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## HYDROGENATION OF AROMATIC HYDROCARBONS IN GASOLINE FRACTIONS OVER SUPPORTED CATALYSTS UNDER PRESSURE

**Abstract.** The aim of the work was to study the process of hydrodearomatization of gasoline fractions under increased hydrogen pressure. It has been used Rh-Pt (9:1)/Al<sub>2</sub>O<sub>3</sub> catalyst in the work. It has been studied the hydrogenation of two gasoline fractions of "Atyrau Oil Refinery" LLP (Hydrogenizate KU GBD and Stable Catalysate LG). Technological parameters of the process of hydrodearomatization for the production of environmentally friendly fuels, containing no benzene and low in aromatic hydrocarbons have been worked out (pressure, temperature). Data on group composition of organic substances in gasolines demonstrate that after catalytic hydrogenation benzene in final samples of two fractions is absent. For hydrogenizate, the aromatic content decreased from 11.12 weight % to 2.20 weight %. For stable catalysate, the amount of aromatics decreased from 51.5 weight % to 10.96 weight %. The catalyst was studied by BET, porometry and EM methods, which established a uniform formation of nanoscale particles on the catalyst surface.

**Key words:** catalysts, hydrogenation, hydrodearomatization, aromatic hydrocarbons, benzene, gasoline.

### Introduction

Technical progress in various fields of machine building, machine operation and laws for the protection of the biosphere tighten the requirements for the quality of fuels and oils. Continuously deteriorating environmental conditions in the world dictate the need to operate clean and quality fuel. The quality of fuels depends largely on the hydrocarbon composition. Aromatic hydrocarbons in fuels are represented by monoaromatic compounds: benzene, toluene, xylene isomers, and polyaromatic compounds - naphthalene, tetralin and other condensed aromatic compounds.

The product of incomplete combustion of benzene is benzpyrene - a strong carcinogen. When burning 1 liter of gasoline in the exhaust gas, benzpyrene is formed up to 81 µg and in case of 1 liter of diesel fuel - up to 170 µg. By the standards of gasoline EURO-6, it is provided the benzene content is less than 0.1% and amounts of aromatic hydrocarbons up to 11%.

When moving to new standards, the problem arises of removing benzene from gasoline, which can not be separated by conventional physical methods. One of the methods for improving the operational properties of fuels is their hydrodearomatization, which consists in the hydrogenation of benzene and polycyclic aromatic hydrocarbons contained in gasoline fractions in the presence of effective catalysts.

Catalytic hydrogenation of aromatic hydrocarbons allows changing the chemical structure of hydrocarbons in the desired direction and improving the performance of motor fuels [1-8]. Development and introduction of technology of hydrodearomatization of fuel fractions of oils and fuels will improve the operational properties of domestic gasolines and the ecological situation in the Republic.

In the process of exploitation of fuels and low quality oils, except harmful emissions into the atmosphere, there is also rapid wear of the equipment due to the deposition of carbon from polycyclic aromatic hydrocarbons and the service life of the equipment reduces approximately to 30%. Reducing the amount of aromatic hydrocarbons in car fuels will extend the service life of the machines.

Leading countries are engaged in the problem of hydrodearomatization of refined products. These countries are: Russian Federation, USA, Great Britain, France, Germany, Poland, Japan, China [9-15]. In the oil refining industry, hydrogenation processes are carried out under harsh conditions (high temperature and hydrogen pressure) on metal oxide catalysts, where Co, Mo, Ni, Cu, W and other transition metals are used as the metal. Catalytic systems based on platinum group metals, especially Pt, Pd, Rh and Ru are the most effective and selective catalysts for hydro - dehydrogenation reactions [11-20].

In the industry use both catalysts on the basis of metals of group VIII and sulphidic, however on the last one the hydrogenation of benzene and aromatic hydrocarbons is carried out in more stringent conditions.

Recently Pt-Pd catalysts have been intensively used for the hydrotreating of petroleum products, especially for the reduction of benzene in gasolines and aromatics in diesel fuels, so they are closely watched by researchers. The addition of Pd to Pt/Al<sub>2</sub>O<sub>3</sub> leads to an increase not only the activity, but also the stability in the hydrogenation of benzene. By varying the nature of the carrier, modifying the catalysts reach a uniform distribution of the metals on carriers, optimum acidity and stability against sulfur-containing compounds.

Taking into account the toughened measures currently applied to motor fuels, the transition to European quality standards for fuels, as well as the modernization of Kazakhstan's oil refineries, carrying out research in this area is relevant and timely.

The purpose of this work is to study the catalytic hydrodearomatization of two gasoline fractions of "Atyrau Oil Refinery" LLP to reduce the content of benzene and aromatic hydrocarbons by hydrogenating them on supported catalysts based on Group VIII metals.

## Experimental

In this work it has been used a catalyst based on the metals of the platinum group Pt and Rh. At preparation of catalysts were used RhCl<sub>3</sub>·3H<sub>2</sub>O, H<sub>2</sub>PtCl<sub>6</sub>·6H<sub>2</sub>O of "chemically pure" mark. Solutions of these compounds were applied by the adsorption method on the prepared carrier Al<sub>2</sub>O<sub>3</sub>. A mixture of aqueous solutions of two metals was applied at preparation of bimetallic catalysts. The catalyst samples were filtered off and dried at 100-110°C to constant weight. The reduction of supported catalysts was carried out in a quartz tube with electrical heating in a hydrogen stream at 200°C for 4 hours, then the catalysts were cooled in a hydrogen stream until room temperature.

The experiment was carried out on a kinetic installation - the autoclave of "Amar Equipment" in the isobaric-isothermal regime. Analysis of the initial compounds and reaction products was carried out on the Crystallux 4000M chromatograph: column Zebron ZB-1 filled with dimethylsiloxane, column length is 30 mm, column diameter is 0.53 mm.

The catalysts were studied by physicochemical methods of analysis: measurement of the catalyst surface (BET), electron microscopy (EM). The surface of the catalysts was examined by the BET method on nitrogen adsorption by "Accusorb" instrument. Electron microscopy of samples was studied by means of electron microscope EM-125K by replica with extraction using microdiffraction (160,000 magnification).

## Results and discussion

It has been studied the hydrogenation of benzene and aromatic hydrocarbons contained in two gasoline fractions of "Atyrau Oil Refinery" LLP - Hydrogenizate KU GBD and Stable Catalysate LG. The content of benzene in them is 2.54% and 5.17%, and aromatics are 11.12% and 51.5%, respectively.

As the catalyst used Rh-Pt(9:1)/Al<sub>2</sub>O<sub>3</sub>, which showed the greatest efficiency in the hydrogenation of individual benzene and toluene [2]. The effect of temperature and hydrogen pressure was studied on the process of hydrogenation of benzene and aromatics for two gasoline fractions.

Data on hydrogenation of sample of gasoline №1, the hydrogenizate KU GBD in the temperature range of 25-200°C are presented in table 1. At 25°C after the experiment, the catalysate contains 0.05% benzene, at higher temperatures benzene is absent, i.e. its conversion reached to 100%. The content of aromatic hydrocarbons decreased from 11.12% to 2.30-3.05%.

According to the results of table 1, it can be concluded that, on the effective catalyst Rh-Pt (9:1)/Al<sub>2</sub>O<sub>3</sub>, benzene can be hydrogenated at low temperatures, which is important for the industrial conduct of such a process, since the industry uses temperatures 150-400°C.

Table 1 - Hydrogenation of the fraction of Hydrogenizate KU GBD at various temperatures and 3 MPa

Temperature	The content of benzene, %		Aromatic content, %	
	Initial	After experiment	Initial	After experiment
25	2,54	0,03	11,12	3,05
50		-		2,30
100		-		2,63
150		-		2,20
200		-		2,53

When the hydrogen pressure is changed from 2.5 to 4.0 MPa, the time of the catalytic treatment of the Hydrogenizate KU GBD decreases from 65.0 to 32 minutes (picture 1). The presence of benzene in this case after the experiment in the amount of 0.06% was observed at 2.5 MPa, with other hydrogen pressure values there are no traces of benzene. The content of aromatic compounds after hydrogenation of gasoline on this catalyst varies between 1.23 - 4.65%.

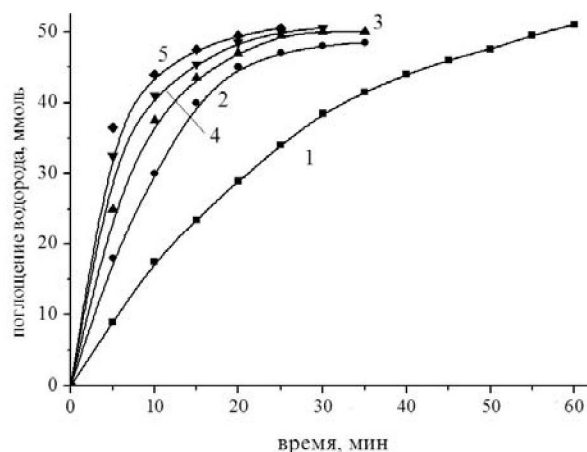


Figure 1 - Hydrogenation of gasoline (Hydrogenizate KU GBD) at different hydrogen pressures on Rh-Pt (9:1)/Al<sub>2</sub>O<sub>3</sub> catalyst at 50°C. a - 1 - 2.0 MPa; 2-2.5 MPa; 3 - 3.0 MPa; 4 -3.5 MPa; 5 - 4.0 MPa

At hydrogenation of the second sample of gasoline (Stable catalysate LG) in the range of hydrogen pressures from 0.5 to 4.0 MPa, the reaction order of the hydrogenation of gasoline on Rh-Pt(9:1)/Al<sub>2</sub>O<sub>3</sub> catalyst is on hydrogen, calculated from the bilogarithmic dependence of the rate from the pressure is equal to 1 (Figure 2, a). Benzene with initial content of 5.17% was completely hydrogenated and removed from this gasoline fraction, the aromatic content decreased from 51% to 10-14%.

An increase in temperature from 25 to 150°C increases the rate of hydrogenation of the second sample of gasoline (Figure 2,b) and the apparent activation energy is 40.8 KJ/mol. Benzene with initial content of 5.17% was completely removed from this gasoline fraction, and the aromatic content decreased from 51% to 10-15%. It should be noted for Stable catalysate LG, as well as for Hydrogenizate KU GBD, that is already at 50°C benzene is fully hydrogenated.

Studies of the influence of temperature and hydrogen pressure made it possible to select the optimal parameters for the production of environmentally friendly fuels without benzene and with a low content of aromatic hydrocarbons - temperature 50°C and pressure 3 MPa.

Data on group composition of organic substances in two gasolines of initial fractions and after hydrogenation on Rh-Pt(9:1)/Al<sub>2</sub>O<sub>3</sub> catalyst are presented in table 2, P = 3 MPa, T = 50°C.

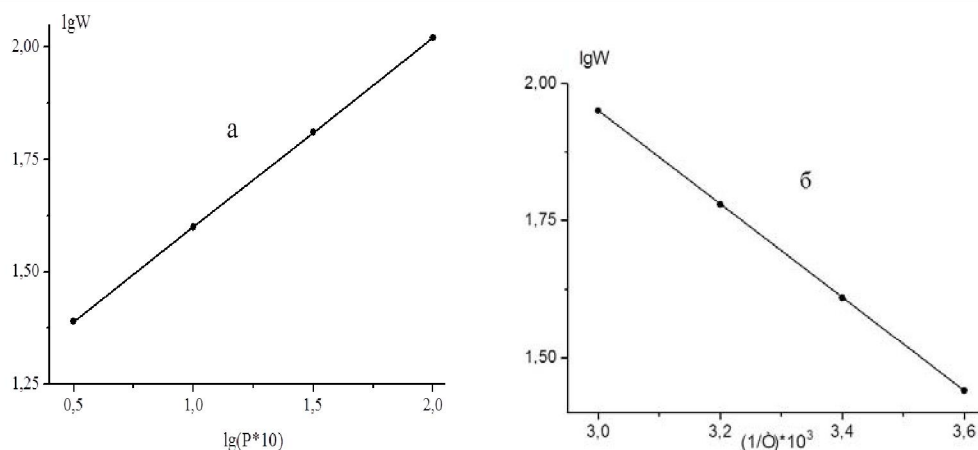


Figure 2 - Hydrogenation of Stable catalyst LG on Rh-Pt (9:1)/Al<sub>2</sub>O<sub>3</sub> at 4.0 MPa. a - bilogarithmic dependence of rate from pressure; b - dependence of the logarithm of rate from reciprocal temperature

In the initial Hydrogenizate KU GBD benzene was 2.22 wt.% (2.54 vol.%), after hydrogenation benzene was not detected in the samples - i.e. it was completely hydrogenated, the amount of toluene was decreased from 2.81 wt.% (3.25 vol%) to 0.54 wt.% (0.64 vol%). The aromatic content in the initial gasoline was 13.70 wt.% (11.12% by volume), after hydrogenation it became 2.26 wt.% (2.07% by volume).

It should be noted that the amount of olefins decreased almost 2 times from 0.47 wt.% (0.50% by volume) to 0.28 wt.% (0.28% by volume), which is very favorable for gasoline, since the presence of olefins leads to instability (in chemical terms, polymerization and oligomerization reactions proceeds).

In addition, the content of isoparaffins increased from 35.79 wt.% (28.94% by volume) to 44.26 wt.% (45.04% by volume), which is favorable for the octane number. It is possible that the hydroisomerization reaction proceeds on this catalyst.

For the Stable catalyst LG, the benzene content in the initial state was 4.53 wt.% (5.17% by volume), after the reaction the benzene content was 0.15 wt. % (0.19% by volume), i.e. conversion of benzene equal to 97%. The content of toluene after hydrogenation decreased from 19.65 wt.% (22.63% by volume) to 12.99 wt.% (15.01% by volume). The amount of aromatics decreased from 51.5 wt. % (49.19% by volume) to 10.96 wt. % (11.32% by volume).

Table 2 - Group composition of gasoline fractions of initial and after hydrogenation on Rh-Pt (9:1)/Al<sub>2</sub>O<sub>3</sub> catalyst at 3 MPa, 50°C

Sample Name	Unit of measure	The content of the defined indicator						
		Paraffins	Isoparaffins	Olefins	Naphthenes	Aromatics	Benzene	Toluene
Hydrogeni-zate KU GBD initial)	% weight	30,44	35,79	0,47	30,03	13,70	2,22	2,81
	% volume	31,97	28,94	0,50	27,46	11,12	2,54	3,25
Rh-Pt (90:10)/ Al <sub>2</sub> O <sub>3</sub>	% weight	29,47	44,26	0,28	23,77	2,26	-	0,54
	% volume	30,46	45,04	0,28	22,19	2,07	-	0,64
Stable catalyst (initial)	% weight	16,17	29,57	0,78	1,95	51,5	4,53	19,65
	% volume	19,14	33,66	0,84	1,90	49,19	5,17	22,63
Rh-Pt (90:10)/ Al <sub>2</sub> O <sub>3</sub>	% weight	14,71	51,75	0,74	12,08	11,96	0,15	12,99
	% volume	16,12	54,51	0,75	11,78	10,32	0,19	15,01

The amount of paraffins also decreased from 16.17 wt.% (19.14% by volume) to 14.71wt. % (16.12% by volume). And the content of isoparaffins increased from 29.57 wt.% (33.66% by volume) to 51.75



wt.% (54.51% by volume). Apparently there has been a reaction of isomerization of paraffins into isoparaffins. The amount of olefins practically did not change. The content of naphthenes increased sharply from 1.95 wt. % (1.90% by volume) to 12.08 wt. % (11.78% by volume).

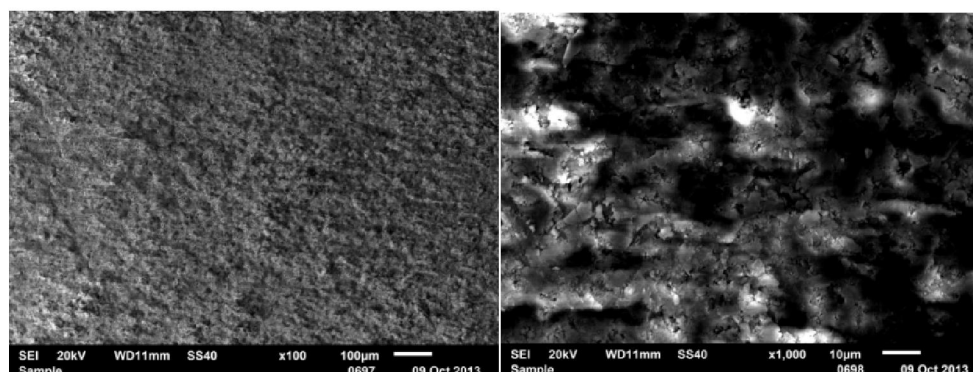
Gasoline fraction Stable catalyst LG treated in the process of hydrodearomatization was studied for octane number in LLP "Independent Expertise Center for Oil Products ORGANIC". The data on the analysis are given in Table 3. The octane number by the research method (RM) after treatment of Stable catalyst was unchanged and equal to 94 units. And the octane number by the motor method (MM) increased from 82.6 to 82.7. This indicates that the catalytic treatment of gasoline does not decrease the octane number.

Table 3 - Octane number of gasoline fraction of Stable catalyst LG before and after catalytic treatment

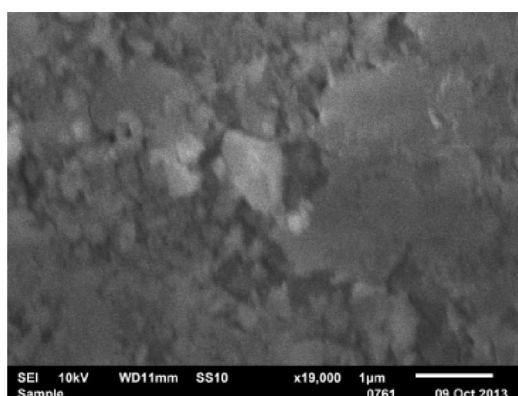
Gasoline sample	Octane number RM	Octane number MM
Initial (sample 1)	94	82,6
After experiment (sample 2)	94	82,7

The characteristics of the catalyst determined by method BET and porometry showed a well developed surface - the surface area of the Rh-Pt(9:1)/Al<sub>2</sub>O<sub>3</sub> catalyst - 138 m<sup>2</sup>/g, the pore diameter - 4-14 Å, the catalyst pore volume - 313.28 ml/g.

The surface of catalysts was studied by scanning electron microscopy. In pictures of different increase of the Rh-Pt (9:1)/Al<sub>2</sub>O<sub>3</sub> catalyst (fig. 3) is shown the uniform surface of the carrier, on which agglomerates of active metals are viewed.



2



3

Figure 3 - EM images of Rh-Pt (9:1)/Al<sub>2</sub>O<sub>3</sub>

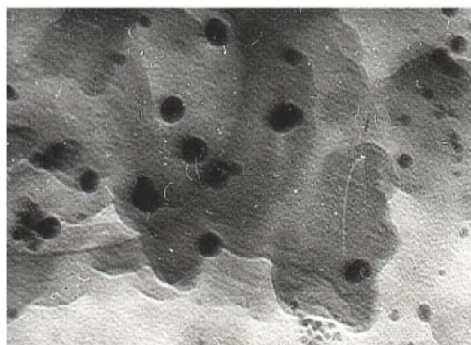


Figure 4 - EM image of 2% Rh-Pt (9:1)/Al<sub>2</sub>O<sub>3</sub> (increase 160,000)

In the transmission electron microscopy photo, Rh-Pt(9:1)/Al<sub>2</sub>O<sub>3</sub> catalyst is represented by finely dispersed particles of 2-2,5 nm in size and a small number of denser and larger particles of 50 nm, microdiffraction pictures of which are represented by diffuse rings corresponding to metals (Fig. 4). Metal particles are distributed on the surface of oxide of aluminum and represent the mixed bimetallic Rh-Pt agglomerates together with Rh particles. The high activity of the catalyst is perhaps caused by formation of alloys of these metals.

### Conclusions

Thus, a highly efficient catalyst for the hydrogenation of gasolines has been synthesized, which makes it possible to completely remove benzene, and also 2-4 times to reduce the content of aromatic compounds. The catalyst is tested in the process of hydrogenation of gasolines of two fractions of LLP "Atyrau Oil Refinery": Hydrogenizate KU GBD and Stable catalysate LG. By method of studying the effect of temperature and hydrogen pressure for two gasoline fractions were chosen the optimal parameters (temperature 50°C and pressure 3 MPa) for the production of environmentally friendly fuels, which do not contain benzene and with low aromatic hydrocarbons content.

Data on the group composition of organic substances in gasolines indicate that for hydrogenizate, the aromatic content decreased from 13.70 wt.% to 2.26 wt.%. For stable catalysate, the amount of aromatics decreased from 51.5 wt.% to 10.96 wt.%. The amount of olefins decreased almost 2 times, and the content of isoparaffins increased from 35.79 wt.% to 44.26 wt.%, which is favorable for the octane number. Furthermore, the content of isoparaffins also increased from 29.57 wt.% to 51.75 wt.%.

By BET method, porometry and EM was established a uniform formation of metal particles in zero degree of valence on the surface of catalysts, the composition and nano-sized particles of metals of group VIII were determined.

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### **БЕНЗИН ФРАКЦИЯЛАРЫНДАҒЫ ХОШ ИІСТІ КӨМІРСУТЕКТЕРДІ ЖОҒАРЫ ҚЫСЫМДА ОТЫРҒЫЗЫЛҒЫН КАТАЛИЗАТОРЛАРДЫ ҚОЛДАНУ АРҚЫЛЫ СУТЕКТЕНДІРУ**

**Аннотация.** Жұмыстың мақсаты - бензин фракцияларын сутектендіру процесін сутегінің жоғары қысымында зерттеу. Жұмыс барысында Rh-Pt(9:1)/ $\text{Al}_2\text{O}_3$  катализаторы қолданылды. ЖШС «Атырау мұнай өңдеу зауытының» екі бензин фракциялары сутектендіру процесі бойынша зерттелінді (Гидрогенизат КУ ГБД және Тұрақты катализат ЛГ). Құрамында бензол жоқ және төмен мөлшердегі хош иісті көмірсутектері бар экологиялық таза жанармайлар өндіру үшін гидродеароматизация процесінің технологиялық параметрлері жасалынды (қысым, температура). Жанармайдағы органикалық қосылыстардың топтық құрамының көрсеткіштері бойынша каталитикалық сутектендіру процесінен соң бензол компоненті екі бензин фракцияларының құрамынан толығымен жойылды. Гидрогенизат фракциясы бойынша хош иісті көмірсутектер мөлшері 11,12 мас.%-дан 2,20 мас.%-ға дейін төмендеді. Тұрақты катализат фракциясы бойынша хош иісті көмірсутектер мөлшері 51,5 мас. %-дан 10,96 мас. %-ға дейін төмендеді. Катализаторлар БЭТ әдісі, порометрия және электрондық микроскопта зерттелінді.

**Түйін сөздер:** катализаторлар, сутектендіру, гидродеароматизация, хош иісті көмірсутектер, бензол, бензин.

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### **ГИДРИРОВАНИЕ АРОМАТИЧЕСКИХ УГЛЕВОДОРОДОВ В БЕНЗИНОВЫХ ФРАКЦИЯХ НА НАНЕСЕННЫХ КАТАЛИЗАТОРАХ ПОД ДАВЛЕНИЕМ**

**Аннотация.** Целью работы являлось изучение процесса гидродеароматизации бензиновых фракций при повышенном давлении водорода. В работе использовался Rh-Pt(9:1)/Al<sub>2</sub>O<sub>3</sub> катализатор. Изучено гидрирование двух бензиновых фракций ТОО «Атырауский нефтеперерабатывающий завод» (Гидрогенизат КУ ГБД и Стабильный катализат ЛГ) Отработаны технологические параметры процесса гидродеароматизации для производства экологически чистых топлив, не содержащие бензол и с низким содержанием ароматических углеводородов (давление, температура). Данные по групповому составу органических веществ в бензинах свидетельствуют о том, что после каталитического гидрирования бензол в конечных пробах двух фракций отсутствует. Для гидрогенизата содержание ароматики снизилось с 11,12 мас.% до 2,20 мас.%. Для стабильного катализата количество ароматики уменьшилось с 51,5 мас.% до 10,96 мас.%. Катализатор исследован методами БЭТ, порометрии и ЭМ, которые установили равномерное образование на поверхности катализаторов наноразмерных частиц.

**Ключевые слова:** катализаторы, гидрирование, гидродеароматизация, ароматические углеводороды, бензол, бензин.

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