

10-4-2024

## STUDY OF TECHNOLOGICAL PARAMETERS ON CATALYTIC HYDROISOMERISATION OF AROMATIC HYDROCARBONS INTO ALKYL NAPHTHENIC HYDROCARBONS

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### Recommended Citation

Ra'no, Habibullaeva Mirzaxmatovna; Torayeva, Nodira Turobjonovna; Daminov, Temurbek Z.; and Eshmukhamedov, Murod Azimovich (2024) "STUDY OF TECHNOLOGICAL PARAMETERS ON CATALYTIC HYDROISOMERISATION OF AROMATIC HYDROCARBONS INTO ALKYL NAPHTHENIC HYDROCARBONS," *Technical science and innovation*: Vol. 2024: Iss. 3, Article 6.

DOI: <https://doi.org/10.59048/2181-0400>

E-ISSN: 2181-1180

.1640

Available at: <https://btstu.researchcommons.org/journal/vol2024/iss3/6>

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UDC 665.662.2.

## STUDY OF TECHNOLOGICAL PARAMETERS ON CATALYTIC HYDROISOMERISATION OF AROMATIC HYDROCARBONS INTO ALKYL NAPHTHENIC HYDROCARBONS

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Received: Aug 23, 2024; Accepted: Sep 30, 2024; Online: October 04, 2024.

**Annotation.** The article presents experimental results of the catalytic hydroisomerization of aromatic hydrocarbons into alkyl naphthenic hydrocarbons. Aluminum col-molybdenum with silicon oxides has been studied as a catalytic system, which imparts acidic properties to the catalytic system and stimulates isomerization.

The dehydrogenation reaction of cyclohexane has been studied, which occurs on the active surface of one of the metal oxides to cyclohexene, then the resulting cycloolefin diffuses to the acidic active center - aluminosilicate, where skeletal isomerization occurs and then the isomerized cycloolefin again returns to the active center of the surface of the catalytic system, where it is hydrogenated to saturated hydrocarbon.

The influences of temperature, pressure, and the rate of transmission of raw materials through a stationary layer of the catalytic system into the reactors were studied. The optimal parameters for the catalytic transformations of benzene into methylcyclopentane have been determined: starting at 325°C, with increasing temperature the content of five-membered naphthene increases and the content of cyclohexane decreases.

By clarifying the influence of pressure, it was determined that the hydroisomerization reaction with increasing pressure first increases to a maximum and at a value of 8.0 Mn/m<sup>2</sup> decreases and at higher pressures the maximum formation of cyclohexane and methylcyclohexane is observed and it was found that the amount of decomposition products (pentane and hexane) in the catalyzate at all parameters are insignificant, i.e. does not exceed 0.5% mass.

For the first time, a comprehensive study was carried out on the synthesis with specified physicochemical and operational parameters in the form of a new catalytic system obtained with a specified stabilization of activity by introducing technological changes and additives both into the raw materials and into the composition of the catalyst.

**Key words:** petroleum products, isomerization, dehydrocyclization, skeletal isomerization, aluminosilicate, bifunctional catalyst, gas condensate, incomplete combustion, alkylnaphthenic hydrocarbons.

**Annotatsiya.** Maqolada aromatik uglevodorodlarning naften uglevodorodlariga katalitik gidroizomerizatsiya usuli bilan aylantirishning eksperimental tadqiqot natijalari keltirilgan. Bunda katalitik tizim sifatida kremniy oksidi bilan modifikatsiyalangan alyumonikelmolibden oksidli aralashmalari sinovdan o'tkazilgan.

Siklogeksanni degidirlash reaksiyasi o'rganilgan bo'lib, bu jarayon metall oksidli katalizator faol sirtida siklogeksengacha o'zgarishi va hosil bo'lgan sikloolefin ayumosilikatning kislotali markaziga diffuziyalanish tufayli singishi natijasida skelet izomerlanishi sodir bo'lishi va izomerlangan siokloolefin katalitik sistemaning markaziga qaytib kelib to'yingan uglevodorodgacha gidrirlanishi tadqiq etilgan.

Katalitik reaksiyani olib borishda reaksiya muxitidagi temperatura, bosim va shu bilan birga katalitik tizimning turg'un qatlami ustidan o'tkazilayotgan masulotning tezligining ta'siri o'rganilgan va olingan eksperimental natijalar qayd etilgan.

Benzolning metilsiklopentangacha kaatlitik o'zgarishi optimal paarmetrlari o'rganilib, harorat 325°C dan oshishi bilan besh xalqali natenni miqdori oshishi va siklogeksanning miqdori kam bo'lishi ma'lum bo'lgan.

Bosimning ta'siri aniqlangach, bosim oshgan sari gidroizomerizatsiya reaksiyasi avvaliga maksimumgacha yetishi va bosim qiymati 8,0 Mn/m<sup>2</sup> bo'lganida kamayishi va yanada yuqori

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*bosiimlarda siklogeksan va metilsiklogeksanlarning hosil bo'lishi maksimumga yetishi kuzatilgan va barcha parametrlarda katalizatda parchalanish mahsulotlari (pentan va geksan) miqdori 0,5% dan oshmasligi o'rganilgan.*

*Birinchi marotaba aktivligini barqarorlashtirish maqsadida katalizator tarkibiga va xom ashyoga qo'shimcha qo'shishda texnologik o'zgartirishlar qo'llash bilan olingan ma'lum fizik-kimyoviy va ekspluatatsion parametrlarga ega bo'lgan yangi katalitik sistemani kompleks tadqiqoti bajarilgan.*

**Kalit so'zlar:** *neft mahsulotlari, izomerlanish, degidrotsikllanish, skelet izomerizatsiyasi, aluminosilikat, ikki funksiyali katalizator, gaz kondensati, to'liq yonmaydigan, alkilnaftienik uglevodorodlar.*

**Аннотация** *В статье представлены экспериментальные результаты каталитической гидроизомеризации ароматических углеводородов в алкил нафтеносодержащие углеводороды. В качестве каталитической системы изучена алюмоникельмолибденовая с оксидами кремния, придающие каталитической системе кислотоустойчивые свойства стимулирующую изомеризации.*

*Исследована реакция дегидрирования циклогексана, которая происходит на активной поверхности одного из оксидов металла до циклогексена, затем образовавшийся циклоолефин диффундирует к кислотному активному центру-алюмосиликату, где и происходит скелетная изомеризация и далее изомеризовавшийся циклоолефин снова возвращается на активный центр поверхности каталитической системы, где и гидрируется до предельного углеводорода.*

*Изучены влияния температуры, давления, скорость пропускания сырья через стационарный слой каталитической системы в реакторы. Определены оптимальные параметры каталитических превращений бензола на метилциклопентан и начиная 325°C, с повышением температуры увеличивается содержание пятичленного нафтена и уменьшается содержание циклогексана.*

*Выяснением влияние давления определено, что реакция гидроизомеризации с повышением давления сначала увеличивается до максимума и при значении 8,0 Мн/м<sup>2</sup> уменьшается и более повышенных давлениях наблюдается максимального образование циклогексана и метилциклогексана и установлено, что количество продуктов разложения (пентана и гексана) в катализате при всех параметрах незначительно, т.е. не превышает 0,5 % масс.*

*Впервые выполнены комплексное совокупное исследование по синтезу с заданными физико-химическими и эксплуатационными параметрами виде новой каталитической системы, полученной с заданной по стабилизации активности посредством внесения технологических изменений и добавок как в сырье, так и в состав катализатора.*

**Ключевые слова:** *нефтепродукты, изомеризация, дегидроциклизация, скелетная изомеризация, алюмосиликат, бифункциональный катализатор, газоконденсат, неполный сгорания, алкилнафтеносодержащие углеводороды.*

### Introduction

Among various chemical-catalytic processes, the most intensively developed are those in which a significant role belongs to the reaction of isomerisation of the hydrocarbon skeleton of hydrocarbon molecules, allowing to obtain raw materials for petrochemical synthesis, cheap high-octane fuels on the basis of low-octane gasolines of direct distillation, highly stable reactive and low-freezing diesel fuels, etc. Therefore, the most important task for the nearest future is to ensure advanced development of production facilities for catalytic cracking, reforming and isomerisation, which allow to significantly improve the properties of refined petroleum products. Chemical properties of aromatic, paraffinic, olefinic, naphthenic hydrocarbons allow us to consider the great potential of these hydrocarbons in various syntheses and catalytic transformations. That is why at present the problem of reforming hydrocarbon structures - isomerisation of hydrocarbons becomes an important state task.

In connection with the increasing demand and tightening on the quality of commercial gasoline fractions is in the focus of technologist isomerisation of aromatic hydrocarbons and production of cyclopentadiene - a hydrocarbon with a high value of octane number, with the simultaneous possibility of obtaining commercial gasoline with a minimum content or without aromatic hydrocarbons. Production of cyclopentadiene and its homologues can be practically carried out on the basis of dehydrocyclisation of aromatic and dehydrogenation of pent membered oils, which can be obtained either by separation from narrow fractions of selected oils, or by catalytic transformation of cyclohexane and benzene hydrocarbons.

### Research methods and results obtained

The results of studies have shown that the proposed oxide catalyst, prepared by applying nickel and molybdenum salts on aluminosilicate, allows high selectivity, productivity and stability of active

action. In the experimentally found optimum conditions of the process of hydroisomerisation of benzene, the production of methylcyclopentane is achieved, and the possibility of formation of by-products is excluded. The catalyst has increased mechanical strength, multiple heat treatment does not affect its catalytic and mechanical properties.

Many works have been devoted to separation of pure naphthenes from light fractions of oils. However, the isolation of individual hydrocarbons is associated with certain difficulties and limited product yields. Therefore, other catalytic methods are used to obtain alkylcyclopentanes.

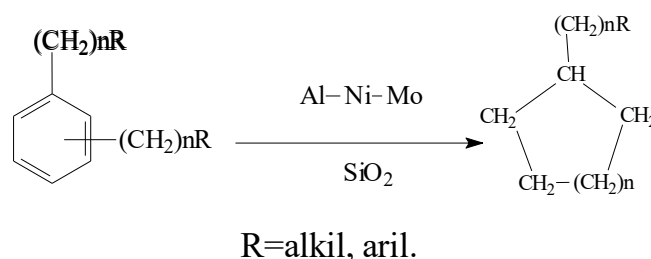
The processes of hydrocarbon processing in the oil refining and petrochemical industries are usually complex and consist of many interrelated and mutually influencing reactions. For improvement and intensification of processes it is necessary to obtain data on the mechanism and nature of interrelation of such reactions, since the mechanism characterises the specificity of catalyst influence and creates prerequisites for process control and therefore the study of the mechanism, kinetics and thermodynamics of isomerisation of individual hydrocarbons is of considerable theoretical and practical interest.

According to the well-known classical scheme of the mechanism, isomerisation of cyclohexane into methylcyclopentane proceeds in the liquid phase in the presence of halide salts of aluminium. In this case cyclohexane in the presence of aluminium chloride reversibly isomerises and at 80° C the isomerisation process is not accompanied by side reactions. It was clarified that the isomerisation reaction proceeds through an intermediate stage of dehydrogenation of the initial hydrocarbon with formation of cycloolefin and hydrogen. It was found that isomerisation of n-hexane and cyclohexane in the presence of aluminum chloride is strongly retarded if carried out under high hydrogen pressure.

This circumstance is explained by the fact that the initial stage of the process is the dehydrogenation reaction proceeding with the release of hydrogen. Therefore, the equilibrium in this stage with increasing pressure should shift towards the initial substances, which leads to a decrease in the equilibrium concentration of dehydrogenation products with increasing pressure and a significant decrease in the rate of further stages of the isomerisation process. The conducted study confirmed the proposal about the presence of the initial stage of isomerisation of saturated hydrocarbons in the presence of aluminum chloride dehydrogenation with hydrogen release. However, the nature of the remaining stages of the reaction and the structure of intermediate products were not clarified by these studies.

The isomerisation process was carried out in the presence of saturated some catalytically active metal oxides on a multicomponent system. It was found that the isomerisation reaction proceeds on two different active centers of the catalyst surface, unsaturated hydrocarbons being intermediate products existing in the gas phase. The dehydrogenation reaction of cyclohexane occurs on the active surface of one of the metal oxides to cyclohexane, then the formed cycloolefin diffuses to the acidic active centre-aluminosilicate, where skeletal isomerisation takes place. The isomerised cycloolefin returns to the active center on the surface of the catalytic system, where it is hydrogenated to a limiting hydrocarbon. It is considered expedient to combine both functions in one and the same active center of the catalyst and it is shown that a simple mechanical displacement of both functions allows to carry out the isomerisation reaction. Thus, under the applied conditions catalytically active oxides with pure silica gave very small yield of isomerisation products. When silica was replaced by aluminosilicate, the isomerising fraction of the catalyst was high.

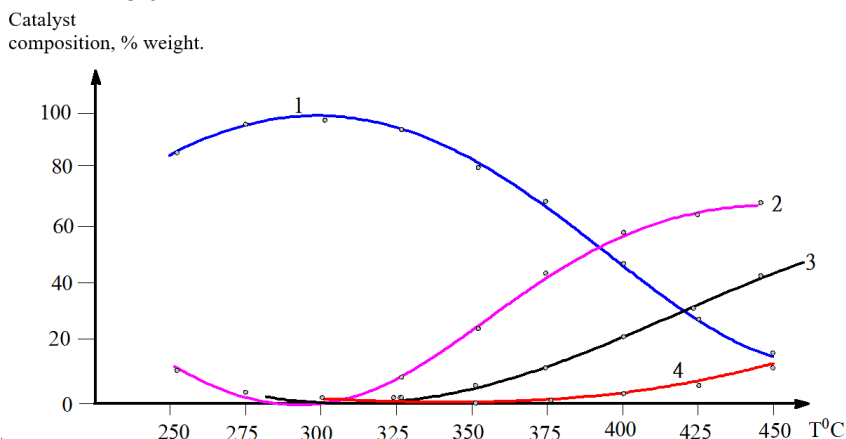
Investigation of the influence of temperature. The results of experiments to find out the influence of reaction temperature on the depth of hydroisomerisation of benzene into methylcyclopentane at a hydrogen pressure of 8.0 MN/m<sup>2</sup> and a volume rate of 0.25<sup>h-1</sup> are as follows. (Fig.1). at a temperature of 250-300°C only hydrogenation of benzene into cyclohexane occurs. Moreover, the depth of conversion of benzene into cyclohexane increases as the temperature increases and reaches a maximum at 300° C, and then begins to fall. Starting from 325° C, the content of five-membered naphthene increases with increasing temperature and the content of cyclohexane decreases. The optimum temperature should be considered to be 400°C. Since at this temperature the content of five-membered naphthene in the catalyst is about 43 % wt %. And the amount of decomposition does not exceed 0.5 wt. %. At 300° C on this catalyst from benzene it is possible to obtain cyclohexane with purity up to 99 wt. % from benzene.



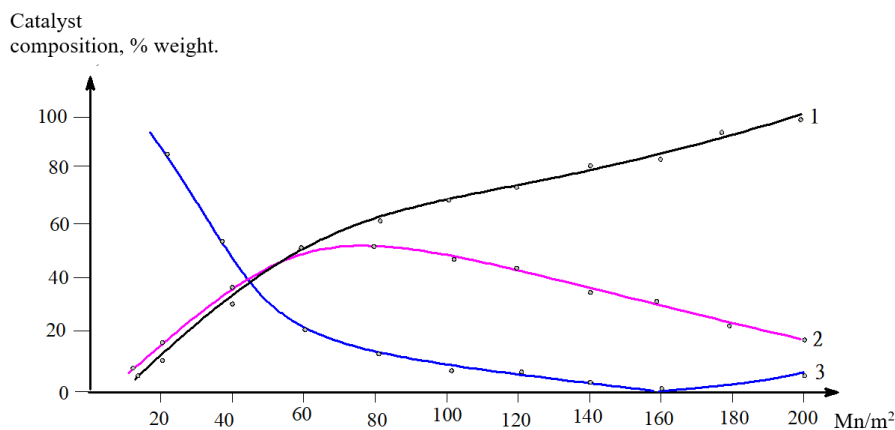
**Influence of pressure.** To find out the optimum pressure, experiments on hydroisomerisation of benzene into

methylcyclopentane were carried out at an optimum temperature of 400°C. And the volumetric rate of feedstock passing through the stationary catalyst layer 0,25h<sup>-1</sup> and different hydrogen pressures: 1,5; 2,5; 5,0; 7,0; 9,0; 12,5; 15; 17,5; 20,0 MN/m<sup>2</sup> (Fig.2). It was found that the hydroisomerisation reaction with increasing pressure first increases, and then, having reached the maximum at 8.0 MN/m<sup>2</sup>, starts to

decrease. however, it should be noted that the depth of benzene conversion into naphthenes continuously and smoothly increases. At pressure more than 14.0 MN/m<sup>2</sup> it actually transforms into cyclohexane and methylcyclopentane. The amount of decomposition products (pentane and hexane) in the catalyst at all parameters is insignificant, i.e. does not exceed 0.5 wt%.



**Fig.1. Depth of benzene hydroisomerisation as a function of temperature variation.**  
 1- Cyclohexane; 2- Methylcyclopentane; 3- Benzene; 4- Hydrocracking products.



**Fig.2. Depth of benzene hydroisomerisation as a function of pressure variation.**  
 1 - Cyclohexane; 2 - Methylcyclohexane; 3 - Benzene.

Table-1. Depth of benzene conversion to methylcyclopentane at recirculation of feedstock through a stationary catalyst bed (pressure 8.0 MN/m<sup>2</sup>, temperature 395-400o C, volume velocity 0.25) hour<sup>-1</sup>

**Table 1**

| Nº                          | Indicators                         | 1-cycle | 2-cycle |
|-----------------------------|------------------------------------|---------|---------|
| 1                           | Yield of reaction products, % vol. | 96      | 94      |
| Product distribution, % wt. |                                    |         |         |
| 2                           | Methylcyclopentane                 | 39,8    | 68,2    |
| 3                           | Cyclohexane                        | 45,5    | 25,9    |
| 4                           | Benzene                            | 14,2    | 5,2     |
| 5                           | Hydrocracking products             | 0,5     | 0,7     |

Thus, based on the presence of two types of active centres on the surface of the bifunctional catalyst, the migration of the converted substance from the hydrodehydrating centre to the acidic centre and back is assumed to be necessary; apparently, the migration proceeds with intermediate desorption into the volume. The mechanism of mutual transformation of benzene, cyclohexane and methylcyclopentane on the surface of bifunctional catalyst proceeds in the most complicated scheme.

It is assumed that the presence of two kinds of active centres on the surface of the catalyst - hydrodehydrating and isomerising - migrates the reacting molecule from one centre to the other. At mutual transformation of the mentioned hydrocarbons formation of cycloolefin is always

obligatory. The same fact is confirmed by experimental studies. It is observed that in the process of mutual transformation of cyclohexane, benzene and methylcyclopentane cyclohexane is not a precursor of reaction products. As part of the study of the mechanism of destructive hydrogenation of benzene in the presence of polyfunctional sulfide-tungsten catalysts, it is found that a significant part of methylcyclopentane is formed directly from benzene without desorption into the volume of intermediates from the catalyst surface. The apparent consecution of the process is assumed. At short contact time, when the concentration of benzene and cyclohexane are close, most of the benzene is converted into cyclohexane, i.e. the benzene hydrogenation reaction prevails, since the latter concentrates most of the activity of the converted benzene, and at the same time the gyroisomerisation reaction takes place. At long contact time, as benzene is used up, its transformation into methylcyclopentane prevails, and in addition, the latter starts to be formed from cyclohexane in increasing amounts. On the basis of the material balance it is possible to determine the optimum ratio between the intensity of these transformations at the moment of almost equal concentration of benzene and cyclohexane. By comparing experimental data with calculated data, the presence of both direct - without desorption of intermediate products into the volume - transformations of benzene into methylcyclopentane or methylcyclopentane into benzene, and consecutive ones through cyclohexane was found out.

The automotive industry has expanded significantly in recent years - both in production volume and in the number of different new vehicle models. The new models are designed to provide higher performance and better environmental and fuel efficiency for the vehicle fleet. These powerful and high-speed engines are designed to use higher-quality petroleum products, especially higher-octane petrol. In this connection, the issues of ensuring maximum combustion of automotive petrol in internal combustion chambers have acquired a completely new significance and have become one of the most important scientific tasks, the solution of which determines the technical progress and development of the country's economy. Since motor petrol take the first place among petroleum products in terms of production and consumption. The demand for petrol of the economy sectors has already in 2010 increased more than 2 times in comparison with 1995. The required volume can be met only by further increase in the volume, deepening and chemicalisation of the processing of feedstock. At the same time, the chemical composition of motor fuels is becoming more diverse and complex, active chemical components appear in them and as a result their properties change significantly.

This scientific information presents the results of research on the transfer of chemically passivated oxygen catalysed to the very final stage of combustion of hydrocarbon fuel particles in a closed system, providing complete oxidation of hydrocarbons. Scientific issues related to ensuring complete combustion of hydrocarbon fuels in internal combustion engines have been studied. The degrees of combustion of hydrocarbon fuel microparticles during their short stay in internal combustion chambers have been studied. Scientific novelty consists in conducting research - experimental works on studying the form and content of chemically bound and free oxygen by applying elements of modern achievements of science and technology. In modern internal combustion engines, the particles of the combustible component are in the combustion chambers not more than 0.2 seconds. During this time interval, complete combustion of hydrocarbon particles to the nucleus does not always occur. The reasons for such incomplete combustion are; very short residence time of particles in combustion chambers. Insufficiency of chemical conditions for complete combustion of hydrocarbon feedstock in a closed system. Limited possibility of ensuring the full course of the hydrocarbon combustion reaction due to the difficulty of regulating the supply of the stoichiometric ratio in the combustion chamber "fuel-oxygen (air)" and others. The above-mentioned technological limitations can be solved by increasing the content of absorbed chemically passivated oxygen in the composition of hydrocarbon fuel. Some mechanisms of hydrocarbon fuel combustion in a closed system have been studied. Technological novelty consists in application of mechanisms of introduction of chemically passivated oxygen in volume of hydrocarbon fuel microparticles, and on possibility to increase oxygen in a core of microparticles by application of achieved scientific successes of modern science and technology, because, increase of concentration of oxygen content in a core of hydrocarbon fuel microparticles in combustion chambers stimulates full combustion by internal explosion, that certainly, positively influences on engine power, environment protection (decreases the end of combustion of hydrocarbon fuels), as well as on combustion of hydrocarbon fuel. Consequently, the realisation of the studied mechanism can affect the reduction of emissions of some types of gases with greenhouse effect, and this can be an important step towards solving the problem of all mankind.

Automotive petrol, due to the peculiarities of the composition of source oils and different technological schemes differ greatly from each other. The composition of petrol A-80 of domestic local origin includes from 10 to 80% of gas condensate and products of thermal cracking, high molecular weight

oil fractions of catalytic refining of hydrocarbon raw materials. These new grades of commercial petrol due to the complexity of chemical composition complicate the reaction of complete oxidation of hydrocarbon raw materials in closed systems for a fixed period of time of fuels stay in the combustion chamber of engines. According to materials of analytical researches in internal combustion chambers there is a combustion from 35,5% to 55% of hydrocarbon fuel, and the rest part continues to burn out in an emission pipe until the products of fuel combustion are released into the atmosphere. Increasing the content of the combustible part of hydrocarbon fuel in the combustion chamber itself is naturally of economic and environmental interest.

Methods of organic catalysis are becoming increasingly important in industry due to the need to solve the problems of development of secondary catalytic processing of hydrocarbon raw materials. In fact, without the development of the latest methods of organic catalysis, many processes underlying the catalytic transformation of hydrocarbon raw materials are unthinkable. Based on these considerations, the direction of selection of new types of catalysts for catalytic processes should be developed, as well as the development of the most effective methods of processing of low-nomenclature organic raw materials and a variety of chemicals with different target purposes. One of the main successes of chemistry and technology of fuels and gas is the development of chemical methods of processing fuels, combining their refining, which gives them specific qualities of synthetic fuels with simultaneous obtaining of chemical products that are raw materials for chemical and petrochemical industries. The use of various high-performance catalysts has made it possible to intensify such catalytic processes as cracking, reforming, hydrotreating, hydroisomerisation, etc., which is very urgent. The catalytic mutual transformation of benzene, cyclohexane and methylcyclopentane and their homologues has been known for a long time. Studies in this direction have been developed with the use of various types of catalysts based on both noble and base metals, on carriers and without them. The use of catalysts containing noble metals, despite their efficiency, is always considerably limited by their high cost, scarcity and sensitivity to contact poisons. Broader possibilities are inherent in catalysts and catalytic systems based on metals of group VIII of the periodic system, which probably explains the extensive literature material on their basis. Studies by a number of scientists in the reaction of destructive hydrogenation of benzene, toluene and ethylbenzene revealed that hexamethylene hydrocarbons into pent methylene hydrocarbons and recycling of the latter into paraffin and isoparaffinic hydrocarbons. Due to the presence of sulphury in oil feedstock, deactivating

components of catalytic systems, the development of poison-resistant catalysts is of great importance in industry and research practice. There are numerous scientific-technological and scientific-analytical information, aimed at reforming the structure of hydrocarbon compounds, providing improvement of ecological and operational characteristics of fuel fractions. It should be noted that these promising, put forward as newly synthesised catalytic systems with improved efficiency, currently cannot go beyond laboratory studies, which stimulates the continuation of research work in this direction.

The present analytical and research information presents the results of isomerisation of benzene fractions component structure in the presence of polyfunctional and polymetallic catalytic systems in hydrogen medium. The main objective is to synthesis components that improve the environmental and performance characteristics of fuel mixtures, meeting the requirements of international standards "Euro-4" and "Euro-5", which strictly regulate both the component composition of fuel fractions and the emission products from internal combustion engines. Such strict regulation of the content of benzene series compounds and other cyclic unsaturated compounds with heteroatoms in the composition of liquid fuel mixture is explained by procedural measures aimed at preventing the possibility of formation of (PAHs) in the emission composition as products of incomplete combustion. Decontamination of petroleum refined products is almost entirely associated with hydrogenation reactions. Fuel fractions as a whole and their individual components are subjected to hydrogenation purification from undesirable compounds. The selection of appropriate catalytic systems is also connected with the study of new reactions with two or more reactions in one reaction zone. For the first time we have carried out a complex cumulative study of synthesis of a new catalytic system with specified physicochemical and operational parameters, obtained with a given stabilisation activity by means of technological changes and additives both in the raw materials and in the catalyst composition.

Thus, the results on the chemistry of catalytic hydrogenation transformations of hydrocarbons of different structures, used in practice, can be applied with the same success for hydrogenation refining of hydrocarbons of both traditional and alternative origin.

### Conclusion

1. The possibility of hydroisomerisation of monocyclic aromatic hydrocarbons in the presence of a catalytic system consisting of a mixture of nickel, aluminium, molybdenum and silicon oxides has been studied.

2. The effect of temperature, pressure and rate of feedstock passage through the stationary layer of the catalytic system was studied.

3. The optimal parameters of hydroisomerisation of aromatic hydrocarbons to alkyl naphthenic hydrocarbons have been found. In the found optimum conditions of the process: temperature 375°C, pressure 160 MN/m<sup>2</sup> and volumetric speed 0.5 h<sup>-1</sup>, the content of aromatic hydrocarbons in the catalyst decreases to almost zero.

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