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ABSTRACT

of the dissertation for the degree of Doctor of Philosophy

**STUDY OF LOW-TEMPERATURE ISOMERISATION-
DISPROPORTIONATION PROCESS OF CONVERSION OF
STRAIGHT-RUN GASOLINES WITH THE PARTICIPATION
OF COMPOSITE CATALYSTS BASED ON ZEOLITE**

Speciality: 2314.01 – Petrochemistry

Field of science: Chemistry

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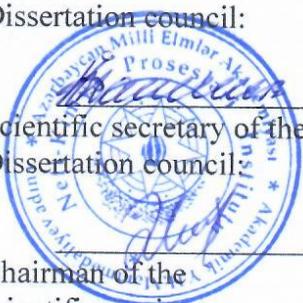
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GENERAL CHARACTERISTICS OF THE WORK

The relevance of the work. Modern stringent environmental requirements for gasolines imply the ultimate limitation of aromatic hydrocarbons in them, while maintaining their high anti-knock characteristics. One of the solutions to this problem is the transfer of high-temperature dehydrocyclization processing of straight-run gasoline to low-temperature isomerization processing. Isomerization of C_{7+} hydrocarbons, limited by the fact that they are converted into by-product C_1 - C_4 hydrocarbons, contribute to catalyst deactivation.

Isomerization of n-alkanes can proceed by means of mono - or bimolecular intermediates, their formation, and cleavage. The use of high pressures of hydrogen and the introduction of platinum into the isomerizing industrial catalyst block the formation of bimolecular intermediates, which accumulate on the catalyst surface at low temperatures and lead to its deactivation. However, carrying out the process through the formation of a bimolecular intermediate and its isomerization and disproportionation can make it possible to involve in the process both C_{7+} components of straight-run gasolines and gaseous alkanes. The occurrence of such reactions, with the formation of C_5 - C_6 alkanes with a high content of isocomponents, is shown by examples of the conversion of hexane-butane and heptane-butane mixtures with the participation of composite catalysts, the components of which are Co (Ni) H-zeolite (MOR or ZSM-5) and zirconium dioxide modified with the SO_4^{2-} or WO_4^{2-} anion.^{1,2}

The need to develop methods for processing straight-run gasolines into high-octane gasolines that do not contain aromatic components requires the involvement of C_{7+} in low-temperature isomerization processing and is an urgent problem in petrochemistry.

1. Abasov, S.I. Joint conversion of n-hexane and n-butane on zirconium-zeolite catalysts / S. I. Abasov, S. B. Agaeva, R. V. Starikov, M. T. Mamedova, A. A. . Iskenderova, E. S. Isaeva, A. A. Imanova, D.B. Tagiev // Catalysis in industry, -2015, - vol.15, No. 4, - p.73-78.

2. Abasov, S.I. Conversion of n-heptane, n-butane and their mixtures on catalytic systems of Al_2O_3/WO_4^{2-} - ZrO_2 and $HMOR/WO_4^{2-}$ - ZrO_2 / S. I. Abasov, S. B. Agaeva, M. T. Mamedova, E. S. Isaeva, A.A. Imanova A.A. Iskenderova, A.E. Alieva, R.R. Zarbaliev, D.B. Tagiev // Applied Chemistry, - 2018, -vol.91, Issue 6, -p. 838-845.

Solving this problem by directly involving straight-run gasoline in the low-temperature process of coupled isomerization and disproportionation using composite catalytic systems, the components of which have similar functions, will increase the resources of C₅-C₆ hydrocarbons with a high octane number.

Subject and object of research. The object of research is straight-run gasolines, associated oil gases, gaseous alkanes, a mixture of butane and heptane. Involvement of straight-run gasolines, accompanying oil gases, gaseous alkanes, butane and heptane mixtures in a low-temperature isomerization-disproportionate catalytic process with the participation of composite catalytic systems.

The purpose and objectives of the work. Research and development of the process of increasing the resource of high-octane components of modern gasolines that do not contain aromatic hydrocarbons by processing straight-run gasolines in the presence of composite Me (Co, Ni) H-zeolite (ZSM-5, H-MoR)/SO₄²⁻/ZrO₂ catalysts .

✓ Composite catalysts, the components of which are Ni or Co / H zeolite HZSM, MoR and sulfated zirconia have been synthesized and selected for the low-temperature isomerization-disproportionate transformation of straight-run gasoline with the formation of C₅-C₆ alkanes;

✓ conditions of the involvement of straight-run gasoline in the reaction of low-temperature isomerization-disproportionation transformation on composite catalytic systems have been established

✓ the influence of the composition of the composite catalyst on the isomerization-disproportionation conversion of straight-run gasoline was revealed;

✓ substantiated the mechanism of conversion of straight-run gasoline on a composite catalytic system

✓ the isomerization-disproportionation properties of the composite catalyst were estimated by the example of a model reaction of the transformation of mixtures n-C₄H₁₀: n-C₇H₁₆.

✓ the main regularities of the conversion of straight-run

gasoline and propane-butane fraction on a composite catalytic system have been revealed.

Research methods. Gas chromatographic analysis, XRD, EPR, and IR spectroscopy were used to accurately determine the composition of the feedstock, products of isomerization-disproportionation transformation, and composite catalytic systems.

The reliability of the results. The degree of reliability of the results obtained is confirmed by the use of modern spectral and analytical methods of analysis.

Basic provisions of defence:

✓ contacting straight-run gasoline with composite catalytic systems, the components of which are modified Co or Ni H-zeolite (MOR; ZSM-5) and sulfated zirconium dioxide, promoted the formation of C₅-C₆ paraffins;

✓ primary activation of C₇₊ alkanes and the formation of bimolecular intermediates proceeds with the participation of active (oxidative) centers of the SO₄²⁻ - ZrO₂ component, and hydro-cleavage into final products by active (acidic) centers of H-zeolite;

✓ conditions for the implementation of the process of converting straight-run gasoline into C₅-C₆ in the presence of a composite catalytic system have been established;

✓ kinetically and experimentally substantiated of the assumptions about the isomerization-disproportionate transformation of C₇₊ components;

✓ the possibility of involving the propane-butane fraction in the isomerization-disproportionate transformation with straight-run gasoline.

Scientific novelty:

- For the first time, the possibility of direct involvement of straight-run gasoline in the low-temperature isomerization disproportionate process of obtaining C₅-C₆ alkanes with a high content of structurally branched components has been established.

- Found the conditions for the conversion of straight-run gasoline on a composite catalytic system with the formation of C₅-C₆ components.

- On the basis of kinetic studies of straight-run gasoline and a model reaction of butane and heptane, the regularities of the formation and decomposition of a bimolecular intermediate into final products have been established.

- It has been shown that the primary activation of the isomerization-disproportional process of conversion of gaseous C_4 - and C_{7+} proceeds with the participation of SZ oxidative centers, and the stabilization of the bimolecular intermediate and its decomposition into final products with the participation of the zeolite component.

- For the first time, the possibility of a joint process of converting straight-run gasoline and gaseous alkanes into C_5 - C_6 alkanes with a high content of branched components has been established.

Theoretical and practical value.

- conjugation of redox and acidic properties on the developed composite catalytic systems allows the formation of a bimolecular intermediate with the participation of liquid C_{7+} and gaseous C_4 hydrocarbons and their splitting into the target C_5 - C_6 alkanes;

- the ability to transfer the process of converting straight-run gasoline from high-temperature dehydrocyclization processing to low-temperature isomerization-disproportionate processing on a composite catalytic system, which allows increasing the resource of C_5 - C_6 paraffins with a high content of branched components which are necessary for the production of modern high-octane gasolines;

- allows to involve in the process of conversion of straight-run gasoline components of accompanying oil gases, and also eliminates the need for fractionation stages of straight-run gasoline used to obtain benzene-free components and regulating the content of aromatic hydrocarbons;

- simplification of the processing of straight-run gasoline using industrial technology isomerization of C_5 - C_6 alkanes will reduce the energy and material consumption of the production of high-octane gasolines that do not contain aromatic hydrocarbons.

Approbation and application of the work.: According to the

data of the dissertation work, 21 works were published, 9 of which are articles, and 12 of them are abstracts of reports of international and republican scientific conferences.

The results of the work were published in republican and international journals: Processes of Petrochemistry and Oil Refining (Azerbaijan) - 4 articles; Chemical problems-1 article (Azerbaijan); Applied Chemistry (Russia) - 2 articles; Refining and Petrochemistry (Russia) -1 article; Young scientist (Russia) -1 article.

The main results of the dissertation were reported, discussed in the form of oral and poster presentations at international and republican conferences: X International Conference "Mechanisms of Catalytic Reactions" (Svetlogorsk, Russia, 2016); V Russian Conference (with international participation) Actual problems of petrochemistry. Dedicated to the memory of Academician V.N. Ipatieva .; V Russian scientific conference "Actual problems of petrochemistry" (Zvenigorod, 2016). ; IX Baku International Mammadaliyev Conference on Petrochemistry (Baku, 2016); III Russian Congress on Catalysis (Nizhny Novgorod, 2017); 3rd International Turkic World Confection on Sciences and Technologies (Baku, 2017); International Scientific and Technical Conference "Petrochemical Synthesis and Catalysis in Complex Condensed Systems" dedicated to the 100th anniversary of Academician B.K.Zeynalov (Baku, 2017); Scientific Conference dedicated to the 110th anniversary of Academician M. Nagiev (Baku, 2018); IV International Turkic World Conference on Chemical Sciences and Technologies (Ukraine, Kiev 2018); International scientific-practical conference dedicated to the 110th anniversary of academician V.S. Aliyev (Baku, 2018); IV All-Russian (with international participation) scientific symposium (Ivanovo-Suzdal, 2019); IV Scientific - Technological Symposium Catalytic Hydroprocessing in Oil Refining (Novosibirsk, 2021); IV Russian Congress on Catalysis "Roscatalys" (Kazan, 2021).

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Place of the dissertation work. The research presented in this dissertation was carried out in accordance with the research program of the Institute of Petrochemical Processes of ANAS 2016-2019.

Personal contribution of the author. Statement of problems, collection and generalization of literature data, development and implementation of experiments, preparation of samples for their further study, systematization of results, compilation of articles and abstracts, as well as interpretation and generalization of data from physicochemical analyzes were carried out mainly by the author.

The structure and scope of the dissertation. The dissertation work is presented on 143 pages of computer text and consists of an introduction - 6 pages (10639 characters); four chapters: literary review - 30 pages (54597 characters), experimental part - 13 pages (15309 characters), discussion of the results (3 and 4 chapters) - 73 pages (86414characters); conclusions - 2 pages (2159characters); list of used literature, consisting of 157 bibliographic titles. The thesis includes 32 tables and 18 figures. The total volume of the dissertation is 169048 characters (without tables, figures, annexes bibliography).

In the first chapter of the dissertation, analyzes of the information available in the literature on the conversion of straight-run gasoline on zeolite catalytic systems of various types are given, as well as areas of application based on the results obtained.

The second chapter presents methods for the synthesis of catalysts, experiments and physicochemical studies.

The third chapter presents the main research results obtained in the course of the work carried out on the isomerization-disproportionate conversion of straight-run gasoline in the presence of composite catalytic systems. The influence of the composition of straight-run gasoline, the composition of composite catalysts, and modification of catalyst components on the conversion of straight-run gasoline is shown. The kinetic features and the mechanism of low-temperature isomerization-disproportionate transformation of straight-run gasoline on a composite catalytic system are discussed.

In the fourth chapter, the main results of the study of the joint

conversion of straight-run gasoline and the propan-butane fraction, as well as the model reaction of the conversion of n-butane and n-heptane and their mixtures on composite catalyst systems are presented.

MAIN CONTENT OF WORK

Conversion of straight-run gasolines with the participation of composite catalytic systems. The synthesized composite catalysts and their components were tested in contact with straight-run gasolines in the temperature range 180-200⁰C. The main technological parameter of the reaction is the temperature of its realization. Studies have shown that at temperatures below 220 ° C, individual components of the composite catalytic system do not show any noticeable activity in the conversion of straight-run gasoline. In contrast to individual components, contacting straight-run gasoline with a composite catalytic system leads to a significant change in the distribution of hydrocarbons in the catalyzate. From the obtained data (Table 1), it follows that the content of higher molecular weight paraffins of C₇ + decreases monotonically with an increase in temperature from 140 to 200⁰C.

The conversion products of these paraffins are C₆- alkanes. The distribution of these alkanes in the catalyzate has a complex temperature dependence.

An analysis of the data (Table 1) shows that in the temperature range 140–160⁰C, a total increase in C₅ – C₆ alkanes is observed in the reaction products at an almost constant ratio of isomeric and normal alkanes.

With increasing temperature, the distribution of hydrocarbons in the catalyzate changes significantly. Along with a decrease in the yield of iso-(C₅ – C₆), the yields of n-C₅ and unwanted C₄- paraffins increase. A change in the nature or composition of the components of the composite catalyst leads to changes in the composition of the catalyzate. When straight-run gasoline interacts with a composite catalyst with a high content of SO₄²⁻ (6%), the distribution of hydrocarbons in the products changes significantly.

Table 1
Temperature dependence of the conversion of straight-run gasoline on the catalyst M-11. (0.4%Co(HZSM)/SO₄²⁻(2%)-ZrO₂)
v_{H2}=40ml/min, WHSV = 2.5h⁻¹

T, °C	Initial composition, wt%											
	C _{1-C₂}	C ₃	iso-C ₄	C ₄	iso-C ₅	C ₅	iso-C ₆	DMB*	C ₆	iso-C ₇	C ₇	8+
	Hydrocarbon composition, wt%											
	-	-	-	1.0	1.0	1.2	5.0	-	3.8	22.0	6.5	59.5
140	-	-	-	-	24.3	2.8	22.6	23.6	3.6	3.2	5.7	37.8
160	-	-	0.9	-	29.3	4.6	28.7	23.0	3.4	4.5	3.4	25.2
180	0.5	2.2	15.2	3.0	24.6	9.7	23.5	18.5	3.3	2.4	2.4	13.8
200	4.8	25.3	27.5	2.4	8.3	12.4	7.9	0.7	2.8	1.8	1.8	6.0

* Total content of dimethylbutane (DMB) in isohexane

Contacting straight-run gasoline with a composite catalyst in the temperature range 180-220°C leads to a decrease in the content of its high-temperature C₈₊ component by 18.9-27.5%, corresponding to an increase in the concentration of C₅-C₆ components. The conversion of C₈₊ with the participation of composite catalysts proceeds with an insignificant change in the content of gaseous C₄-paraffins, i.e. practically without the formation of by-products of the composition C₁-C₃ (Table 2).

Table 2
Conversion of straight-run gasoline on catalyst M-12
(0.4%Co(HZSM)/SO₄²⁻(6%)-ZrO₂) τ=30 min, CH/H₂ 1:3; WHSV = 2.5h⁻¹

T, °C	Initial composition, wt%							
	C ₄	iso-C ₅	C ₅	iso-C ₆	C ₆	iso-C ₇	C ₇	C ₈₊
	1.0	1.0	1.2	5.0	3.8	22.0	6.5	59.5
Hydrocarbon composition, wt%								
180	0.6	2.5	4	11.2	10.9	21.8	8.4	40.6
220	1.5	5.2	2.9	12.6	10.6	17.8	17.0	32.0

The decrease in the content of C₈₊ components (i.e., the conversion of higher molecular weight components of straight-run benzene) on B-166 is higher than on M-12, but lower than on M-11.

Content of hydrocarbons iso-C₆ and n-C₆, are also higher. It should be noted that the ratio of iso-C₆/n-C₆ in the case of catalyst B-166 significantly exceeds the analogous value on M-12. However, the main products of C₈₊ conversion are also C₅ – C₆ paraffins. Replacement of metal-HZSM-5 in a composite catalyst with metal-HMOR does not change the general nature of changes in the composition of straight-run gasoline when in contact with a similar composite catalyst (B-166).

Table 3

**Conversion of straight-run gasoline on catalyst
B-166 1%Ni (HMOR /SO₄²⁻ (6%)-ZrO₂), τ=30min, CH/H₂ 1:3,
WHSV = 2.5h⁻¹.**

T, °C	Initial composition, wt%							
	C ₄	iso-C ₅	C ₅	iso-C ₆	C ₆	iso-C ₇	C ₇	C ₈₊
	1.0	1.0	1.2	5.0	3.8	22.0	6.5	59.5
Hydrocarbon composition, wt%								
180	2.6	7.8	5.8	21.0	13.0	20.0	7.5	21.8
200	1.5	5.2	4.5	19.2	15.3	21.0	8.0	24.8
220	2.4	5.5	4.2	17.5	14.2	21.0	9.0	26.2

From a comparison of tables 1-3, it follows that on a catalyst containing a smaller amount of SO₄²⁻ - ions, an increase in temperature from 180 to 200°C leads to both an increase in the conversion of C₈₊ hydrocarbons and a noticeable increase in the conversion of iso-C₇ and n-C₇. However, an increase in temperature within the shown limits does not significantly affect the overall change in the products of the process, the main products of which are C₅ – C₆ alkanes, with a significant content of iso-components.

Considering the difficulty of the structural isomerization of n-paraffins on unmodified HZSM-5 zeolites and vice versa, the ease of such a transformation on HM, such a difference in the distribution of the products of contacting straight-run gasoline with composite

catalysts indicates the conjugate nature of the functioning of SO_4^{2-} - ZrO_2 and metal/H-components in the process of converting straight-run gasoline.

Taking into account that the main products of the conversion of straight-run gasoline with the participation of composite catalysts are $\text{C}_5 - \text{C}_6$ alkanes and the dependence of their content in the catalyzate, other things being equal, on the component composition of the catalyst, it can be concluded about generality of process regularity on composite catalysts metal/H-zeolite/ SO_4^{2-} - ZrO_2 and the objective reality of the conversion of C_{7+} components of straight-run gasoline into C_5 - C_6 alkanes with a high content of branched molecules. Theoretical calculations show an increase in the octane number of straight-run gasoline in one pass over the composite catalyst by 15-20 points.

Influence of conditions on the conversion of straight-run gasoline.

The content of $n\text{-C}_5\text{-C}_6$ and $n\text{-C}_4$ components in the catalyzate can be conditionally associated with a decrease in the isomerizing activity and an increase in the cracking activity of the composite catalytic system.

The temperature dependence of the distribution of the components of the catalyzate of straight-run gasoline as a result of its contact with the composite catalytic system is generally limited by the processes of formation of a branched hydrocarbon intermediate and its decomposition (hydrogenolysis/ hydrocracking). An increase in temperature to $T \geq 180^\circ\text{C}$ leads to an increase in the cracking activity of the composite catalytic system, and the content of undesirable C_4 - alkanes at 200°C reaches 60%.

At a certain temperature, the distribution of hydrocarbons in the catalyzate is affected by the conditions of contact of straight-run gasoline with a composite catalytic system. Varying the volumetric velocity of straight-run gasoline leads to changes in the conversion of C_{8+} paraffins. In general, with an increase in the volumetric velocity, the conversion of C_{8+} components of straight-run gasoline and the yield of gaseous C_4 - alkanes decrease.

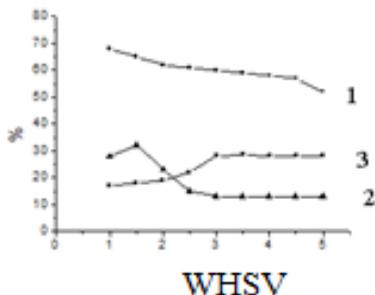


Figure. Influence of the volumetric feed velocity of straight-run gasoline on its conversion. Catalyst M-11, T = 180 ° C.

- 1- conversion of C₈₊;**
- 2- yield C₄₋**
- 3- DMB content in isohexane**

The data obtained show (Figure) that the conversion of C₇ paraffins is practically independent from the volumetric velocity variation, and the C₈₊ conversion remains constant in the velocity range 2.0–4.0 h⁻¹.

A change in the hydrogen – hydrocarbon ratio is also a factor in the redistribution of the hydrocarbon composition of straight-run gasoline. It was found that the conversion of straight-run gasoline upon contact with a composite catalytic system at a constant volumetric velocity (WHSV = 2.5 h⁻¹) with an increase in the linear hydrogen supply rate - v (H₂) leads to an increase in the conversion of C₇₊ components of straight-run gasoline and is accompanied by a similar increase in the yield of undesirable gaseous alkanes (Table 4).

The transformation of straight-run gasoline with an increase in v_{H_2} from 10 to 30 ml • min⁻¹ leads to an intensive increase in the conversion of high-molecular components of straight-run gasoline and the formation of gaseous alkanes, an other words, to an increase in hydrocracking; with an increase in $v_{H_2} \geq 40$ ml • min⁻¹, the noted parameters continue to increase slightly.

Table 4

Effect of hydrogen on the conversion of straight-run gasoline on the catalyst M-11. T = 180°C, WHSV = 2.5 h⁻¹, τ = 30 min.

ν _{H2} , ml/min	Conversion, wt%	Composition of catalyzate, wt%			
		C ₄₋	C _{5-C₆}	C ₇	C ₈₊
0	58.1	7.9	59.6(20.6)*	7.6	24.9
20	74.4	13.0	67.2(19.4)*	4.6	15.2
30	76.1	18.0	63.0(18.5)*	4.3	14.2
40	77.3	20.9	61.1(18.0)*	4.2	13.8
50	76.4	21.0	60.8(17.6)*	4.2	14.0

* total content of dimethylbutane in hexane

The accumulation of C₅ – C₆ alkanes in the catalysis has an extreme character and, with an increase in ν_{H2}, passes through the maximum value, which is in the range of 20–30 ml • min⁻¹. An increase in temperature leads to the intensification of the formation of gaseous C₄₋ alkanes (Table 3). The yield of these products also depends on ν_{H2} (Table 4). An increase in the yield of C₄₋ in the range of 10 ≤ ν_{H2} < 30 ml • min⁻¹ indicates the role of hydrogen as a co-reactant for hydrocracking of C₇₊ paraffins and a carrier gas that promotes the removal of products from the reaction zone. The extreme dependence of the C₅ – C₆ yield from ν_{H2} indicates a possible superposition of routes leading to their formation.

Based on the results obtained, it was assumed that the intermediate intermediates of these routes can be both monomolecular intermediates, formed with the participation of C₇₊ components of straight-run gasoline, and bimolecular, formed with the participation of both C₇₊ and the products of their transformations.

Influence of the composition of the catalyst components on the conversion of straight-run gasoline. The transformation of C₈₊ components and the formation as products of C₅-C₆ paraffins containing a significant amount of branched molecules suggests skeletal isomerization of the resulting intermediates and their hydro-degradation.

Table 5

Influence of Co HZSM-5/SO₄⁻²/ZrO₂ components on the conversion of straight-run gasoline. T = 180°C

2% SZ: Co/ HZSM	Conversion of C ₈₊ , %	Selectivity,%		6%SZ: Co/HZSM	Conversion of C ₈₊ , %	Selectivity,%	
		C ₅ -C ₆	Iso-C ₅ -C ₆			C ₅ -C ₆	iso-C ₅ -C ₆
1:1	31.0	19.5	61.0	1:1	13.1	6.7	72.0
1:3	47.0	29.6	59.0	1:3	16.3	8.3	64.0
1:9	59.0	37.2	56.0	1:9	40.8	18.2	61.0

Variations in the content of the zeolite component in the composite catalyst with the participation of SZ containing 2 and 6% sulfate ions showed that with an increase in the SZ : Co/HZSM ratio from 1 : 1 to 1 : 9 wt. leads to an increase in the conversion of C₈₊. Similarly, the yield of C₅-C₆ increases, but the content of isomeric C₅-C₆ paraffins decreases monotonically (table 5).

With a constant ratio of zeolite and SZ components, a change in the concentration of the modifying anion SO₄⁻² showed that the most effective samples are those containing from 2 to 4% SO₄⁻² (table 6).

Table 6

Influence of concentration of SO₄⁻² on the conversion of straight-run gasoline on Co/HZSM-5/SZ systems.

SO ₄ ⁻²	T, °C	Conversion of C ₈₊ , %	Distribution of products,%	
			Выход C ₅ -C ₆ (и-C ₅ -C ₆)	C ₇
2	180	59.0	37.2(56)	59.0
	200	55.8	30.4(52)	55.8
	220	52.8	28.6(54)	52.8
4	180	63.4	36.6(60.5)	63.4
	200	58.3	33.2(58.0)	58.3
	220	56.0	30.4(55.0)	56.0
6	180	40.8	18.2(61)	40.8
	200	50.4	20.8(67)	50.4
	220	71.6	35.9(69)	71.6
10	180	31.8	17.6(48.6)	31.8
	220	46.2	20.6(59)	46.2

These samples are characterized by a high yield of C₅-C₆ paraffins and the presence of isocomponents in them. Processes involving samples containing 6-10% higher concentrations of SO₄²⁻ ions require higher temperatures. However, the stability of these catalysts is not high.

The activity of composite catalysts in the studied process depends on the nature and conditions of reduction of the modifying metal (Table 7).

Table 7

The influence of modification of elements of the VIII group on conversion of C₈₊ on various catalysts. T = 180^oC, H₂/CH = 3; WHSV = 2.5h⁻¹.

time, min	Conversion of C ₈₊ , %		
	Co(HZSM)/SO ₄ ²⁻ (2%)-ZrO ₂ (reduction with hydrogen at 380 ^o C)	Ni, HZSM-5/SO ₄ ²⁻ (2%)-ZrO ₂ (reduction with hydrogen at 380 ^o C)	Co(HZSM)/SO ₄ ²⁻ (2%)-ZrO ₂ (reduction with hydrogen at 500 ^o C)
15	76.0	83.0	52.0
60	72.0	83.6	52.5
120	68.0	83.7	52.8
180	62.0	84.0	53.0
240	54.0	84.3	54.0
300	48.0	84.5	55.0

A sample containing Ni, all other condition being equal, is stable and more active, but less selective than containing Co. The low stability of the cobalt-containing sample is eliminated by increasing the temperature of the reduction treatment of the sample. However, this decreases both the activity of the sample, due to the partial reduction of SO₄²⁻, and the yield of the target C₅-C₆ alkanes, while the gaseous ones increase.

A sample containing Ni, all other condition being equal, is stable and more active, but less selective than containing Co.

The low stability of the cobalt-containing sample is eliminated by increasing the temperature of the reduction treatment of the sample. However, this decreases both the activity of the sample, due to the partial reduction of SO_4^{2-} , and the yield of the target $\text{C}_5\text{-C}_6$ alkanes, while the gaseous ones increase.

From a comparison of the data characterizing the conversion of straight-run gasoline on Ni- and Co-containing samples, it follows that elements of VIII B group are responsible for the formation of hydrogenolysis/hydrocracking products.

Kinetic study of the conversion of straight-run gasoline

Mathematical modeling of the optimization of simultaneously occurring reactions using kinetic data obtained in the study of the transformations of individual alkanes, their mixtures and straight-run gasoline directly with the participation of the M-11 composite catalyst was carried out using regression equations describing the yield of reaction products from the input parameters with appropriate restrictions.

Solved on the basis of the compiled statistical model of the process, the problem of optimizing the yields of pentane and hexane isomers

$$1.140 \leq X_1 \leq 200$$

$$0,8 \leq X_2 \leq 1,2$$

$$F_{\max} = 30.1\% \text{ at } X_1 = 143^{\circ}\text{C}; X_2 = 1.001 \text{ for } i\text{-C}_5\text{H}_{12}$$

$$1. 140 \leq X_1 \leq 200$$

$$3,7 \leq X_2 \leq 4,0$$

$$F_{\max} = 3,9\% \text{ at } X_1 = 141^{\circ}\text{C}; X_2 = 3,8 \text{ for } i\text{-C}_6\text{H}_{14}$$

where F-yield of reaction products

satisfactorily, describes the obtained experimental data.

The obtained mathematical models allow not only to predict the value of the response function for the given conditions of the experiment, but also provide information on the shape of the

response surface. The study of these surfaces allows to select the optimal mode of the technological process.

From the data obtained E_{act} and K_{rat} (table 8) it follows that the limiting stage of the conversion of straight-run gasoline is the more favorable primary isomerization of $C_8 +$ components, which is facilitated by low activation energies.

Table 8

The values of the calculated rate constants and activation energies.

hydrocarbons	t, °C	K_1 (formation rate constant)	K_2 (pre-exponential factor).	E_{act} , kJ/mol
C_{7+}	130	0.0593	1197.80	25.0
	190	0.020858		
	250	0.00144		
	310	0.001064		
i - C_5H_{12}	130	0.1555	2248	24.0
	190	0.025075		
	250	0.0094		
	310	0.001756		
$\Sigma(C_5-C_6)$	130	0.0593	16.05	7.0
	190	0.020858		
	250	0.00144		
	310	0.001064		
I - C_6H_{14}	130	1.04	41.62	9.0
	190	0.946529		
	250	0.3222		
	310	0.254593		

Model reaction of conversion of a mixture of butane and heptane in the presence of composite catalysts. The conversion of a mixture of n- C_4H_{10} : n- C_7H_{16} differs from the individual transformation of these substances. The introduction of n- C_4H_{10} into the reaction has a significant effect on the distribution of the conversion products of n- C_7H_{16} on the catalyst M-6a (Co/Al₂O₃/ SO₄²⁻ - ZrO₂). As a result, the content of C_5-C_6 alkanes in the catalyzate significantly increases. An

increase in temperature from 140 to 180°C leads to a monotonic decrease in the yield of C₅-C₆ and an increase in the yield of iso-C₇ alkanes (table 9). The yield of C₃ in this temperature range is significantly lower than that of the conversion of individual n-C₇H₁₆.

Table 9
Conversion of n-C₇H₁₆ : n-C₄H₁₀ (1: 1 mol) mixture on M-6a
(Co/Al₂O₃/SO₄²⁻ - ZrO₂)

T, °C	Distribution of products						
	Conversion, %			Selectivity, %			
	n-C ₄ H ₁₀	n-C ₇ H ₁₆	C ₄ + C ₇	C ₃	C ₄	C ₅ -C ₆	iso - C ₇
140	2.7	27.1	25.0	2.2	-	56.2	41.6
160	4.6	40.3	30.0	8.2	-	44.9	46.9
180	7.5	45.4	55.0	13.6	-	36.8	49.6
200	-	57.6	57.6	28.3	30.2	23.6	17.9

The presence of C₄H₁₀ in the reaction mixture reduces the stability of the Co/Al₂O₃/SZ functioning. During the conversion of n-C₇H₁₆, the catalyst under the experimental conditions remains stable for 120 minutes, but during the conversion of n-C₄H₁₀: n-C₇H₁₆, by the 30th minute of the experiment, the catalyst noticeably decreases its activity. Replacing the Co/Al₂O₃ component with Co/HZSM-5 affects the conversion of n-heptane.

Table 10
Effect of temperature on the conversion of n-heptane

T, °C	Co /Al ₂ O ₃ /SZ				Co/HZSM-5/ SZ			
	Conversion, %	Yield, wt%			Conversion, %	Yield, wt%		
		C ₃ -C ₄ *	C ₅ -C ₆	iso-C ₇		C ₃ -C ₄	C ₅ -C ₆	iso-C ₇
140	56.0	7.5	2.1	46.4	43	4.5	5.3	33.2
160	78.0	35.8	2.1	40.1	64	29.2	6.5	28.3
180	84.0	53.8	1.8	28.4	72	36.7	7.3	28.0
200	95.0	83.2	1.6	10.2	93	80.5	2.5	10.0

* C₁-C₂ content does not exceed 1%.

The conversion of n-C₇H₁₆ in the presence of Co/HZSM-5/SZ in the range 140-180⁰C is noticeably lower (~ 12%) compared to Co/Al₂O₃/SZ and leads to a decrease in the yield of C₃-C₄ and an increase in the yield of C₅-C₆ alkanes (table 10). Raising the temperature to 200⁰C practically eliminates the noted features of the n-C₇H₁₆ conversion in the presence of Co/ HZSM-5/SZ.

As a result of using the zeolite component, the involvement of n-C₄H₁₀ in the process sharply increases, the conversion of which exceeds the conversion of individual n-C₄H₁₀ on both SZ-containing catalysts. Under the influence of n-C₄H₁₀, the conversion of n-C₇H₁₆ on these samples decreases. Moreover, the dependence of the conversion of n-C₇H₁₆ from temperature becomes more complex, consisting in a decrease in this parameter with an increase in temperature above 160⁰C (table 11).

Table 11

**Conversion of n-C₇H₁₆: n-C₄H₁₀ (1 : 1) on a composite catalyst
Co/HZSM-5/SZ**

T, ⁰ C	Distribution of products						
	Conversion, %			Selectivity, %			
	n-C ₄ H ₁₀	n-C ₇ H ₁₆	n-C ₄ H ₁₀ + n-C ₇ H ₁₆	C ₃	C ₅ -C ₆	iso-C ₇	C ₈ +
140	21.5	28.2	26.0	21.0	54.2	24.8	-
160	30.6	41.0	37.2	28.2	40.6	28.6	2.6
180	43.4	36.7	39.2	19.0	36.6	39.9	4.5
200	5.0	23.5	16.6	46.9	30.3	22.8	-

A similar dependence on temperature is observed for the yield of C₅-C₆ products of conversion of a mixture of n-C₄H₁₀: n-C₇H₁₆.

Taking into account the absence of C₁-C₂ in the products, the involvement of n-C₄H₁₀ in the process, as well as the high values of C₃ yields on Co/HZSM-5/SZ as compared with Co/Al₂O₃/SZ, it is highly probable that C₃ and C₈ reaction products are the result of hydro degradation of bimolecular intermediates [C₄+ C₇]. The absence of C₈ in the reaction products at 140⁰C and low yields in the temperature range 160-180⁰C may result from both their higher boiling temperature and higher reactivity. The result of these effects

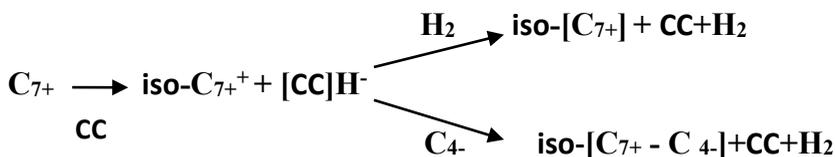
is a monotonic increase in the conversion of $n\text{-C}_4\text{H}_{10}$ in the range 140-180⁰C. A sharp decrease in the conversion of $n\text{-C}_4\text{H}_{10}$ during the transformation of the mixture at 200⁰C is a consequence of a decrease in the probability of the formation of bimolecular intermediates [$\text{C}_4 + \text{C}_7$].

The effect of $n\text{-C}_4\text{H}_{10}$ on the conversion of $n\text{-C}_7\text{H}_{16}$ is most pronounced on the stability of the catalyst functioning. From the data obtained, it follows that if the conversion of $n\text{-C}_7\text{H}_{16}$ on M-11 and Co / Al_2O_3 /SZ is sufficiently stable for 120 min, then upon the conversion of the $n\text{-C}_4\text{H}_{10}$: $n\text{-C}_7\text{H}_{16}$ mixture under the same conditions, the catalysts begin to lose activity already after 30 minutes of work. The use of a zeolite component in the catalyst leads to stabilization of the conversion of $n\text{-C}_4\text{H}_{10}$: $n\text{-C}_7\text{H}_{16}$. The reason for catalyst deactivation is blocking of its active surface with hydrocarbon deposits (HCD).

Comparison of the data characterizing the stability of the catalyst functioning with the data on the accumulation of HCD during the transformation of the mixture with their participation showed that the accumulation of 1.31% of HCD corresponds to the onset of inhibition of the process. Additional accumulation of HCD (1.51%) leads to a sharp decrease in the activity of the catalyst. As a result of replacing the alumina component with a zeolite one, the accumulation of HCS sharply decreases: in 120 min. reaches 1.05%., and the sample of Co/HZSM-5/SZ is stable during this time. Thus, the presence of an acidic component in the SZ containing catalyst contributes to a significant decrease in the accumulation of HCD. Elemental analysis of HCD shows that the H/C ratio falls within the range of 0.16 -0.18. and corresponds to the formula $(-\text{CH}_2-)_n$. It is assumed that HCD are formed stabilized intermediates, which are saturated hydrocarbons, the boiling temperature of which are higher than the temperature of the experiments. The introduction of a zeolite component increases the cracking activity of the catalyst. Effective cleavage of the [$\text{C}_4 + \text{C}_7$] intermediate into C_7 . products reduces the accumulation of HCD and stabilizes the process.

The formation of the conversion products of the n-C₄H₁₀: n-C₇H₁₆ mixture is an isomerization-disproportionation of the bimolecular intermediate and is a consequence of the synergism of the catalytic properties of SZ and Co/HZSM-5 components of the composite catalyst arising in the temperature range 140- 180⁰C.

The role of monomolecular and bimolecular activation in the conversion of a mixture of C₇₊ : C₄₋ paraffins in the presence of a composite Co/HZSM-5/SZ catalyst. Hydro-cleavage of high-molecular-weight intermediates of straight-run gasoline or a mixture of straight-run gasoline and propane-butane fraction can be represented by the following scheme.



where CC – composite catalyst

Under the influence of gaseous alkanes on the composite catalyst, intermediate substances with molecular weight [C₇₊ C₄₋] are formed, which are more active than C₇₊, isomerized and hydrocracked with the formation of C₅ – C₇ components with a high content of branched paraffins. At the same time, the yield of conversion products of straight-run gasoline or a mixture of straight-run gasoline : propane-butane fraction is the result of both monomolecular C₇₊ and bimolecular [C₇₊ C₄₋] transformations of reactant components, i.e. is an isomerization – disproportionation process. The distribution of the products of this transformation of a mixture of straight-run gasoline : propane-butane fraction can have a complex temperature dependence due to the possibility of the formation of additional intermediates [(C₇₊ + C₄₋) + C₄₋], due to further aggregation of intermediates (C₇₊ + C₄₋).

Physicochemical characteristics of catalysts. The maxima of intense absorption bands (ab) of IRS of composite catalysts and

Co/HZSM-5 contain a set of absorption bands, similar to those contained in the IR spectra of Co/HZSM-5 and SZ. Taking into account the satisfactory compliance of these as presented in table 11 with those described in the literature for IRS ZSM-5 and SZ, it can be assumed that there is no direct interaction between the Co/HZSM-5/SZ components.

From the X-ray diffraction pattern of sample M-11, it follows that the sample contains a zeolite phase characteristic of the structure ZSM-5 and tetragonal and monoclinic phases of zirconium dioxide.

Taking into account the results of IR spectral analysis, it seems that the compositional catalytic system, by analogy with the phase states established for the Pt / SZ-Al₂O₃ system, can be considered as a pseudo-solid solution of HZSM-5-SZ.

From the data presented above and their interpretation, it follows that the initial activation of the isomerization-disproportionate process of conversion of n-C₄H₁₀, n-C₇H₁₆ and their mixtures occurs with the participation of Lewis acid or redox centers localized on SZ. On the other hand, due to the zeolite component, the carrier of Bronsted acid sites, the yield of products of disproportionation of the intermediate substances of the transformation of n-C₄H₁₀, n-C₇H₁₆ and their mixtures increases sharply. The change in the selectivity of the composite catalyst as a result of replacing Co/Al₂O₃ with Co/HZSM-5 shows that due to the phase state close to the state of a solid solution, the centers located on its various components are conjugated. Thus, the effect of conjugation of centers located on different carriers predetermines the subsequent experimental behavior of the composite catalyst.

Influence of gaseous alkanes on the conversion of straight-run gasoline in the presence of a composite catalytic system.

Contacting a mixture of straight-run gasoline and propane-butane fraction with the synthesized composite catalysts at 160-200⁰C leads to a significant change in the distribution of paraffins contained in the initial mixture (table 12).

Table 12

**Conversion of a mixture of straight-run gasoline :
propane-butane fraction (1: 1) on catalyst M-11. T = 180°C.**

CH	time, min	Raw material composition, wt%							
		C ₄ -	iso-C ₅	C ₅	iso-C ₆	C ₆	iso-C ₇	C ₇	C ₈₊
		47.7	4.3	4.2	3.0	2.0	10.0	3.0	25.8
Composition of catalyzate, wt%									
2	15	15.7	10.7	4.0	15.7	5.7	16.4	18.9	12.9
	30	17.4	10.4	4.2	16.0	4.3	14.2	22.0	11.5
	45	17.9	10.8	4.9	15.9	6.9	14.4	17.3	11.9
	60	20.2	10.3	4.6	15.4	6.0	14.5	17.7	11.3
4	30	4.5	10.5	9.5	26.2	8.5	13.7	11.5	15.6
	45	4.3	11.2	8.7	26.4	8.3	14.0	11.4	15.7
	60	4.7	10.8	8.1	25.5	7.5	13.5	12.9	17.0

Gaseous C₄- and high-molecular weight C₈₊ components of the mixture are significantly reduced. An increase in the content of C₅-C₇ paraffins is observed in the catalyzate. Consequently, under the influence of high-molecular C₈₊ paraffins, which are capable of undergoing transformations at these temperatures on composite catalysts, low-active gaseous paraffins of the propane-butane fraction are involved in the formation of products with a large number of carbon atoms in the chain (C₅ - C₇).

The absence of the mixture of C₁ – C₂ hydrocarbons in the conversion products shows that the formation of C₅ – C₇ paraffins is preceded by the formation of bimolecular intermediates with the participation of C₈₊ and C₄ hydrocarbons and their subsequent hydro cleavage.

The increased content of C₅-C₇ alkanes with a branched structure indicates skeletal isomerization of bimolecular intermediates in the process of their formation, which is a factor facilitating the hydro cleavage of the intermediate. The involvement of C₄ hydrocarbons in the process under the influence of C₈₊ indicates the identity of the initial activation of C₈₊ components both

through the formation of a bimolecular intermediate $[C_{7+} C_4]$ and a monomolecular $[C_{8+}]$.

CONCLUSIONS

1. It was found that contacting straight-run gasoline in a hydrogen atmosphere at an atmospheric pressure of 0.1 MPa with composite catalytic systems, the components of which are H-zeolites (MOR; ZSM-5) modified with Co or Ni and sulfated zirconia (tetragonal structure) leads to the selective formation of C₅-C₆ paraffins [1].
2. The conversion of C₇₊ components of straight-run gasoline into C₅-C₆ paraffins with a high content of branched molecules is realized in the temperature range 140-200°C, volumetric velocities WHSV = 1-4h⁻¹ and hydrogen/hydrocarbon ratios = 2 - 4. It was found that on an effective catalyst 0.4%Co/HSZM-5/2%SO₄²⁻-ZrO₂ at a temperature of 180°C, WHSV = 2.5 h⁻¹ and H₂/CH = 3, the conversion reaches 77%, and the total concentration of C₅-C₆ paraffins containing up to 78% of branched components in the catalysate increases from 11% to 61% [4].
3. An assumption was made about the isomerization-disproportionation conversion of C₇₊ components of straight-run gasoline in the participation of M/H-zeolite/SO₄²⁻ - ZrO₂ catalytic systems by the formation of a bimolecular intermediate, its isomerization and hydro cleavage (mainly on the β-bond) [5].
4. It has been shown that the primary activation of C₇₊ alkanes and the formation of bimolecular intermediates occurs with the participation of active (oxidative) centers of the SO₄²⁻ - ZrO₂ component, and the hydro cleavage into final products by active (acidic) centers of the H-zeolite[2].
5. On the example of the model reaction of isomerization-disproportionation interaction of butane with heptane, was substantiated the possibility of involving the accompanying oil gas components in isomerisation - disproportionation conversion of straight-run gasoline into C₅-C₆ paraffins, necessary for the

production of clean, high-octane gasolines and this process was carried [8].

6. It was found that an effective catalytic system for the combined processing of straight-run gasoline and propane-butane fraction 0.5%Co/HZSM-5/SO₄²⁻(2%) - ZrO₂ provides the conversion of C₄ and C₈₊ components of the mixture at the level of 63 and 55% and the yield of C₅-C₆ and C₇ paraffins at the level of 21 and 23 wt.% [9].

7. The replacement of the zeolite component of the composite catalyst with alumina leads to a loss of activity of the catalytic system, and the replacement of Co/HZSM with Co/HMOR or a change in the concentration of sulfate ions on zirconia contributes to a change in the activity of the composite catalyst and the distribution of conversion products of straight-run gasoline and propane-butane fraction [7].

The main content of the dissertation is published in the following works.

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