Effect of gasoline composition on the absorption and retention of fuel vapor by activated carbon of the automobile adsorber

A A Khodyakov, S V Khlopkov, A V Azanov and R Kh Abu-Nidzhim
Peoples’ Friendship University of Russia (RUDN University), 6 Miklukho-Maklaya Street, Moscow, 117198, Russian Federation
Email: khlopkov-sv@rudn.ru

Abstract. The physicochemical characteristics of petrol samples are studied. It is established that the fuel purchased at fuel stations corresponds to the standard parameters presented in the EURO standard for unleaded petrol. The presence of saturated hydrocarbons in petrol is determined using concentrated sulfuric acid, which reacts with unsaturated (alkenes, olefins) and aromatic hydrocarbons, without reacting with saturated hydrocarbons. The fractional composition of petrol samples is determined from the distillation temperatures measured by a mercury thermometer included in the automatic oil distillation apparatus ARNS-1E, acidity titration with an alcohol solution of potassium hydroxide gasoline mixtures with 80% ethanol. An acid-base phenolphthalein indicator (pH = 8.0-9.6) is used to indicate an excess of OH-. The density of the fuel samples is measured with oleometers (densimeters), octane numbers (ON) - indicator (Octanometer) OKTAN-IM. The limit of the permissible absolute error of the measurement of ON is ± 2 octane units. It is established that all petros without exception contain saturated, unsaturated and aromatic hydrocarbons. It is shown that of the seven samples studied, two samples have some peculiarities. It is concluded that when controlling the quality of fuel, it is necessary to take into account not only the conformity of the determined value to the parameter of the norm, but also the differences in the values of the indices obtained when testing a series of fuel samples.

1. Introduction
Modern automotive gasolines are a mixture of components obtained by using various technological processes. Depending on the composition of the raw materials and the synthesis technology gasoline can contain a large number of individual hydrocarbons. The increased content of aromatic hydrocarbons in this fuel requires not only the engine performance improving, but also the physicochemical properties control of the gasoline fractions compound. The group hydrocarbon composition also affects the gasoline octane number. Each hydrocarbon has its own blending value, the blending octane number. The mathematical models adequacy to calculate octane numbers is questionable. Nowadays refractometry, densimetry, dielectricity and method of magnetic birefringence are currently used to assess the motor fuels performance characteristics. These rapid analysis methods can be an obstacle to the market penetration of counterfeit and improper fuels.

The purpose of this work was to study the fuel samples chemical activity and determine the content of saturated, unsaturated and aromatic hydrocarbons in petrol. The octane numbers of gasoline samples and saturated hydrocarbons can be measured using the dielectricity method. To obtain data that allowing drawing reliable conclusions, we used a system approach, consisted not in the study of one or two
gasoline samples, but of a whole series of fuel samples. As an illustration of the need to control the differences between the parameters characterizing the physicochemical petrol qualities, the experiments were conducted on the absorption of fuel vapor by activated carbon.

It is known that both performance indicators and environmental characteristics of fuel characterize the quality of petrol. In addition, the introduction of modern norms and standards that are associated with the modification of the component composition of fuel sets the task of controlling the physicochemical characteristics of petrol [1-6].

2. Experimental
The presence of saturated hydrocarbons in petrols was determined using concentrated sulfuric acid, which reacts with unsaturated (alkenes, olefins) and aromatic hydrocarbons without reacting with saturated hydrocarbons [7, 8]. The experiments were carried out in a separatory funnel. First, the petrol (30 ml) was poured into the funnel, followed by concentrated sulfuric acid (40 ml).

The mass of the objects of the study was measured on a VLTE-150 portion scale. According to the GOST (national standard) technique, experiments with sulfuric acid are carried out by shaking the petrol samples with H₂SO₄ concentrated for 30 minutes one time [9]. The one-fold interaction of the components of petrol with H₂SO₄ concentrated was replaced by a four-fold treatment of fuel samples with sulfuric acid. In such experiments, the ratio of the mass of fresh portions of acid to fuel increased from 3.5:1 to 9:1.

The total time of reaction was 40 minutes. The measurement of the saturated hydrocarbons mass forming the upper layer in the separatory funnel was carried out after shaking (for 10 minutes), settling, separation of the mixture, and draining the sulfuric acid layer located in the lower part of the funnel. Before placing saturated hydrocarbons in the measuring container, the funnel with the remaining fuel was washed (three times) with distilled water. The presented experiment process with sulfuric acid is associated with a high content of aromatic hydrocarbons in modern petrols, as well as using benzol and monomethylaniline (C₆H₅NHCH₃) containing benzol ring [1, 10-12] as octane promoter.

Oxygen-containing compounds—alcohols and esters (oxygenates) can be presented in petrols [1, 10-12]. To determine the possibility of formation (similar to alkenes and aromatic hydrocarbons) of oxygenates of the sulfuric acid layer, the effect of H₂SO₄ concentrated on the mixture of n-heptane with isopropyl (absolute) alcohol (IPA) and methyl tertiary butyl ether (MTBE) was studied. The content of IPA and MTBE in such two-component mixtures was ~ 33-39%.

The fractional composition of the petrol samples was determined from the distillation temperatures measured by a mercury thermometer included in the ARNS-1E automatic oil distillation apparatus, acidity-titration with alcoholic potassium hydroxide blends of petrol with 80% ethanol. An acid-base phenolphthalein indicator (pH = 8.0-9.6) was used to determine an excess of OH.

The density of the fuel samples was measured with oleometers (densimeters), octane numbers (octanometer) OKTAN-IM. The range of measurement of octane numbers (octane number by the research method - RON, octane number by the motor method - MON) is from 67 to 98. The limit of the permissible absolute error of the measurement of ON was ± 2 octane units (o.u.). Measurements of the GCR were carried out using the factory calibration of the octanometer.

The objects of the study were samples of petrols (B-1, B-2, B-3, B-4, B-5, B-6) AI-95, AI-98, purchased at different times at gas stations in Russia and Spain (Sample B-7). The volumes of liquids, depending on the test methods used, ranged from 1 to 100 ml.

3. Result and Discussion
The comparison of the data and the normative in Table 1 values shows that parameters such as density, acidity, boiling point temperature of petrol samples, the remainder in the flask correspond to those presented in footnotes of Table 1 standards. However, petrols B-1 and B-2 have, in comparison with other samples, the lowest and highest value of the remainder in the flask, lower values of fₜ₄₀ and high acidity. A similar regularity is observed in the value of the octane number. So, for example, in B-2 this figure was 100 o.u. (Table 3).
Table 1. Density ($\rho$), acidity ($K$), temperature of the boiling point of fuel samples ($t_{kk}$), and remainder in flask

| Petrol | $\rho$, kg·m$^{-3}$ at $15^\circ$C$^a$ | the remainder in the flask, %$^b$ | $t_{kk}$, $^\circ$C$^c$ | $K$, mg of potassium hydroxide na 100 ml of petrol$^d$ |
|--------|----------------------------------|----------------------------------|----------------|----------------------------------|
| B-1    | 738                              | 1.9                              | 175             | 2.6                              |
| B-2    | 743                              | 0.7                              | 176             | 2.8                              |
| B-3    | 754                              | 1.5                              | 206             | 1.1                              |
| B-4    | 748                              | 0.9                              | 187             | 1.7                              |
| B-5    | 754                              | 1.0                              | 201             | 1.2                              |
| B-7    | 736                              | 0.9                              | 208             | 2.2                              |

$^a$Density at $15^\circ$C according to GOST 32513-2013 is 720-780 kg·m$^{-3}$.

$^b$The balance in a flask, % (in terms of volume) no more 2 (GOST 32513-2013).

$^c$The boiling point, $^\circ$C not higher than 215$^\circ$C (GOST 32513-2013).

$^d$Acidity, mg potassium hydroxide per 100 cm$^3$, not more than 3 (TU 38.001.165-87).

It was noted in experiments with concentrated sulfuric acid, that in the primary and secondary treatments of samples B-1 and B-2, the upper layer of liquid in the separatory funnel acquires a reddish tint. Mixtures of fuel with acid (the first treatment), in comparison with the mixtures $\text{H}_2\text{SO}_4$ concentrated-B-3-7, not only, strongly foams, but also heat, burning hands when shaking liquids. This may be due to the high content of B-1 and B-2 in the petrol of a single component. Moreover, the interaction of such components with sulfuric acid, in contrast to alkenes and aromatic hydrocarbons, proceeds violently, with a large release of heat. A similar course of the process was observed under the action of $\text{H}_2\text{SO}_4$ concentrated on a mixture of n-heptane with isopropyl alcohol. This may indicate the presence of monohydric alcohols in B-1 and B-2. It should be noted that isopropyl alcohol and MTBE, reacting with sulfuric acid, form (alogically alkenes and aromatic hydrocarbons) in the separatory funnel sulfuric acid layer.

Table 2. Mass content of saturated (C$_{ch}$) hydrocarbons and substances that form sulfuric acid layer (C$_{sulf}$) in experiments with $\text{H}_2\text{SO}_4$

| Sample | B-1 | B-2 | B-3 | B-4 | B-5 | B-6 | B-7 |
|--------|-----|-----|-----|-----|-----|-----|-----|
| C$_{ch}$, % | 41.9 | 42.1 | 48.0 | 48.1 | 46.1 | 51.7 | 59.7 |
| C$_{sulf}$, % | 58.1 | 57.9 | 52.0 | 51.9 | 53.9 | 48.3 | 40.3 |

Table 2 shows the mass content of saturated hydrocarbons in fuel samples. The low values of C$_{ch}$ in B-1 and B-2, do not reflect, in contrast to other samples, the true value of the concentration of limiting hydrocarbons, since the rapid flow of the interaction of B-1 and B-2 with $\text{H}_2\text{SO}_4$ concentrated can lead to a significant loss of hydrocarbons in presented petrol. Therefore, it is reasonable to analyze the concentrations of the ultimate hydrocarbons in samples B-3-7. Such analysis shows that the content of saturated hydrocarbons in petrols varies from 46.1% to 59.7%, i.e., it is different.

The evidence of differences in the content of saturated hydrocarbons is the data presented in the literature, including petrol tests of the EURO standard [13-16]. Thus, the average value of the volumetric content of isoalkanes, n-alkanes and cyclanes in the samples of catalytic reforming petrol is 46.8% [15]. In petrol (AI-93) catalytic cracking (without octane promoter additives), the concentration of the ultimate hydrocarbons is 56.9% vol. %, in commodity fuel, 58.3-59.5 vol. % [13]. In the end, the different content of saturated hydrocarbons is determined by the type of technology for producing petrol components and the compounding variant used to produce petrol of the required grades [14].
Alkenes, interacting with H₂SO₄ concentrated, form the lower sulfuric acid layer in the separatory funnel (as well as arenes). The volume fraction of olefinic hydrocarbons according to the standards for petrol must not exceed 18.0% [1]. The tests presented in the literature testify both to the presence and absence of olefins produced by various manufacturers [10-12]. Therefore, a qualitative determination was made of the presence of hydrocarbons in the samples of petro-ols, in the structure of which, as in alkenes, a double chemical bond is realized between the carbon atoms. For this kind of tests, potassium permanganate (KMnO₄) was used, which reacts effectively with such hydrocarbons.

It is determined that after the reagent introduction into the fuel samples, the color of the aqueous solution of KMnO₄ changes with time. The violet color of potassium permanganate disappears, and brown flakes appear in the solution which turns brown. After a day, the brown flakes do not disappear in the samples prepared in this way, the solution becomes colorless. The effects of changing the color of the aqueous solution and the formation of brown flakes observed in experiments with potassium permanganate indicate that hydrocarbons with a double chemical bond are present in petro-ols. Such hydrocarbons in petro-ols are primarily olefinic hydrocarbons.

The content of hydrocarbons reacting with sulfuric acid and forming a sulfuric acid layer is different in mass (expressed as a percentage) of the fuel samples before and after their treatment with H₂SO₄. According to the data presented in Table 2 it follows that this parameter was 40.3-53.9%. The values of C_sulf in B-1 and B-2 due to the loss of hydrocarbons caused by the rapid reaction of the reaction of fuels with sulfuric acid should be considered, in comparison with other samples, significantly overestimated, i.e., not representative. In addition to olefins, as noted earlier, aromatic hydrocarbons are also presented in the sulfuric acid layer. C_sulf values confirm this conclusion, which are in order of magnitude identical to the average value of the content in the produced petrol aromatic hydrocarbons, equal to ~ 34.0 vol. % [10-12].

The determination of the octane number by the OCTAN-IM indicator is based on the measurement of the complex permittivity of the petrol (ε_comp) [13, 16-20]. This method (dilometry) refers to an indirect one-factor method for determining the ON. The mathematical model describing the connection between octane numbers and ε_comp is not only nonlinear, but also contains internal contradictions. Such contradictions include a large contribution to the dielectric permittivity of arenes (having a higher ε value than that of other hydrocarbons) and a significant dependence of the ON on the content of these substances in petro-ols [20]. It is established that the dielectric constant has a maximum sensitivity for petrol with octane numbers of 88-93 o.u. The sensitivity of the method for a fuel having an ON above 95 reduces. Difficulties in recognizing noted grades are associated with the presence of isooctane, isopentane and butanes in high-octane petro-ols, which have a low dielectric constant at a high octane number of compounds [16].

Table 3. Octane numbers (ON) of saturated hydrocarbons (RONₕ, R-1) and RONₕₑₚ (R-2) of fuel samples

| Petrol | B-1 | B-2 | B-3 | B-4 | B-5 | B-6 | B-7 |
|--------|-----|-----|-----|-----|-----|-----|-----|
| R-2    | 96.7| 100.0| 96.2| 98.7| 97.7| 97.0| 96.1|
| R-1    | 79.1| 80.3| 80.9| 76.4| 76.2| 81.0| 80.2|

Table 3 shows the octane numbers of petrol (ONB) and saturated hydrocarbons (ONHS) measured by the OCTAN-IM indicator. The octane numbers of petro-ols (excluding ONB = 100 in B-2) differ by 0.3-1.3 o.u. from the value of the octane number declared by the fuel manufacturer. In fuel B-2, the value of ONB exceeds the declared AI-95 by 5 o.u., which is higher than the error of measurement of ON (~ 2 o.u.). The increase in ONB with increasing ε_comp is usually associated with an increased content of substances in the fuel, in which molecules there are polar bonds [18]. Such a substance may be for example a monohydric alcohol.
Thus, the physicochemical characteristics of the studied fuel samples correspond to the standard parameters presented in the EURO standard for unleaded petrol. All petrols contain saturated, unsaturated and aromatic hydrocarbons. Among the seven studied samples, two samples have certain peculiarities. Checking the quality of fuel, one should take into account not only the compliance of the determined value with the standard parameter, but also the differences in the values of the indices obtained during testing of a series of fuel samples, i.e., it is reasonable to carry out serial tests. This conclusion can be justified by the temperatures of the boiling point of the petrol samples. This parameter corresponds to the norm for all petrols without exception. However, in the two previously mentioned samples, the boiling-point temperatures have lower values, compared with other fuel samples.

To illustrate the need to control the differences in the parameters characterizing the physicochemical properties of gasolines, experiments were conducted on the absorption of fuel vapors by activated carbon (AC). An activated carbon filter (absorbent carbon) of the CITROEN C4 adsorber was used.

![Figure 1](image)

Figure 1. Scheme of experiments using: 1-rotameter; 2-drexler's bottle (barbater); 3-the stabilizer of pressure (a trap with a hammer stone); 4-dynamic tube.

In Figure 1 shows the scheme of experiments. At a temperature of 20°C air by the compressor, passing through the rotameter (1), is supplied to the barbater (2) and with vapors of the investigated liquid goes to the pressure stabilizer (3) and then into the dynamic tube (4) filled with activated carbon. The inner diameter of the column was 0.02 m, the height of the sorbent layer was 0.01 m. In all the experiments, the AC mass was ~ 1 g, the volume of liquid placed in the barbater - 20 ml. The air flow rate (Q) containing the fuel vapor was measured by a froth flowmeter. Parameter Q at the entrance to the dynamic tube was 0.45 L·min⁻¹, at the output from the column - 0.05 L·min⁻¹. Moreover, as the pore space of the AC filled with adsorbent molecules, flow out of column decreased to values much lower than 0.05 L·min⁻¹ and was not fixed by a foamy flowmeter. Experiments on the molecules desorption from the AC surface were carried out with an airflow at the inlet to the dynamic column equal to 0.45 L·min⁻¹. The weight of the tube with the sorbent was measured at the beginning and after the experiment with a time interval of 10 to 40 minutes. The total time was 100 min. Measurement of the mass of the sorbent and liquids were carried out on the scales VLTE-150. It should be noted that the scheme for the experiments (Figure 1) is a simplified prototype of the experimental setups presented in scientific works [21, 22].

Before refilling in a dynamic tube, the carbon filter was subjected to regeneration, consisted in heating the carbon to temperatures of 250-300°C and forcing it to swing through a container with an air sorbent. Such AC treatment was carried out for several hours until the mass of the annealing object became close to a constant value.
The table 4 shows the masses of gasoline calculated as the difference between the masses of liquids before and after the experiment for 100 minutes, the mass of steam absorbed by the activated carbon formed as a result of air bubbling through the fuel sample layer.

**Table 4. Masses of gasoline ($\Delta_{zh}$), calculated as the difference in mass of these liquids before and after the experiment, the mass of fuel vapor absorbed by activated carbon ($\Delta_{p}$)**

| Gasoline | $\Delta_{zh}$, g | $\Delta_{p}$, g | $\Delta_{p}$, %$^c$ |
|----------|-----------------|----------------|-------------------|
| B-1      | 7.964           | 0.520          | 6.5               |
| B-2      | 9.607           | 0.598          | 6.2               |
| B-4      | 6.413           | 0.518          | 8.1               |
| B-5      | 7.554           | 0.560          | 7.4               |
| B-6      | 7.575           | 0.498          | 6.6               |
| B-7      | 7.293           | 0.512          | 7.0               |

$^c$Calculated by the equation: $\Delta_{p} = (\Delta_{p} / \Delta_{zh}) \cdot 100\%$.

From the comparison of the data given in Table 4 it follows that of 9.607 g of fuel steam B-2, activated carbon absorbed 6.2%. A lower value of $\Delta_{p}$ (%) is observed for gasoline vapors B-1 as compared to samples B-4-7. These differences are in full agreement with the peculiarities of the physico-chemical properties of the B-1 and B-2 gasolines presented earlier. An analogous regularity is also observed in experiments of desorption of gasoline components from the surface of activated carbon (Table 5). Thus, the extraction ($\Delta_{d}$) from the surface of the AC components of gasoline B-1 and B-2 into the flow of air through the dynamic tube was 35.6% and 44.1%, respectively. These values are minimum and maximum in relation to the parameter $\Delta_{d}$ of gasoline B-4-7.

**Table 5. Ratio of the mass of the fuel components desorbed from the AC surface to the mass of the vapor absorbed by the carbon filter ($\Delta_{d}$,%)**

| Gasoline | $\Delta_{d}$, % |
|----------|-----------------|
| B-1      | 35.6            |
| B-2      | 44.1            |
| B-4      | 40.7            |
| B-5      | 41.6            |
| B-6      | 43.2            |
| B-7      | 39.1            |

Experiments on the adsorption of AC vapors of ethanol, absolute isopropyl alcohol, n-heptane and isooctane were carried out to determine the coupling of the absorptivity of a carbon filter with dimensions (radius, $r$) and the dipole moment ($\mu$) of the components of gasolines (Table 6).

**Table 6. Radius ($r$) and dipole moment ($\mu$) of a molecule of ethanol, isopropyl alcohol, n-heptane and isooctane**

| Parameter | Ethanol | Isopropyl alcohol | Isooctane | N-heptane |
|-----------|---------|-------------------|-----------|-----------|
| $r \cdot 10^{-10}$, m | 1.8     | 2.1               | 2.7       | 2.4       |
| $\mu$, D  | 1.63    | 1.66              | 0         | 0         |

From the presented experimental results in Table 7, the values of $r$ and $\mu$ in Table 6 it follows that the correlation of the activated carbon absorptivity with the dimensions and dipole moment of molecules of gasoline components is quite real. For example, vapor of ethanol and isopropyl alcohol, which molecules have a smaller size and a high value of $\mu$, are absorbed more efficiently by the carbon filter than in n-heptane and isooctane (Table 7). A confirmation of this connection is given by the data of dielectric measurements, indicated that the measured value of the octane number of gasoline is significantly influenced by the presence in the fuel of substances in which molecules polar bonds are present [18].
Table 7. Mass of ethanol, isopropyl alcohol, n-heptane and isooctane, calculated as the mass difference ($\Delta_{zh}$) of these liquids before and after the experiment, the mass of steam absorbed by the activated carbon

| Liquid         | $\Delta_{zh}$, g | $\Delta_p$, g | $\Delta_p$, %$^e$ |
|----------------|------------------|---------------|-------------------|
| Isopropyl alcohol          | 2.365            | 0.459         | 19.4             |
| Ethanol                  | 2.468            | 0.448         | 18.2             |
| isooctane                | 4.324            | 0.525         | 12.1             |
| n-heptane                | 3.730            | 0.519         | 13.9             |

$^e$Calculated by the equation: $\Delta_p = (\Delta_p / \Delta_{zh}) \cdot 100\%$.

4. Conclusion
Summarizing the data presented, it is concluded that the specific composition of gasoline B-1 and B-2 are a reflection of the increased content of organic compounds with a higher molecular weight in the fuel (compared to B-4-7). Such substances may be aromatic hydrocarbons and a series of compounds containing polar. This conclusion confirms the value of the Evaluations of gasoline B-2 ($\text{ONB} = 100$ o.u.) and the evidence that the degree of adsorption on activated carbon decreases in the series: aromatic hydrocarbons, paraffins (alkanes) with a regular chain, cycloparaffins (cycloparaffins), paraffins with a branched chain. An increase in the ONB with an increase in $\varepsilon_{\text{comp}}$ is usually associated with a high content of aromatic hydrocarbons and MTBE in fuel.

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