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OPTIMIZATION OF THE OPERATION OF AN ISOMERIZATION INSTALLATION FOR LIGHT PETROL FRACTIONS

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The interest in this process lies in the fact that it has great value since low-octane components are used as raw materials - the n fraction. temperature – 62 °C and catalytic reforming raffinates. This raw material contains mainly pentane and hexane fractions. This raw material, as well as fractions C_5 and C_6 obtained from a gas fractionation unit (GFU), and a central gas fractionation unit (CGFU), are isomerized in a hydrogen environment in the presence of a catalyst. Hydrocarbons with a relatively high octane number are obtained. When isomerizing the pentane fraction, a product with a higher octane number is obtained. Isomerization of n-pentane is of interest not only for the oil refining industry, but also for the petrochemical industry, since isopentane can be converted by dehydrogenation into isoprene, a raw material for rubber. The article conducted a study on the selection of the optimal catalyst for the light gasoline fraction isomerization unit located at the Atyrau Oil Refinery. The catalytic activity of the catalyst samples was assessed in a laboratory flow-type installation. Determination of the mass fraction of C_2-C_5 hydrocarbons in C_5 hydrocarbon fractions (in raw materials and catalyst) was carried out by a method based on the separation of mixture components by gas-liquid chromatography. The components leaving the chromatographic column were detected using a thermal conductivity detector. The calculation of the mass fractions of components was carried out using the normalization method, taking into account correction factors. The minimum concentration of C_2-C_5 hydrocarbons, determined by the method used, is 0.01-0.05% wt.

Key words: isomerization process, catalyst, fraction, isomerate, chromatographic study, motor gasoline, isomerization unit.

ОПТИМИЗАЦИЯ РАБОТЫ УСТАНОВКИ ИЗОМЕРИЗАЦИИ ЛЁГКИХ БЕНЗИНОВЫХ ФРАКЦИЙ

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Интерес к данному процессу заключается в том, что он имеет большую ценность так, как в качестве сырья используются низкооктановые компоненты – фракция н. к. – 62 ° С и рафинаты каталитического риформинга. В этом сырье содержится в основном пентановая и гексановая фракции. Это сырье, а также фракции C_5 и C_6, получаемые с газофракционирующей установки (ГФУ), и центральная газофракционирующая установка (ЦГФУ), изомеризуются в среде водорода в присутствии катализатора. Получают углеводороды со сравнительно высоким октановым числом изостроения. При изомеризации пентановой фракции получают продукт с более высоким октановым числом. Изомеризация н-пентана представляет интерес не только для нефтеперерабатывающей, но и для нефтехимической промышленности, так как изопентан дегидрированием можно превратить в изопрен – сырье для каучука. В статье проведено исследование по подбору оптимального катализатора для установки изомеризации легких бензиновых фракции, расположенной на

Атырауском НПЗ. Оценку каталитической активности образцов катализатора проводили на лабораторной установке проточного типа. Определение массовой доли C_2-C_5 -углеводородов в C_5–фракциях углеводородов (в сырье и катализате) проводили методом, основанном на разделении компонентов смеси методом газожидкостной хроматографии. Фиксирование компонентов, выходящих из хроматографической колонки, производили с помощью детектора по теплопроводности. Расчет массовых долей компонентов производили методом нормирования с учётом поправочных коэффициентов. Минимальная концентрация углеводородов C_2-C_5, определяемая по используемой методике, составляет 0,01-0,05 % масс.

Ключевые слова: процесс изомеризации, катализатор, фракция, изомерат, хроматографическое исследование, автомобильный бензин, установка изомеризации.

ЖЕҢІЛ БЕНЗИН ФРАКЦИЯЛАРЫН ИЗОМЕРЛЕУ ҚОНДЫРҒЫСЫНЫҢ ЖҰМЫСЫН ОҢТАЙЛАНДЫРУ

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Бұл процеске деген қызығушылық оның құндылығы жоғары, өйткені шикізат ретінде төмен октанды компоненттер қолданылады-н. к. фракциясы – 62 °С және каталитикалық риформинг тазартқыштары. Бұл шикізатта негізінен пентан және гексан фракциялары бар. Бұл шикізат, сондай-ақ ГФҚ және ОГФҚ-ден алынған C_5 және C_6 фракциялары катализатордың қатысуымен сутегі ортасында изомерленеді. Көмір сутектер изостроениянның салыстырмалы түрде жоғары октан санымен алынады. Пентан фракциясы изомерленген кезде октан саны жоғары өнім алынады. Н-пентанның изомерленуі тек мұнай өңдеу үшін ғана емес, сонымен қатар мұнай-химия өнеркәсібі үшін де қызығушылық тудырады, өйткені сусыздандыру арқылы изопентанды резеңке үшін изопрен шикізатына айналдыруға болады. Мақалада Атырау МӨЗ-де орналасқан жеңіл бензин фракцияларының изомерленуін орнату үшін оңтайлы катализаторды таңдау бойынша зерттеу жүргізілді. Катализатор үлгілерінің каталитикалық белсенділігін бағалау ағынды типтегі зертханалық қондырғыда жүргізілді. Көмірсутектердің C_5 фракцияларындағы (шикізат пен катализатта) C_2-C_5–көмірсутектердің массалық үлесін анықтау газ–сұйық хроматография әдісімен қоспаның компоненттерін бөлуге негізделген әдіспен жүргізілді. Хроматографиялық бағаннан шығатын компоненттерді бекіту жылу өткізгіштік детекторының көмегімен жүзеге асырылды. Компоненттердің массалық үлестерін есептеу түзету коэффициенттерін ескере отырып, нормалау әдісімен жүргізілді. Қолданылатын әдіс бойынша анықталған C_2–C_5 көмірсутектерінің ең аз концентрациясы массалардың 0,01-0,05% құрайды.

Түйін сөздер: изомерлеу процесі, катализатор, фракция, изомерат, хромато-графикалық зерттеу, автомобиль бензині, изомерлеу қондырғысы.

Introduction. Large hydrocarbon deposits are located in the territory of the Caspian Sea, which are characterized by special geostrategic, geographical, military and political features. In addition to oil and gas resources, the location between the main markets in the West and East is of great strategic importance [1]. The ways of realization of Kazakhstan oil today depend on complex processing of productions, where oil is a raw material for the final product. This is due to the fact that the capacities and technological equipment of the relevant refineries are not oriented to extract the full range of products contained in the feedstock [2].

Crude oil is a complex raw material from which a wide range of petroleum products can be obtained. According to the technological schemes of production, processing into fuel, oil and mixed directions prevails [3]. The introduction of the process of isomerization of straight-run gasoline fractions into the practice of oil refineries determines the relevance of studying this process, as well as the need for scientific research in the field of development of technology of isomerization processes and selection of catalysts that provide optimal conditions for isomerization reactions [4].

The catalysts used in various isomerization technologies have a common kinetic pattern: at high temperatures, the yield of isoalkanes is limited by thermodynamic equilibrium, and at low temperatures, the reaction rate decreases. The selection of a catalyst that provides a minimum yield of aromatic compounds and olefins is important [5].

The purpose of isomerization processes in oil refining is to improve the anti-knock properties of aviation and motor gasoline. In the oil refining industry, they began to be used to produce isobutane from *n*-butane. Isobutane was further alkylated with butylenes to give 2,2,4-trimethylpentane (isooctane).

The great value of the isomerization process lies in the fact that low-octane components are used as raw materials - the n fraction. temperature – 62 °C and catalytic reforming raffinates. This raw material contains mainly pentane and hexane fractions. This raw material, as well as fractions C_5 and C_6 obtained with HFCs and CHFCs, are isomerized in a hydrogen environment in the presence of a catalyst. Hydrocarbons with a relatively high octane number are obtained. When isomerizing the pentane fraction, a product with a higher octane number is obtained. Isomerization of n-pentane is of interest not only for the oil refining industry, but also for the petrochemical industry, since isopentane can be converted by dehydrogenation into isoprene, a raw material for rubber. Thus, isomerization can serve both for the production of high-octane gasoline and for the production of valuable synthetic rubbers [6,7].

The isomerization of paraffin hydrocarbons on a solid catalyst proceeds in two directions: hydrogenation-dehydrogenation and isomerization itself. When a hydrocarbon molecule reaches the catalyst, one of the hydrogen atoms of that molecule is adsorbed on the metal site, and the associated carbon atom is adsorbed on the acid site. The adsorbed molecule isomerizes and leaves the catalytic surface under the action of molecular hydrogen [8-10].

Catalysts can be divided into five main groups: Friedel-Crafts catalysts, tungsten sulfide, bifunctional catalysts, synthetic noble metal zeolites (including rare earth metal additives) and complex catalysts (combining bifunctional and zeolite catalysts with Friedel-Crafts catalysts).

Friedel-Crafts catalysts in most cases contain anhydrous aluminum chloride (sometimes with antimony trichloride) in the form of a complex with hydrocarbons activated by hydrogen chloride. Isomerization using Friedel-Crafts catalysts can be carried out at 20 *atm* and 40-120 °C and even at 24-50°C (catalysts based on bromine chloride).

Tungsten sulfide from sulfide catalysts turned out to be most active and selective at moderate reaction temperatures (about 400 °C). The degree of isomerization of c-hexane on it reached 60% with insignificant hydrocarbon splitting. To ensure stable operation of the catalyst in the system, it is necessary to maintain a pressure of at least 40 atm.

It is important to note that the presence of naphthenic hydrocarbons (up to 20-25%), sulfur compounds and moisture in the raw material does not deteriorate the process performance. The use of hydrogen, despite the fact that it inhibits the isomerization reaction on tungsten sulfide, is necessary because it prevents coking of the catalyst [11].

Bifunctional catalysts are a type of reforming catalyst - platinum or palladium on alumina. However, sometimes aluminosilicate or a mixture of aluminum and boron oxide is used as a carrier. These catalysts have sufficient selectivity for the isomerization of C_5-C_6 paraffinic hydrocarbons, which distinguishes them from Friedel-Crafts catalysts, but their activity is very low, so the temperature has to be increased

Increasing the acidity of a bifunctional catalyst is also achieved by treating it with chlorine-containing organic compounds, and maintaining acidity by introducing HC1 into the system [12].

Synthetic zeolites with noble metals - platinum or palladium, as well as with additives of rare earth metals are based on the use of crystalline aluminosilicates - zeolites. These catalysts are very active in the isomerization of n-peitane and n-hexane. Platinum or palladium deposited on Y-type zeolite allows the isomerization process to be carried out at 315-343 °C, i.e. approximately 150°C lower than when using alumina as a carrier. Experiments on catalysts with different platinum contents showed that even a minimal concentration of platinum ensures high efficiency of isomerization of paraffinic hydrocarbons. Typically the platinum or palladium content ranges from 0.3 to 0.6% [13].

Complex catalysts combine the advantages of bifunctional and zeolite catalysts (often called hydroisomerization catalysts) with the advantages of Friedel-Crafts catalysts, which allow the process to be carried out at lower temperatures. Complex catalysts can be used at 90-200 °C. With their help, almost equilibrium yields of isopentanes and isohexanes are

achieved. Their selectivity is high: only a small amount of C_1-C_4 paraffins is formed as a by-product. However, it should be borne in mind that aluminum chloride, which is usually included in complex catalysts, is hygroscopic; sooner or later it hydrolyzes, and the resulting aluminum hydroxide is deposited inside the pores of the catalyst, reducing their volume and complicating regeneration. In this regard, complex catalysts have not found application in isomerization processes carried out at oil refineries [14-16].

Recently, platinum treated with chlorine-containing organic compounds has been used as a catalyst. The activity of the catalyst is maintained by additional input of HC1, as well as by a gradual increase in temperature to 160 °C.

For the first time, this work proposes replacing the proprietary UOP 1-8+/UOP I-82 catalyst (USA) with a less expensive SI-2 catalyst [17]. This catalyst allows the isomerization process to be carried out at the inlet of reactors with a high degree of isomerization of both pentanes and hexanes. It consists of platinum evenly distributed over the surface of sulfated zirconium oxide modified with aluminum. It has high activity in the isomerization reactions of low-boiling hydrocarbons under thermodynamically favorable conditions, not inferior in efficiency to chlorinated catalysts [17]. At the same time, its resistance to catalytic poisons is higher, and it also tolerates their short-term breakthroughs without reducing activity [18]. In addition, additional advantages of such catalysts are their long service life (up to 10 years) and the possibility of regeneration.

Materials and methods. The catalytic activity of the SI-2 catalyst samples was assessed in a laboratory flow-type installation (Figure 1).

The normal pentane fraction from the raw material tank is drained by gravity into a dosing pump, from where it is dosed into the evaporator. The recycle fraction of normal pentane is similarly fed through a parallel feed dosing line. Gas streams of fresh (concentrated) and recycle fractions are mixed with each other and with hydrogen and sent to the reactor. The catalyst is loaded into the reactor in a stationary bed. The reactor is placed in a laboratory furnace, which heats the reaction stream to the reaction temperature.

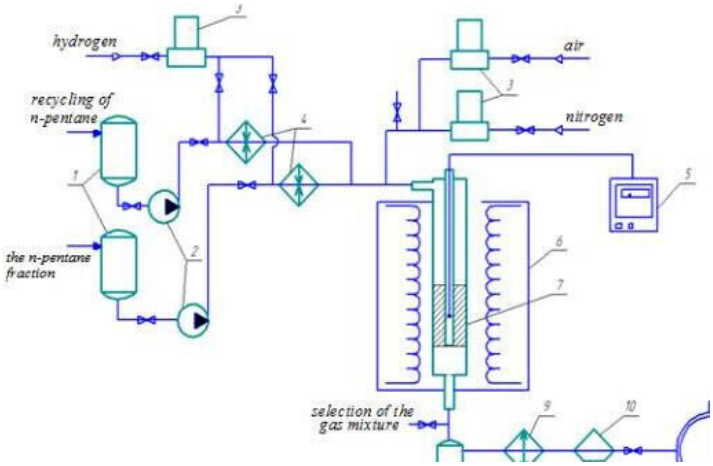

Figure 1 - Diagram of a laboratory setup for testing a catalyst in the process of isomerization of n-pentane to isopentane:

1 – containers for raw materials; 2 – dosing pumps; 3 – gas flow meters; 4 – raw material evaporators; 5 – temperature meter; 6 – laboratory single-zone furnace; 7 – laboratory reactor; 8 – separator; 9 – catalytic condenser; 10 - catlysate separator; 11 – drum type gas meter

In the catalyst layer, pentane of normal structure is isomerized into pentane of iso-structure. The reaction products are sent to a separator, in which the mixture of C_5 hydrocarbons is condensed. The gas part of the flow, consisting mainly of hydrogen and partly of cracking products, is taken into account in a drum gas meter.

The catalyst is loaded into a pre-prepared reactor. A catalyst with a volume of 10 ml is loaded into

an isothermal reactor, and palladium is reduced in a hydrogen flow of 375 h^{-1} at a temperature of 145 °C and a pressure of 1 atm for 1 hour.

Determination of the mass fraction of C_2–C_5 hydrocarbons in C5 hydrocarbon fractions (in raw materials and catalyst) was carried out by a method based on the separation of mixture components by gas-liquid chromatography. The components leaving the chromatographic column were detected using a thermal conductivity detector.

The calculation of the mass fractions of components was carried out using the normalization method, taking into account correction factors. The minimum concentration of C_2–C_5 hydrocarbons, determined by the method used, is 0.01-0.05 % wt.

The preparation of the stationary phase and filling of the chromatographic column was carried out as follows. Diatomite brick was ground in a mortar, sifted on sieves with opening sizes of 0.16 and 0.25 mm, selecting a fraction of 0.16-0.25 mm, it was poured with concentrated nitric acid in an evaporation (porcelain) cup and kept under the acid for 3 hours. After that, the diatomite brick was washed from the acid with distilled water until a neutral reaction (the last portion of water should not cause a change in the color of methyl orange).

The washed diatomite was dried at a temperature of 100-120 °C for 1 hour and calcined at a temperature of 900 °C for 3 hours. Cooling was carried out in a desiccator, after which it was again sieved through sieves with opening sizes of 0.16 and 0.25 mm.

The prepared diatomite brick (chamotte) was impregnated with a stationary phase - triethylene glycol dibutyrate in an amount of 15-20% by weight of diatomite. A sample of 15–20 g of triethylene glycol dibutyrate was measured into a beaker with a capacity of 250 cm^3 on a laboratory scale, dissolved in 200 cm^3 of ethyl ether and transferred quantitatively into a round-bottomed flask with a capacity of 500 cm^3. 100 g of prepared diatomite (chamotte) was poured into the solution. The mixture was mixed well and kept for 20–30 minutes. The flask was sealed with a stopper with two siphons for purging, and the contents were shaken for an additional 5–6 minutes. Ethyl ether was distilled off in a water bath at a temperature of 35–40 °C with simultaneous purging with nitrogen until the smell of ethyl ether disappeared.

Before filling the chromatographic column with the finished sorbent, the column was washed successively with ethyl alcohol, acetone and then purged with air pressure for 20 minutes. A clean and dry column was filled with the prepared stationary phase using a funnel and a water jet (oil) pump. One of the ends of the column was previously covered with a glass wool swab and connected to the pump. The nozzle was compacted with a vibrator. The column was also tapped with a wooden stick to compact it. The other end of the column was also covered with a glass wool swab.

Chromatographic analysis was carried out under the following conditions:

Column temperature, °C 20–40

Chromatograph operating mode isothermal

Carrier gas speed, cm^3/min 25–30

Chart tape speed, mm/hour 240–720

Объём вводимой жидкой пробы, mkl 2–3

Volume of injected vapor-gas mixture, cm^3 1–2

The identification of components C_2-C_5 in the hydrocarbon mixture using the chromatogram was carried out taking into account the relative retention volumes given in Table 1.

Table 1 – Relative retention volumes of raw material components on a 15% dibutyrate triethylene glycol column on diatomite brick and sensitivity correction factors for the thermal conductivity detector

Component name	Vrel.	K
Ethane + ethylene	0,01	0,8
Carbon dioxide	0,02	1,36
Propane	0,04	0,92
Propylene	0,06	0,92
Isobutane	0,09	1,02
n-Butane	0,13	1
Butene-1 + isobutylene	0,18	1,01
Butene-2 – trans	0,23	0,99
Butene-2-cis	0,27	1,01
Isopentane	0,29	1,07
Butadiene	0,32	1,02
3-methylbutene-1	0,33	1,11
n-Pentane	10,38	1,05

The peak area of all components was calculated as the product of the peak height and its width, measured at half the height (half-width). The resulting value of the area of each peak was multiplied by the sensitivity scale of the device. The location of the peaks on the chromatographic diagram is shown in Figure 2.

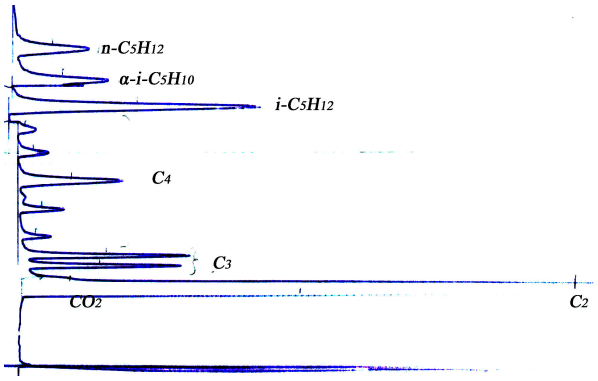

Figure 2 – Location of the chromatographic response of each component on the diagram

The volume fraction of each component in the contact gas (X) in percent was calculated using the formula:

$$X_i = \frac{K_i \times S_i\,(100 - \sum X)}{\sum (S_i \times K)}$$

где K is the molar correction factor of the component (Table 1); S_i - component peak area, cm^2, $\sum S_i$ - sum of the areas of all components multiplied by correction factors, cm^2, $\sum X$ - the sum of the volume fractions of the components of permanent gases, determined by the method of analysis of H_2, O_2, N_2, CH_4 CO in the composition of the contact gas.

The result of the analysis is taken as the result of one determination.

From the data of gas chromatographic analysis, the conversion of n-pentane and the selectivity of isopentane formation are calculated.

Results and discussion. Table 2 presents the results of isomerization of the pentane-hexane fraction with an estimate of the yield of isopentane.

Table 2 – Effect of temperature on the yield of isomerization products on the SI-2 catalyst (space velocity of the raw material in liquid 1.5 h-1)

Test temperature, °C	Isopentane yield, %	Selectivity, %	Equilibrium concentration of isopentane, %
110	27,4	99,1	42
120	30,8	99	39
130	33,5	99	37
140	35,9	89,7	35
150	33,1	89,7	34
170	30,2	89,1	31

As can be seen from Table 2, the highest yield of isopentane with a selectivity of 89.7 % is observed at a temperature of 140 °C.

Table 3 shows the effect of space velocity on the yield of isopentane. As can be seen from the table, the most effective space velocity is 1.0 h-1, at which an increase in isopentane yield to 36.4 % and selectivity to 89.8 % is achieved.

Table 3 – Effect of space velocity on the yield of isomerization products on the SI-2 catalyst (process temperature 140 °C)

Volumetric velocity of raw materials (liquid), hour-1	Isopentane yield, %	Selectivity, %
1.0	36.4	89.8
1.5	35.9	89.7
2.0	30.1	89.0

In addition to pentane isomerization, it is necessary to evaluate the yields of isohexane. Figure 3 shows the results of a study of the dependence of the isomerate yield on temperature (for comparison, production data for the operating catalyst I-82 are presented).

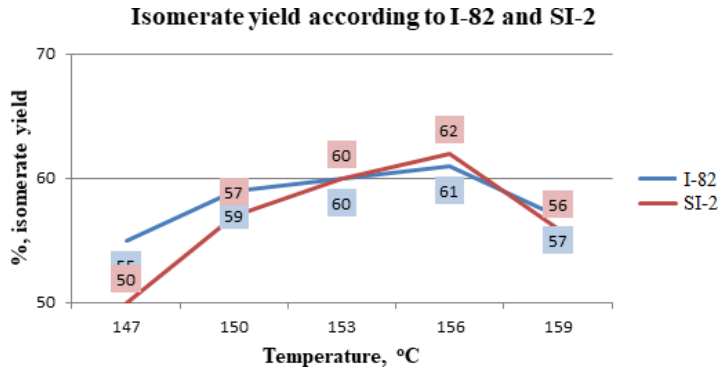

Figure 3 - Ratio of isomerate yield to temperature

As can be seen from the graph of the dependence of isomerizate yield on temperature, the highest isomerizate yield is observed on the SI-2 catalyst (62 %) at a temperature of 156 °C, at which the isomerizate yield on the I-82 catalyst was 61 %.

Figure 4 shows octane number data at various temperatures (for comparison, production data for the current I-82 catalyst is presented).

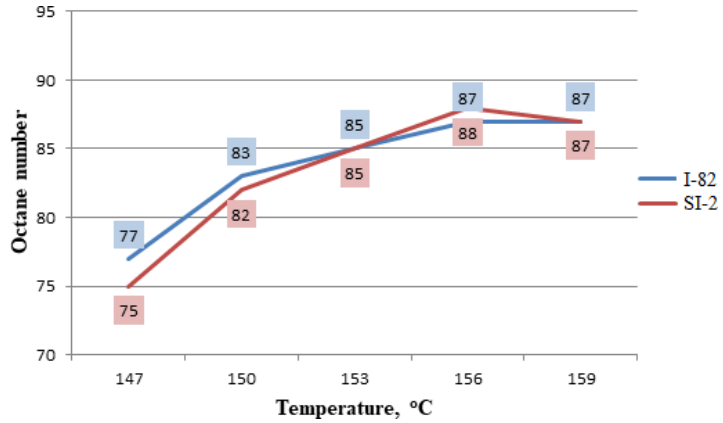

Figure 4 - Ratio of octane number to temperature

As can be seen from Figures 3 and 4, catalysts SI-2 and I-82 at the same temperature ranges show almost identical performance.

Conclusions. Thus, the catalytic activity of the SI-2 catalyst was evaluated at the light gasoline fractions isomerization unit. A feature of this catalyst is its high isomerizing activity, which is practically not inferior to chlorinated aluminum oxide catalysts with significant resistance to the effects of catalytic poisons [18]. As a result of the study of the process of isomerization of light gasoline fractions on the SI-2 catalyst, it was revealed that the catalytic performance of the proposed SI-2 catalyst and the I-82 catalyst used in production have almost the same performance. It is necessary to take into account that the cheaper SI-2 catalyst has many of the above effective properties.

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