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Compositional analysis of associated petroleum gas

using gas chromatography

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This educational-methodical manual is compiled in accordance with the program of practical classes of the course "Physical chemistry of hydrocarbons" for full-time undergraduates of the direction 21.04.01 Petroleum Engineering. An important component of the course is learning to determine the component composition of associated petroleum gas, for subsequent numerical modeling of the composition of the reservoir fluid and familiarizing students with the gas chromatography method.

The educational-methodical manual provides a methodology for analysis with the processing of the results, calculation of the molar mass of the mixture and recalculation for an airless mixture.

This manual will be useful for undergraduates when writing graduation, scientific qualification and research papers.

Considered competencies in the course of work:

The ability to search, critically analyze and synthesize information, apply a systematic approach to solving tasks.

Is able to solve production and/or research tasks based on fundamental knowledge in the oil and gas industry.

Is able to find and process information required for decision-making in scientific research and in practical technical activities.

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Terms and definitions *

Sample – substance or mixture of substances introduced into a column in one chromatographic cycle

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

Packed column – gas chromatographic column filled with sorbent

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

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

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

Gas chromatographic detector – a conversion element of a gas chromatographic detection system, in which the change in the composition of the gaseous mixture passing through it is converted into a change in the output signal

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

Gas chromatographic thermal conductivity detector (TCD) – gas chromatographic detector, the output signal of which functionally depends on the difference in thermal conductivity of the analyte and carrier gas

* Terms and definitions are given according to [1-3].

Abbreviations

GOST - state standard;

FID - flame ionization detector;

APG - associated petroleum gas;

TCD - thermal conductivity detector;

MS - measurement system

Symbols

$x_{N_2,c}$ — nitrogen molar fraction value after mole fraction correction due to air pollution, %;

x_{N_2} — nitrogen molar fraction value in the sample after normalization, %;

x_{O_2} — oxygen molar fraction value in the sample after normalization, %;

$x_{j,c}$ — normalized molar fraction value of the j-th component in the sample, %;

$x_{j,s}$ — unnormalized molar fraction value of the j-th component in the sample, %;

n — total number of components;

$(n - 2)$ — total number of components excluding oxygen and nitrogen;

M_c — molar mass of the mixture, g/mol;

M_j — molar mass of the j- th component;

w_j — mass fraction of the j-th component, % mass.

1. Summary of test method

To determine the mole fraction of associated petroleum gas (APG) components, the sample is analyzed on a gas chromatograph with a flame ionization detector (FID) and thermal conductivity detectors (TCD) in the column thermostat temperature programming mode.

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;

3. Measuring instruments, auxiliary devices, materials and reagents

3.1 Basic measuring instruments

3.1.1 Laboratory gas chromatographs

General characteristics:

Chromatographs must be equipped with:

a) control panel;

b) columns (packed and capillary) made of a material inert with respect to APG components and filled with a sorbent that ensures a satisfactory separation of specific components of a gas sample under the conditions of analysis. To separate hydrocarbon gases a DB-1 column is used, carbon dioxide and hydrogen sulfide are retained and separated on the HayeSep R column, oxygen and nitrogen are separated on the NaX column (2m*3mm), helium, hydrogen are separated on the NaX column (3m*2mm).

c) set of detectors. A set of detectors should be formed from one or more thermal conductivity detectors or a combination of one or more TCD and a flame ionization detector FID, the sensitivity of which ensures a signal/noise ratio in the operating measurement range of at least 10;

Detection limits of TCD and FID detectors must comply with the requirements of GOST 26703;

d) a column thermostat that provides programmable control of the rate of temperature rise and maintenance of the set temperature with a tolerance limit of not more than 0.5 °C in the entire range of operating temperatures;

e) sample loop of automatic or non-automatic action (preferably heated), allowing the introduction of a gas sample. The volume of the dosed sample is selected depending on the specific configuration of the chromatograph (a set of detectors, the type of chromatographic columns used, etc.) and the content of components in the gas sample; the number of dosing devices may be more than one;

f) built-in or external flow rate regulator of the analyzed gas and calibration mixture;

g) external or built-in software for processing, transferring and storing chromatographic information;

h) backflush valve (if required).

3.1.2 Means of calibration

Calibration mixtures are natural gas composition standard samples used for calibration. Several calibration mixtures can be used in methane for calibration.

3.2 Auxiliary measuring instruments, devices and materials

– measurement system (MS) of environmental parameters (temperature, pressure, humidity) with appropriate measurement ranges;

– temperature MS with a measurement range from 0 °C to 100 °C with a margin of error of no more than ± 0.3 °C, used when bringing the volume of a gas sample to standard conditions (for example, a mercury glass thermometer of Class II according to GOST 28498);

– pressure MS with a measurement range from 610 to 790 mm Hg. Art. with limits of permissible error no more than ± 0.8 mm Hg. Art., used when bringing the volume of a gas sample to standard conditions;

– gas flow MS with limits of the main permissible error of not more than $\pm 4\%$ of the upper measurement limit (for example, according to GOST 13045); or built-in electronic gas flow meter;

– manometer according to GOST 2405;

– portable samplers or stationary sampling systems in accordance with GOST 31370;

– gas flow fine adjustment valve or pressure regulator with purge valve;

– balloon made of carbon steel grade 150u with a capacity of 40 dm³ in accordance with GOST 949;

– balloon reducer;

– filter to remove mechanical impurities;

- gaseous helium grade A with the main component volume fraction of at least 99,995 %;
- high purity gaseous argon with the main component volume fraction of at least 99,998 %;
- hydrogen generator (high purity gaseous with the main component volume fraction of at least 99,995 %);
- air, compressed class 0 according to GOST 17433.

It is allowed to use other auxiliary measuring instruments and devices of a similar purpose, the technical and metrological characteristics of which are not inferior to those indicated, as well as materials that ensure accuracy standards during measurements.

It is allowed to use hydrogen generators and air compressors that provide the supply of clean gases and air with characteristics in accordance with the above regulatory documents.

Auxiliary gases (air, helium, argon and hydrogen) are supplied to the chromatograph through a filter system.

4. Preparing to take measurements

Turn on the compressor to supply air to the FID, check the working air pressure (0.17-0.64 MPa). Open the cylinder with helium and argon and check the working pressure (0.4 - 0.44 MPa). Turn on the hydrogen generator and wait until the operating mode is reached. Turn on the gas chromatograph. Check for tightness and bring to the mode in accordance with the chromatograph operating manual.

Set up the experimental program and perform a blank analysis. The procedure for conducting a blank experiment is described in section 5.2. (Table 1).

Table 1.

Measurement conditions

Parameter	Value			
Column	DB-1	HayeSep R	NaX	NaX
Column length, m	100	3	2	3
Column diameter (internal), mm	0,25	2	3	2
Detector	FID	TCD	TCD	TCD
Carrier gas	Helium	Helium	Argon	Helium
Carrier gas consumption, cm ³ /min	25	15	15	15
Port temperature, °C	250	-	-	-
Detector temperature, °C	250	200	200	200
Hydrogen consumption, cm ³ /min	25	-	-	-
Air consumption, cm ³ /min	250	-	-	-
Blow gas consumption, cm ³ /min	25	-	-	-
Stream division	1:10	-	-	-
Initial column oven temperature (hold time – 5 min)	60			
Column oven heating rate, °C/min	10			
Final thermostat temperature (holding time 10 min), °C	200			

5. Measurements

5.1 Measurement procedure

After introducing the calibration mixture (during a blank experiment) or the analyzed sample into the chromatograph, start the temperature program of the column thermostat. The conditions for performing measurements (Table 1) and their duration during a blank experiment, chromatograph calibration, and sample analysis should be the same.

Before introducing a gas sample into the sample loop, evacuate it for 1-2 minutes by first opening the ВН2 valve. After closing the ВН2 valve, turn off the compressor and purge the sample for 15-20 seconds. Start analysis.

5.2 Conducting a blank experiment

After the chromatograph enters the operating mode, perform a blank analysis under the conditions given in Table 1.

A blank experiment allows one to obtain additional information about the operation of the equipment and to control the residual content of the components of the previous sample in the system. Perform a blank experiment again if the components of the previous sample are present. When performing a blank analysis, pay attention to the curves of the peaks of the output of the components - they should be smooth, solid. A chromatograph performance check is performed to assess its readiness for analysis, as well as to identify the need for some form of maintenance or repair.

The recommended daily practice for checking is as follows:

- turn on the chromatograph and visually check for the absence of extraneous noise (from working mechanisms, gas leaks);
- before setting the operating modes, control the temperature of the thermostats according to the display indications of the control panel or PC monitor (the indications must correspond to the temperature in the room) and the gas flow rates (there must be "zero" readings, the permissible value is 0.3 ml / min);
- set the analysis methodology and control the stability the specified operating modes (maintain the thermostat temperatures, flow rates or gas pressures).
- during calibration and analysis it is necessary to monitor the stability of the detector baseline (detector background, noise amplitude, baseline drift, baseline rise amplitude when programming the temperature of the column oven) and the separation efficiency of the components, to ensure the convergence (repeatability) of the analysis results.

If necessary (in case of suspected contamination of detectors, evaporators; in case of decrease in sensitivity, etc.), as well as in case of long breaks in work, it is recommended to carry out an assessment in accordance with the operation manual

214.2.840.030PЭ Part 1 Description and operation. General information with subsection 6.2 "Testing" of the verification procedure 214.2.840.043Д. A blank should be performed before the calibration mixture and samples are analyzed, but it is also recommended to perform it at the end of a series of analyzes to clean the columns from heavy components.

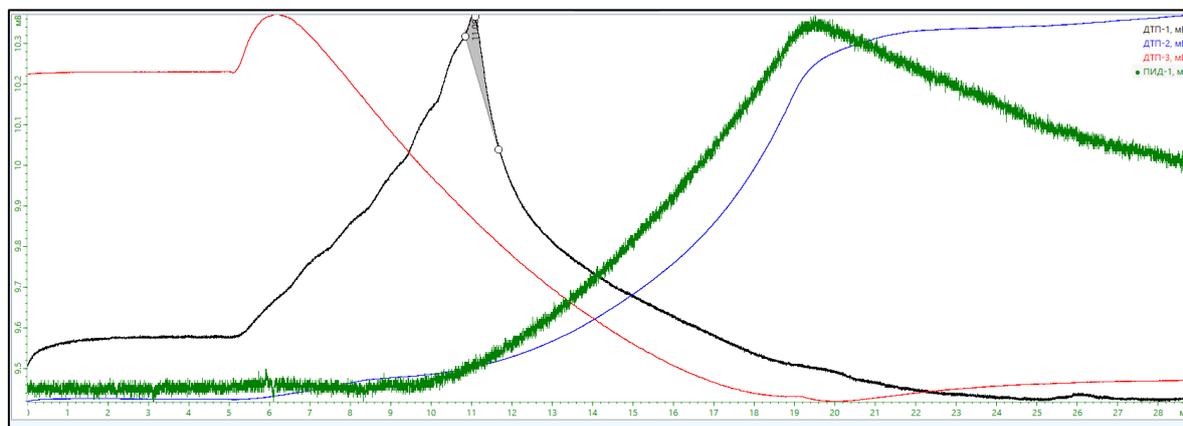


Fig. 1. Blank Chromatogram Example

5.3 APG sampling for analysis

Gas sampling is carried out using the point sampling method, in cylinders made of materials inert with respect to natural gas components. It is also possible to draw gas into gas-tight syringes with a shut-off valve Hamilton 1025 SL 25 ml (22/2/2). In the latter case, to collect the required sample volume fill the syringe with a sample volume slightly larger than required. When injecting sample into the vaporizer, carefully puncture the vaporizer septum to avoid damaging the syringe needle. Injection should be carried out at the same speed from repetition to repetition, avoiding unnecessary delays when introducing the sample. Correct and well-established input technique allows to obtain good convergence of results between analyses.



Fig. 2. View of a gas-tight syringe with a shut-off valve brand Hamilton 1025 SL 25 ml (22/2/2)

5.4 Injection a gas mixture sample into the chromatograph

a) Injection the calibration mixture

Absolute calibration (external standard). The calibration dependence is automatically calculated for all components for which the concentration is known. For components that are not in the SSS, the relative sensitivity factor of n-butane is used (comparative component according to GOST 31371.1-2020).

Carry out calibration of the chromatograph through absolute calibration, using standard samples of the composition of natural gas - gas mixtures in cylinders under pressure. Standard samples must not be expired and have a passport, which indicates the components and their concentration in the cylinder.

Verification of calibration dependences should be carried out at the initial creation of the method, and then each time before performing a series of analyzes of unknown samples, as well as in case of any doubts about the reliability of the analysis results.

Connect the cylinder with the calibration mixture to the chromatograph inlet. The bottle must be closed.

Before calibration, evacuate the sample loop to remove air in the system. Close all valves (BH1, BH2, BH3) to set constant pressure. Turn on the vacuum pump. Open valve BH2 (Fig. 3). Vacuum for 3-4 minutes. The pressure value on the pressure gauge

should reach - (96-100) kPa. Close valve Вн2. The pressure on the pressure gauge should be constant - (96-100) kPa until the sample is injected.

Then, blow off the sample loop with the calibration mixture. All valves (Вн1, Вн2, Вн3) must be closed. Open the valve on the cylinder. Wait until the pressure on the pressure gauge shows +10 kPa or more. Open valves Вн1 and Вн3. Purge the system for 3-4 minutes until the pressure gauge reaches 1 kPa. After purging, supply the calibration mixture to the dosing loop, stopping the flow for no more than 2 seconds to equalize the gas pressure in the dosing loop with atmospheric pressure. To do this, tightly close the cylinder with the calibration mixture and switch the dosing device for introducing gas into the chromatographic columns.

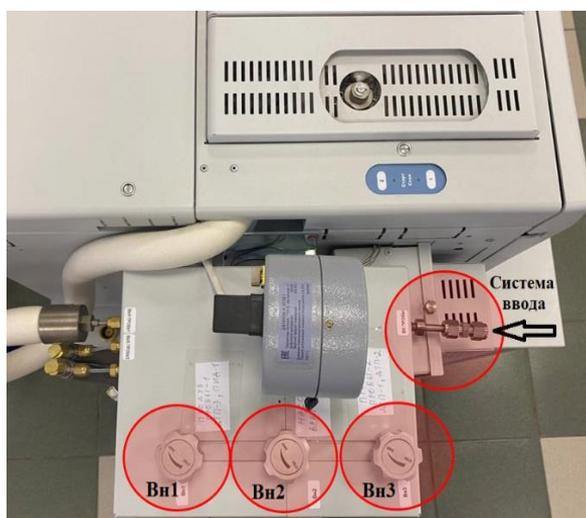


Fig. 3. Gas supply system to the chromatograph (top view)

When calibrating the chromatogram, register the result at least three times. A blank should be run between each injection.

Check the acceptability of the calibration in the chromatograph software.

Save the established equations of calibration characteristics in the software of the chromatograph. The calibration algorithm is described in detail in the «Chromatek 214.00045-51И» user manual.

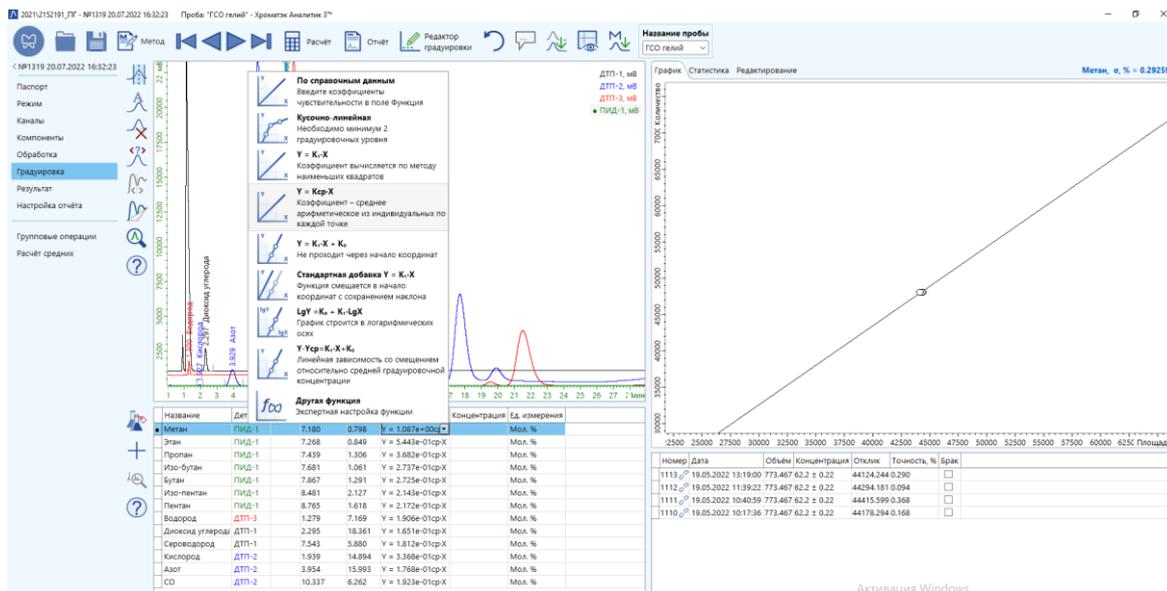


Fig. 4. Example of an absolute chromatograph calibration

Результат анализа							
Компонент	Время (мин)	Площадь (мВ*с)	Высота (мВ)	Концентрация	Ед. измер.	Детектор	
Водород	1,291	405,684	75,453	0,10008	Мол. %	ДТП-3	
Кислород	1,936	214,996	19,429	0,093433	Мол. %	ДТП-2	
Оператор: Питуганова А.Е. Страница 1 из 4							
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Результат анализа							
Компонент	Время (мин)	Площадь (мВ*с)	Высота (мВ)	Концентрация	Ед. измер.	Детектор	
Диоксида углерода	2,293	11925,174	1445,879	2,5386	Мол. %	ДТП-1	
Азот	3,948	6557,616	268,251	1,4985	Мол. %	ДТП-2	
Метан	7,182	44294,181	23588,003	62,119	Мол. %	ПИД-1	
Этан	7,27	17409,188	10620,31	12,269	Мол. %	ПИД-1	
Пропан	7,441	25230,91	16007,342	12,03	Мол. %	ПИД-1	
Сероводород	7,535	6995,742	384,995	1,6364	Мол. %	ДТП-1	
Изо-бутан	7,682	5714,41	3743,938	2,0259	Мол. %	ПИД-1	
Бутан	7,868	8519,873	5433,728	3,0064	Мол. %	ПИД-1	
Изо-пентан	8,481	3575,593	2195,811	0,99294	Мол. %	ПИД-1	
Пентан	8,765	5313,856	3206,58	1,4966	Мол. %	ПИД-1	
СО	10,32	807,769	27,037	0,19251	Мол. %	ДТП-2	
Сумма		136964,992	67016,756	100			

Fig. 5. Example of SSS calibration report

б) APG sample injection for analysis.

After obtaining positive results of the chromatograph calibration (criterion is the consistency of the compositions with the passport data during subsequent injections), proceed to measure the analyzed gas components molar fraction.

Before injection a gas sample into the sample loop, evacuate it for 1-2 minutes.

Dose at least 25 ml of the analyzed natural gas sample into the chromatograph with a gas-tight syringe through a septum or connect a sampler with a gas sample to the inlet (Fig. 6).



Fig. 6(a). Sampler connected to the chromatograph inlet



Fig.6(b). APG sampler

Perform a purge according to paragraph 4.3, and then run analysis under the same conditions under which a blank experiment and analysis of the calibration mixture were carried out (table 1).

After completing the registration of the chromatogram using the software, automatically calculate the measured values of the molar fraction of the components in the chromatograph software.

For hydrocarbons after n-pentane, the area is calculated from the number of carbon atoms in the molecule, i.e. as the sum of the peak areas of an n-alkane and the peaks between that n-alkane and the n-alkane with one less carbon atom (according to ASTM D7169-11)

Typical gas sample chromatograms are shown in figures 7-10.

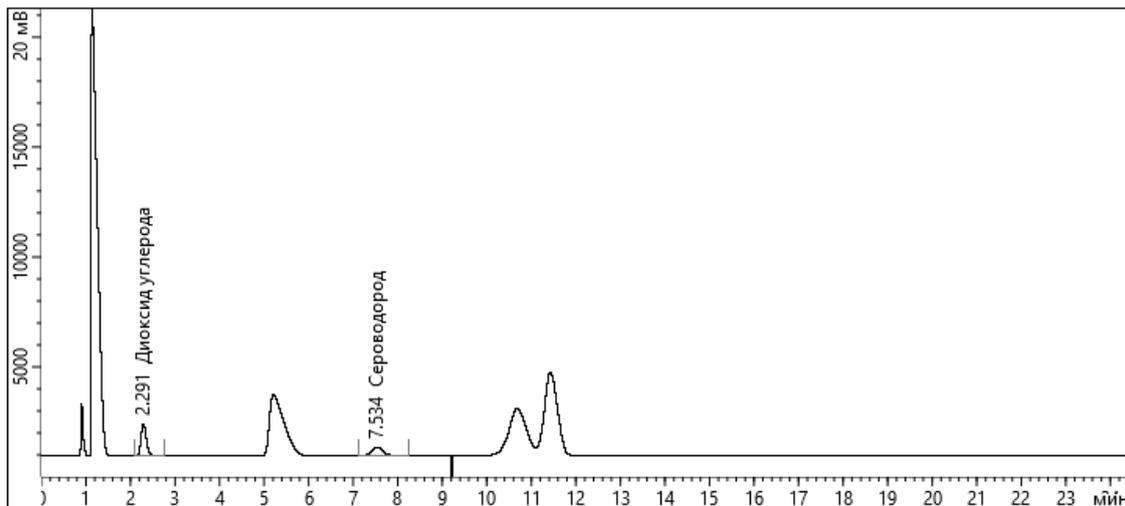


Fig. 7. Chromatogram recorded by the TCD-1 detector

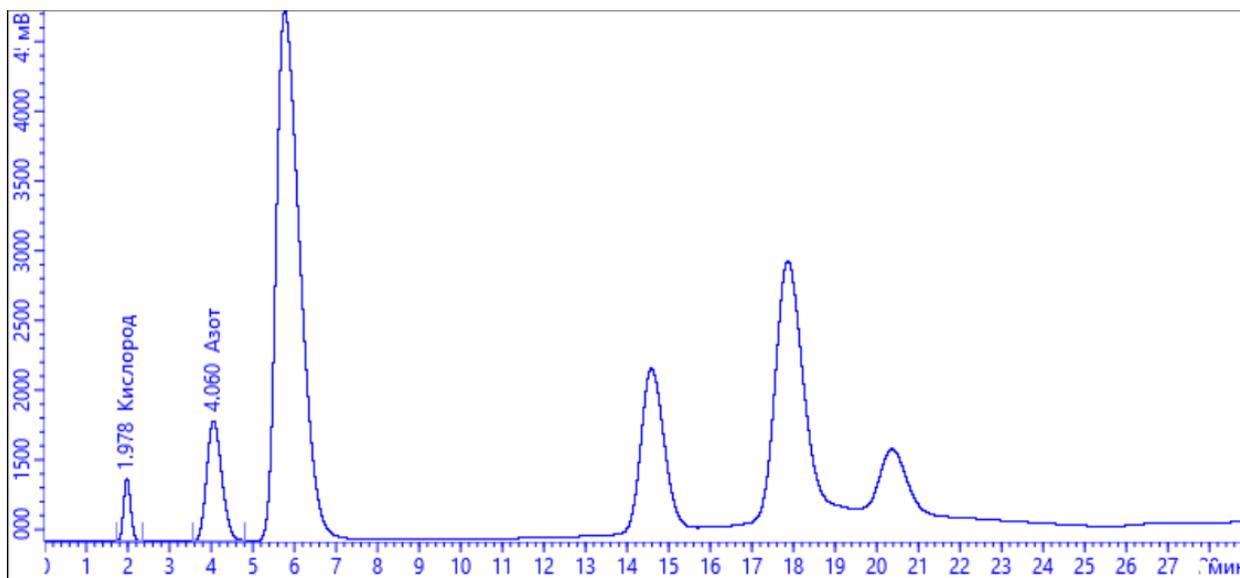


Fig. 8. Chromatogram recorded by the TCD-2 detector

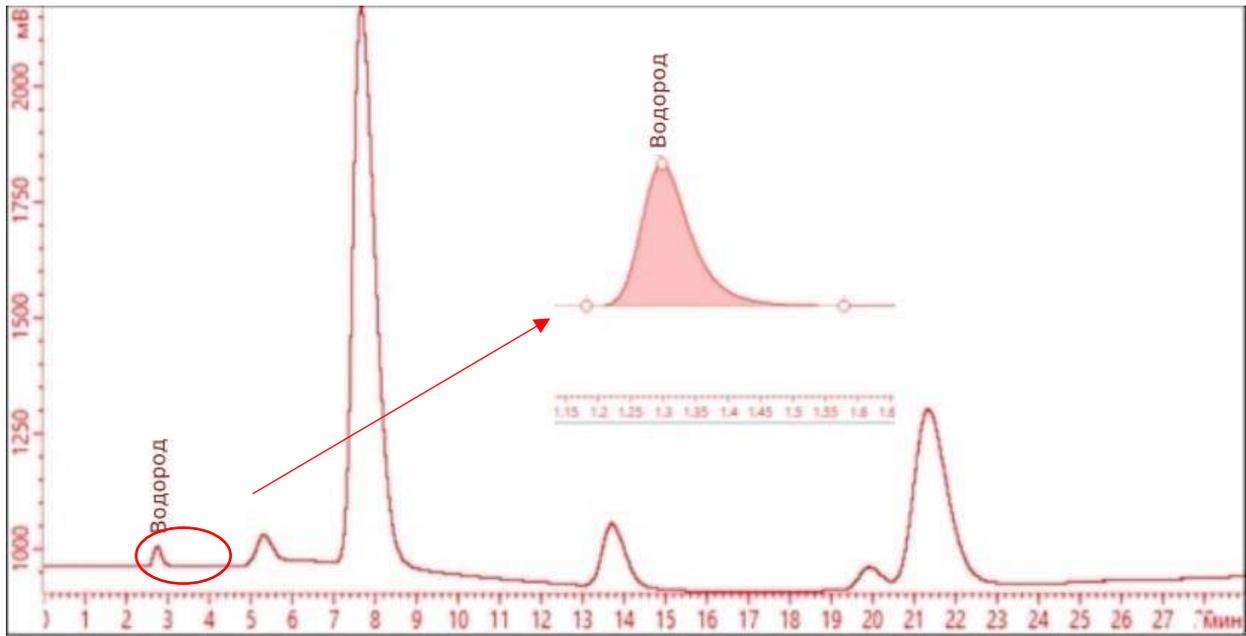


Fig. 9. Chromatogram recorded by the TCD-3 detector

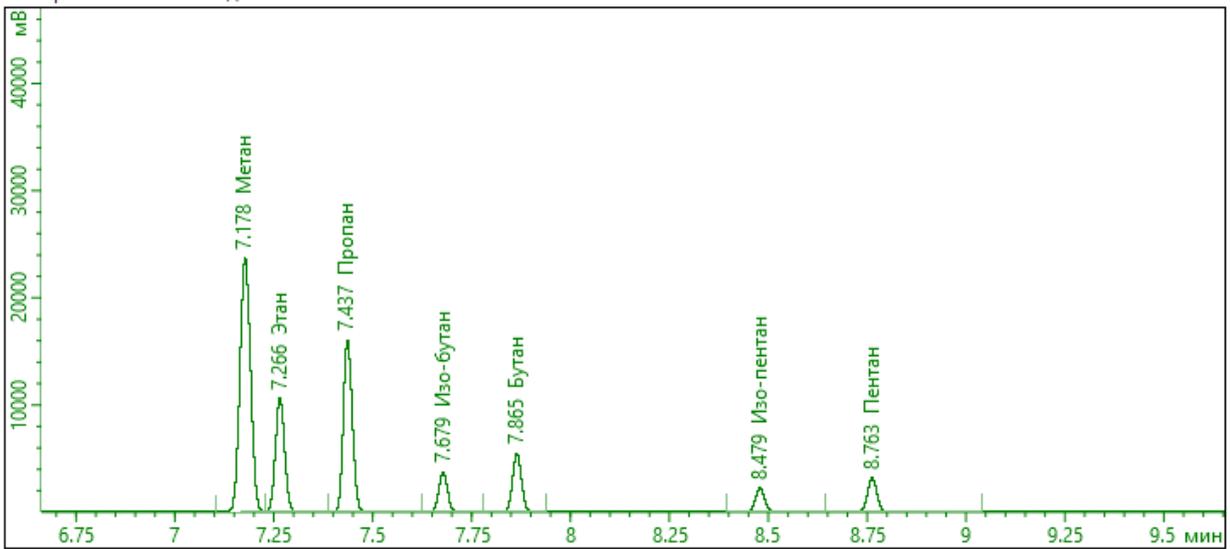


Fig. 10. Chromatogram recorded by FID detector

6. Presentation of the results

Parallel detection. The sample is injected and separated in the chromatographic column. At the exit from the column, the sample is divided into four streams, each of which enters an independent detector.

6.1 Correction for the air presence

Normally, natural gas does not contain oxygen. However, if natural gas samples are found to contain oxygen due to incorrect gas sampling, correct the molar fraction of nitrogen and all other components according to the following procedure:

a) if the sample contains more than 0.02% oxygen, according to GOST 31371.3-2020, the corrected nitrogen mole fraction value is calculated by the formula (1):

$$x_{N_2,c} = x_{N_2} - \frac{78}{21} \cdot x_{O_2}, \quad (1)$$

where $x_{N_2,c}$ — nitrogen mole fraction value after mole fraction correction due to air pollution, %;

x_{O_2} — oxygen molar fraction value in the sample after normalization, %.

b) if the sample contains less than 0.02% oxygen, the corrected value of the nitrogen molar fraction is calculated by the formula (2):

$$x_{N_2,c} = x_{N_2} - \frac{100}{21} \cdot x_{O_2}, \quad (2)$$

In the case when the corrected nitrogen molar fraction value $x_{N_2,c}$ is less than zero, then we equate its value to zero.

The molar fraction of the j-th component value in the sample x_j , expressed as a percentage, corrected for the oxygen content, is normalized to 100 % in accordance with the formula (3):

$$x_{j,c} = \frac{x_{j,s}}{\sum_{j=1}^{n-2} x_{j,s} + x_{N_2,c}} \cdot 100 , \quad (3)$$

where $x_{j,c}$ — normalized mole fraction value of the j-th component in the sample, %;

$x_{j,s}$ — unnormalized mole fraction value of the j- th component in the sample, %;

n — total number of components;

(n – 2) — total number of components excluding oxygen and nitrogen.

6.2 Molar mass of the gas mixture

Gas mixture is a mixture of pure substances that do not enter into a chemical reaction with each other, all of which are in a gaseous state. The molar mass (M_c) of a given mixture is calculated using formula (3):

$$M_c = \sum_{j=1}^N [x_{j,c} \cdot M_j] / 100 , \quad (3)$$

where M_c — molar mass of the mixture, g/mol;

M_j — molar mass of the j-th component (table 2).

Table 2.

Molar masses of gas mixture components

Components	M _j , g/mol
H ₂	2.016
He	4.003
N ₂	28.014
CO ₂	44.010
H ₂ S	34.082
C ₁	16.043
C ₂	30.070
C ₃	44.097
i-C ₄	58.123
n-C ₄	58.123
i-C ₅	72.150
n-C ₅	72.150
C ₆	*84.00
C ₇	*96.00
C ₈	*107.00
C ₉	*121.00
C ₁₀	*134.00
O ₂	*31.999

**The molecular weight is given by Katz, D. L., and Af Firoozabadi. "Predicting phase behavior of condensate/crude-oil systems using methane interaction coefficients". Journal of Petroleum Technology 30, № 11 (1978): 1-649.*

6.3 Mass fraction

Mass fraction (w_j) – the ratio of the component mass to the mass of the mixture, calculated by the formula (4):

$$w_j = \frac{x_{j,c} \cdot M_j}{M_c}, \quad (4)$$

where w_j — mass fraction of the j-th component, % mass.

Practical part

The laboratory work is carried out in teams formed by the lecturer after mastering the theoretical lecture material.

The lecturer issues a task to each formed group, with which the chromatographic analysis of the gas sample will be performed. The results are presented in the form of the Final Protocol.

Table 3

Example of the final protocol

Analyte components	Molar mass, g/mol	Initial mixture	Airless mixture *	
		% /mol.	% mol.	% mol.
H ₂	2.016	-	-	-
He	4.003	-	-	-
N ₂	28.014	2.3847	-	-
CO ₂	44.010	0.2254	0.2330	0.3074
H ₂ S	34.082	-	-	-
C ₁	16.043	43.1670	44.6288	21.4637
C ₂	30.070	16.0710	16.6152	14.9776
C ₃	44.097	18.5770	19.2061	25.3894
i-C ₄	58.123	5.5789	5.7678	10.0499
n-C ₄	58.123	6.9093	7.1433	12.4466
i-C ₅	72.150	2.1037	2.1749	4.7042
n-C ₅	72.150	1.5114	1.5626	3.3797
C ₆	84.000	1.3683	1.4147	3.5624
C ₇	96.000	0.8862	0.9162	2.6368
C ₈₊	107.000	0.3264	0.3374	1.0823
O ₂	31.999	0.8910	-	-
Summ	-	100.00	100.00	100.00
Molar mass, g/mol			33.3577	

* calculated according to paragraph 6.1 and 6.3

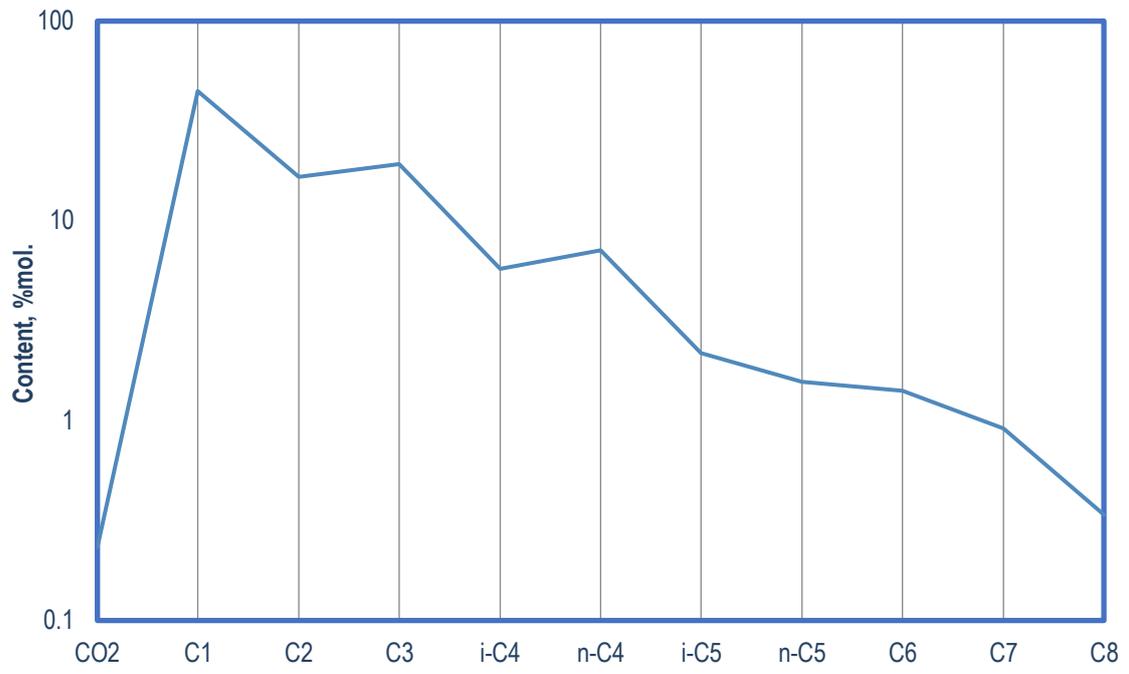


Fig. 8. Example of an APG sample component composition

Review questions

- 1) Gas chromatography – is it?
- 2) Chromatogram – is it?
- 3) Carrier gas – is it?
- 4) Peak area – is it?
- 5) How the chromatograph is calibrated?
- 6) Calibration mixtures – is it?
- 7) The frequency of checking the calibration dependence?
- 8) Measurement conditions?
- 9) Capillary gas chromatographic column – is it?
- 10) Packed gas chromatographic column – is it?
- 11) Methodology for conducting chromatographic analysis?
- 12) Correction for the presence of oxygen?
- 13) Mass fraction – is it?
- 14) Molar mass – is it?

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