

CATALYTIC REFORMING OF HYDROGENATED SHALE OIL NAPHTHA

By

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INTRODUCTION

Catalytic reforming is a process for converting low-octane naphthas or gasolines into high-octane products. Most catalytic reforming processes require very clean feedstocks to avoid deactivating the catalysts. Raw shale-oil naphthas produced during retorting or by subsequent thermal cracking of the crude shale oil have poor color and oxidation stability; they turn dark in color and form large amounts of gum soon after they are prepared. Their instability and their high contents of sulfur and nitrogen compounds make them unsuitable as feedstocks to modern noble-metal catalytic reforming processes.

To overcome the problems associated with upgrading shale-oil naphthas, production of stable naphthas by catalytic hydrogenation of crude shale oil or by coking of crude shale oil, followed by hydrogenation of the coker distillate, has been investigated (1,6). A few results on catalytic reforming of naphtha from hydrogenated coker distillate have been published (6), but little information has been published on catalytic reforming of the naphtha obtained by hydrogenating crude shale oil. The present study was undertaken to provide information on reforming the naphtha from hydrogenated crude over a broad range of reforming temperatures and pressures.

Cyclohexane was reformed at the same conditions as those used for the hydrogenated shale-oil naphtha so a comparison could be made between the results obtained with the hydrogenated naphtha and those obtained with this good reforming feedstock.

LABORATORY PROCEDURES

Naphtha Feedstock Preparation

Shale oil was prepared by the gas-combustion process from Green River oil shale at the Bureau of Mines facility near Rifle, Colo. (10). The oil was filtered to remove spent shale dust and was separated from water by decantation and distillation. It was then hydrogenated in a once-through operation with a cobalt-molybdenum-carrier catalyst at a temperature of 790°F, a pressure of 1,000 psig, a liquid hourly space velocity (LHSV) of 0.1 volume of oil per volume of catalyst per hour, and a hydrogen feed rate of 6,000 scf/bbl. The hydrogenation removed over 90 percent of the nitrogen and sulfur from the oil. The hydrogenated oil was fractionated with a true boiling point apparatus, and the fraction that distilled between 160° and 400°F was further hydrogenated with a nickel tungsten catalyst at a temperature of 700°F, pressure of 1,500 psig, LHSV of 1.0, and hydrogen feed rate of 5,000 scf/bbl to reduce its nitrogen content to 7 ppm and its sulfur content to less than 0.001 weight percent. The light ends of the liquid product from the second hydrogenation step were distilled

off with a cut point of 160°F, and the higher boiling portion of this product (amounting to 32 volume-percent of the crude oil) was used as the hydrogenated naphtha feedstock.

The two-stage hydrogenation procedure was used because earlier experiments had shown that naphtha with a nitrogen content of less than 50 ppm was not produced in a single stage with either catalyst. The cobalt molybdate catalyst produced lower catalyst deposits than the nickel tungsten catalyst when hydrogenating crude shale oil, but the nickel tungsten catalyst appeared to effect better nitrogen removal in the second stage where a relatively clean naphtha feed was used. Thus, the two-stage hydrogenation with two catalysts appeared more desirable than a two-stage hydrogenation using the same catalyst in both stages.

Variables of Reforming

The experiments described here were made for the purpose of evaluating hydrogenated shale-oil naphtha as a feedstock for established reforming processes. However, finding the best operating conditions for a process is an important part of research; failure to properly control the operating variables may result in an uneconomical process. In the instance of reforming, variables such as those shown in table 1 determine the yield and quality of the products (7).

An exhaustive investigation of the reforming variables was not possible with the small laboratory-size quantities of feedstock that were available, so a selection of the variables to be investigated was necessary. Changes in temperature and pressure have more pronounced effects on the yield and quality of products than changes in the other variables; therefore, to approximate the best set of operating conditions with a commercial reforming catalyst, preselected ranges of temperature and pressure within the usual commercial reforming ranges were chosen for investigation, with the other variables held constant. Of the two variables, temperature and pressure, a proper selection of operating temperature is probably more important. As the temperature is increased, the equilibrium is shifted toward an increase in the production of aromatics; also, hydrocracking is increased. Operation under hydrogen pressure is necessary because at low pressure catalytic activity is quickly lost owing to coke formation. As the partial pressure of hydrogen is lowered, hydrocracking rates and gasoline yields decrease; aromatization rates, octane number, and hydrogen production increase.

Apparatus and Operating Procedure

Figures 1 and 2 show the flow diagram and the equipment of the reforming unit. The reactor was the fixed-bed type, equipped for downward flow, with an internal diameter of 5/16 inch, an outside diameter of 9/16 inch, and an internal length of 38.5 inches. The reactor was charged with 8.6 ml of catalyst in the form of 1/16-inch extrusions. The catalyst occupied 6.9 inches of the reactor length, and the bottom of the catalyst bed was approximately 8.5 inches from the bottom of the reactor. The upper part of the reactor was used to preheat the feed and hydrogen. The reactor was inserted in a 36- by 3-inch aluminum cylinder that was heated by a four-element furnace controlled by a four-point electronic controller.

Temperatures were measured by eight thermocouples in two vertical thermowells in the aluminum cylinder next to the reactor. The thermocouples were spaced at 3.5-inch intervals along the length of the reactor with the bottom thermocouple 5 inches above the bottom of the reactor and the top thermocouple 9 inches from the top of the reactor. Only the temperatures measured by the three thermocouples in the catalyst region were used for the reaction temperatures.

For convenience, hydrogen (99.9+ pct purity) from standard shipping cylinders was substituted for the highly concentrated hydrogen recycle gas that is normally used, to keep coke laydown on the catalyst to a minimum. At the beginning of each run, before

liquid feed was started, the system was pressurized with helium, and helium was fed through the system at a slow rate for several hours while the furnace was heating the reactor to reaction temperature. After the reactor reached the desired reaction temperature, the helium was replaced by hydrogen and the feed pump was started. The feed was pumped by a positive displacement pump at the rate of 25 cc/hr, and the hydrogen was metered through a rotameter at the rate of 3,000 scf/bbl. After passing through the reactor, the oil and gas were cooled through a double-pipe heat exchanger and then passed into a low-pressure receiver. Outlet gases were reduced to atmospheric pressure, metered through a positive-displacement dry meter, and stored in a small stainless steel tank from which samples were taken for analysis. The liquid product was separated into stable and heavy reformates at a cut point of 400°F at 760 mm pressure using a Vigreux column and a condenser. There was a trace of heavy reformat in all samples.

A total of 27 tests was run. The first nine tests were run while some minor revisions to the new equipment were being made, and data from these runs are not reported. Nine tests were made with cyclohexane and nine with shale-oil naphtha. All tests were carried out at a nominal LHSV of 3 volumes of oil per volume of catalyst per hour and a hydrogen feed rate of 3,000 scf/bbl. The experiments were run for onstream test periods of 17 to 24 hours, with total throughputs of 50 to 70 volumes of feed per volume of catalyst. Operating pressures of 200, 400, and 600 psig were used at nominal temperatures of 850°, 875°, and 900°F. The furnace surrounding the reactor permitted the tests to be made essentially isothermally. Specific details for the individual runs are given in tables 2 and 3.

Catalyst

The catalyst was commercial platinum-on-silica-alumina in the form of 1/16-inch-diameter extrusions. It had been treated with a halide during manufacture to promote acid-catalyzed reforming reactions. Fresh catalyst was used for each reforming run.

RESULTS AND DISCUSSION

Catalytic reforming improved the quality of motor fuel rather than its yield. Reforming differs from cracking processes because the charge stocks are already in or near the boiling range of gasoline (6). The main reactions of catalytic reforming are shown in figure 3 (9).

Several samples of the stable reformat were submitted for mass spectral group-type analysis. The method, which is limited to samples of low olefin content (3 percent or less), determines six classes of hydrocarbons: Paraffins, monocycloparaffins, dicycloparaffins, alkylbenzenes, indanes and tetralins, and naphthalenes. The analytical results are presented in table 3 and shown graphically in figure 4.

From the mass spectral group-type analysis, it is apparent that naphthene dehydrogenation is the dominant reaction. This occurs more easily with an increase in temperature and a decrease in pressure, and it increases the octane number by a very noticeable extent.

The paraffin carbon numbers and the alkylbenzene carbon numbers were also determined from the spectral group-type analysis. These numbers are the average number of carbon atoms per molecule in the straight chain paraffins and the alkylbenzenes. The spectral analyses in table 3 show that the average paraffin carbon number decreased in all but one run and the mole percent of paraffins increased in all runs. This indicates that there was some hydrocracking of the large straight chain paraffins into smaller straight chain paraffins. Hydrocracking makes shorter paraffins which have a higher octane, but severe hydrocracking is undesirable because it gives poor yields caused by the light gases that it makes.

A composite sample of the spent catalyst from all of the reforming runs with hydrogenated naphtha was analyzed and found to contain 1.12 weight-percent hydrogen, 4.76 weight percent carbon, and 87.23 weight percent ash. Most of the carbon probably came from cracking of the feedstock.

Figures 5-A and 5-B show that as temperature was increased the API gravity decreased, but the increase of cracking, ignoring other changes in composition, should have made the API gravity increase. The decrease was probably caused by the increase of aromatics as shown in figures 5-C and 5-D.

Figure 6 shows that as temperature increases the product yield decreases quite rapidly and the octane increases quite rapidly. Aromatics have very high octane numbers, and a comparison of figures 5-C and 5-D with figures 6-C and 6-D, and an examination of the mass spectral analyses, show that the increase in octane was caused mostly by an increase in aromatics.

The optimum operating condition for each pressure occurs at the point where the yield-octane number, defined as the product of the volume-percent yield and the octane number divided by 100, is at a maximum. Yield-octane numbers were calculated for the reforming experiments and are given in tables 2 and 3 and figure 7.

The best quality reformat, with an 89 clear research octane number and a yield of 79.9 volume percent, had the highest yield-octane number (71.1) and was obtained at the highest temperature (900°F) and lowest pressure (200 psig). This suggests that the optimum operating condition would be at a higher temperature than those investigated. At the higher temperatures, octane numbers would be expected to increase further but would eventually level off while gas production would continue to increase, causing the curve for yield-octane number versus temperature to go through a maximum and curve downward. The limited amount of feedstock available did not permit the determination of the temperatures at which these effects would occur.

To determine where the curves should be drawn in figures 5, 6, and 7, all nine sets of data in each part of each figure were used to derive one equation of the form (4):

$$Y = A + bt + cP + d \frac{P}{t}$$

where Y = dependent variable
 t = temperature, °F
 P = pressure, psig
 a, b, c, d = constants

Thus, the data obtained at all three pressures influenced the position of each curve at any one pressure. For this reason the curve at any individual pressure does not necessarily go through the data points for that particular pressure. Although temperature and pressure had considerable effect on the dependent variables, the interaction terms ($d \frac{P}{t}$) in the equations had little effect.

SUMMARY AND CONCLUSIONS

Good quality reformates were produced from the naphtha prepared by hydrogenation of crude shale oil. The best quality reformat was produced at 900°F and 200 psig, which were the highest temperature and lowest pressure tested. This reformat had a clear research octane number of 89 and a yield of 80 volume percent. Results indicated that a higher operating temperature might give a better yield octane relationship.

ACKNOWLEDGMENTS

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TABLE 1. - Reforming variables

Catalyst	Time
Pressure	Naphtha
Temperature	Impurities (S,N,O, metals)
Space velocity	Regeneration
H ₂ /naphtha ratio	Startup procedure

TABLE 2. - Operating conditions, product yields, and properties of liquid products for catalytic reforming of cyclohexane

Run number	Feed	10	11	12	16	17	18	13	14	15
Temperature, ° F:										
Average	-	849	875	899	843	874	901	854	879	907
Maximum	-	862	891	910	862	888	915	864	889	922
Pressure, psig	-	200	200	200	400	400	400	600	600	600
LESV, V ₁ /V ₂ /hr./	-	2.94	3.07	2.92	2.92	2.93	2.94	2.94	2.94	2.93
Throughput, V ₁ /V ₂	-	69.47	65.70	61.76	59.48	57.72	65.36	64.33	65.53	63.49
Moles hydrogen feed/mole liquid feed	-	2.43	2.43	2.43	2.43	2.43	2.43	2.43	2.43	2.43
Hydrogen feed, scf/bbl	-	3000	3000	3000	3000	3000	3000	3000	3000	3000
Hydrogen produced, scf/bbl	-	2835	3028	3089	2083	2399	2464	1132	1375	1702
Liquid yield, vol pct of feed	-	5.82	5.88	5.99	4.05	4.46	4.78	2.20	3.06	3.30
Liquid yield, wt pct of feed	-	76.86	76.66	75.98	80.99	80.30	79.30	86.30	85.95	85.35
Research octane number, clear	-	84.78	84.99	84.24	85.13	84.61	84.17	85.74	86.61	85.67
Research octane number, with 3 cc TEL/gal	-	85	103	105	109	96	98	89	90	88
Yield-octane, vol fraction x R.O.N., clear	-	79.2	80.5	82.8	77.8	77.1	77.7	76.8	77.4	75.1
Properties of stable reformate:										
Specific gravity, 60°/60° F	0.782	0.863	0.867	0.867	0.822	0.824	0.830	0.778	0.785	0.788
Gravity, ° API	49.4	32.5	31.7	31.7	40.6	40.2	39.0	50.4	48.8	48.1
Average molecular weight	84	75	76	77	76	75	74	78	78	73
Characterization factor, K	11.0	10.0	9.9	9.9	10.4	10.4	10.3	11.0	10.8	10.9
Heat of vaporization, Btu/lb	155	170	169	168	165	166	167	158	160	163
ASTM distillation, ° F:										
IBP	176	169	169	168	161	160	158	159	156	146
10 pct recovered	177	173	172	172	165	165	165	161	161	157
50 pct recovered	177	174	174	175	167	167	168	163	163	161
90 pct recovered	177	177	177	177	171	173	174	166	168	169
End point	178	187	201	193	161	162	179	179	199	179
Rec., vol pct	99.0	99.0	99.0	99.0	98.0	98.0	99.0	99.0	99.0	98.0
Res., vol pct	nil	nil	nil	nil	nil	nil	nil	nil	nil	nil
Loss, vol pct	1.0	1.0	1.0	1.0	2.0	2.0	1.0	1.0	1.0	2.0
Carbon, wt pct	85.72	-	-	92.04	-	-	-	87.14	85.43	87.62
Hydrogen, wt pct	14.28	-	-	8.00	-	-	-	12.55	12.66	11.84

1/ Volume of feed/volume of catalyst/hour.

TABLE 3. - Operating conditions, product yields, and properties of liquid products for catalytic reforming of hydrogenated naphtha

Run number	Feed	19	20	21	25	26	27	22	23	24
Temperature, ° F:										
Average		847	871	903	849	875	900	844	876	904
Maximum		860	866	917	860	887	912	856	884	915
Pressure, psig		200	200	298	400	400	400	600	600	600
LESL, Vi/Vc/hr		2.99	3.00	2.98	2.99	2.96	2.96	2.99	2.98	2.99
Throughput, Vi/Vc		68.32	70.92	70.36	52.74	51.33	55.86	60.39	59.99	55.14
Moles hydrogen feed/mole liquid feed		3.50	3.50	3.50	3.50	3.50	3.50	3.50	3.50	3.50
Hydrogen feed, scf/bbl		3000	3000	3000	3000	3000	3000	3000	3000	3000
Hydrogen produced, scf/bbl		1184	1459	1756	1219	1179	1497	818	1218	1393
Hydrogen produced, wt pct of feed		2.24	2.91	3.50	2.43	2.35	2.98	1.63	2.43	2.77
Liquid yield, vol pct of feed		87.80	84.32	79.90	88.38	85.40	81.95	90.60	85.91	82.20
Liquid yield, wt pct of feed		91.02	87.89	84.41	91.28	88.31	85.18	92.38	88.59	85.00
Research octane number, clear	40	69	78	89	70	79	85	59	70	82
Research octane number, with 3 cc TEL/gal	64	88	94	99	89	92	80	88	88	95
Yield-octane, vol fraction x R.O.N., clear		60.6	65.8	71.1	61.9	67.5	69.7	53.4	60.1	67.4
Properties of stable reformate:										
Specific gravity, 60°/60° F	0.762	0.790	0.794	0.805	0.787	0.788	0.792	0.777	0.784	0.788
Gravity, API	54.2	47.6	46.7	44.3	48.3	48.1	47.2	50.6	49.0	48.1
Average molecular weight	118	108	110	108	119	116	110	119	117	111
Characterization factor, K	11.9	11.4	11.3	11.2	11.5	11.4	11.4	11.6	11.5	11.4
Heat of vaporization, Btu/lb	130	140	138	140	133	133	137	131	133	136
ASTM, distillation, % F:										
IEP	225	161	153	148	179	144	137	177	180	142
10 pct recovered	256	218	230	220	243	226	220	242	238	214
50 pct recovered	297	290	288	285	294	287	283	293	291	277
90 pct recovered	350	352	350	340	350	344	344	350	350	342
End point	371	390	406	411	391	394	395	383	389	397
Rec., vol pct	99.0	97.0	98.0	98.0	99.0	98.0	98.0	98.0	98.0	99.0
Loss, vol pct	nll	nll	nll	nll	nll	nll	nll	nll	nll	nll
Carbon, wt pct	85.90	87.30	87.29	86.16	86.90	87.28	87.60	86.10	86.92	87.24
Hydrogen, wt pct	14.02	12.69	12.44	13.30	12.92	12.68	12.24	13.32	12.92	12.67
Nitrogen, ppm	7.14	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5
Sulfur, wt pct	<0.001									
Alkybenzene carbon number	8.40	8.44	8.01	8.24	7.72	7.98	7.95	8.17	8.14	
Paraffin carbon number	8.87	8.99	8.81	8.68	8.59	8.30	8.10	8.59	8.48	
Hydrocarbons, mole pct										
Paraffins	42.1	53.9	50.9	47.9	59.5	55.2	49.9	61.8	62.7	
Monocycloparaffins	38.4	0.3	2.1	0	1.3	1.7	0.5	9.0	5.7	
Dicycloparaffins	7.0	0.7	0.1	0.2	0.8	0.3	0.3	1.2	0.7	
Alkybenzenes	9.1	41.4	42.3	48.8	37.2	41.7	48.7	28.6	28.5	
Indanes-tetralins	1.7	1.9	3.0	2.0	0.4	0.6	0.2	0.7	1.7	
Naphthalenes	1.7	1.8	1.6	1.1	0.8	0.5	0.4	0.7	0.7	

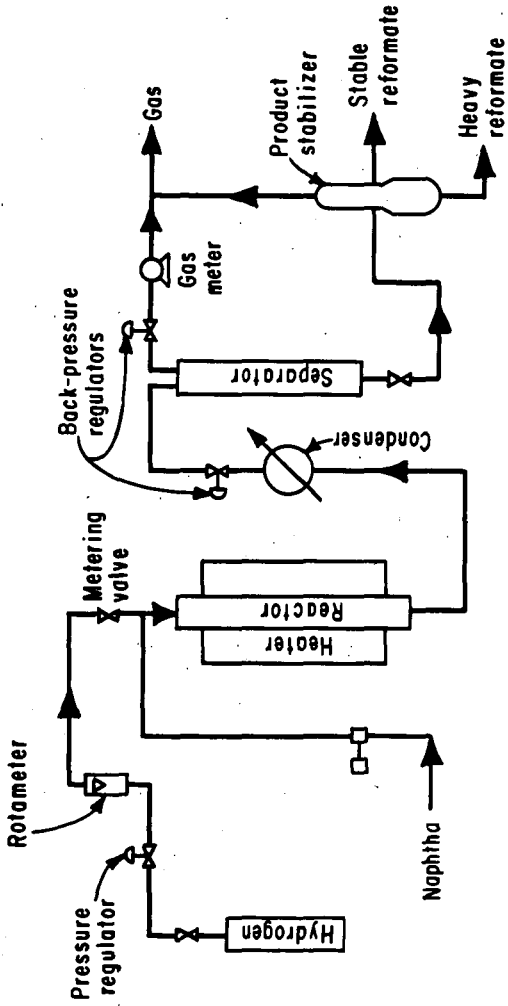


FIGURE 1.-Simplified Flow Diagram of Catalytic Reforming Unit.

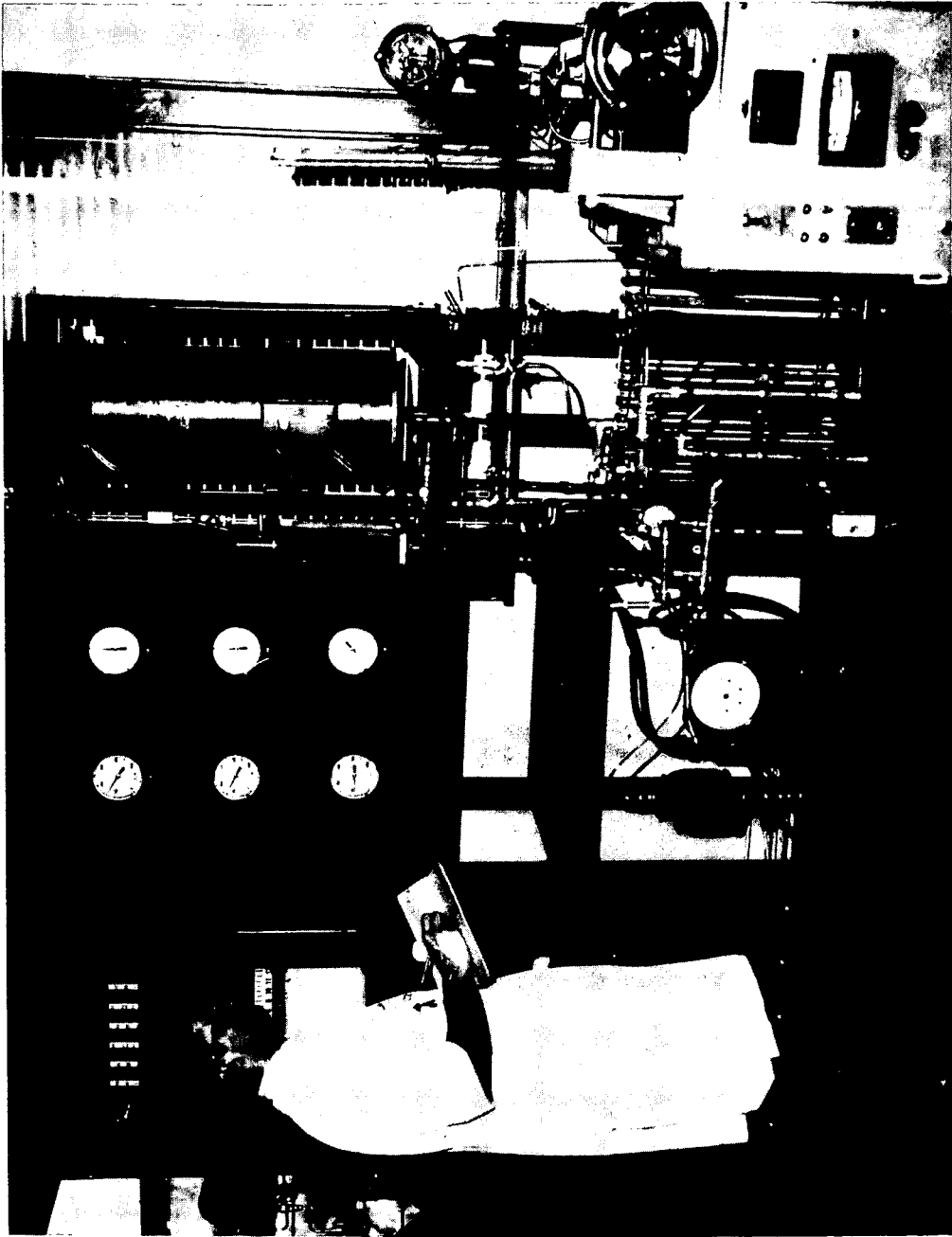
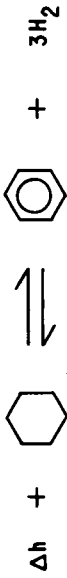
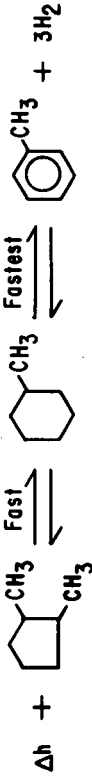


Figure 2.

1. Naphthene dehydrogenation; an endothermic reaction



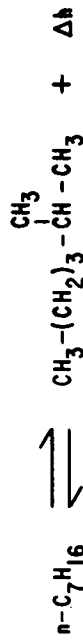
2. Naphthene dehydroisomerization; an endothermic reaction



3. Paraffin dehydrocyclization; an endothermic reaction



4. Paraffin isomerization; a mildly exothermic reaction



5. Hydrocracking; a markedly exothermic reaction

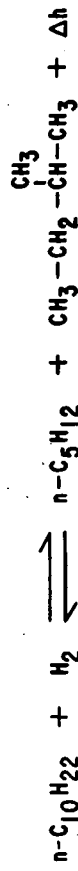


FIGURE 3.-Catalytic Reforming Reactions.

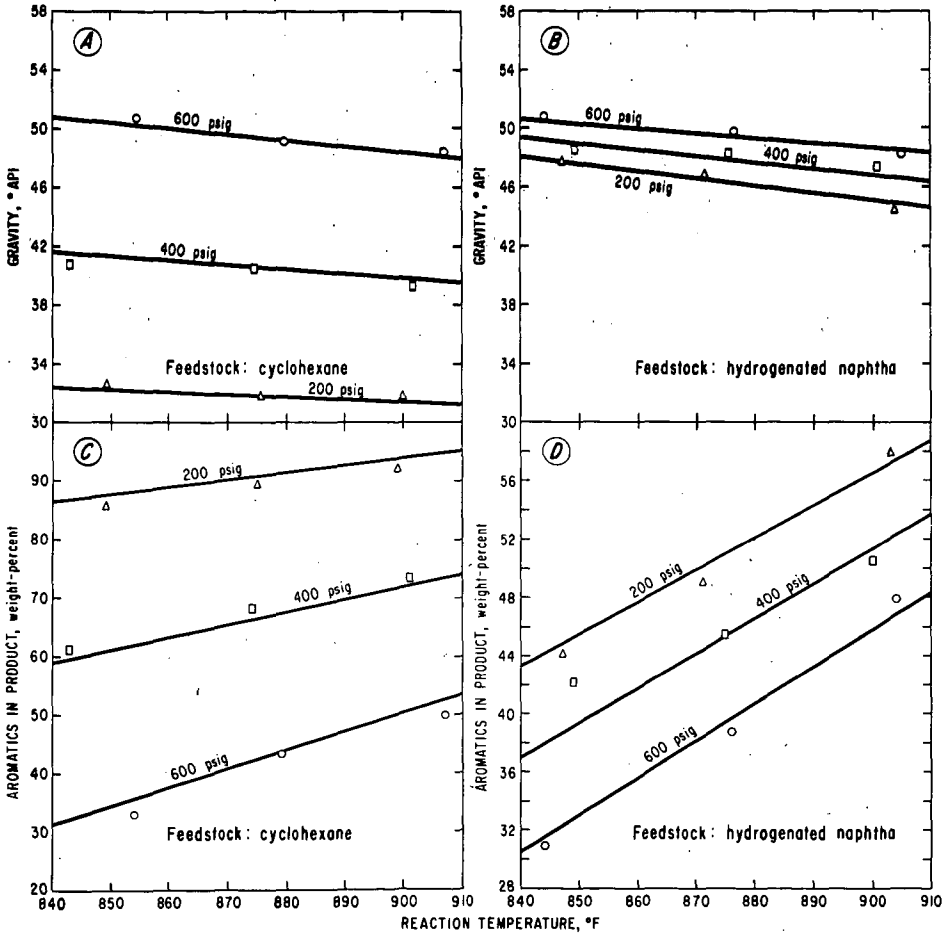


FIGURE 5.—Effect of Temperature and Pressure on Results from Catalytic Reforming.

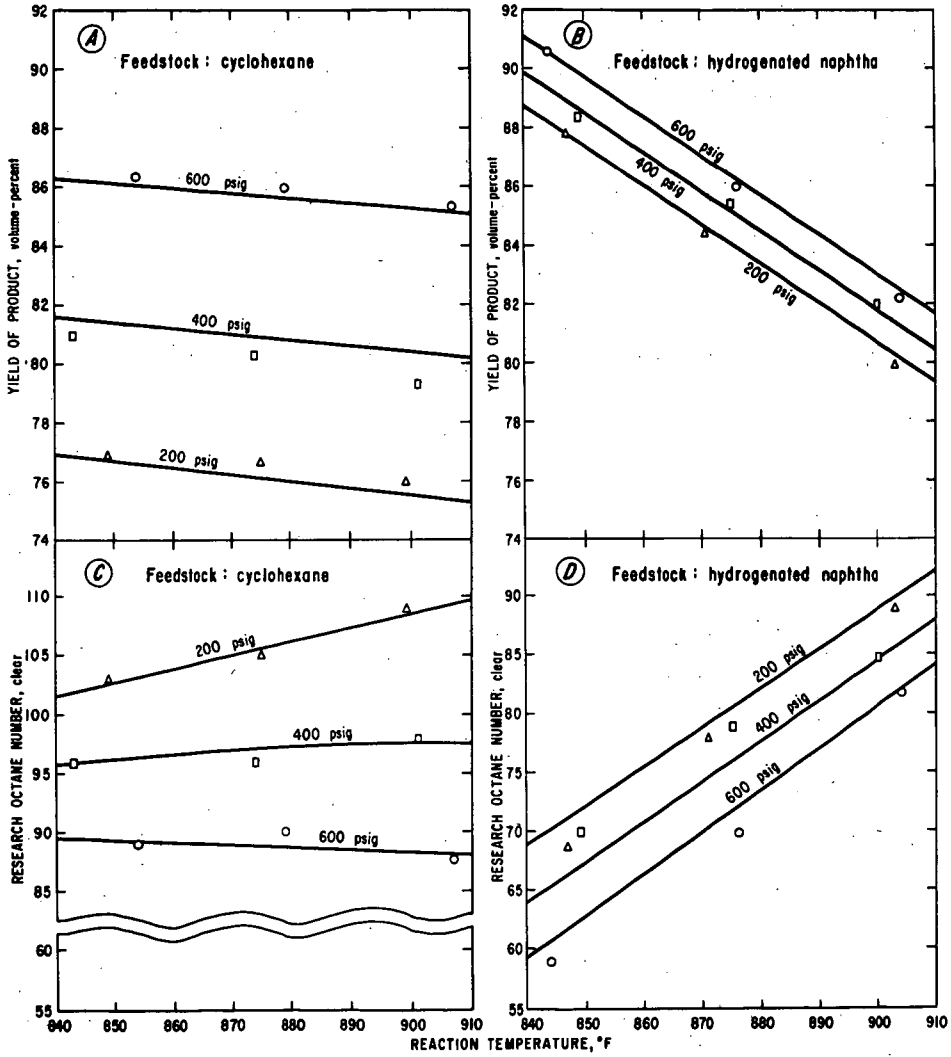


FIGURE 6-Effect of Temperature and Pressure on Results from Catalytic Reforming.

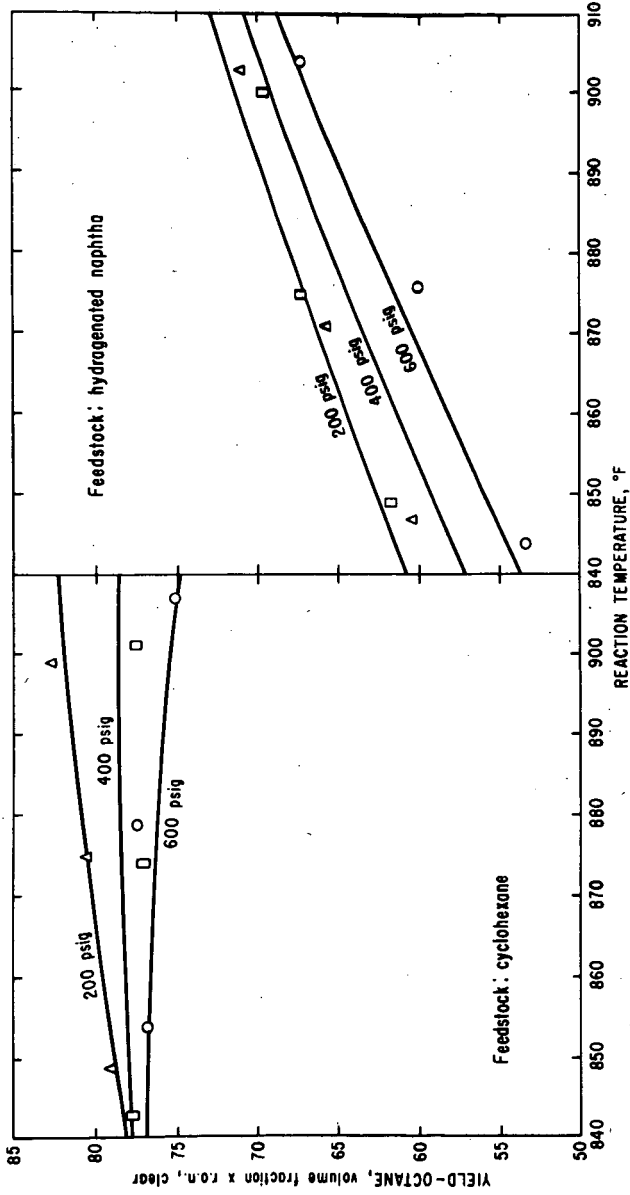


FIGURE 7.—Effect of Temperature and Pressure on Yield-Octane from Catalytic Reforming.

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Vol. 14, No. 2	Symposium on Coal Combustion in Present and Future Power Cycles	Toronto, Canada May, 1970
Vol. 14, No. 3	Synthetic Fuels Symposium No. 3 - Economics of Solid Fuel Conversion Processes General Papers	Chicago, Illinois September, 1970
Vol. 14, No. 4 Parts I and II	Symposium on Hydrogen Processing of Solid and Liquid Fuels	Chicago, Illinois September, 1970
Vol. 14, No. 5	Symposium on High Temperature and Rapid Heating Reactions of Fuels	Chicago, Illinois September, 1970
Vol. 15, No. 1	Symposium on Shale Oil, Tar Sands and Related Materials	Los Angeles March, 1971
Vol. 15, No. 2	Symposium on Combustion Symposium on Pollution Control in Fuel Combustion, Mining and Processing	Washington, D. C. September, 1971
Vol. 15, No. 3	Symposium on Gasification of Coal General Papers	Washington, D. C. September, 1971
Vol. 16, No. 1	Symposium on Quality of Synthetic Fuels, Especially Gasoline and Diesel Fractions, and Pipeline Gas	Boston, Mass. April, 1972
Vol. 16, No. 2	Symposium on Preparation and Properties of Catalysts for Synthetic Fuel Production General Papers	Boston, Mass. April, 1972
Vol. 16, No. 3	Symposium on Modern Methods of Fuel Analysis	Boston, Mass. April, 1972
Vol. 16, No. 4	Symposium on Non-Fossil Chemical Fuels	Boston, Mass. April, 1972

DIVISION OF FUEL CHEMISTRY

PROJECTED PROGRAMS

Environmental Pollution Control - Part I. Removal of Oxides of Sulfur and Nitrogen from Combustion Product Gases

Robert M. Jameson

New York, N. Y.
August, 1972

Environmental Pollution Control - Part II. Removal of Sulfur from the Fuel

Robert M. Jameson

New York, N. Y.
August, 1972

Storch Symposium

New York, N. Y.
August, 1972

General Papers

Wendell H. Wiser

New York, N. Y.
August, 1972

Symposium on the Power Industry of the Future - Fossil and Fission Fuels

Joint with IEC Division - Develop by IEC

New York, N. Y.
August, 1972

Novel Combined Power Cycles

S. Fred Robson

Dallas, Texas
April, 1973

Fuel from Waste Products

H. R. Appell

Dallas, Texas
April, 1973

Carbon Monoxide Production and New Uses

J. S. Mackay

Dallas, Texas
April, 1973

Synthetic Fuel Gas Purification

H. S. Vierk

Dallas, Texas
April, 1973

Coal Gasification

L. G. Massey

Dallas, Texas
April, 1973

General Papers

F. Schora

Dallas, Texas
April, 1973