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EFFECT OF REACTION TEMPERATURE AND SULFATE ANION CONCENTRATION ON THE CONVERSION OF GAS GASOLINE OVER A COMPOSITE CATALYST

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Catalysts based on sulfated zirconium dioxide and cobalt-modified HZSM-5 zeolite were synthesized and the conversion of gas gasoline with their participation was studied. It was found that during the conversion of gas gasoline with the participation of these catalysts, its C_{4-} and C_{7+} components are converted into i-C_5, n-C_5 and i-C_6 alkanes. The bimolecular intermediate [C_{4-} - C_{7+}] formed during the process decomposes in two directions and two processes occur in the system: isomerization and hydrocracking. The rate of these processes depends on the reaction temperature and the amount of sulfate anions. The production of iso-alkanes passes through a maximum at 180^{0}C and the best isomerizing catalyst is a catalyst with 2% sulfate anions. It was found that the synthesized catalysts allow converting gas gasoline into high-octane gasoline components.

Keywords: *gas gasoline, sulfated zirconium dioxide, zeolite, isomerization, high-octane gasoline components, bimolecular intermediate*

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Introduction

The requirements for fuel composition have made the isomerization process of n-alkanes an integral part of the oil refining industry. Alkane isomerization reactions are reversible exothermic processes and proceed without volume change. Therefore, thermodynamic equilibrium depends only on temperature. Low temperatures create favorable conditions for the release of isomers, especially highly branched isomers with high octane numbers. In industry, the isomerization process is carried out near thermodynamic equilibrium, therefore its efficiency depends largely on the activity of the catalyst. Existing isomerization catalysts can be divided into the following groups [1]: a) homogeneous isomerization catalysts; b) ionic liquid catalysts (PF_6^-, BF_4^-, alkyl-substituted pyridinium ion, etc.); c) heteropolyacid catalysts; d) hydroisomerization catalysts (aluminum chloride, zeolite, metal-promoted zeolite, etc.), f) solid superacid catalysts (halogenated alumina, La_2O_3-TiO_2, etc.).

One of the solid superacid catalysts is sulfated zirconium dioxide (SZ) catalysts. These catalysts are distinguished by their high activity, resistance to poisons, and operate in a favorable temperature range (140-220^{0}C) [2-4]. Such catalysts have been widely studied since the 1960s. Studies are being conducted to determine the structure, nature, and activity of their active centers [5-8], to study their acid-base properties [9, 10], and to improve and further develop their catalytic properties. Such studies also include the placement of SZ on the surface of various carriers [11-14], the addition

of various metals and metal oxides to the SZ system [15-17], and the replacement of the sulfate anion with other oxyanions [18-20].

In all these works, sulfated zirconium dioxide-based solid superacid catalysts were studied only in the isomerization process of individual alkanes, their activity in the conversion of alkane mixtures and the effect of the amount of sulfate anions on the activity of these catalysts have not been thoroughly studied. In this regard, the aim of the present work is to synthesize composite catalysts consisting of sulfated zirconium dioxide and HZSM-5 zeolite modified with cobalt metal and to study the activity of these catalysts in the conversion of natural hydrocarbon mixture gas gasoline into high-octane gasoline components and the effect of the amount of sulfate anions and reaction temperature on this isomerization activity.

Experimental part

The modified zeolite component of the composite catalyst was prepared by keeping decationized HZSM-5 (Si/Al = 23) in a $Co(NO_3)_2$ solution of a given concentration for 24 h, followed by evaporation of the aqueous phase, drying at 120 and 350°C (3 h) and 550°C (5 h), and treatment in a hydrogen stream (40 $ml \cdot min^{-1}$, 3 h) at 380°C.

The sulfated zirconium dioxide (SZ) component of the catalyst was synthesized by hydrolyzing $ZrOCl_2$, taken in a given amount, with a 25% ammonia solution at a pH of 8-9. For this purpose, a solution containing 100 g of $ZrOCl_2 \cdot 8H_2O$ in 300 ml was added. Ammonia solution was added dropwise to H_2O heated to 800°C while stirring. The resulting gel was kept in the solution for 2 hours at the above temperature, then filtered, washed with distilled water, and dried at 100°C for 24 hours. The resulting zirconium hydroxide gel was sulfated with a solution of $(NH_4)_2SO_4$ (with stirring for 2 hours), and then the aqueous portion was evaporated to a wet state [21, 22].

The resulting zirconium hydroxide was mixed with the powder of the corresponding zeolite component until visually homogeneous. The mixture was dried at 120°C (3 hours), calcined at 550°C and 600°C (5 hours), then ground into powder and granulated with a binder - Al_2O_3 hydrogel into 1.5×(3-4) mm granules. The above-described heat treatment was repeated.

The composition of the synthesized catalysts is shown in Table 1.

Table 1. Composition of the synthesized catalysts*.

Catalyst	Composition			
	HZSM-5	Co	ZrO_2	SO_4^{2-}
C- 2	62.6	0.4	10	2
C - 4	60.6	0.4	10	4
C- 6	58.6	0.4	10	6
C - 8	56.6	0.4	10	8

*all samples contain 25% Al_2O_3

Before conducting experiments, samples of catalysts were placed in the reactor and subjected to standard (regeneration) treatment with nitrogen, and then with air at 500^0C (2h) and hydrogen at 380^0C (2h) with a linear gas supply rate of 2 l/h.

As raw material, we used gas gasoline (GG) of Garadag Gas Refinery (Baku), hydrogen electrolytic.

Catalytic transformations of GG were studied at atmospheric pressure in a flow-type catalytic installation equipped with a quartz reactor with a stationary catalyst bed of 3 cm^3 (ratio H2/hydrocarbon=3), with a volume velocity of the liquid of 2 h^{-1}. The installation scheme includes two gas supply lines with dryers, regulators and gas flow switches (nitrogen, air and hydrogen) and one line for supplying liquid, equipped with a dosing pump DZN-2 (OKBA NPO Chimavtomatika).

Reaction product analysis was performed both directly after the reactor and from receivers cooled with dry ice. An LHM-80 chromatograph with a flame ionization detector was used for online analysis of samples of the gas mixture conversion products. The column, 3 m long and 3 mm internal diameter, was filled with Chromosorb M coated with squalane (10 wt%). The thermostatting temperature was 343 K, and the carrier gas (nitrogen) pressure was

0.8-0.9 kgf/cm^2. The analysis results were XL chromatograph, Perkin Elmer (capillary column (100 m long), methylphenylsiloxane phase), which allowed for a more detailed identification of the hydrocarbon composition of the conversion products using a computer program.

The conversion (α) of the reactants was calculated:

$$\alpha = \frac{m - m_K}{m} \times 100\% = \frac{\Delta m}{m} \times 100\%$$

where m is the initial amount of reactant; m_k is the residual amount of reactants in the catalysate.

The selectivity for product formation was calculated taking into account their content in the catalysate (l_i):

$$Sel = \frac{l_i}{\sum l_i} \times 100\%$$

additionally monitored using an Auto System

Results and discussion

The obtained results show that the main converted components of GG on composite catalysts are C_{4-} and C_{7+} hydrocarbons of GG, and the obtained products are mainly n-C_5, i-C_5 and i-C_6 (Table 2). This shows that, during the conversion of GG, first C_{4-} and C_{7+} hydrocarbons form the bimolecular intermediate [C_{4-} - C_{7+}], and then this intermediate is divided into i-C_5, n-C_5 and i-C_6. Thus, high and low molecular weight components of GG are converted into medium molecular weight components. This averaging proceeds with superior formation of isomers on synthesized catalysts.

Table 2. Temperature dependence of GG conversion on C-6 catalyst.H_2/CH=1 : 3, WHSV = 2h^{-1}.

T, ^{0}C	$\sum C_{4-}$	iC$_5$	C$_5$	iC$_6$	C$_6$	iC$_7$	$\sum C_{7+}$
	Composition of raw material (GG) , %						
	5.5	**25.2**	**19.2**	**18**	**8.4**	**5.4**	**18**
	Composition of catalysate, %						
140	4.5	28	20.3	20.2	6.3	4.2	16.5
160	1.8	34	24.4	21.6	2.8	3.6	11.8
180	1.0	37.5	26	22.6	2.6	4.5	5.8
200	0.4	26.6	32.2	19.8	2.7	7.9	10.4
220	1.0	26	31.0	19.4	1.5	9.0	12.2
250	2.0	25.4	27	20.0	2.0	10	14.0

Based on the obtained results, the reaction temperature can be divided into two ranges: 140 - 180^{0}C and 200 - 250^{0}C (Table 2). Intensive accumulation of C_5-C_6 isomers, which are high-octane gasoline components and consumption of C_{4-} - C_{7+} components are observed in the first temperature interval. This indicates that in the temperature range of 140 - 180^{0}C, hydroisomerization, which is one of the two main conversion pathways of the intermediates formed in the system, prevails.

In the second temperature interval, there is a marked decrease in the conversion of both C_{4-} and C_{7+}, and accumulation of i-C_7 occurs. The appearance of C_1-C_2 hydrocarbons in the products indicates the dominance of hydrocracking in the temperature range of 200 - 250^{0}C. The simultaneous change of C_{4-} and C_{7+} indicates their joint participation in the formation of intermediates that form the conversion products of GG.

Thus, as the temperature increases, the isomerization activity of the catalyst decreases and the hydrocracking capacity increases. This is shown by the fact that the ratio of selectivity for n-pentane to selectivity for iso-pentane and iso-hexanes (n-C_5/i-C_5-C_6) approaches unity with increasing temperature (table 3).

Table 3. Temperature dependence of gas gasoline conversion over C - 6 catalyst. H_2/CH = 1 :3;WHSV = $2h^{-1}$, τ=30 min.

T, ^0C	Conversion of C_{4-} və C_{7+} components of GG, %	Selectvity for izocomponents, %	n-C_5/i-C_5-C_6
180	74	69	0.44
200	75	66	0.51
220	79.6	50	1

In order to study the effect of the amount of sulfate anions, the activity of C-2, C-4, C-6 and C-8 catalysts with 2, 4, 6 and 8% SO_4^{2-} anions in the conversion of GG were studied. The results of the studies are given in Table 4 and Figures 1 and 2. As can be seen from Table 4, significant conversion of the C_{4-} and C_{7+} components of GG occurs on all four catalysts at 180^0C within 30 – 45 minutes, and then the activity of the catalysts gradually stabilizes. Although the difference in the amount of sulfate anions does not affect the overall course of GG conversion, it manifests itself in the conversion of the C_{4-} and C_{7+} components of GG, selectivity for isomers and the yield of n-C_5 (Figures 1 and 2).

Table 4. Dependence of the conversion of gas gasoline on the reaction time over composite catalysts. H_2/CH = 1 : 3, WHSV = $2h^{-1}$, T = 180^0C.

Reaction time, min.	∑C_{4-}	iC_5	C_5	iC_6	C_6	iC_7	∑C_{7+}
	Composition of raw material (GG) , %						
	5.5	**25.2**	**19.2**	**18**	**8.4**	**5.4**	**18**
	Composition of catalysate , %						
	C– 8 (8%SO_4^{2-})						
15	1.2	25.5	24.6	22	3.2	8	15.5
30	1	26	25	24	2.8	6.0	15.2
45	1.4	28	24	20	3.0	10.2	13.4
C– 6 (6%SO_4^{2-})							
15	1.2	35	24	21	4.6	4.8	9.4
30	0.8	37.5	26	23.3	2.6	4.0	5.8
45	1.0	28	28	26.4	3.3	4.5	8.8
60	2.0	27	29.4	24.6	3.9	4.1	9
C – 4 (4%SO_4^{2-})							
15	1.2	28	21.3	24.4	3.2	5.8	16.1
30	0.8	31.8	22	26.3	1.9	6.4	10.8
45	1.0	32.5	23.3	27.5	2.0	7.2	6.5
C – 2 (2%SO_4^{2-})							
15	1	26	19.8	24	2	11.2	16
30	0.9	30.0	20.8	25	1.0	7.2	15.1
45	1.3	33.5	17.8	21.1	2.7	9.2	14.4

Figure 1 shows the conversion of C_{4-} and C_{7+} components of GG over C-2, C-4, C-6 and C-8 at different temperatures. As can be seen from the figure, the conversion of C_{4-} and C_{7+} components of GG over the catalysts increases as the temperature increases.

In the range of 180-220^0C, regardless of the temperature, the conversion of C_{4-} and C_{7+} components of GG passes through a maximum depending on the amount of sulfate anions. Thus, when the amount of sulfate anions increases from 2 to 6%, the conversion of C_{4-} and C_{7+} components of GG increases, and when the amount reaches 8%, the conversion decreases (Figure 2).

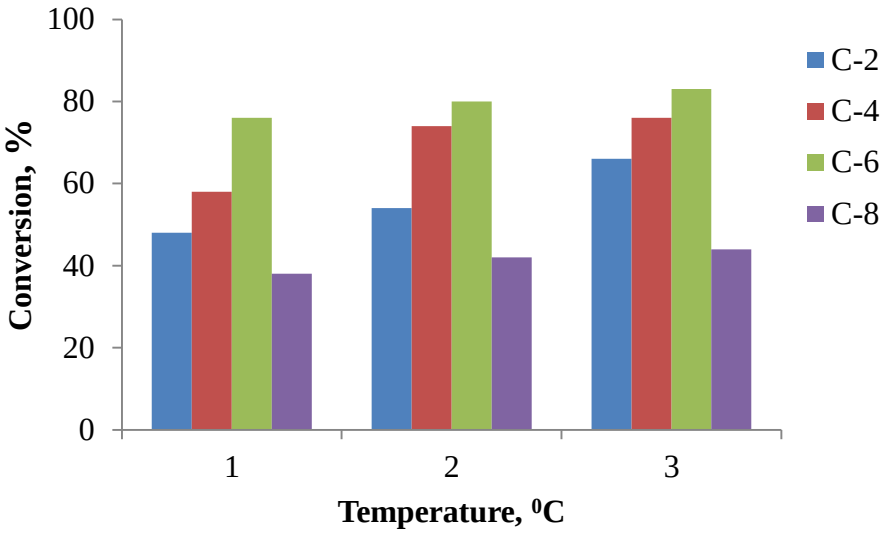

Fig. 1. Conversion of C_{4-} and C_{7+} components of GG on catalyst samples. H_2/CH = 1 : 3, WHSV = $2h^{-1}$. 1- 180^0C; 2- 200^0C; 3- 220^0C.

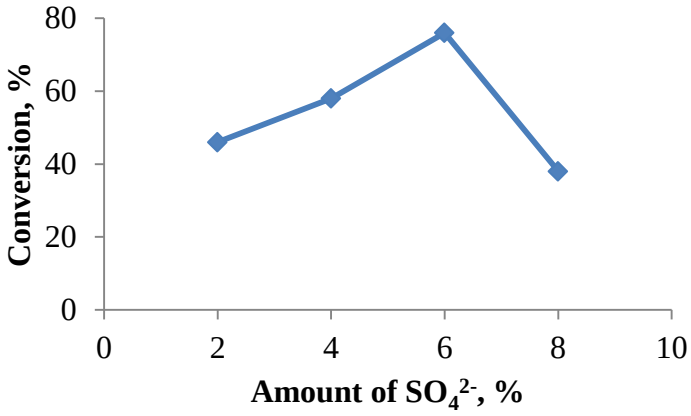

Fig.2. The dependence of the conversion of C_{4-} and C_{7+} components of GG on the amount of SO_4^{2-} ions. H_2/CH = 1 : 3, WHSV= $2h^{-1}$, T = 180^0C.

Such dependence of the catalyst activity on the amount of sulfate anions is due to the state of sulfate groups on the surface of the catalyst and its acidic properties. Thus, as the amount of SO_4^{2-} anions increases, the isolated tridentate sulfate groups on the surface are converted into binuclear bidentate $S_2O_7^{2-}$ groups, which is accompanied by a significant increase in the strength of Bronsted acid centers. With a further increase in the amount of SO_4^{2-} anions, the strength of Bronsted acid centers and their amount on the surface practically do not change, and at values of the amount of SO_4^{2-} ions above 6%, the catalytic activity decreases [23].

The selectivity for isomers also depends on the amount of sulfate ions and temperature, as shown in Figure 3. Thus, as the temperature increases, the selectivity for isomers on the catalysts decreases. However, the selectivity of C-2, which is more selective in the range of 180-220^0C, decreases sharply at 220^0C and becomes lower than the selectivity of C-4, C-6 and C-8. At this temperature, the C-4 catalyst with 4% sulfate anions becomes more selective. The decrease in isoselectivity with an increase in the amount of sulfate anions is associated

with an increase in hydrocracking as a result of increased acidity. As it is known, these processes proceed with the formation of a carbenium intermediate ion, and the superiority of isomerization and cracking reactions depends on the residence time of these ions on the catalyst. If the acidity is not very strong, the residence time of these intermediate ions on the catalyst surface is short and they quickly convert to isomers, hydridize and leave the surface in the form of isomers. When the acidity of the surface is strong, the intermediate carbenium ions formed can remain on the surface longer, which allows them to undergo hydrolysis, i.e., hydrocracking [24].

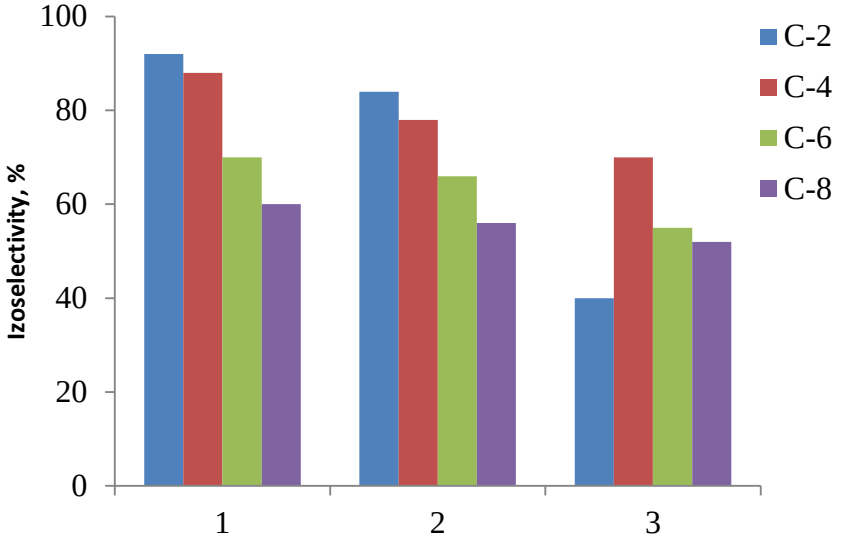

Fig.3. Temperature dependence of selectivity for isomers in the conversion of GG over catalyst samples. H_2/CH = 1:3, WHSV = $2h^{-1}$. 1- 180^0C; 2- 200^0C; 3- 220^0C.

The above ideas are proven by the dependence of n-pentane yield on the amount of SO_4^{2-}anion and temperature (Figure 4).

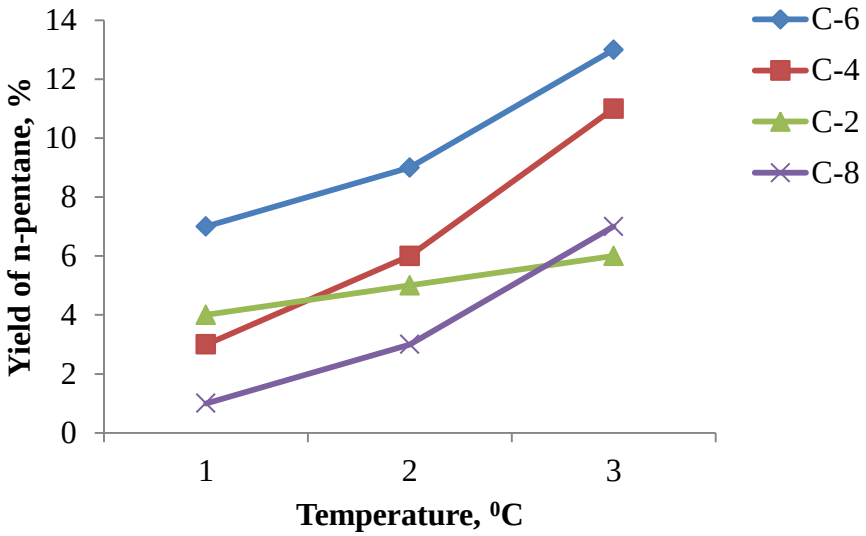

Fig.4. Temperature dependence of n-C_5H_{12} yield on C-2, C-4, C-6 and C-8 samples. H2/CH = 1 : 3, WHSV = $2h^{-1}$. 1- 180^0C; 2- 200^0C; 3- 220^0C.

As can be seen from the figure 4, the yield of n-C_5H_{12} increases with increasing temperature on all four catalysts. The dependence of the yield of n-pentane on the amount of sulfate anions corresponds to the dependence of isoselectivity on the amount of sulfate anions. Thus, the yield of n-pentane is highest on the C- 6 sample, where the conversion of the C_{4-} and C_{7+} components of the GG is highest at 180^0C, and the selectivity for isostructured hydrocarbons is low (except for C – 8). As the temperature increases, this yield increases, hydrocracking accelerates on all catalysts, and isomerization weakens. This is also shown by the ratio of the selectivity of normal pentane to the selectivity for isoalkanes (isopentane + isohexane + isoheptane) (n-C5/i-C5-C7) calculated for the catalysts (table 5).

Table 5. Temperature dependence of the ratio $n - C_5 / \sum iC_5 - iC_7$ in the conversion of GG over C-2, C-4, C-6 and C-8 catalyst samples. H_2/CH = 1 : 3, WHSV = $2h^{-1}$.

Temperatur, ^{0}C	C – 8	C - 6	C- 4	C - 2
180	0.75	0.45	0.16	0.1
200	0.9	0.52	0.37	0.2
220	1.4	1	0.57	1.9

As can be seen from Table 5, the smallest $\frac{\mathrm{n-C_5}}{\sum \mathrm{iC_5}} - \mathrm{iC_7}$ ratio at 180 – 200^0C belongs to C – 2. This proves that the more isoselective catalyst is the C - 2 catalyst with 2% of SO_4^{2-} anions. Although this catalyst is more selective, its selectivity is more sensitive to temperature and decreases more rapidly with increasing temperature. On the other hand, the conversion and yield of n-pentane on it are small. Although the C-6 catalyst lags behind C-4 and C-2 in terms of selectivity, its selectivity is less sensitive to temperature, since when the temperature changes from 180^0C to 200^0C, there is a very slight change in its isoselectivity. However, it is superior to all three remaining catalysts in terms of the yield of n-pentane and the conversion of the C_{4-} and C_{7+} components of the GG. Thus, C-6 catalyst is an effective catalyst for high conversion of GG and obtaining n-pentane, and C-2 catalyst is an effective catalyst for high isoselectivity.

Conclusion

Thus, composite solid superacid catalysts synthesized based on sulfated zirconium dioxide and cobalt-modified HZSM-5 zeolite allow the conversion of gas gasoline into high-octane gasoline components at low temperature (180^0C) and atmospheric pressure.

The intermediate compound formed during the process undergoes transformation in two directions: isomerization and hydrocracking. The rate of these processes depends on the reaction temperature. Isomerization prevails in the range of 140-180^0C, and hydrocracking in the range of 200-250^0C. The isomerizing activity of the catalyst also depends on the amount of sulfate anions: at low concentrations (2 wt.%) the conversion is relatively low, but the yield of isomers is high; at higher concentrations (6%), the opposite is true.

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REAKSİYA TEMPERATURUNUN VƏ SULFAT ANİONLARININ QATILIĞININ QAZ BENZİNİNİN KOMPOZİSİYA TƏRKİBLİ KATALİZATOR ÜZƏRİNDƏ ÇEVRİLMƏSİNƏ TƏSİRİ

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Sulfatlaşdırılmış sirkonium dioksid və kobaltla modifikasiya olunmuş HZSM-5 seoliti əsasında kompozisiya tərkibli katalizatorlar sintez edilmiş və onların iştirakı ilə qaz benzininin çevrilməsi öyrənilmişdir. Məlum olmuşdur ki, bu katalizatorların iştirakı ilə qaz benzininin çevrilməsi zamanı onun C_{4-} və C_{7+} komponentləri i-C_5, n-C_5 və i-C_6 alkanlarına çevrilir. Proses zamanı əmələ gələn [C_{4-} - C_{7+}] bimolekulyar intermediatı iki istiqamətdə parçalanır və sistemdə iki proses gedir: izomerləşmə və hidrokreking. Bu proseslərin sürəti reaksiyanın temperaturundan və sulfat anionlarının miqdarından asılıdır: izo-alkanların alınması 180^0C-də maksimumdan keçir və ən yaxşı izomerləşdirici katalizator 2% sulfat anionlarına malik katalizatordur. Məlum olmuşdur ki, sintez olunmuş katalizatorlar qaz benzinini yüksək oktanlı benzin komponentlərinə çevirməyə imkan verir.

Açar sözlər: *qaz benzini, sulfatlaşdırılmış sirkonium dioksid, seolit, izomerləşmə, yüksək oktanlı benzin komponentləri, bimolekulyar intermediat*