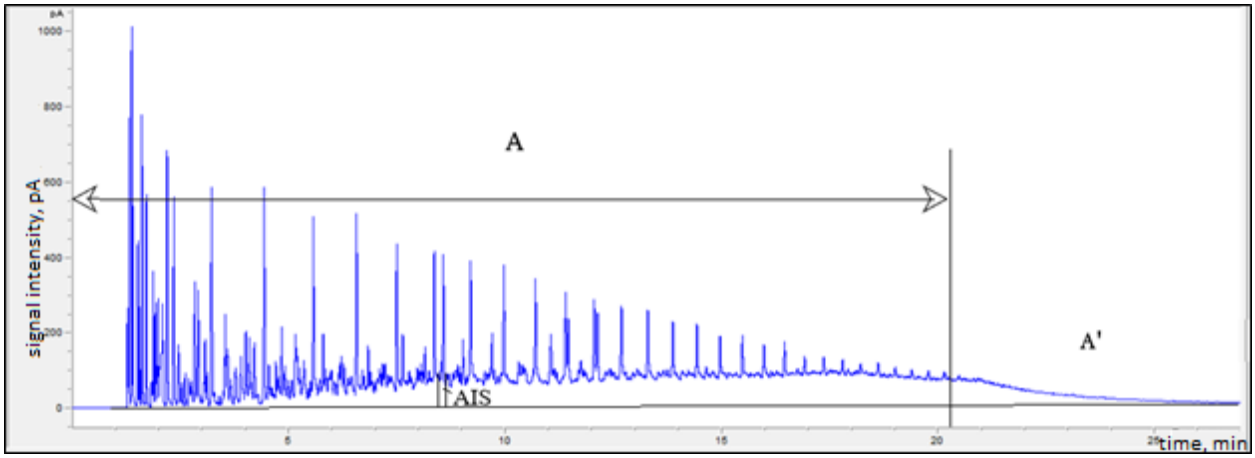

Figure 2 – Chromatograms of oil samples with an internal standard iso-C_{16}

8. Processing of results

Calculate the corrected area of the chromatogram of the sample (further referred to as the total corrected area) from the difference in the chromatograms areas of the analyzed sample and the blank experiment. For this, it is necessary that the times of the above chromatograms were the same.

On the obtained chromatograms, identify the peaks by comparing their retention times with the retention times obtained from the analysis of the calibration mixture. Calculate the peak areas after identification.

Select the standard internal component (iso-C16) by using the retention times obtained for the calibration mixture. Next, determine the total area on the chromatogram of the analyzed sample with an internal standard (AIS area, Figure 2). Also determine the corresponding total area on the oil sample chromatogram without an internal standard (BIS area, Figure 1).

Mass fraction of the internal standard in the mixture Xst, calculated by the formula (4):

$$X_{st} = \frac{m_{st}}{m_o + m_{st}}, \quad (4)$$

where

m_{st} – mass of the internal standard, g;

m_o – mass of oil sample, g.

The ratio of the sum of the peak areas of all hydrocarbons on two chromatograms up to the retention time of the corresponding component C_{35}, obtained in the analysis of oil samples with and without adding an internal standard, minus the areas with the peaks of the internal standard Z, is calculated by the formula (5):

$$z = \frac{B - BIS}{A - AIS}, \quad (5)$$

where

B – the sum of the areas of all peaks in the chromatogram (Figure 1) to the component C_{35}, account units;

BIS – area on the chromatogram (Figure 1) corresponding to the position of the peak of the internal standard, account units;

A – the sum of the areas of all peaks in the chromatogram (Figure 2) up to C_{35}, account units;

AIS – area on the chromatogram (Figure 2) with the peak of the internal standard, account units.

The theoretical total area ST of the chromatogram of the oil sample, taking into account the unrecorded components of the non-volatile residue, is calculated by the formula (6):

$$S_T = (AIS*Z - BIS)\frac{1-X_{st}}{X_{st}} \quad (6)$$

Mass fraction of non-volatile residue RES (C_{36+}) account by using formula (7):

$$RES = 100 - (\frac{B}{S_T} * 100) \quad (7)$$

The total content of all hydrocarbons recorded in the chromatogram is assumed to be 100%.

Light components of the mixture: determine the areas of the components 'peaks up to n-pentane obtained on the TG-BOND Q column (methane, ethane, propane, iso-butane, n-butane, iso-pentane, n-pentane). To calculate the content of individual hydrocarbons use the values of the mass sensitivity coefficients (Table 2). The data on the areas of the peaks obtained on two columns is calculated according to the areas of the peak of n-pentane. For hydrocarbons with a higher number of carbon atoms, the area is calculated as the sum of the peak of an n-alkane and the peaks between this n-alkane and an n-alkane with one less carbon atom (according to ASTM D2887).

Calculate the mass fraction of oil components C_1 - C_{35} X_i (%) by normalizing the peak areas of all hydrocarbons on the chromatogram of an oil sample by using the formula (8):

$$X_i = \frac{S_i * K_{mass.i.rel.} * 100}{\sum(S_i * K_{mass.i.rel})}, \quad (8)$$

where

S_i – peak area of the i-component, calculated from the chromatogram without an internal standard (Figure 1), account units;

The obtained values of mass fractions are corrected to take into account the mass fraction of non-volatile residue ω (%) according to the formula (9):

$$\omega = \frac{X_i\ (100 - RES)}{100} \quad (9)$$

The obtained results are presented in the form of Table 4.

Table 4

An example for filling the calculated values of the mass (ω) and a molar fraction (φ) of oil components

Compo-nent	**(A) Mr, g/mol**	**S_i, units**	**(B) S_{i-st}, units**	**$K_{mass.i.rel}$**	**X_i, %**	**ω, %**	**$S_i * K_{mass.i.rel}$**	**(C) ν, mol**	**(D) φ, %**
C_1	-	-	-	-	-	-	-	-	-
…									
(E)C_{36+}									

(A)Mr– take the molar mass of the oil component from the literature [3];

(B)S_{i-st} – the area of the peak of the oil component, calculated from the oil chromatogram with the internal standard, units of account;

(C)ν- the amount of the substance of the oil component, taking into account the content of non-volatile residue, mol;

(D) φ– mole fraction of oil component, taking into account the content of non-volatile residue, %.

(E) – a molar mass of heavy residue Mr_{C36+} *account by using formula* $Mr(oil) = \frac{\omega_{C1}+\omega_{C2}+\cdots+\omega_{C36+}}{\frac{\omega_{C1}}{Mr_1}+\frac{\omega_{C2}}{Mr_2}+\cdots+\frac{\omega_{C36+}}{Mr_{36+}}}$ *,*

where $\omega_{C1}, \omega_{C2}, \ldots, \omega_{C36+}$*–mass fractions of the components of the mixture. The molar mass of oil Mr is determined experimentally by the cryoscopic method.*

According to Table 4 construct the molecular mass distribution of the oil component in the form of a histogram (the ordinate is the mole fraction, the abscissa is the component).

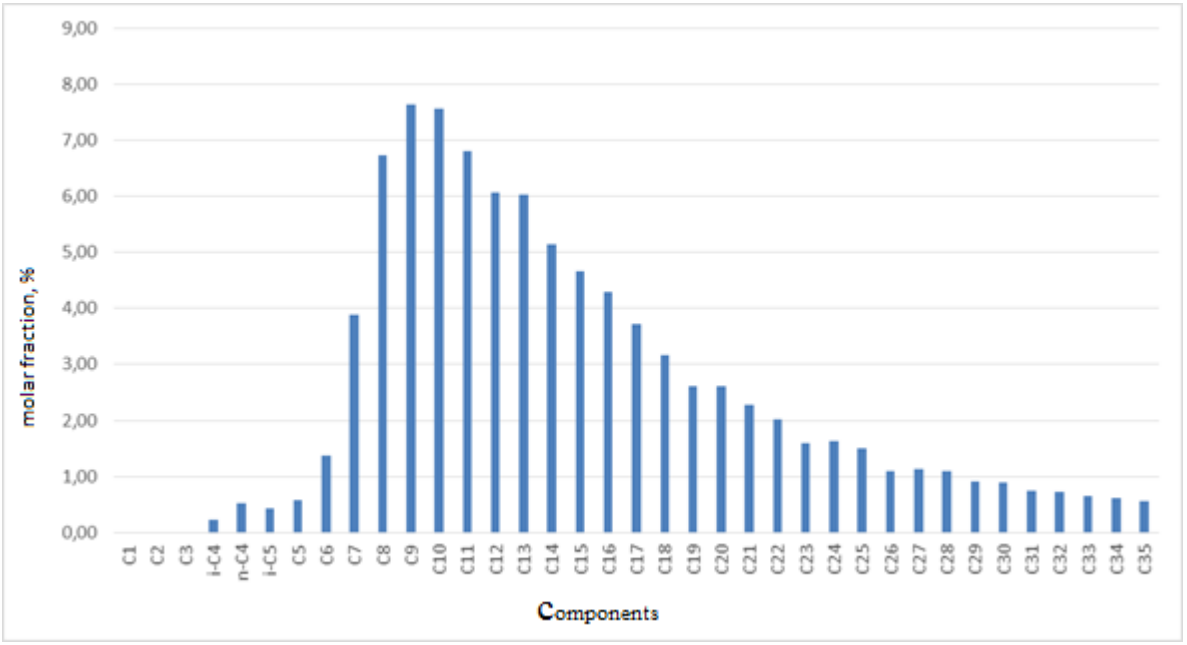

Figure 3 – An example of the molecular mass distribution of oil components

Practical part

Laboratory work is carried out in teams formed by the teacher after studying the theoretical material.

The teacher gives a task to each group, in which the chromatographic analysis of the oil sample will be performed. The results of the component composition of the oil C_{36+} are provided in the form of a report.

Final protocol:

Components	% mass.	% mol.	Mr, g/mol
Methane	0,003	0,045	16,040
Ethane	0,008	0,059	30,070
Propane	0,098	0,520	44,100
iso-Butane	0,063	0,255	58,120
n-Butane	0,324	1,308	58,120
iso-Pentane	0,315	1,024	72,150
n-Pentane	0,575	1,872	72,150
Hexane (C6)	1,372	3,833	84,000
Heptane (C7)	2,856	6,253	107,230
Octane (C8)	3,597	7,359	114,720
Nonane (C9)	3,555	6,620	126,070
Decane (C10)	3,449	6,041	134,000
Hendecane (C11)	3,368	5,379	147,000
Dodecane (C12)	3,304	4,817	161,000
Tridecane (C13)	3,268	4,384	175,000
Tetradecane (C14)	3,207	3,962	190,000
Pentadecane(C15)	3,146	3,584	206,000
Hexadecans (C16)	3,038	3,212	222,000
Heptadecane (C17)	3,004	2,975	237,000
Octadecane (C18)	2,880	2,693	251,000

Nonadecane (C19)	2,757	2,460	263,000
Ecoisane (C20)	2,691	2,297	275,000
Heneicosane (C21)	2,563	2,068	291,000
Docosane (C22)	2,492	1,918	305,000
Tricosane (C23)	2,353	1,737	318,000
Tetracosane (C24)	2,224	1,577	331,000
Pentacosane (C25)	2,103	1,431	345,000
Hexacosane (C26)	2,019	1,320	359,000
Heptacosane (C27)	1,945	1,221	374,000
Octacosane (C28)	1,871	1,132	388,000
Nonacosane (C29)	1,775	1,036	402,000
Triacontane (C30)	1,691	0,954	416,000
Hentriacontane (C31)	1,587	0,867	430,000
Dotriacontane (C32)	1,461	0,772	444,000
Tritriacontane (C33)	1,396	0,715	458,000
Tetratriacontane (C34)	1,298	0,645	472,000
Pentatriacontane (C35)	1,176	0,568	486,000
Hexatriacontane (C36+)	25,173	11,087	533,000
Total	100,000	100,000	
C5+	99,505	97,814	
C6+	98,615	94,918	
C7+	97,243	91,085	
Molecular mass of oil			235

Review questions

1) What is gas chromatography?
2) What is a chromatogram?
3) What is chromatography peak?
4) What is a peak area?
5) What is a peak width?
6) What is a corrected chromatogram area of sample?
7) What is an internal standard method?
8) What is a non-volatile residue?
9) What is a theoretical total chromatogram area?
10) What is a stationary liquid phase?
11) What is a flame ionization detector (FID)?
12) How to determine the mass fraction of an internal standard X_{st}?
13) Methodology for conducting chromatographic analysis?
14) Measurement conditions?
15) How to prepare a calibration mixture?
16) How to prepare an oil sample with and without an internal standard?
17) How to calculate the theoretical total area ST of an oil sample chromatogram?
18) How to calculate the mass fraction of non-volatile residue RES?

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