

## Isomerization of *n*-Heptane on Platinum-Zeolite Catalysts in the Presence of Cyclohexane and Benzene

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**Abstract**—Effect of cyclohexane and benzene on the isomerization of *n*-heptane on Pt/BEA-Al<sub>2</sub>O<sub>3</sub> and Pt/MOR-Al<sub>2</sub>O<sub>3</sub> catalysts was studied. In the presence of catalysts with platinum deposited from a [Pt(NH<sub>3</sub>)<sub>4</sub>]Cl<sub>2</sub> solution, cyclohexane and benzene inhibit the conversion of *n*-heptane. At the same time, the presence of cycloalkanes and aromatic hydrocarbons leads to an increase in the isomerization selectivity due to the suppression of side reactions of hydrocracking. Samples based on the BEA zeolite were found to be more active as compared with similar samples based on mordenite.

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The increasing number of countries annually introduce ecological standards on motor fuels. Among the main requirements imposed on the quality of gasoline are the limited content of sulfur, olefins, metals, aromatic hydrocarbons, and other carcinogenic substances. For example, when penetrating into a human organism via the respiratory channel or skin, aromatic hydrocarbons and, in particular, benzene can accumulate in bone marrow or fat tissues in the form of metabolites [1] and thereby substantially raise the risk of development of diseases of the lymphatic system [2] and malfunction of organs [3]. By June 2017, restrictions on the content of aromatic hydrocarbons and benzene have been adopted in 123 countries all over the world. In 65 of these (53%), the maximum content of benzene is set to be 1 vol % and less [4]. The aforesaid means that the problem of development of new processes and technologies for reducing the content of benzene and aromatic hydrocarbons in gasoline is highly topical at present. This is particularly true for those countries in which putting into operation the corresponding ecological standards requires significant changes in the structure of the already existing oil-processing schemes.

One of promising processes capable of reducing the content of benzene in gasolines is the hydroisomerization of benzene-containing gasoline fractions. As feed for the given process can primarily serve light fractions of the reformat with boiling onset at 85 or 100°C and the direct-run gasoline fraction (70–85°C). As a rule, just these fractions are the main sources of benzene and contain up to 30% this hydrocarbon. Typical compositions of feed for the hydroisomerization process are listed in Table 1.

The benzene-containing gasoline fraction (BGF) from the reforming process and the direct-run gasoline fraction are, on the whole, characterized by low octane numbers because the alkanes they contain are of predominantly normal structure. In the course of hydroisomerization, benzene with research octane number (RON) of 113 is first hydrogenated to cyclohexane (CH) with RON = 83, which is, in turn, isomerized into methylcyclopentane (MCP) with RON = 91.3. It can be seen that the decrease in the octane number, caused by the hydrogenation of benzene, is partly compensated for by the isomerization of CH into MCP. In addition, the total content of branched components in the alkane part of the fraction also

**Table 1.** Typical composition of gasoline fractions

Hydrocarbon, wt %	Fraction 70–85°C (direct-run)	Fraction 85°C (from reformat)	Fraction 100°C (from reformat)
Pentanes	–	10–20	15–20
Hexanes	5–10	20–30	20–30
Heptanes	5–10	20–30	40–50
MCP + CH	70–80	5–10	to 1
Benzene	3–10	10–30	3–5
Toluene	–	–	5–10
RON	75–85	70–80	55–65

increases, which can not only offset the loss of the octane number, but also raise it by 2–3 units. The increase in the octane number of the light and medium-boiling part of the reformat and of the direct-run gasoline fraction (70–85°C) positively affects not only the overall RON, but also the service properties of gasolines because of improving the distribution of octane numbers over the distillation temperature, which has in this range the known dip, “octane hole” [5].

It is known that the isomerization of alkanes is slowed down in the presence of aromatic hydrocarbons and cycloalkanes. This is due to the preferential adsorption of these latter on acid centers of catalysts. This fact was first reported in [6, 7] in which this phenomenon was examined on platinum deposited onto mordenite from a  $[\text{Pt}(\text{NH}_3)_4]\text{Cl}_2$  solution. The introduction in Europe in 2000 of the ecological standard Euro-3, which diminished the acceptable content of benzene in gasoline from 5 to 1 vol %, again revived the interest of European researchers in this problem. In analyses of the reactions of BGF hydroisomerization, the hindrance to the isomerization of alkanes in the presence of aromatic compounds and cycloalkanes has been studied on platinum or palladium supported by MOR, BEA, ZSM-5, USY, and ZSM-12 zeolites [8–10] on tungstated zirconium dioxide [11], and even on homogeneous systems [12]. Also, it has been shown that the inhibition can be reduced on BEA and ZSM-12 zeolites because of the branched structure of wide channels in these zeolites [10]. In 2005, Russia also took the path of reducing the content of benzene, by adopting fuel regulations similar to the Euro-2 standard (not more than 5 vol % benzene).

In 2011, the Technical Regulation of the Customs Union came into force, which finally prohibited in Russia fuels containing >1 vol % benzene and specified a transition to Class 3 fuels (analog of Euro-3). For example, the refineries of Russia and Customs Union encountered the problem of modernization, and researchers, the problem of new technologies, processes, and catalysts, which was manifested in a new explosion of interest in the BGF isomerization process [13, 14] and also in the problem of inhibition of the isomerization of alkanes by aromatic compounds [15]. Large refineries incorporated into structures of vertically integrated companies have already reported reaching the fuel quality satisfying the newest standard (Class 5, July 2016). However, the rapid modernization of plants was primarily due to the redistribution of material flows, lower severity of the catalytic reforming process, and isolation of the excess benzene from the reformat. At the same time, small and medium refineries having only a small number of secondary processes, have, in order to provide the fuel quality and offset the loss of the octane number, to purchase expensive MTBE and ETBE additives. These solutions a priori cannot be effective, and, therefore, development of new processes and catalysts for reducing the content of benzene remains a topical issue.

In earlier studies of our research team [16, 17], we optimized the composition of catalysts for BGF hydroisomerization, based on MOR and BEA zeolites and  $\text{Al}_2\text{O}_3$  binder component. The goal of the present study was to examine the isomerization of paraffin hydrocarbons in the presence of cyclohexane and benzene on Pt/BEA– $\text{Al}_2\text{O}_3$  and Pt/MOR– $\text{Al}_2\text{O}_3$  catalysts.

**Table 2.** Support characteristics according to N<sub>2</sub> adsorption–desorption data and TPD NH<sub>3</sub>

Sample	SiO <sub>2</sub> /Al <sub>2</sub> O <sub>3</sub>	S <sub>BET</sub> , m <sup>2</sup> g <sup>-1</sup>	Amount of desorbed ammonia			
			μmol g <sup>-1</sup>			μmol m <sup>-2</sup>
			weak acid centers 100–300°C	strong acid centers 300– 500°C	total	
ABEA	40	414	354	299	653	1.6
AMOR	20	338	388	355	743	2.2

**Table 3.** Catalytic parameters of the *n*-heptane conversion

Catalyst	T(X50%), °C			Yield of isoheptanes at the temperature of 50% conversion of <i>n</i> -heptane (in terms of <i>n</i> -heptane), wt %			
	Feed	<i>n</i> -heptane	<i>n</i> -heptane + cyclohexane	<i>n</i> -heptane	<i>n</i> -heptane + cyclohexane	<i>n</i> -heptane + benzene	
Pt <sub>Am</sub> /ABEA	<i>n</i> -heptane	246	258	259	24	39	38
Pt <sub>Ac</sub> /ABEA	<i>n</i> -heptane	253	254	263	43	47	45
Pt <sub>Am</sub> /AMOR	<i>n</i> -heptane	277	286	290	32	33	36
Pt <sub>Ac</sub> /AMOR	<i>n</i> -heptane	290	294	291	45	42	44

## EXPERIMENTAL

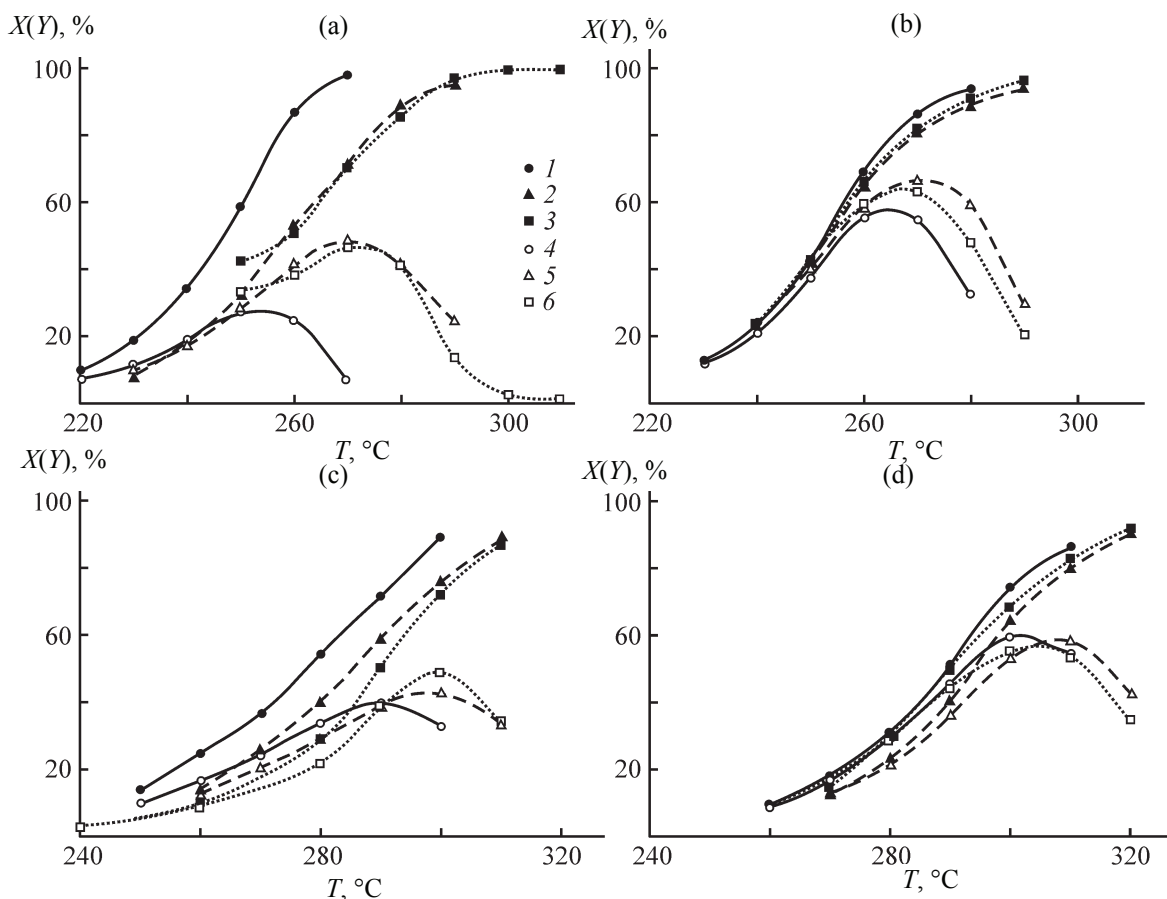
The supports were prepared from zeolites BEA (CP-814E) and MOR (CBV-21A) (Zeolyst International) with SiO<sub>2</sub>/Al<sub>2</sub>O<sub>3</sub> ratio of 40 and 20, respectively. As a binder served γ-Al<sub>2</sub>O<sub>3</sub> produced from Pural SB pseudoboehmite (Sasol Germany GmbH). A zeolite and the binder were mixed in a mass ratio of 30 : 70. Pseudoboehmite was peptized with a mixture of organic acids. The resulting paste was molded on an extruder, dried at 120°C, and calcined at 500°C. Platinum was fixed in amount of 0.3 wt % on a support from an aqueous solution of chloroplatinic acid H<sub>2</sub>PtCl<sub>6</sub> (samples designated as Pt<sub>Ac</sub>/ABEA and Pt<sub>Ac</sub>/AMOR, where A stands for Al<sub>2</sub>O<sub>3</sub>, and MOR and BEA, for the corresponding zeolites) or from tetraammineplatinum chloride [Pt(NH<sub>3</sub>)<sub>4</sub>]Cl<sub>2</sub> (samples designated as Pt<sub>Am</sub>/ABEA and Pt<sub>Am</sub>/AMOR). The samples were dried in a flow of air at 500°C. Prior to being tested, all the catalysts were reduced in hydrogen at 350°C.

The catalytic tests were performed on a catalytic setup with a flow-through isothermal reactor. The charge of the catalyst fraction of 0.2–0.7 mm was 1 g.

As feed served *n*-heptane, a model mixture composed of 20 wt % cyclohexane and 80 wt % *n*-heptane, and a model mixture composed of 20 wt % benzene and 80 wt % *n*-heptane. Test conditions: *p* = 1.5 MPa, *T* = 230–330°C, liquid hourly space velocity (LHSV) = 2 h<sup>-1</sup>, H<sub>2</sub>/feed = 3 mol/1 mol. The composition of the products was determined in the on-line mode with a Tsvet-800 gas chromatograph with PONA/PIONA capillary column (J&W Scientific). The catalyst activities were compared by using the temperature at which the maximum yield of the target products MCP and heptane isomers and the temperature of the 50% conversion of *n*-heptane. The selectivity was calculated as the ratio of the yield of MCP or heptane isomers, calculated per fraction of a component in the model mixture, to the conversion of cyclohexane or *n*-heptane, respectively.

## RESULTS AND DISCUSSION

**Acid characteristics of a support.** The texture and acid characteristics of the ABEA and AMOR supports synthesized in the study were examined by the methods of low-temperature adsorption and desorption of N<sub>2</sub> and



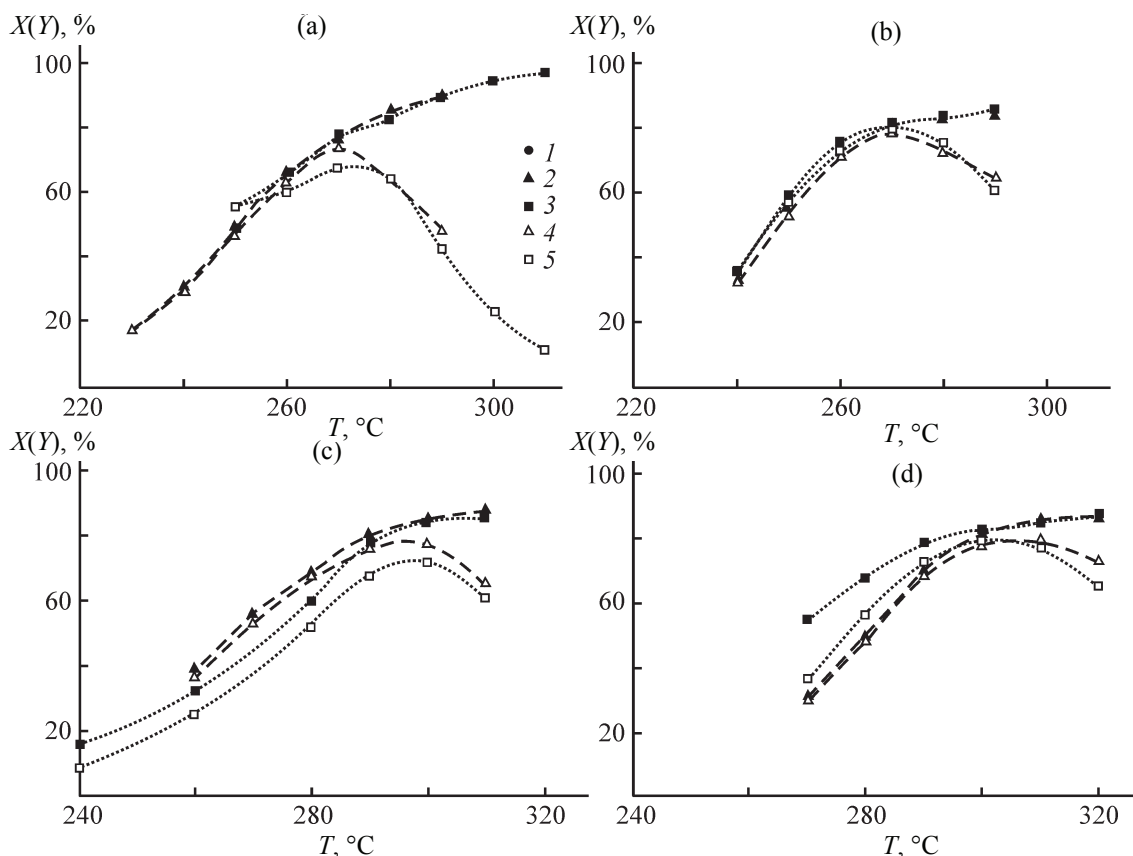
**Fig. 1.** Temperature dependences of the conversion  $X$  of  $n$ -heptane on the catalysts (a)  $\text{Pt}_{\text{Am}}/\text{ABEA}$ , (b)  $\text{Pt}_{\text{Ac}}/\text{ABEA}$ , (c)  $\text{Pt}_{\text{Am}}/\text{AMOR}$ , and (d)  $\text{Pt}_{\text{Ac}}/\text{AMOR}$  for various kinds of feed. (1)  $n$ -heptane, (2)  $n$ -heptane + cyclohexane, (3)  $n$ -heptane + benzene. Yields  $Y$  of isoheptane in terms of  $n$ -heptane for different kinds of feed: (4)  $n$ -heptane, (5)  $n$ -heptane + cyclohexane, (6)  $n$ -heptane + benzene. ( $T$ ) Temperature, ( $X$ ) conversion, ( $Y$ ) yield; the same for Fig. 2.

temperature-programmed desorption of  $\text{NH}_3$ . The results obtained in analyses of the supports are presented in Table 2.

**Isomerization of  $n$ -heptane.** The isomerization of  $n$ -heptane was examined with both pure  $n$ -heptane and its mixtures with cyclohexane and benzene. The catalytic parameters of the conversion of  $n$ -heptane on the catalysts  $\text{Pt}_{\text{Am}}/\text{ABEA}$ ,  $\text{Pt}_{\text{Ac}}/\text{ABEA}$ ,  $\text{Pt}_{\text{Am}}/\text{AMOR}$ , and  $\text{Pt}_{\text{Ac}}/\text{AMOR}$  and various types of feed are shown in Fig. 1. Table 3 list the temperatures of 50% conversion of  $n$ -heptane, found by interpolation, and the yield of branched heptanes under the given conditions.

The catalysts on which platinum was deposited from platinum ammoniate have a higher activity in the reaction of  $n$ -heptane isomerization, compared with the catalysts prepared from hexachloroplatinic acid, for both types of zeolites. This is evidenced by the shift of the temperature dependences of the conversion to

lower temperatures by 5–10°C. The absolute yield of the isomers is higher on  $\text{Pt}_{\text{Ac}}/\text{ABEA}$  and  $\text{Pt}_{\text{Ac}}/\text{AMOR}$  catalysts. The yield per delivered  $n$ -heptane on catalysts of this series is 43–47 wt %, whereas on  $\text{Pt}_{\text{Am}}/\text{ABEA}$  and  $\text{Pt}_{\text{Am}}/\text{AMOR}$  catalysts, the yield of the isomers is lower and, on average, falls within the range 32–38 wt %. The activity of the catalysts prepared with  $[\text{Pt}(\text{NH}_3)_4]\text{Cl}_2$  is probably higher because platinum deposited from a  $\text{H}_2\text{PtCl}_6$  solution is localized on the surface of aluminum oxide, whereas in the case of the  $[\text{Pt}(\text{NH}_3)_4]\text{Cl}_2$  solution, Pt is predominantly fixed within zeolite channels [17]. It is known that, upon introduction of platinum into a zeolite, the metal interacts with Brønsted acid centers [19]. In the process, a proton is transferred from a BAS to a metal particle, which results in its oxidation and fixation in the zeolite channel. Oxidized platinum particles stabilized within the zeolite channels are strong active centers. The high strength of acid centers in the catalysts impregnated



**Fig. 2.** Temperature dependences of the conversion  $X$  of cyclohexane on the catalysts (a)  $\text{Pt}_{\text{Am}}/\text{ABEA}$ , (b)  $\text{Pt}_{\text{Ac}}/\text{ABEA}$ , (c)  $\text{Pt}_{\text{Am}}/\text{AMOR}$ , and (d)  $\text{Pt}_{\text{Ac}}/\text{AMOR}$  for different kinds of feed. (1)  $n$ -Heptane, (2)  $n$ -heptane + cyclohexane, (3)  $n$ -heptane + benzene. Yields  $Y$  of MCP in terms of cyclohexene for different kinds of feed: (4)  $n$ -heptane + cyclohexane, (5)  $n$ -heptane + benzene.

from the ammoniate presumably intensifies the splitting reactions and, as a consequence, diminishes the selectivity of formation of the target reaction products, isomers of heptane. The different localizations of platinum and its different nearest neighbors lead to significantly different catalytic properties of the system.

The catalysts prepared on the basis of BEA zeolite are more active in the reaction of  $n$ -heptane isomerization, compared with the catalysts based on mordenite. This is evidenced by the shift of the conversion plots of  $n$ -heptane by 30–40°C to lower temperatures. These significant differences are attributed [8] to the branched 3D structure of large channels in the BEA zeolite, which are formed by 12-membered rings of silicon or aluminum atoms, whereas the corresponding channels in the MOR zeolite have a 1D structure and are connected by channels with finer inlet windows inaccessible to hydrocarbons.

The introduction of cyclohexane or benzene into the feed makes lower the activity in the reaction of

$n$ -heptane isomerization of the catalysts prepared from  $[\text{Pt}(\text{NH}_3)_4]\text{Cl}_2$ , which is evidenced by the shift of the conversion plots to higher temperatures by 15–20°C. This occurs because benzene and cycloalkanes (CH and MCP) block active centers of the catalysts and thereby inhibit the reaction with alkanes. However, the selectivity of  $n$ -heptane isomerization on these catalysts is substantially higher in the presence of cyclohexane or benzene (Table 4). For example, the highest yield of isoheptanes in the conversion of pure  $n$ -heptane on the  $\text{Pt}_{\text{Am}}/\text{ABEA}$  catalyst is 27 wt %, whereas in the presence of benzene and cyclohexane, it is 46–47 wt % (in terms of  $n$ -heptane. On the  $\text{Pt}_{\text{Am}}/\text{AMOR}$  catalyst, this phenomenon is somewhat less pronounced and the corresponding yields of isoheptanes are 39% against 42–48%. This is possible because the side reactions of hydrocracking of heptanes are suppressed in the presence of cyclohexane or benzene to a greater extent, compared with the isomerization reaction.

Table 4. Yield of products, conversion of the feed, and selectivity of the catalysts in the reactions of isomerization and hydroisomerization, wt %

Zeolite	BEA						MOR							
	H <sub>2</sub> PtCl <sub>6</sub>		[Pt(NH <sub>3</sub> ) <sub>4</sub> ]Cl <sub>2</sub>		[Pt(NH <sub>3</sub> ) <sub>4</sub> ]Cl <sub>2</sub>		H <sub>2</sub> PtCl <sub>6</sub>		[Pt(NH <sub>3</sub> ) <sub>4</sub> ]Cl <sub>2</sub>		[Pt(NH <sub>3</sub> ) <sub>4</sub> ]Cl <sub>2</sub>			
Pt	260	270	270	270	250	270	270	270	300	310	300	290	300	300
Products	<i>n</i> -heptane	<i>n</i> -heptane + cyclohexane	<i>n</i> -heptane + benzene	<i>n</i> -heptane + benzene	<i>n</i> -heptane	<i>n</i> -heptane + benzene	<i>n</i> -heptane + benzene	<i>n</i> -heptane + benzene	<i>n</i> -heptane	<i>n</i> -heptane + cyclohexane	<i>n</i> -heptane + benzene	<i>n</i> -heptane	<i>n</i> -heptane + cyclohexane	<i>n</i> -heptane + benzene
Methane	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.1	0.1	0.1
Ethane	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.1	0.1	0.1	0.1	0.1
Propane	5.0	4.5	5.6	7.2	12.0	7.2	8.0	8.0	6.0	7.0	4.0	11.9	10.0	7.4
Isobutane	6.8	5.7	7.2	16.0	16.0	9.4	10.4	10.4	6.6	8.3	4.6	13.3	11.4	8.4
<i>n</i> -Butane	0.3	0.2	0.2	0.2	0.6	0.4	0.5	0.5	0.8	0.9	0.6	2.4	2.0	1.4
Isopentane	0.6	0.1	0.2	0.2	1.5	0.6	0.5	0.5	0.3	0.3	0.3	1.8	1.3	0.7
<i>n</i> -Pentane	0.1	0.0	0.1	0.1	0.3	0.1	0.1	0.1	0.2	0.2	0.2	0.7	0.5	0.3
Isohexane	0.4	0.2	0.2	0.2	0.97	0.7	0.5	0.5	0.2	0.5	0.3	0.9	0.8	0.7
<i>n</i> -Hexane	0.1	0.1	0.1	0.1	0.33	0.2	0.2	0.2	0.1	0.2	0.1	0.2	0.2	0.2
Isoheptanes	55.3	53.0	48.5	63.2	26.9	37.7	36.1	36.1	59.5	46.4	43.3	39.4	33.8	38.2
Isoheptanes (in terms of <i>n</i> -heptane)	55.3	66.3	63.2	63.2	26.9	47.1	46.3	46.3	59.5	58.0	55.2	39.4	42.2	48.7
<i>n</i> -Heptane	31.1	16.5	14.2	14.2	41.1	23.5	23.4	23.4	25.9	17.1	24.8	28.8	19.8	22.0
MCP <sup>a</sup>	–	15.6	18.2	18.2	–	14.6	14.5	14.5	–	15.7	17.0	–	15.5	15.8
CH <sup>a</sup>	–	3.9	4.5	4.5	–	4.7	4.9	4.9	–	3.0	3.7	–	4.0	3.5
Other hydrocarbons	0.3	0.3	0.4	0.4	0.3	0.8	0.6	0.6	0.3	0.4	0.4	0.5	0.9	0.7
Benzene	–	0	0	0	–	0	0	0	–	0	0	–	0	0
X( <i>n</i> -Heptane)	69.0	80.6	81.5	81.5	58.9	70.7	70.2	70.2	74.1	79.9	68.5	71.2	75.2	72.0
X(CH)	–	80.5	80.3	80.3	–	76.4	77.2	77.2	–	85.2	82.6	–	84.1	84.1
X(Benzene)	–	–	100	100	–	–	100	100	–	–	100	–	–	100
S(Isoheptanes)	80.2	82.2	77.3	77.3	45.7	66.7	65.5	65.5	80.4	72.6	80.6	55.3	56.2	68.1
S(MCP)	–	96.9	95.2	95.2	–	95.9	87.1	87.1	–	92.3	95.7	–	77.4	85.5

<sup>a</sup> (MCP) methylcyclopentane, (CH) cyclohexane.

For the catalysts on which platinum is deposited from  $\text{H}_2\text{PtCl}_6$ , the increase in the isomerization selectivity in the presence of cyclohexane or benzene is weakly pronounced. For example, the increase in the yield of isoheptanes is only 20% on the  $\text{Pt}_{\text{Ac}}/\text{ABEA}$  catalyst, against 74% for the  $\text{Pt}_{\text{Am}}/\text{ABEA}$  catalyst. No such increase in selectivity is observed for the  $\text{Pt}_{\text{Ac}}/\text{AMOR}$  catalyst.

For the catalysts on which platinum is deposited from  $\text{H}_2\text{PtCl}_6$ , the shift of the *n*-heptane conversion plots to lower temperatures is exceedingly weakly pronounced (2–3°C). Thus, the inhibition of the conversion of alkanes on these catalysts is either weakly pronounced, or is absent at all. These data may also be due to the localization of platinum. Presumably, the localization of platinum on the  $\text{Al}_2\text{O}_3$  surface and its remoteness from acid centers of the zeolite affect the selectivity of the reactions in which the mixture components are converted.

#### Hydroisomerization of benzene and cyclohexane.

The catalytic parameters of the conversion of benzene and cyclohexane in a mixture with *n*-heptane on  $\text{Pt}_{\text{Am}}/\text{ABEA}$ ,  $\text{Pt}_{\text{Ac}}/\text{ABEA}$ ,  $\text{Pt}_{\text{Am}}/\text{AMOR}$ , and  $\text{Pt}_{\text{Ac}}/\text{AMOR}$  catalysts are presented in Fig. 2. Table 4 lists the yields of the products, conversions of feed, and selectivities of the catalysts in the reactions of isomerization and hydroisomerization.

For all the samples under study, prepared with  $\text{H}_2\text{PtCl}_6$  and  $[\text{Pt}(\text{NH}_3)_4]\text{Cl}_2$ , the conversion of benzene was 100% in the whole temperature we examined (230–320°C). Therefore, it is convenient, when analyzing the selectivity of MCP formation from the starting feed benzene, to consider the stage of cyclohexane conversion into MCP and calculate the selectivity of MCP formation as the ratio between the MCP yield and the cyclohexane conversion.

According to the plots describing the conversion of cyclohexane and Table 4, the yield of MCP on all the catalysts is independent of which reagent is used (cyclohexane or benzene) and reaches, in terms of CH, a value of about 70–75% on the catalysts prepared from the ammoniate and 80–82% on those prepared from  $\text{H}_2\text{PtCl}_6$ . As also in the case of isomerization of *n*-heptane, the catalysts based on BEA are more active in the reaction of cyclohexane isomerization, compared with the catalysts based on mordenite.

## CONCLUSIONS

(1) Catalysts for hydroisomerization of benzene-containing fractions, based on BEA and MOR zeolites, were examined on model mixtures.

(2) The influence exerted by the platinum precursor was examined and it was shown that the catalysts on which platinum is deposited from platinum ammoniate are more active in the isomerization reactions of heptane and cyclohexane. The catalysts based on BEA zeolite exhibit a higher activity in the reactions of isomerization of *n*-heptane and cyclohexane, compared with the catalysts based on mordenite.

(3) It was demonstrated that cyclohexane and benzene inhibit the conversion of *n*-heptane on the catalysts on which platinum is deposited from a  $[\text{Pt}(\text{NH}_3)_4]\text{Cl}_2$  solution. If platinum is deposited from  $\text{H}_2\text{PtCl}_6$ , no isomerization inhibition is observed and the yield of the target products is higher than that in the case of platinum ammoniate.

(4) It was shown that using the acid  $\text{H}_2\text{PtCl}_6$  as a precursor of platinum provides availability of reagents for preparation of industrial batches of the catalyst and make it cost-attractive for industrial manufacture.

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