OXIDATIVE SULFUR REFINING OF GASOLINE FRACTIONS IN THE PRESENCE OF HYDROGEN PEROXIDE ON A HETEROGENEOUS CATALYST

N.K. Andryushchenko[1], S.G. Yunusov[1], I.A. Khalafova[2], Z.B. Babayeva[1], Kh.I. Talybova[1]

[1]Institute of Petrochemical Processes named after Yu.G. Mamedaliyev of the Ministry of Science and Education of Azerbaijan, Baku
[2]Azerbaijan State Oil and Industry University of the Ministry of Science and Education of Azerbaijan, Baku
khalafova.irada@gmail.com

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Abstract: *This study is aimed at improving the quality of motor fuels, in particular, reducing the total sulfur content by developing alternative methods – the process of oxidative desulfurization with hydrogen peroxide without the use of hydrogenation processes. The process of oxidative desulfurization with hydrogen peroxide of wide and narrow fractions of GCC and GC: b.s.-120 °C and 120 °C-b.e. gasoline of catalytic cracking and b.s.-85 °C and 85 °C-b.e. gasoline of coking on a heterogeneous Mo-containing catalyst was studied. The total sulfur content in the fractions b.s.-120 °C and 120 °C-b.e. GCC was 283 and 636 ppm, respectively, and in the fractions b.s.-85 °C and 85 °C-b.e. GC – 1451 and 1894 ppm, respectively. In the course of the work performed, the optimal conditions for carrying out the process in a flow mode were identified: $T = 100$ °C and $V = 2$ h^{-1}. Subsequently, the obtained fractions were subjected to extraction with ionic liquids (IL) to isolate sulfur-containing compounds. In this case, the degree of desulfurization of the GCC fractions was 73.7%, and that of the GC fractions was 83.6%, with virtually no change in the octane number (ON) of gasoline.*

In the course of the studies on the process of oxidative desulfurization of CCG and CG gasolines, followed by extraction with ionic liquids, the total sulfur content does not exceed 55-60 ppm in the composition of commercial gasoline.

Key words*: oxidative desulfurization, extraction with ionic liquids, hydrogen peroxide, catalytically cracked gasoline, coking gasoline, degree of desulfurization*

Introduction

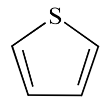
The problem of obtaining environmentally friendly motor gasoline is reduced to reducing the toxic products of their combustion by reducing the proportion of carcinogenic aromatic hydrocarbons and sulfur compounds, as well as introducing high-octane alkylates, isomerizates, and oxygenates into their composition, which will significantly reduce the toxicity of exhaust gases.

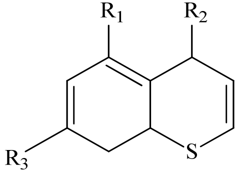
The need to reduce the total sulfur content in fuels arises from the fact that their combustion releases toxic sulfur compounds—primarily sulfur dioxide—into the atmosphere.

The presence of sulfur compounds in petroleum products, including gasoline and oils, significantly deteriorates their performance characteristics. These compounds reduce oxidative stability, increase the tendency to form gums, and negatively affect storage properties. In automotive gasolines, sulfur compounds decrease stability and compatibility with additives, while also enhancing corrosiveness and promoting carbon deposition. It is known that sulfur-containing compounds are also active catalytic poisons in many oil refining processes. With their prolonged action on the catalyst, poisoning is irreversible [1, 2].

Improving the environmental performance of motor fuels to the level of international standards is currently the main task of modern oil refining. To date, according to the specification, fuels can contain no more than 10 ppm sulfur, no more than 35% aromatics, and no more than 1% benzene. Bringing sulfur to such low values is a very difficult task. Therefore, the task of searching for new cost-effective technologies to reduce the content of total sulfur in the products of primary and secondary oil refining, as well as at the stage of its preparation for processing, is very relevant [3].

The distribution of sulfur by fractions depends on the nature of the oil and the type of sulfur compounds. When moving to higher-boiling fractions, the amount of sulfur-containing compounds increases.

The structure of sulfur compounds is represented mainly by mercaptans RSH, thio-ethers R^1SR^2, thiophenes 

, benzothiophenes 

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The hydrotreatment method is widely used in industry, which makes it possible to achieve rather high degrees of desulphurization. But, it should be noted that this method requires modern expensive catalysts and a large consumption of expensive hydrogen. In addition, as a result of desulfurization in a hydrogen environment, olefins are saturated, and, consequently, a significant decrease in the octane number (ON) by 4-6 points, which significantly worsens the quality of motor gasoline, causes corrosion of equipment, and reduces the antioxidant capacity fuel, etc.

In this regard, in order to obtain fuels with a very low (below 10 ppm) sulfur content and minimize ON losses, alternative methods for desulfurization of motor fuels (adsorption, extraction, oxidative desulfurization using alkyl peroxides and hydrogen peroxide, etc.) are being developed without using hydrogen [3]. During oxidative desulfurization, sulfides and disulfides are converted into sulfoxides and sulfones (R_2SO and R_2SO_2) [1-6]. Unlike sulfides, sulfoxides and sulfones have a higher polarity and proton-acceptor capacity, which makes it possible to develop various methods for their isolation from gasoline fractions. The presence of a 35% hydrogen peroxide solution creates good conditions for the catalytic oxidation of gasoline fractions followed by extraction of IL. The main advantages of these processes are that they are carried out at room temperature and atmospheric pressure without changing the octane number of gasoline.

Extensive research is being carried out on the direct oxidation of fuels using hydrogen peroxide in the presence of acids (sulphuric, hydrochloric, acetic, etc.) for the oxidation of sulfides and their subsequent extraction, which makes it possible to extract organosulfur compounds in the form of concentrates of petroleum sulfoxides and sulfones. Sulfide oxidation can be performed without the use of acids, employing 30–35% aqueous hydrogen peroxide as the oxidizing agent. Molybdenum- and cobalt-containing compounds are commonly used as catalysts in this process [6–9].

In study [10], it was shown that catalysts based on molybdenum have extremely high selectivity in the hydrodesulfurization of dibenzothiophene.

Thus, in connection with the increasing quality requirements for motor gasoline, there is an acute problem of finding new oxidizing systems for deep purification of motor fuels from sulfur compounds without reducing the octane number of the latter.

In this study, we consider the possibility of reducing the content of organosulfur compounds in the composition of catalytic cracking and coking gasolines and their narrow fractions as a result of catalytic oxidation with hydrogen peroxide using heterogeneous Co- and Mo-containing catalysts. In the course of our studies, the Mo-containing catalyst showed better results in oxidative desulfurization than the cobalt-containing one. Therefore, the results obtained on the molybdenum-containing catalyst are presented further in the article.

The extraction of sulfur-containing compounds from oxidized gasoline fractions was carried out using ionic liquids, the structural features of which are the asymmetry of the molecule, the distribution of the electrostatic charge of a relatively large group of atoms, and the presence of hydrogen bonds between the cation and anion, which give the IL structure a certain direction. The nature of the IL anion determines their ability to dissolve both polar and nonpolar compounds.

This study presents the results of a study of the process of oxidative desulphurization of gasolines using IL as to produce low-sulfur fuel components that comply with Euro-4 and Euro-5 standards. Nitrogen-containing compounds with high polarity, morpholinium formate (MF) and aniline formate (AF), were used as ILs to reduce the sulfur content in motor gasolines [7].

The use of the method of oxidative desulfurization followed by the extraction of sulfur-oxidized hydrocarbon fractions of IL makes it possible to partially reduce the total sulfur content in catalytic cracking and coking gasolines, which makes it possible to reduce the hydrogen pressure in the hydrotreating process and therefore minimizes the loss of olefins and, as a consequence, the octane number of gasoline.

Experimental part

The main components of motor gasolines are reforming gasoline (RG), CCG, CG, and straight-run gasoline (SRG). The largest amount of sulfur compounds is found in CG and CCG, which are presented in the form of organosulfur compounds – RSH mercaptans, RSR^1 sulfides, C_4H_6S thiophenes, dibenzothiophenes, etc. - which are distributed over all oil fractions of both distillate and thermodestructive origin.

In this work, catalytic cracking and coking gasolines produced at the Heydar Aliyev Refinery were studied. The content of total sulfur in them is 349 and 1791 ppm, respectively.

Due to the fact that the process of oxidative desulfurization of wide gasoline fractions requires a large amount of oxidizing agent (H_2O_2), it was customary to divide CCG and CG into narrow fractions: s.b.-120°C and 120°C-e.b. catalytic cracking gasoline and s.b.-85°C and 85°C-e.b. coking gasoline. The content of total sulfur in fractions s.b.-120°C and 120°C-e.b. of CCG was 283 and 636 ppm, respectively, and in the fractions of s.b.-85°C and 85°C-e.b. of CG – 1451 and 1894 ppm, respectively.

Coking gasoline produced at the refinery named after Heydar Aliyev, contains a large amount of unsaturated hydrocarbons (> 35 %), a high sulfur content (up to 1900 ppm) and a low octane number of 66-67 p. by M.M., which limits its use as a component of motor fuel in the composition of commercial gasoline [9-13]. Table 1 represents physical and chemical characteristics of coking gasoline and catalytic cracking gasoline obtained at the Refinery named after Heydar Aliyev.

Table 1. Physical and chemical characteristics of catalytic cracking and coking gasolines

Indices	CCG	CG
Density at 20 °C, кg/m^3	724	731
Fractional composition, °C		
s.b.	33	42
10 %	43	72
50 %	102	120
90 %	189	174
e.b.	200	180
Boiling-off, %	–	98
Content of total sulfur, ppm	349	1791
Content of mercaptane sulfur, ppm	30	72
Group hydrocarbon composition, % wt.:		
paraffin-naphthene hydrocarbons	59.0	44.9
Including iso-paraffine hydrocarbons	34.8	26.11
unsaturated hydrocarbons	12.7	34.0
aromatic hydrocarbons	28.3	21.1
Octane number by M.M., p.	81	67

As can be seen from Table 1, the content of sulfur and unsaturated hydrocarbons in CG is 1791 ppm and 34%, respectively.

The composition of light s.b.-85°C and heavy 85°C-e.b. fractions isolated from CG was studied for the purpose of further carrying out the process of oxidative desulfurization (Table 2).

Table 2. Qualitative characteristics of CG and its narrow fractions s.b.-85°C and 85°C-s.b.

Indices, % wt.	**CG**	**Fr. s.b.-85 °C CG**	**Fr. 85 °C-e.b.**
Aromatics	21.1	1.79	24.5
Olefins	34.0	43.36	32.35
Paraffin-naphthenes	44.9	3.13	49.14
Refraction index	1.4225	1.3831	1.4556
Iodine number, g I_2/100 g of gasoline	86.3	110.1	82.2
Total sulfur, ppm	1791	1462	1889
Octane number by M.M., p.	67	69	66

As follows from Table 2, sulfur content in CG and its fractions s.b.-85 °C and 85°C-e.b. is 1791, 1462, and 1889 ppm, respectively.

Table 3 shows the qualitative characteristics of CCG and its narrow fractions s.b.-120°C and 120°C-e.b.

Table 3. Qualitative characteristics of CCG and its narrow fractions s.b.-120°C and 120°C-e.b.

Indices	**CCG**	**Fr. s.b.-120 ºC**	**Fr. 120 °C-e.b.**
Density at 20 °C, кg/m^3	800	720	820
Fractional composition, °C			
s.b.	42	43	131
10 %	51	51	152
50 %	75	72	163
90 %	185	115	185
e.b.	200	120	200
Content			
unsaturated hydrocarbons, % wt.	16.9	23.3	5.6
total sulfur, ppm	349	280	641
Octane number by M.M., p.	81	80	83

It can be seen from Table 3 that the sulfur content in CCG and its fractions s.b.-120°C and 120°C-e.b is 349, 280, and 641 ppm, respectively.

Studies on the process of oxidative desulfurization of CCG, CG, and their narrow fractions with hydrogen peroxide were carried out by us both in stationary and in flow modes. A 35% hydrogen peroxide solution was used as an oxidizer, and a heterogeneous Mo-containing catalyst was used as a catalyst [5, 6, 13-16].

The catalyst was synthesized by applying solutions of molybdenum salts (nitrates, bromides) onto carriers of various natures, which were mainly γ-Al_2O_3.

The process of oxidative desulfurization of CCG in an amount of 50 ml was carried out in a stationary mode at low temperatures of 65-70°C with hydrogen peroxide in an amount of 10 ml on a Mo-containing catalyst with a load of 5 g. The experiment time was 2 hours. The sulfur content as a result of the process decreased from 349 to 309 ppm, and the degree of desulfurization was only 11.5%.

In order to increase the degree of desulfurization of the above gasolines, the process was carried out in a flow mode at temperatures of 50-200°C. We have studied the effect of temperature on the degree of desulfurization of gasoline fractions. The volume rate of the raw material was 2.0 h^{-1} [5, 6, 16-18].

Fig. 1 shows a diagram of a laboratory setup for the process of oxidative desulfurization of CCG in a flow system.

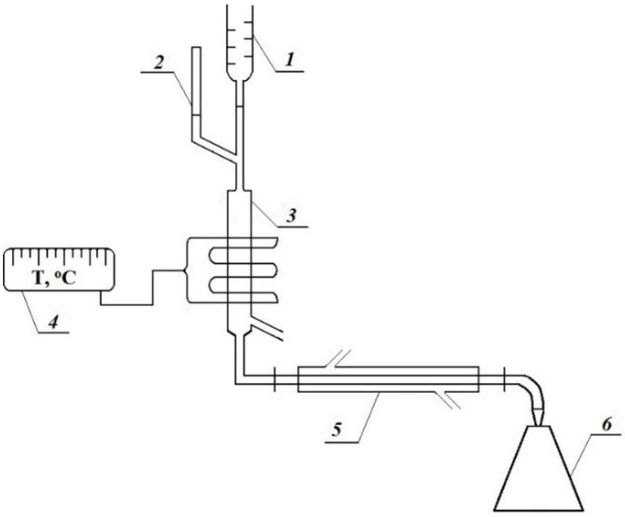

Fig. 1. Scheme of the laboratory installation for the process of oxidative desulfurization of CCG and CG in a flow system. 1 – container for CCG/CG supply; 2 – container for H_2O_2 supply; 3 – flow system reactor; 4 – potentiometer; 5 – refrigerator; 6 – receiver

The laboratory setup consists of a reactor (3) 250 mm high and 30 mm in diameter. The catalyst is placed on the grid. From above, CCG/CG and hydrogen peroxide in the corresponding molar ratio from tanks (1, 2) entered the reactor. The temperature in the reactor was controlled manually using a LATR connected to a potentiometer (4). The resulting product entered the refrigerator (5), then went to the receiver (6). Then the resulting product settles in a separating funnel, where water is separated from gasoline.

The remaining traces of water in the resulting gasoline were absorbed with calcium chloride.

Results and its discussion

Below in Tables 4-5 show the results of the process of oxidative desulfurization in a flow system of a wide fraction of CCG (S – 349 ppm) and its narrow fractions (s.b-120°C (S – 280 ppm), 120°C-e.b. (S – 641 ppm)) on a Mo-containing catalyst.

Table 4. Oxidative desulfurization of CCG (broad fr.) in a flow system

Indices, % wt.	**Temperature, °C**			
	50	**100**	**150**	**200**
Initial sulfur, ppm	349	349	349	349
Sulfur after process, ppm	254	236	299	303
Desulfurization degree, %	27.2	32.4	14.3	13.2
Losses, %	6.5	6.8	7.0	7.2

From Table 4 shows that the optimal temperature is 100°C, at which the degree of CCG desulfurization is 32.4%.

Table 5. oxidative desulfurization of fr. s.b.-120 °c and 120°c-e.b. ccg in a flow system

Indices	**Temperature, °C**							
	Fr. s.b.-120 °C CCG (light)				**Fr. CCG 120 °C-e.b. (heavy)**			
	50	**100**	**150**	**200**	**50**	**100**	**150**	**200**
Initial sulfur, ppm	280	280	280	280	641	641	641	641
Sulfur after process, ppm	243	235	219	233	405	325	315	330
Desulfurization degree, %	13.2	16.1	21.8	16.8	36.8	49.3	50.9	48.5

Losses, %	3.6	3.7	4.0	4.1	3.5	3.8	4.0	4.2

As can be seen from Table 5, with increasing temperature, the degree of desulfurization for fr. s.b.-120°C increases. The maximum degree of desulfurization fr. s.b.-120°C CCG is observed at 150°C and is 21.8%. Table 5 also shows that with increasing temperature, the degree of desulfurization for fr. 120°C-e.b. also increases. Optimal is T = 150°C, at which the degree of desulfurization fr. 120°C-e.b. CCG is 50.9%.

Thus, the use of the oxidative desulfurization method makes it possible to partially reduce the total sulfur content in the CCG, which makes it possible to reduce the hydrogen pressure during subsequent hydrotreatment and minimizes the loss of olefins and, as a result, stabilizes the octane numbers of gasoline [6].

Further studies were carried out on the process of oxidative desulfurization of CG and its narrow fractions. Since coking gasoline contains the largest amount of sulfur (up to 1791 ppm), the process of oxidative desulfurization of CG is of considerable interest in comparison with the hydrotreatment process, when the saturation of olefins significantly reduces the ON of gasoline.

Tables 6-7 show the results of the process of oxidative desulfurization in a flow system of a wide fraction of CG (S – 1791 ppm) and its narrow fractions (s.b.-85°C (S – 1462 ppm), 85°C-e.b. (S – 1889 ppm)) on a Mo-containing catalyst.

Table 6. Oxidative desulfurization of cg (broad fr.) in a flow system

Indices, % wt.	**Temperature, °C**			
	50	**100**	**150**	**200**
Initial sulfur, ppm	1791	1791	1791	1791
Sulfur after process, ppm	1290	1187	1420	1470
Desulfurization degree, %	28.0	33.7	20.7	17.9
Losses, %	5.5	5.8	6.0	6.2

From Table 6 shows that the optimal temperature is 100°C, at which the degree of CG desulfurization is 33.7%.

Table 7. Oxidative desulfurization fr. s.b.-85°C and 85°C-e.b. CG in a flow system

Indices	**Temperature, °C**							
	Fr. s.b.-85 °C CG (light)				**Fr. 85 °C-e.b. CG (heavy)**			
	50	**100**	**150**	**200**	**50**	**100**	**150**	**200**
Initial sulfur, ppm	1462	1462	1462	1462	1889	1889	1889	1889
Sulfur after process, ppm	1230.4	1095.5	1197.1	1166.6	1187.5	920.5	950.8	971.6
Desulfurization degree, %	15.8	25.1	18.1	20.2	37.1	51.3	49.7	48.6
Losses, %	3.0	3.4	3.1	3.5	3.4	3.9	3.7	4.1

As can be seen from Table 7, with increasing temperature, the degree of desulfurization increases. The maximum degree of desulfurization is observed at 100°C and is for fr. s.b.-85°C CG 25.1%, and for fr. 85°C-e.b. CG – 51.3%.

The CCG and CG products oxidized with hydrogen peroxide were further involved in the extraction process with an ionic liquid (morpholinium formate obtained on the basis of morpholine and formic acid) in a ratio of 1:1 at room temperature.

The main indicators of IL morpholinium formate:

Density at 20 °C, кg/m^3	1.07-1.14
Viscosity at 30 °C, Pa·c	6.0-7.0
pH of medium	7-9
Pour point, °C	-5
Electrical conductivity, Cm	$1.1\cdot10^{-3}$-$1.1\cdot10^{-3}$

As is known, the extraction process is based on the better solubility of sulfur-containing compounds compared to non-aromatic compounds.

In Tables 8-9 show the results of extraction of the obtained CCG oxidates from the process of oxidative desulfurization of wide and narrow fractions at temperatures from 50 to 200°C in a flow system. Extraction was carried out using an ionic liquid - morpholinium formate - in a ratio of 1:1 at room temperature for 1 hour [6-8, 19, 20].

Table 8. Extraction of the resulting CCG oxidates (from the broad fraction) by IL

Indices, % wt.	**Temperature, °C**			
	50	**100**	**150**	**200**
Initial sulfur, ppm	349	349	349	349
Sulfur after oxidation and extraction by IL, ppm	104.7	91.78	118.66	121.45
Desulfurization degree, %	70.0	73.7	66.0	65.2

The optimum temperature is 100°C, at which the total sulfur content after IL extraction decreases from 349 to 91.78 ppm and the highest degree of desulfurization of oxidized CCG IL reaches 73.7%. At higher temperatures, the rates of side reactions increase, and the oxidizer is consumed in the process of olefin oxidation to epoxides and alcohols. In this regard, the degree of oxidation of sulfur compounds is significantly reduced.

Table 9. Extraction of the resulting CCG oxidates (from narrow fractions) by IL

Indices	**Temperature, °C**							
	Fr. s.b.-120 °C CCG (light)				**Fr. 120 °C-e.b. CCG (heavy)**			
	50	**100**	**150**	**200**	**50**	**100**	**150**	**200**
Initial sulfur, ppm	280	280	280	280	641	641	641	641
Sulfur after oxidation and extraction by IL, ppm	113.2	101.6	92.8	109.5	235.3	208.6	156.5	195.8
Desulfurization degree, %	59.6	63.7	66.9	60.9	63.3	67.5	75.6	69.5

From Table 9 it can be seen that the maximum degree of oxidation of light s.b. is -120°C and heavy 120°C-e.b fractions of CCG are 66.9% and 75.6% vol. at T = 150°C.

The removal of total sulfur was carried out by the extraction method by IL, which was used as morpholinium formate.

The results of extraction obtained from the process of oxidative desulfurization of oxidates of wide and narrow fractions of CG, obtained in a flow system in the temperature range of 50-200°C, are presented in Tables 10-11. Extraction was also carried out using morpholinium formate IL (ratio 1:1) for 1 hour at room temperature [6, 8-13, 19-21].

Table 10. Extraction of obtained CG oxidates (from the broad fraction) by IL

Indices	**Temperature, °C**			
	50	**100**	**150**	**200**
Initial sulfur, ppm	1791	1791	1791	1791
Sulfur after extraction, ppm	439	419	448	508
Desulfurization degree, %	75.5	76.6	75.0	71.6
Losses, %	3.8	4.0	4.2	4.5

From Table 10 shows that with an increase in temperature from 50 to 200°C, the degree of desulfurization of the wide CG fraction changes from 71.6 to 76.6%. The optimal temperature is 100°C, at which the degree of desulfurization is 76.6%.

Table 11. Extraction of obtained CG oxidates (from narrow fractions) by IL

Indices	Temperature, °C							
	Fr. s.b.-85 °C CG (light)				Fr. 85 °C-e.b. CG (heavy)			
	50	100	150	200	50	100	150	200
Initial sulfur, ppm	1462	1462	1462	1462	1889	1889	1889	1889
Sulfur after extraction, ppm	531	506	544	573	331	310	337	376
Desulfurization degree, %	63.7	65.4	62.8	60.8	82.5	83.6	82.2	80.1
Losses, %	3.3	3.8	4.0	4.3	3.2	3.9	4.1	4.5

As can be seen from Table 11, with the weighting of the fractional composition of coking gasoline, the degree of desulfurization increases (up to 83.6 %).

From Table 11 it can be seen that with an increase in temperature from 50 to 200°C during the treatment of IL, the degree of desulfurization of the light CG fraction (fr. s.b.-85°C) reaches 60.8-65.4%, while the sulfur content decreases by 2.5-3 times. The optimal temperature is 100 °C, at which the degree of desulfurization is 65.4%. The degree of desulfurization of the heavy fraction of CG (fr. 85°C-e.b.) with an increase in temperature from 50 to 200°C during the treatment of IL reaches 80.1-83.6%. The sulfur content is reduced by 5-6 times. The optimum temperature is 100°C.

Conclusion

The process of oxidative desulfurization of the components of commercial gasoline CCG and CG in the flow mode was investigated, and the optimal conditions for the process were identified. At T = 50-200 °C and V = 2 h^{-1}, the maximum degree of desulfurization of the wide fraction of CCG followed by extraction with ionic liquids was 73.7% at T = 100°C. The degree of desulfurization of a wide CG fraction followed by extraction with ionic liquids was 76.6% at T = 100°C. The degree of desulfurization of narrow CG fractions with subsequent extraction of sulfur-containing compounds with ionic liquids reached 83.6%.

In the course of the studies on the process of oxidative desulfurization of CCG and CG gasolines, followed by extraction with ionic liquids, the total sulfur content does not exceed 55-60 ppm in the composition of commercial gasoline. On the basis of the work done, it can be concluded that in order to reduce the sulfur content to the required Euro-4 and Euro-5 standards, it is possible to carry out the process of hydrotreatment of catalytic cracking and coking gasolines at low hydrogen pressures with little or no reduction in octane number.

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