The effect of carbon laydown on catalyst activity
by Huo-Yen Hsieh

A thesis submitted in partial fulfillment of the requirements for the degree of MASTER OF SCIENCE in Chemical Engineering
Montana State University

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Liquid products obtained from the catalytic hydrogenation experiments were analyzed for the sulfur-nitrogen concentrations and for the extent of hydrocracking by the method of ASTM D-86.

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The SRC-II Light Ends Column Feed (LECF) gave better sulfur and nitrogen removal than did SRC-II Vacuum Flash Feed (VFF). SRC-II VFF gives a greater pore volume reduction of the catalyst than does SRC-II LECF.

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Date Dec. 22, 1981

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HUO-YEN HSIEH

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Approved:

Lloyd Berg
Chairperson, Graduate Committee

J. P. McCandless
Head, Major Department

Michael Malone
Graduate Dean

MONTANA STATE UNIVERSITY
Bozeman, Montana

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ABSTRACT

Solvent Refined Coal (SRC-II) from Pittsburg and Midway Coal Mining Company's SRC-II pilot plant was hydrotreated with Montana State University developed catalyst MSU-C-49 combining 4% CoO, 8% MoO_3, 1% NiO and 8% WO_3. Thirty-two batch autoclave tests were performed.

Liquid products obtained from the catalytic hydrogenation experiments were analyzed for the sulfur-nitrogen concentrations and for the extent of hydro-cracking by the method of ASTM D-86.

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INTRODUCTION

America has more energy in coal than the Middle East has in oil. It is estimated that coal accounts for 80 percent of the fossil fuel resources in the U.S.(1). For many uses, however, the coal must be turned into liquid and gaseous fuels. It has become increasingly obvious that it is vital for this country to develop as quickly as possible effective means of converting coals to convenient non-polluting fuels.

According to estimates made by the U.S. Department of Energy (DOE), coal, natural gas, and nuclear power usage will trend upward, while crude oil and gasoline consumption will decline. In the coming years, coal consumption will increase to 750 million tons annually from 1980's 705 million tons. On the down side, petroleum consumption will drop by 400,000 bbl/d to 16.6 million bbl/d(2).

Several coal liquefaction processes and technologies have been developed. These processes can be divided into three general categories: pyrolysis, extraction-hydrogenation, and indirection liquefaction. At present there are a number of processes that might be developed into commerial demonstration plants but the manufacturing cost

seems to be greater than those for conventional petroleum fuel.

It is the purpose of this research to investigate the effect of operating conditions on the catalyst activity and to provide the information on the life of the catalyst before the continuous long run tests are made.

BACKGROUND

Coal is an abundant resource -- it constitutes about 77 percent of U.S. conventional energy reserves, but currently supplies less than 20 percent of energy consumption(3). Moreover, coal can supply all energy demands since it can be burned directly or transformed into liquid, gas, or feedstock. But its potential can not be realized until a number of problems are solved. Coal burning can be a major source of pollution in the form of sulfur dioxide, nitrogen oxides, particulates, and solid waste. The processes for making liquids and gas from coal have yet to be fully developed.

The principal objectives of the resource program are organized around the extraction, processing, and utilization of coal in an environmently acceptable manner. In order to meet the environmental requirements, significant sulfur and nitrogen reduction must be obtained and the mineral substances in the liquid phase must be reduced to a low concentration. These are accomplished by the Solvent Refined Coal (SRC)-II process.

Chemical Structure of Coal

Coal is comprised of carbon, hydrogen, oxygen, sulfur, nitrogen and inorganic substances. The structure of coal is composed chiefly of condensed, aromatic rings of high molecular weight. Figure 1 shows the representative coal structure as given by Hill and Lyon (4).

SRC (Liquid) Process - SRC-II

The SRC project was begun in 1962 and the process was successfully demonstrated in a 50 lb/hr continuous flow unit in 1965. The SRC-II process is operated by Pittsburg and Midway (P&M) Coal Mining Company. A combustion test of the SRC solid product performed by the Southern Company Services in a 22.5 MW utility plant demonstrated its capability to meet current emission standards for sulfur and nitrogen oxides. Consolidated Edison Company operating a combustion test of the SRC distillate fuel in a utility boiler has successfully demonstrated that emissions will comply with Environmental Protection Agency, (EPA), standards for sulfur and nitrogen oxides. Rust Engineering Company began building the pilot plant in 1972 at Ft. Lewis, Washington. A schematic diagram of SRC-II process is shown in Figure 2(5).

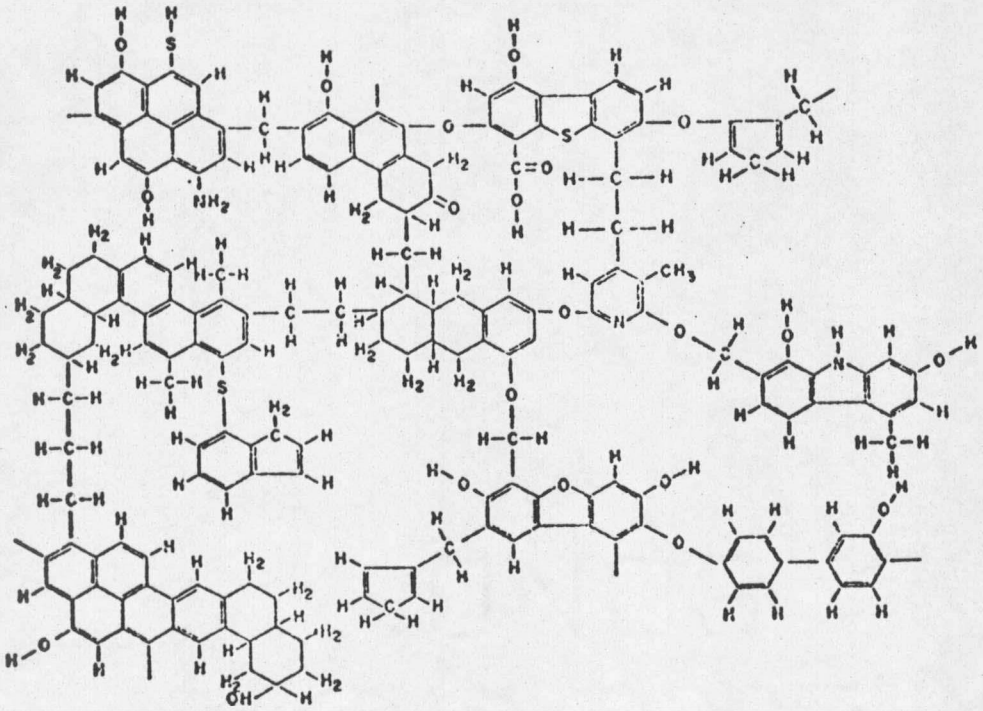


Figure 1. Coal Structure

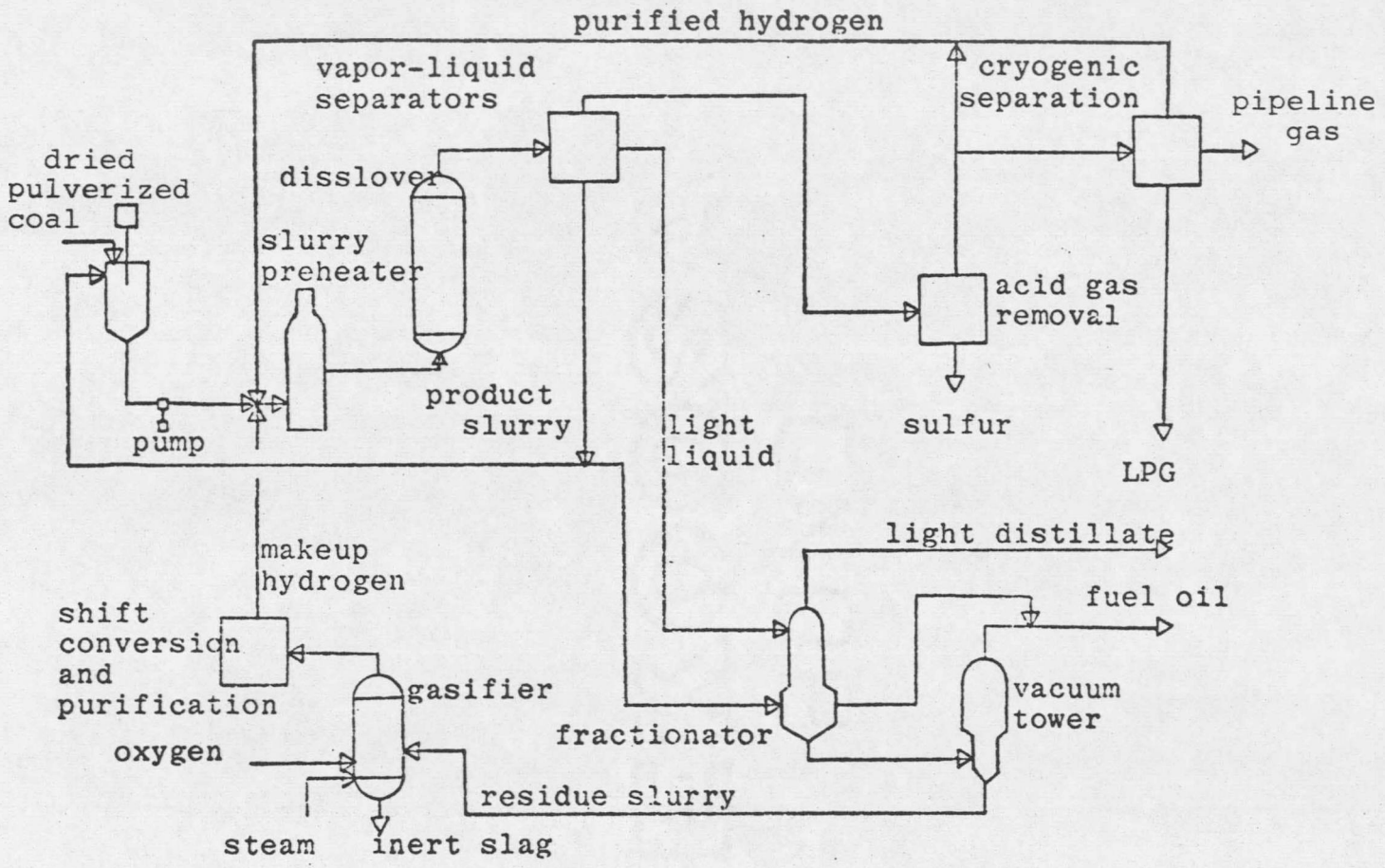


FIGURE 2. SRC-II PROCESS SCHEMATIC

Chemical and Physical Properties of SRC-II Product

The SRC process is not considered as a single product process. Table I(6) shows the gas and liquid yields of SRC. The SRC-II product used in this research was made from Kentucky #9 coal. This is shown in Table II(7).

Catalytic Hydrotreating for Upgrading

The hydrogenation, desulfurization and denitrogenation are the major coal conversion and upgrading problems. Sulfur, nitrogen and oxygen contents of coal are often greater than 1 wt% and the hydrogen to carbon atoms ratio of coal is too low. The maximum sulfur level is determined from the current EPA standard(8). For liquid products from the SRC-II, it is easier to meet the minimum standard for sulfur than for nitrogen content. The major steps in upgrading are to reduce the nitrogen and sulfur levels to less than 0.3 wt% and to increase the hydrogen to carbon ratio of the fuel. Some hydrocracking processes (the Standard Oil of Indiana Ultracracking process and Union Oil Unicracking process) can tolerate feedstock with a nitrogen content of as high as 0.3 wt% (9,10). The reason for the requirement of low nitrogen content is to prevent catalyst poisoning during conventional hydrocracking.

TABLE I

SRC Process Gas and Liquid Yields*

C_1 - C_4 gas, scf	3130
CH_4 gas	2100
C_5 - 350 °F gal	32
bbl	0.762
350-750 °F distillate, gal	38
bbl	0.904
Total liquid, gal	70
bbl	1.666

Approximate analysis of C_1 - C_4 gas cut:

	Vol.%	BTU value/ft^3
CH_4	67.0	680
C_2H_6	19.3	340
C_3H_8	10.0	260
C_4H_{10}	3.7	120
	100.0	1400

* Per ton of SRC

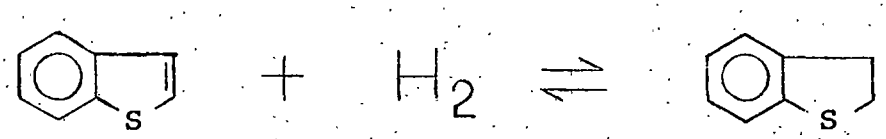
TABLE II

SRC Feed Coal Analysis, June 1979

Average Raw Coal Analysis (wt%)	
Ash	9.55
Moisture	6.14
Average Analysis of Forms of Sulfur (wt% on Coal)	
Pyritic Sulfur	2.03
Sulfate Sulfur	0.27
Organic Sulfur	1.27
Total	3.57
Average Dried Pulverised Coal Analysis (wt%)	
Carbon	70.76
Hydrogen	5.18
Nitrogen	1.53
Sulfur	3.57
Oxygen (by difference)	8.60
Ash	9.97
Moisture	6.39

Hydrodesulfurization (HDS) and Hydrodenitrogenation (HDN)

There are many sulfur and nitrogen compounds in coal liquids of which benzothiophene and quinoline are typical. The proposed mechanisms for the desulfurization and denitrogenation of coal liquids have been studied in microreactors(11-14). In the HDS of benzothiophene, it was found that hydrogenation of the double bond in the triophene ring took precedence over the removal of sulfur.

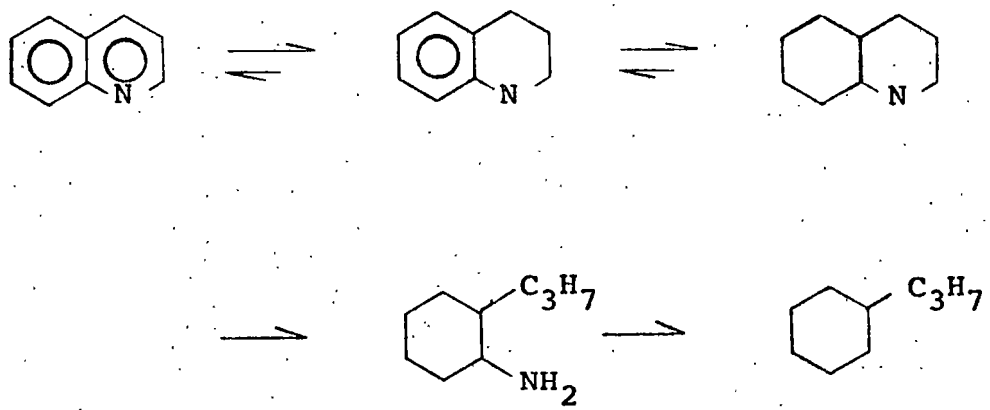


It was also found that the two compounds above desulfurized at the same rate when both were reacted separately, so it is not certain whether one is an intermediate of the other in the reaction(12,13).

Usually nitrogen is more difficult to move than sulfur. Model nitrogen compounds that are present in coal liquids are pyridines and substituted

pyrroles (indole) as the basic nitrogen heterocyclics.

It has been shown that the total rate of HDN shows a maximum with respect to hydrogen partial pressure. The only individual reaction which decreases in rate with increasing hydrogen partial pressure is the conversion of 1,2,3,4-tetrahydroquinoline to ortho-n-propylaniline. This rate determining step dominates the overall network at high temperature(11-15).



RESEARCH OBJECTIVE

The object of this research was to optimize the operating conditions for upgrading the SRC-II products.

The upgrading consisted of the reduction of nitrogen and sulfur contents of the feedstock and an increment of clean product recovered in the ASTM D-86 distillation test.

A catalyst designated MSU-C-49 with a metal combination of 4% CoO, 8% MoO_3, 1% NiO and 8% WO_3 was fabricated and evaluated in a batch autoclave reactor.

The objective was to investigate the effect of operating conditions on the catalyst activity. Catalyst deactivation was determined by the pore volume measurement.

This research is expected to provide information of the activity and life of the catalyst before a continuous long run test is made.

MATERIALS, EQUIPMENT AND PROCEDURES

Feedstock

The SRC-II liquid products made by using Kentucky #9 coal from the Colonial Mine were supplied by the Pittsburg and Midway Coal Mining Company (P&M). In this research there are two SRC-II products, P&M's SRC-II Vacuum Flash Feed (VFF) and Light Ends Column Feed (LECF), which were used as feedstocks.

The VFF containing 1.17 wt% N and 0.72 wt% S is a slurry at room temperature and the LECF containing 0.88 wt% N and 1.21 wt% S is a mixture of 15% SRC Naphtha, 40% SRC Middle Distillate and 45% SRC Heavy Distillate. These analyses are shown in Table III.

Catalyst Preparation

MSU fabricated catalyst, MSU-C-49, with 4% CoO, 8% MoO_3, 1% NiO and 8% WO_3 was prepared by impregnating the active metal compounds on a commercial base, Nalco extrudates. The MSU-C-49 combination of metal oxides was reported by Montana State University to have the highest HDN activity(16). Four metal salts, ammonium molybdate, nickelous nitrate, cobalt nitrate and ammonium metatungstate, were selected for their solubilities in

TABLE III

PROPERTIES OF SRC-II FEEDSTOCKS

	Vacuum Flash Feed	Light Ends Column Feed
% Carbon	87.43	-*
% Hydrogen	7.15	-*
% Nitrogen	1.17	0.88
% Sulfur	0.72	1.21
% Oxygen	3.72	-*
% Ash	0.246	0.02
Sp. Gravity 60/60°F	1.08	0.983
ASTM D-86 Distillation, °F		
IBP	408	122
5		217
10	445	288
20	485	381
30	544	446
40	598	488
50	642	541
60		577
70		611
80		660
90		727
95		795
End Point	684	956

* Data not available

water which is needed in the wetness method of preparation.

The procedure used was as follows:

1. The support was dried in oven at 110 ^{o}C for 8 hours.
2. The support was calcined at 450 ^{o}C for 8 hours to be sure that the support could be weighed correctly.
3. Temperature was cooled in a desiccator to room temperature.
4. Weight of the support was recorded.
5. The support was impregnated in a slowly rotating jar with a specific metal solution, the concentration of which is calculated by the formulation (17):

   Metal oxide percent in the support
   = Conc. of solution x pore volume /
   (1 + (pore volume x conc. of solution)).
6. The catalyst was dried in an air stream of 3 psig.
7. The steps from 1 to 4 were repeated for the air dried catalyst and record the weight increase after impregnation.

This procedure could be repeated as needed to obtain

the desired percentages of metal oxides.

The catalyst carrier, Nalco-78-6008C-1/32", obtained from Nalco company was used in this research. The properties of this catalyst carrier are 98% Al_2O_3 and 2% SiO_x, and possessing a surface area of 214.6 m^2/gm, an average pore diameter of 156.5 Å, a median pore volume of 156.5 Å and a pore volume of 0.84 ml/gm. Average pore diameter (Å) is equal to 40,000 (pore volume / surface area).

Catalyst Pretreatment

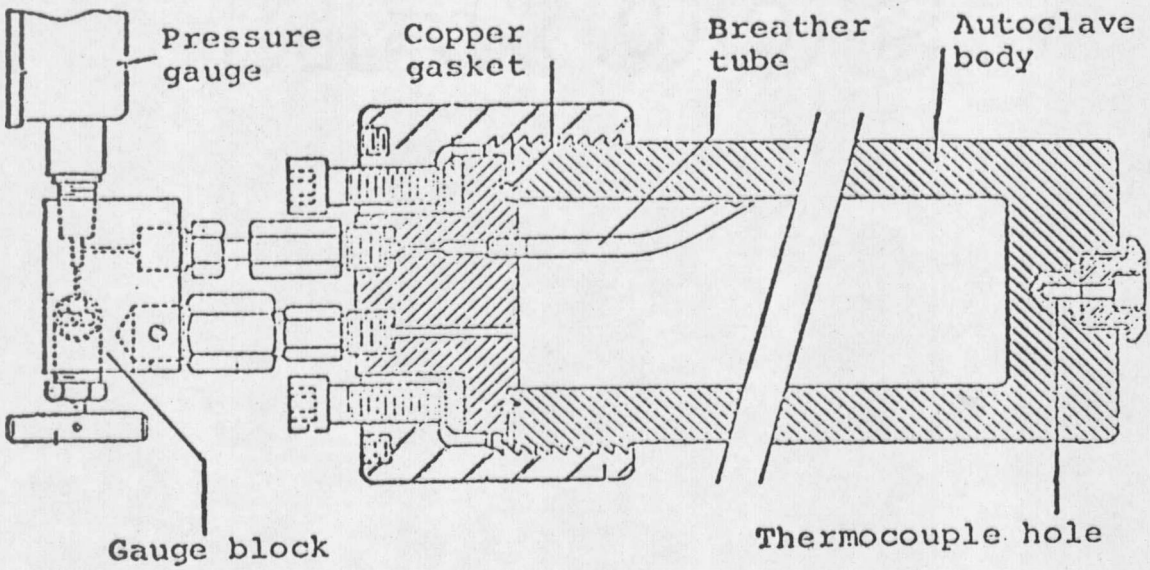
All catalysts were pretreated by the following procedure which was used to activate the catalyst and to prevent the reduction of catalyst activity(18,19). The catalyst was treated with a 10% hydrogen sulfide in hydrogen mixture for twelve hours. Temperature was maintained at 325 ^oC by the use of a powerstat on the electric pipe heater. Exit gases from the sulfides were scrubbed with water and a 20% sodium hydroxide-water solution before venting to the hood. Extreme caution should be taken whenever using hydrogen sulfide because it can cause collapse, coma and death within a few seconds

after one or two inspirations. Hydrogen sulfide is extremely hazardous because it fatigues the sense of smell in high concentration, therefore giving no warning(20).

Batch Autoclave Tests

Batch autoclave tests were made in a Parr Series 4000 pressure reaction apparatus(21,22). The Parr autoclave and heater-rocker are shown in Figure 3.

The autoclave was charged with 30 ml of catalyst, together with 200 ml of SRC-II product. The copper head gasket and autoclave head were secured using a torque wrench. New copper head gaskets were torqued to 60 ft-lbs. Subsequent runs with the same head gasket were torqued higher until the copper gasket was replaced. The pressure gauge and gauge block were attached to the autoclave head. The autoclave was pressurized to 1,100 (or 2,000) $\pm$ 50 psig hydrogen gas from a high pressure cylinder or using a Haskel gas booster air-driven compressor(23) and checked for leaks. The autoclave was heated to 400 (or 425) $\pm$ 10^{o}C, which usually took about 50 minutes. The run time, 60 (or 120) minutes, was measured from the



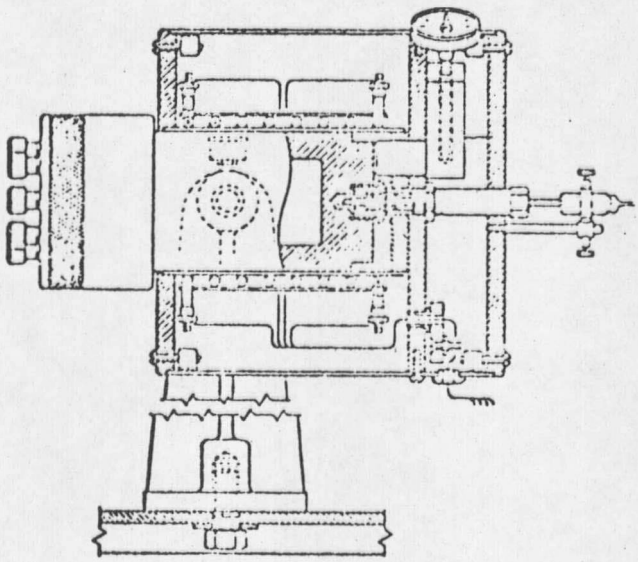


FIGURE 3. ROCKING AUTOCLAVE ASSEMBLY DETAILS

time the target temperature was reached. An iron-constantan thermocouple placed in the base of the autoclave connected to a single point Micromax recorder(24) monitored the temperature of reaction. Reaction temperature was controlled by manual adjustment of a powerstat variable transformer. Pressure and temperature were recorded at 15 minute intervals during each run. Upon completion of the run, the autoclave was removed from the heater-rocker and allowed to cool to room temperature. The gas in the autoclave was then vented in a hood by opening the needle value in the autoclave gauge block. After the autoclave head and gauge block were removed, the liquid product was then filtered from the catalyst and analyzed.

Operation of Catalyst Regeneration

The regeneration and the sulfiding processes were carried in the same reactor. The premixed gas, 40% oxygen in nitrogen, stored in

the cylinder, was fed into the top of the reactor. The outlet gases passed through a wet test meter used for measuring the flow rate. The burn-off process had to be carried out at 550 ^{o}C. The catalyst was checked to determine the completeness of burn-off. After completing the process, the catalyst was resulfided prior to reuse.

Analytical Procedure

Liquid products from all runs were analyzed for nitrogen and sulfur contents and for the extent of hydrocracking.

The Macro-Kjeldahl method was used to determine nitrogen content using approximately 0.5 grams of product sample(25,26,27). The weight percent denitrogenation (% DN) was calculated as follows:

$$\%DN = (\text{wt\% N of feed} - \text{wt\% N of product}) / (\text{wt\% N of feed})$$

The sulfur analysis was performed on approximately 0.3 grams sample by the quartz tube combustion method using a Bico-Brown Shell design sulfur apparatus(28,29). The desulfurization(% DS) was calculated similarly to

the denitrogenation(% DN).

The extent of hydrocracking was determined by ASTM D-86 atmospheric distillation(30). Fifty milliliters of composite sample representative of each run were used as a standard test volume, if possible. This technique measured the cumulative amount of product which boiled below 650 ^{o}F (434 ^{o}C) or when decomposition began, whichever occured first.

The pore volume of the catalyst was estimated by the water saturation method(31). It gave a relative value rather than absolute. The procedure for pore volume measurement is as follows:

1. Dry the catalyst at 100 ^{o}C for two hours.
2. Cool the catalyst to room temperature in a desiccator.
3. Weigh the catalyst, then immerse the catalyst in the boiling water for 5 minutes.
4. Decant the excess water and widely spread the catalyst on a sheet of paper, then proceed to air dry for 35 minutes.
5. Weigh the saturated catalyst.

The pore volume was calculated by the grams of water absorbed per gram of catalyst assuming the density of water at room temperature to be 1.0 gram/ml.

RESULTS AND DISCUSSIONS

Twenty-seven runs have been carried out with the batch autoclave reactor using SRC-II Vacuum Flash Feed (VFF), and five runs using SRC-II Light Ends Column Feed (LECF). Catalyst, MSU-C-49, that was fabricated at Montana State University was tested in these runs except two runs which tested blank catalyst bases.

The data for each batch run are presented in the Appendix. The samples taken from all runs were analyzed for nitrogen and sulfur content and for the amount of distillable liquids. The pore volume measurements of catalysts were tested after each regeneration. The effect of operating conditions was also examined. The operating conditions for batch runs are shown in Table IV.

Batch Autoclave Runs

Batch tests were performed on SRC-II product to evaluate the effect of carbon laydown on catalyst activity under different operating conditions. As continuous runs had been very long, it was considered that batch runs could provide relatively fast catalyst testing. In an attempt to investigate the activity of the catalysts

TABLE IV

Operating Conditions for Batch Runs

Run	Feedstock*1	Run Temp., °C	Time at Run Temp., min.	Hydrogen Pressure, psig
U-1 to U-5*2	VFF	400	60	1100
H-1 to H-5	VFF	400	60	1100
H-6 to H-10	VFF	400	120	1100
H-11 to H-15	VFF	425	60	1100
H-16 to H-20	VFF	400	60	2000
N-1 to N-5	LECF	400	60	1100

*1 VFF contains 1.17 wt% N and 0.72 wt% S; LECF contains 0.88 wt% N and 1.21 wt% S.

*2 Runs U-1 to U-5 were made with unsulfided catalyst; H-1 to H-20 and N-1 to N-5 were made with sulfided catalyst.
All runs were followed by regeneration.

influenced by operating conditions, the following procedure was performed. To test operating temperature, time at run temperature, hydrogen pressure, and influence of two different feedstocks, SRC-II (VFF) and SRC-II (LECF), thirty batch autoclave runs were tested using the same catalyst, MSU-C-49, and which are designated H-1 to H-20, U-1 to U-5, and N-1 to N-5.

There were two blank runs, designated B-1 and B-2, made with blank catalyst carrier. The detailed data with a summary of operating conditions, analytical results, and content of the reactor from these tests are presented in the Appendix. Table V summarized the catalyst activity for denitrogenation and desulfurization as well as calculated pore volume. All catalysts were pretreated by sulfiding except the five runs made with unsulfided catalysts which were compared with the five runs made with sulfided catalysts under the same operating conditions and feedstocks.

The first two runs, B-1 and B-2, were made using blank catalyst carriers as the catalysts and were carried out to determine a better operating time at run

TABLE V

Batch Run Data Summary

Run	Catalyst	Wt% DS	Wt% DN	Pore Volume*
B-1	Carrier	4.17	0	- -
B-2	Carrier	6.67	0	- -
U-1	MSU-C-49	19.44	3.42	0.56
U-2	MSU-C-49	20.80	5.98	0.53
U-3	MSU-C-49	11.10	11.97	0.52
U-4	MSU-C-49	16.70	1.74	0.52
U-5	MSU-C-49	6.94	2.56	0.51
H-1	MSU-C-49	41.67	13.60	0.32
H-2	MSU-C-49	41.67	16.23	0.49
H-3	MSU-C-49	8.33	3.41	0.51
H-4	MSU-C-49	12.50	2.56	0.49
H-5	MSU-C-49	9.72	0	0.51
H-6	MSU-C-49	44.44	25.64	0.42
H-7	MSU-C-49	37.50	18.80	0.41
H-8	MSU-C-49	41.67	23.08	0.42
H-9	MSU-C-49	44.44	11.11	0.51
H-10	MSU-C-49	47.22	11.11	0.47
H-11	MSU-C-49	48.61	11.97	0.42
H-12	MSU-C-49	58.33	14.53	0.34
H-13	MSU-C-49	48.61	14.53	0.38
H-14	MSU-C-49	44.44	11.11	0.49
H-15	MSU-C-49	45.83	14.53	0.43
H-16	MSU-C-49	66.67	21.37	0.56
H-17	MSU-C-49	68.06	15.38	0.52
H-18	MSU-C-49	44.44	11.97	0.51
H-19	MSU-C-49	37.50	9.40	0.50
H-20	MSU-C-49	19.44	5.13	0.51
N-1	MSU-C-49	84.30	17.05	0.55
N-2	MSU-C-49	76.86	13.64	0.55
N-3	MSU-C-49	79.34	10.23	0.50
N-4	MSU-C-49	71.90	7.95	0.55
N-5	MSU-C-49	75.21	5.68	0.55

*Calculated Pore Volume, ml./gm. of Catalyst.

temperature for feeding the SRC-II VFF. Their operating conditions were 1,100 psig and 400 ^{o}C. Run B-1 was operated for 60 minutes at run temperature, while run B-2 was 90 minutes. Both run B-1 and run B-2 gave zero wt% DN. Run B-1 has 4.17 wt% DS, while run B-2 has 6.67 wt% DS. Figure 4 shows the distillation data of runs B-1 to B-2 products. It indicates a slight improvement in products compared with SRC-II VFF. It also seems that there was not much difference between run B-1 and run B-2. These runs had two different operating times, 60 and 90 minutes. In order to evaluate the effect of operating conditions on catalyst activity, it was desirable to select the best operating conditions. The operating conditions, 60 minutes, 1,100 psig and 400 ^{o}C, of runs H-1 to H-5 were taken to be the best. Sets of five batch runs with the same operating conditions and catalyst were carried out with regenerations after each run. There were six such sets: U-1 to U-5, H-1 to H-5, H-6 to H-10, H-11 to H-15, H-16 to H-20, and N-1 to N-5.

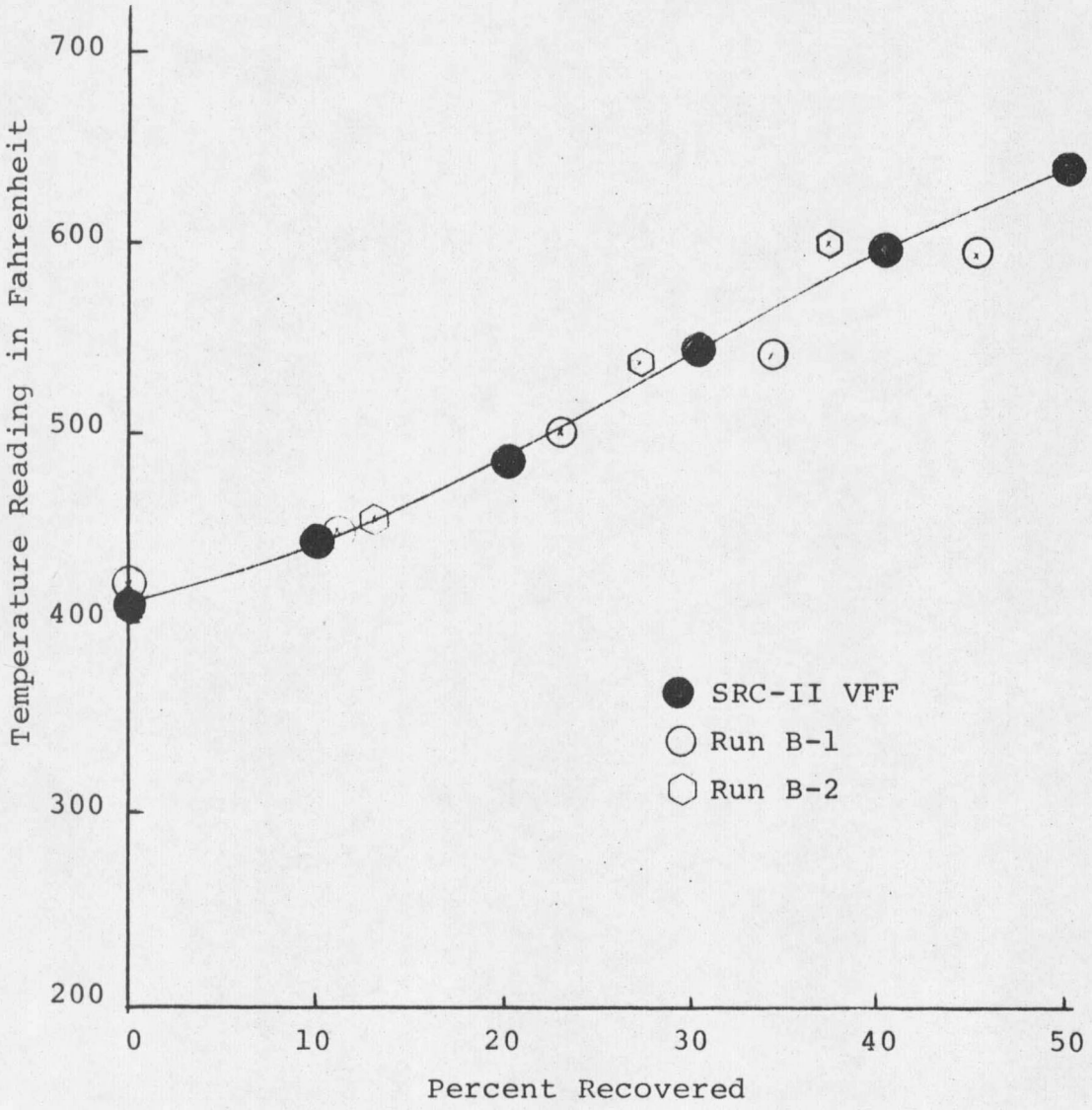


Figure 4. Distillation Data of the Products of Runs B-1 and B-2.

Effect of Sulfiding on Catalyst Activity

The operating conditions of runs H-1 to H-5 were 400 ^{o}C, 1,100 psig and 60 min. The catalyst for these runs was sulfided after each regeneration. Runs U-1 to U-5 used unsulfided catalysts after each regeneration but had the same operating conditions as runs H-1 to H-5. The activities of runs U-1 to U-5 and runs H-1 to H-5 for desulfurization and denitrogenation are compared in Figure 5. Sulfiding of catalysts has been shown to improve hydro-desulfurization activity of catalysts(32). As Figure 5 denotes catalysts H-1 to H-5 showed increased desulfurization activity during runs 1 and 2, whereas remaining data showed no significant difference in desulfurization or denitrogenation between sulfided and unsulfided catalysts. The difference may be seen in a continuous reactor. The comparison of the distillation results shown in Figure 6 and Figure 7 shows little difference between runs U-1 to U-5 and runs H-1 to H-5.

Effect of Operating Time on Catalyst Activity

Five experiments, runs H-6 to H-10, were made to investigate the effect of operating time on catalyst activity. The operating conditions of these runs were

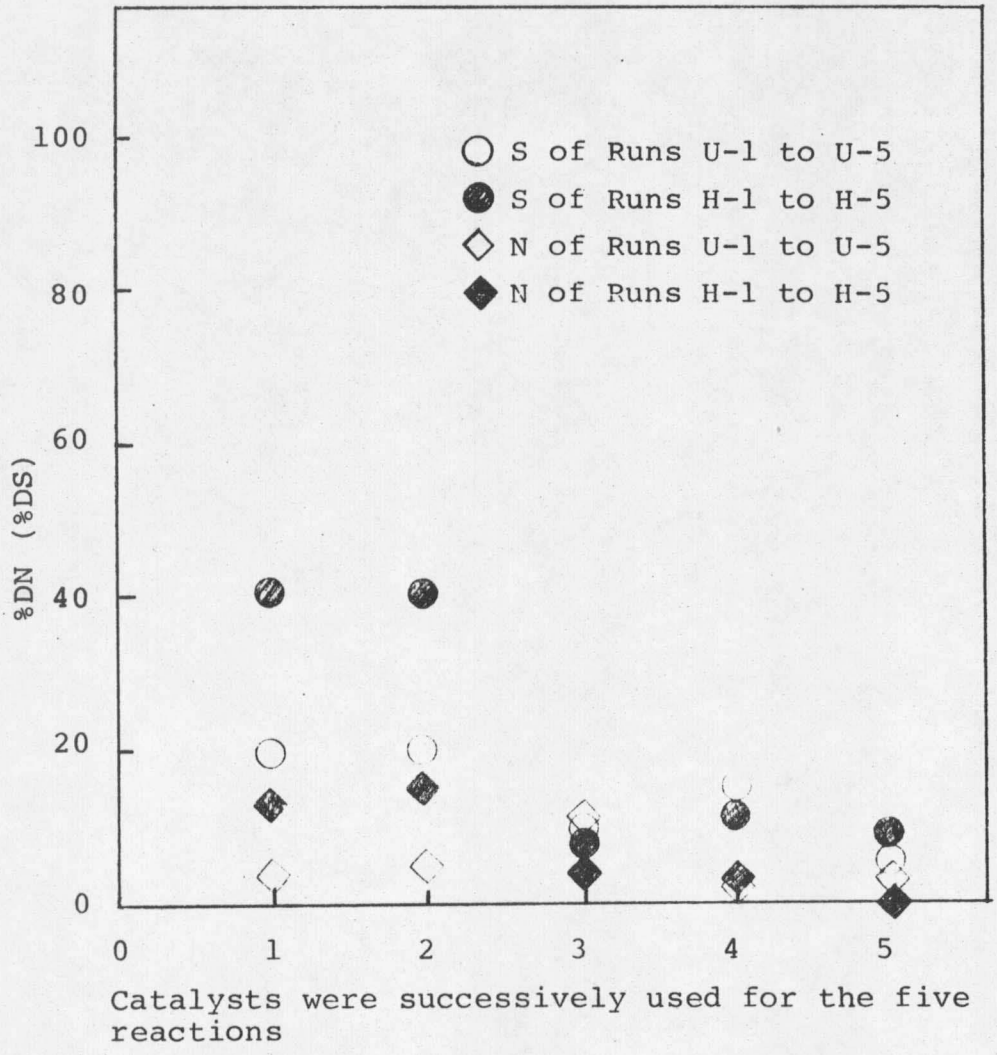


Figure 5. Effects of unsulfided and sulfided catalysts on nitrogen and sulfur removal, data obtained from runs U-1 to U-5 and H-1 to H-5.

Operating conditions: 400 ^{o}C, 1100 psig, 60 min.

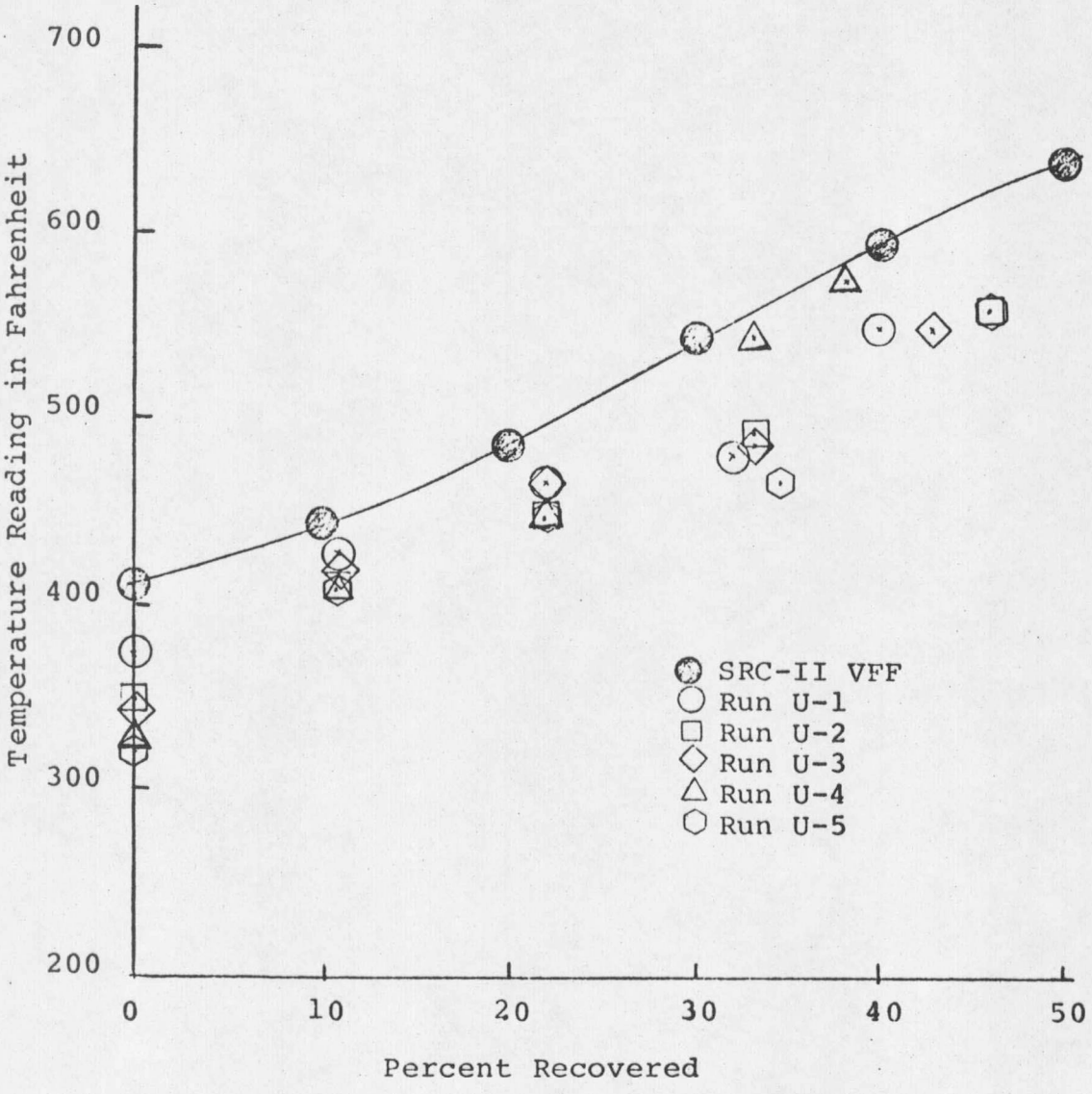


Figure 6. Distillation Data of the Products of Runs U-1 to U-5.

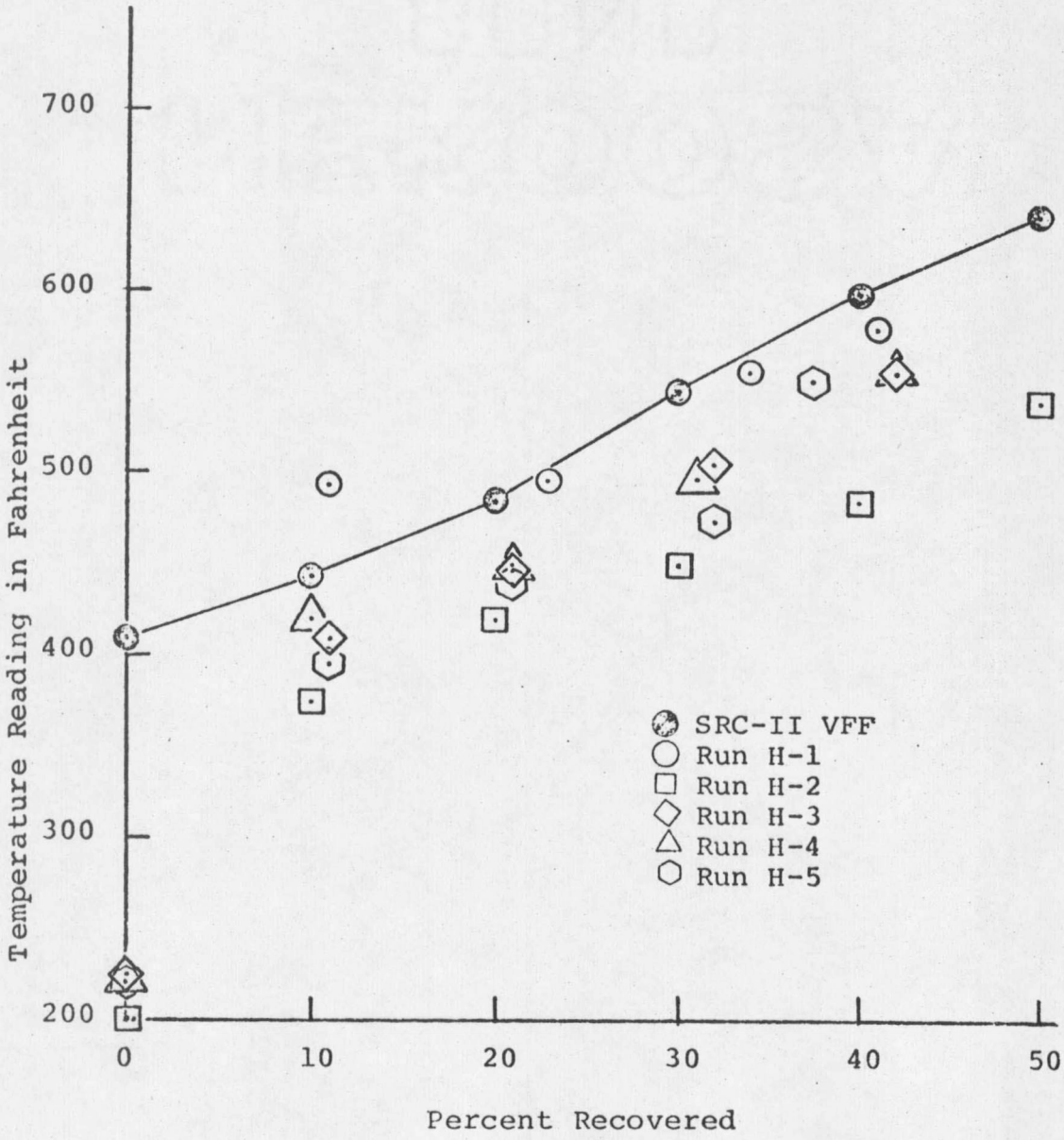


Figure 7. Distillation Data of the Products of Runs H-1 to H-5.

400 °C, 1,100 psig and 120 minutes, and the catalyst was sulfided after regeneration in these runs. Figure 8 shows the catalyst activity in runs H-1 to H-5 and runs H-6 to H-10 relative to desulfurization and denitrogenation. There was an improvement in desulfurization for runs H-6 to H-10 over runs H-1 to H-5. A slight improvement in denitrogenation was noted for runs H-6 to H-10 compared with runs H-1 to H-5. It can be seen from Table V that there is only a slight difference in the pore volume from runs H-1 to H-10. The distillation of the products of runs H-6 to H-10 is shown in Figure 9.

Effect of Operating Temperature on Catalyst Activity

Runs H-11 to H-15 were made at 425 °C, while the other operating conditions in these runs were the same as runs H-1 to H-5. The data in Figure 10 shows that runs H-11 to H-15 gave better desulfurization. It also shows there is no significant improvement in nitrogen removal for these runs compared with runs H-1 to H-5. Figure 11 plots the distillation data of runs H-11 to H-15. There is a slight difference in the pore volume of runs H-11 to H-15 compared with that of runs H-1 to H-5.

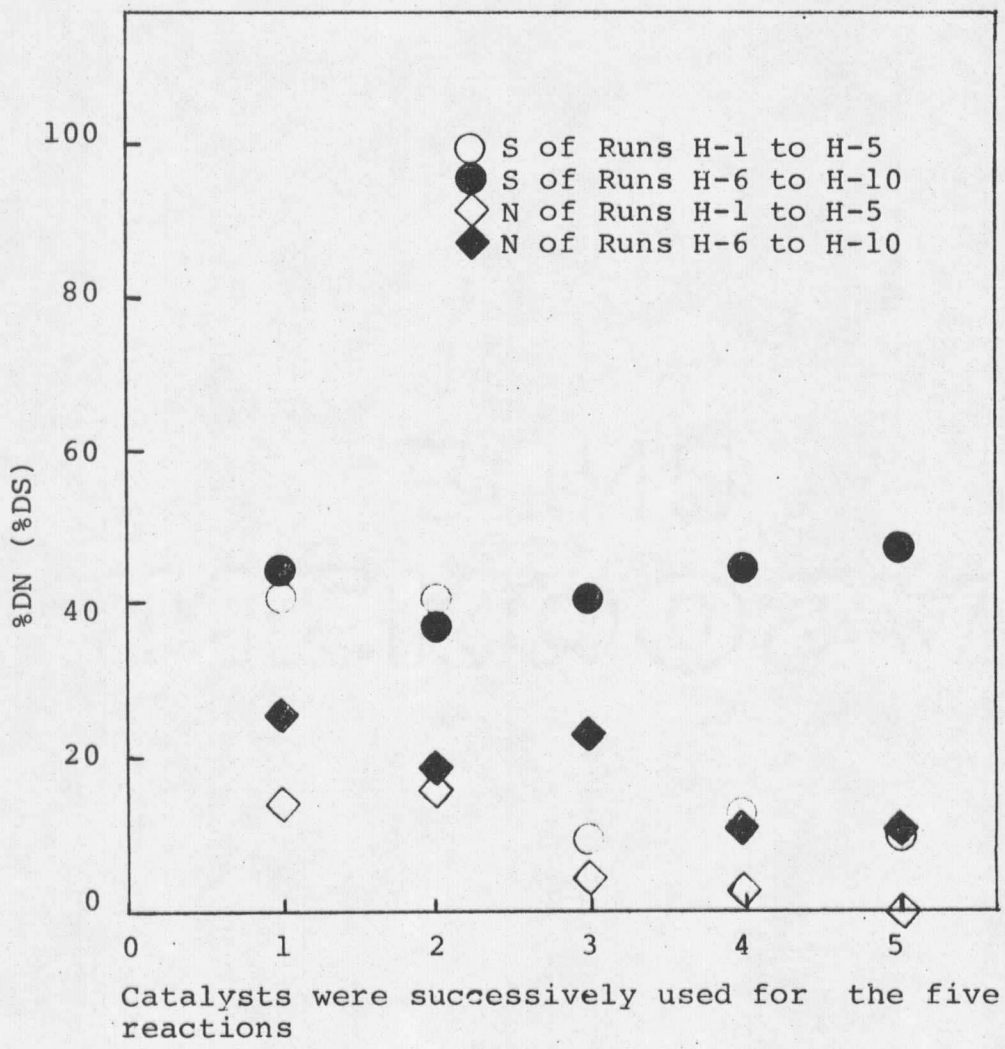


Figure 8. Activity versus operating time for nitrogen and sulfur removal comparing runs H-1 to H-5 with runs H-6 to H-10

Operating conditions:
Runs H-1 to H-5 : 400 °C, 1100 psig, 60 min.
Runs H-6 to H-10 : 400 °C, 1100 psig, 120 min.

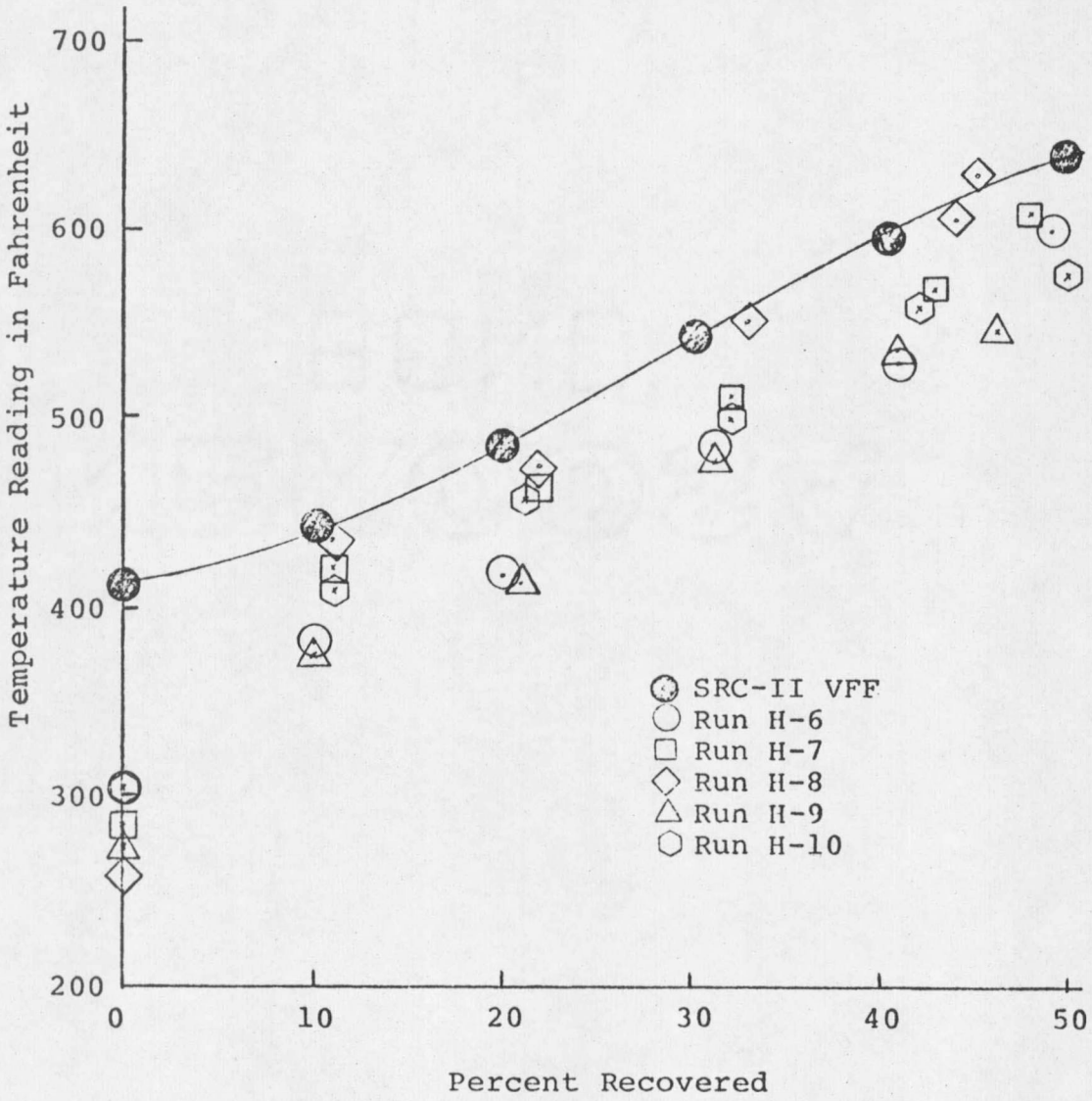


Figure 9. Distillation Data of the Products of Runs H-6 to H-10.

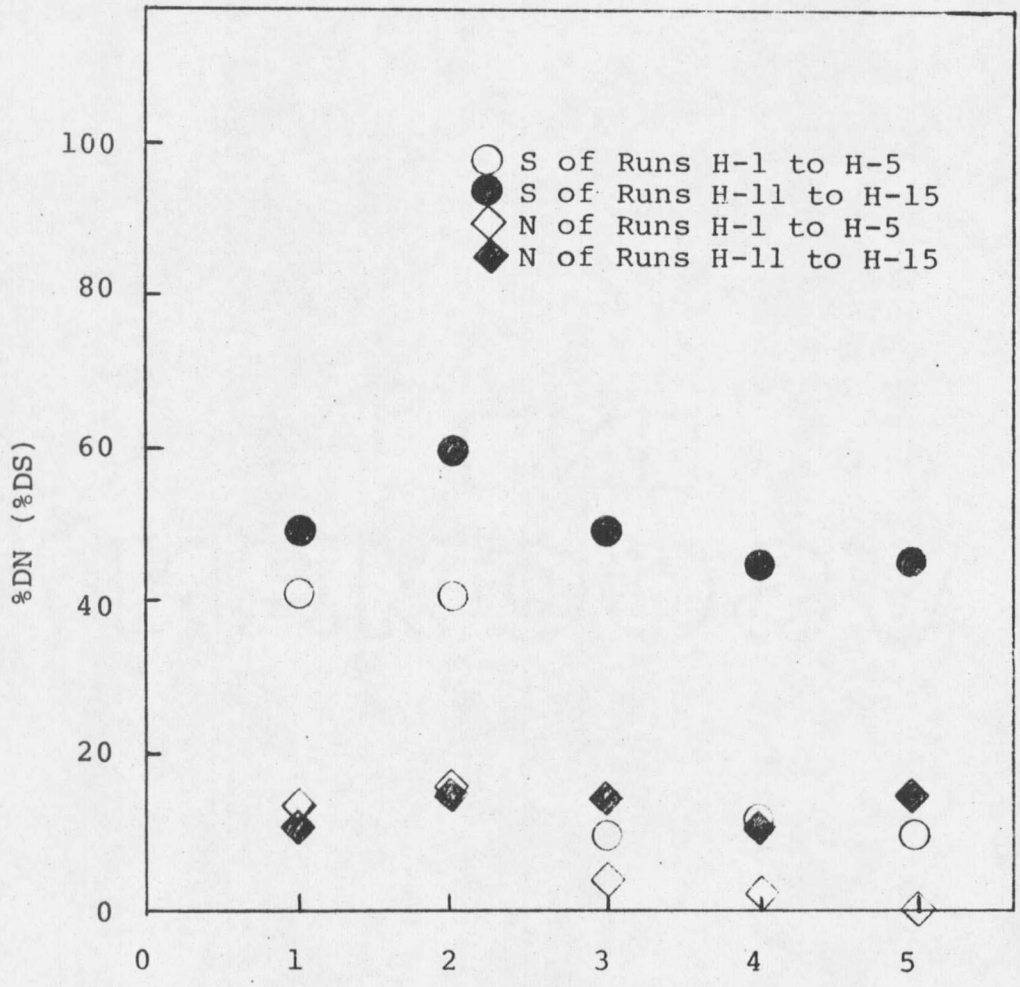


Figure 10. Activity versus operating temperature for nitrogen and sulfur removal comparing runs H-1 to H-5 with runs H-11 to H-15.

Operating conditions:
Runs H-1 to H-5 : 400 ^{o}C, 1100 psig, 60 min.
Runs H-11 to H-15 : 425 ^{o}C, 1100 psig, 60 min.

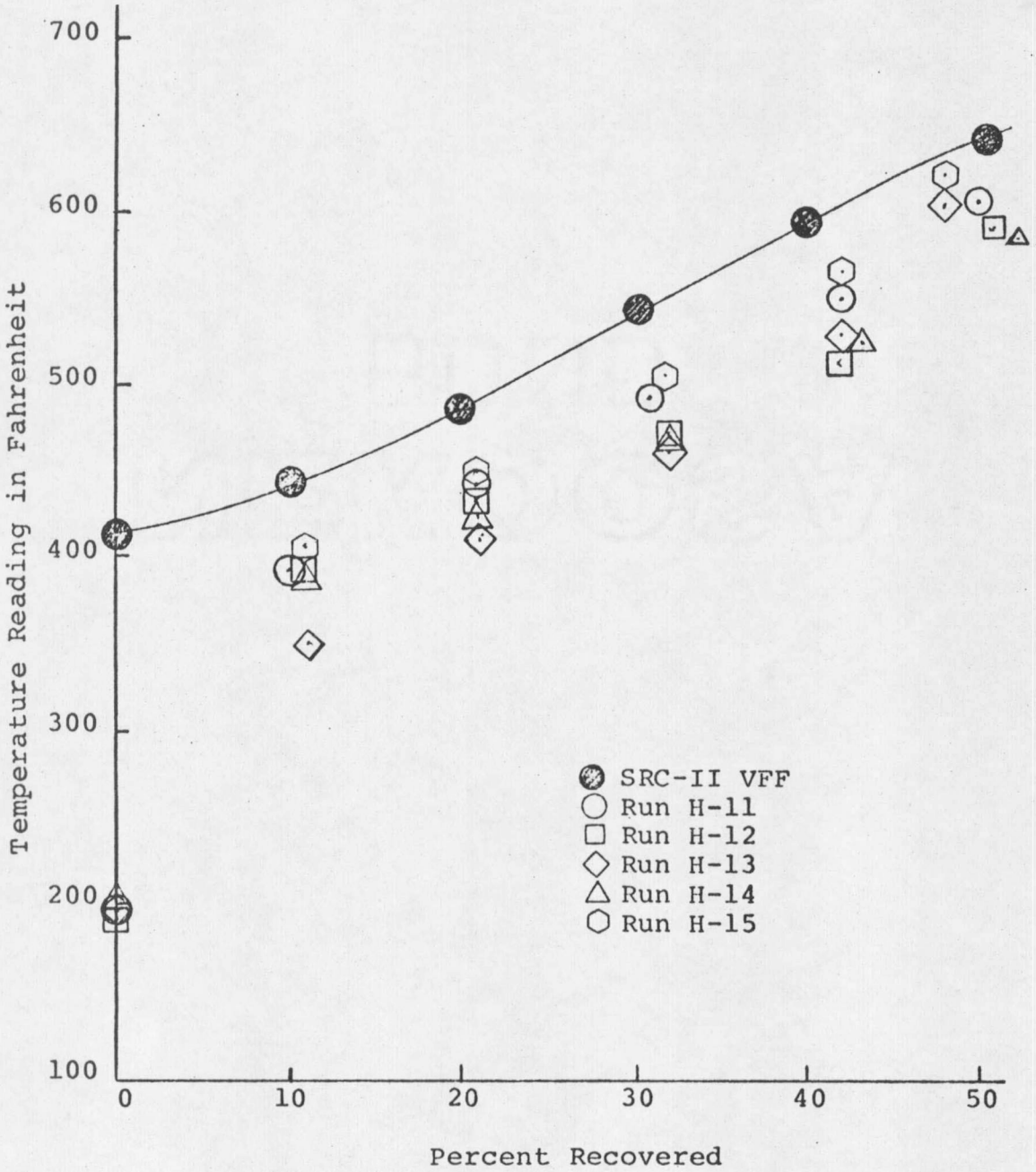


Figure 11. Distillation Data of the Products of Runs H-11 to H-15.

Effect of Initial Hydrogen Pressure on Catalyst Activity

The operating conditions of runs H-16 to H-20 were 400 ^{o}C, 2,000 psig and 60 minutes. Figure 12 shows that runs H-16 to H-20 had much better desulfurization than runs H-1 to H-5. It also shows a slight improvement in denitrogenation. The catalyst pore volume reduction of runs H-16 to H-20 was less than that of runs H-1 to H-5. The distillation data of runs H-16 to H-20 is shown in Figure 13.

Effect of Feedstock on Catalyst Activity

SRC-II LECF served as the feedstock for runs N-1 to N-5, while SRC-II VFF served for runs H-1 to H-5. Runs N-1 to N-5 and H-1 to H-5 had the same operating conditions. The results in Figure 14 show that runs N-1 to N-5 gave much better desulfurization than runs H-1 to H-5. There was no significant improvement in denitrogenation. Figure 15 plots the distillation results of runs N-1 to N-5. It shows an interesting result, that the initial boiling point of the products of these runs is higher than those from SRC-II LECF.

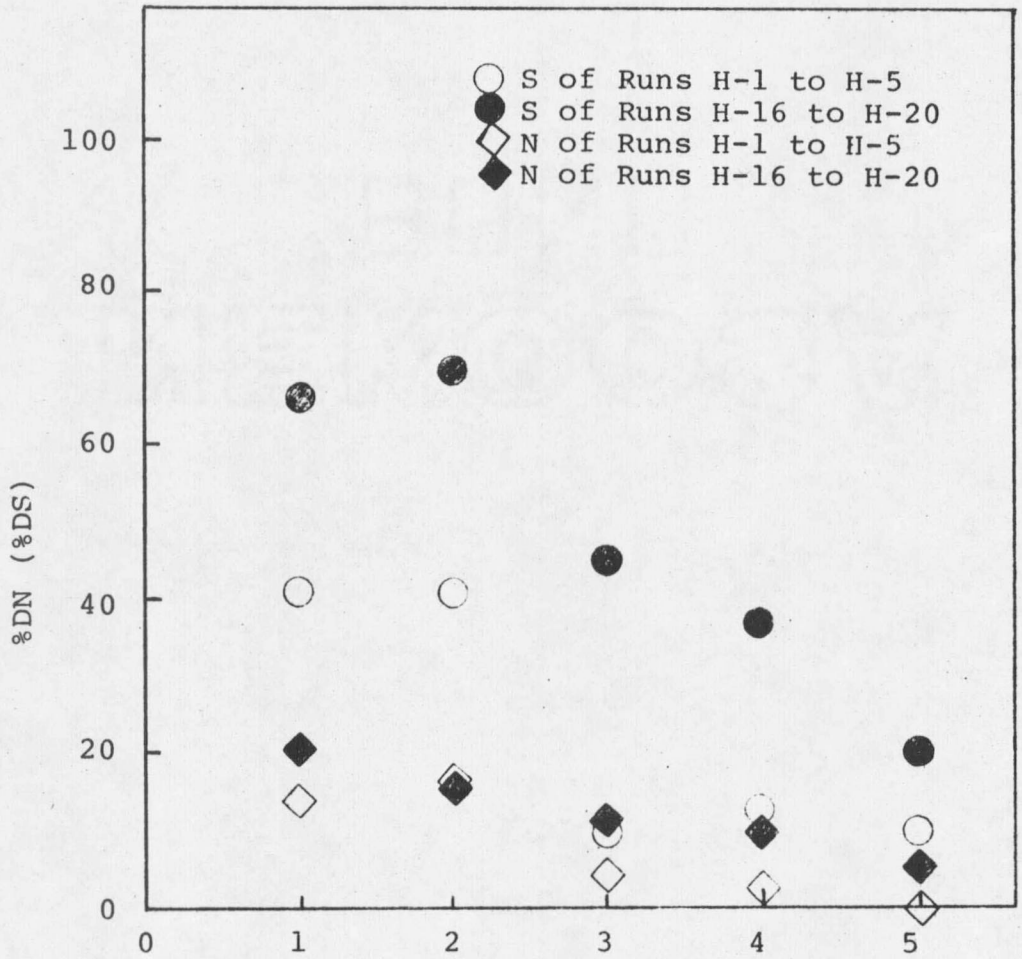


Figure 12. Activity versus operating pressure for nitrogen and sulfur removal comparing runs H-1 to H-5 with runs H-16 to H-20.

Operating conditions:
Runs H-1 to H-5 : 400 °C, 1100 psig, 60 min.
Runs H-16 to H-20 : 400 °C, 2000 psig, 60 min.

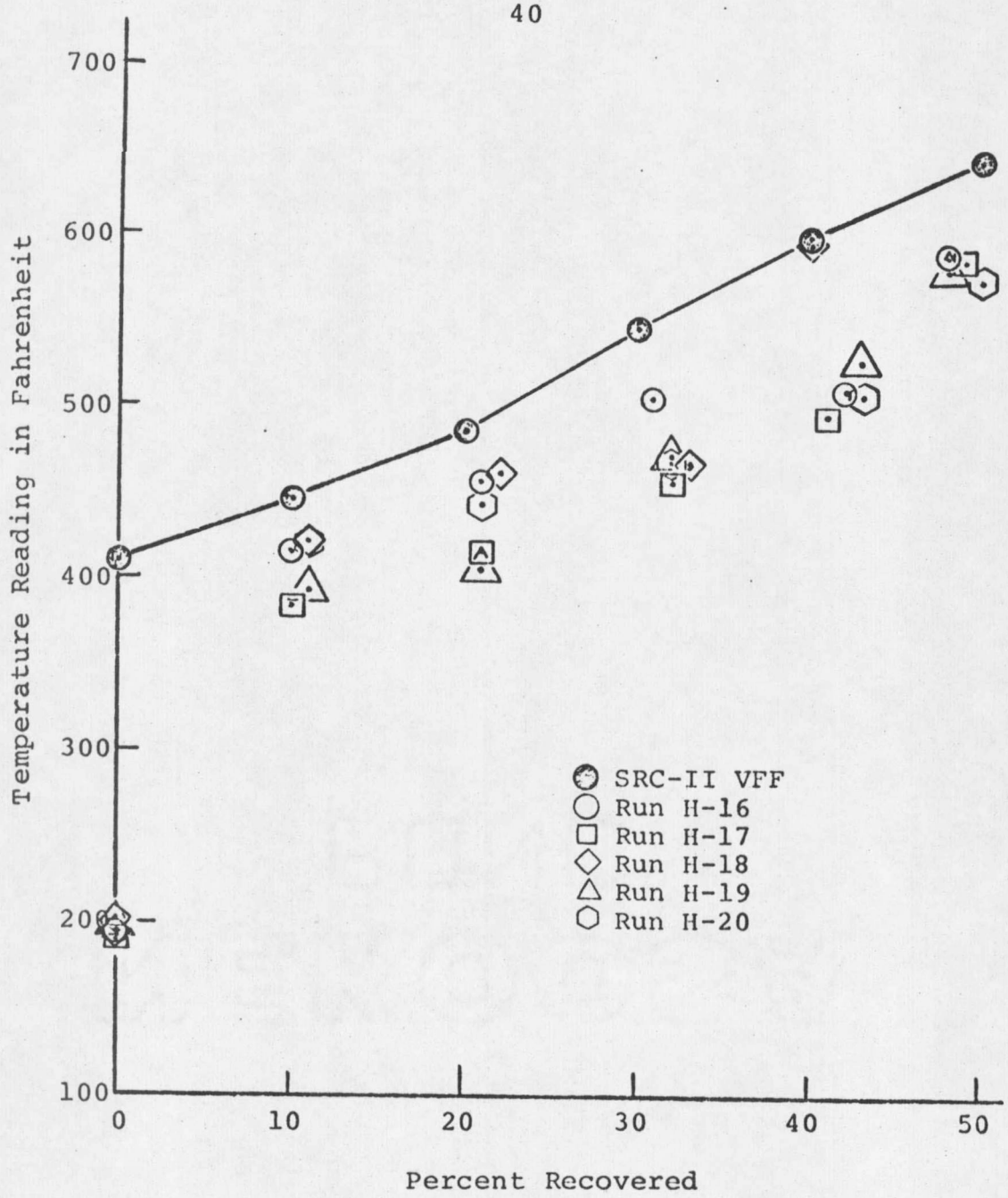


Figure 13. Distillation Data of the Products of Runs H-16 to H-20.

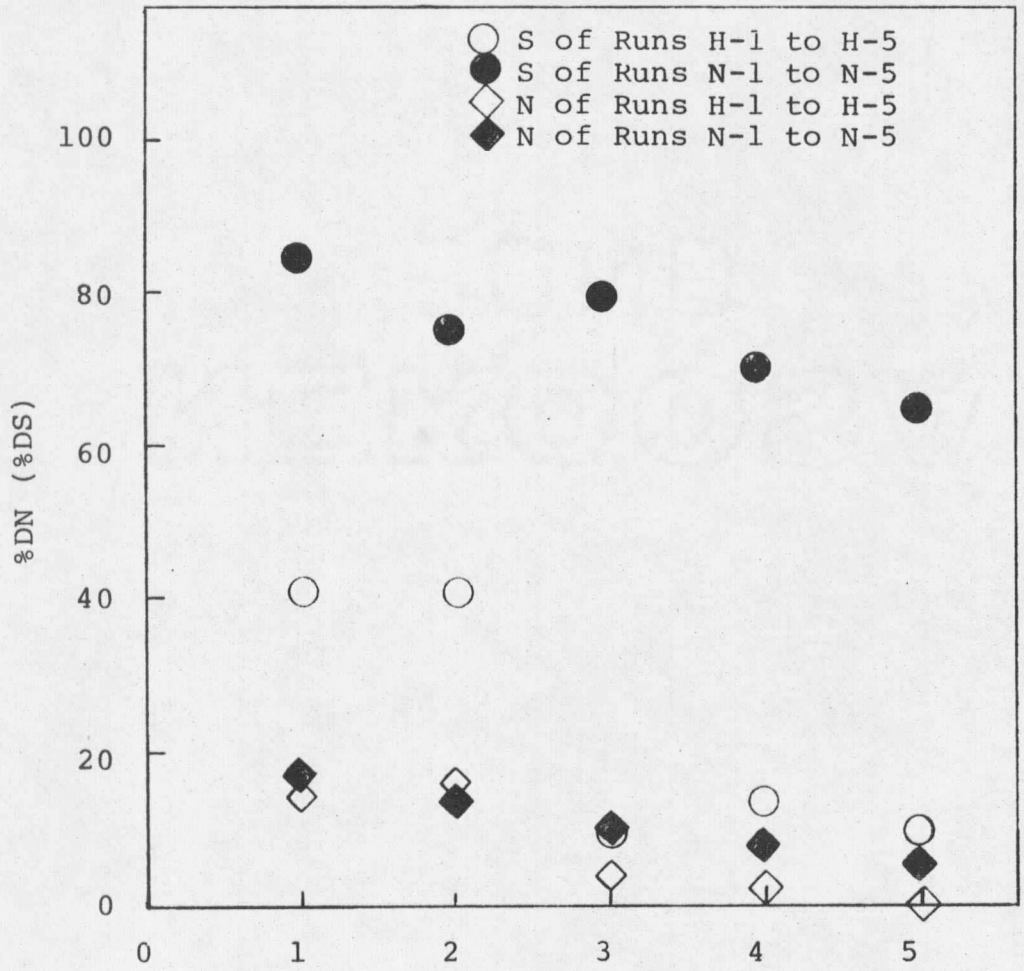


Figure 14. Activity versus feedstock for nitrogen and sulfur removal comparing runs H-1 to H-5 with runs N-1 to N-5.

Operating conditions: 400 °, 1100 psig, 60 min.
Feedstocks:
Runs H-1 to H-5 : SRC-II VFF
Runs N-1 to N-5 : SRC-II LECF

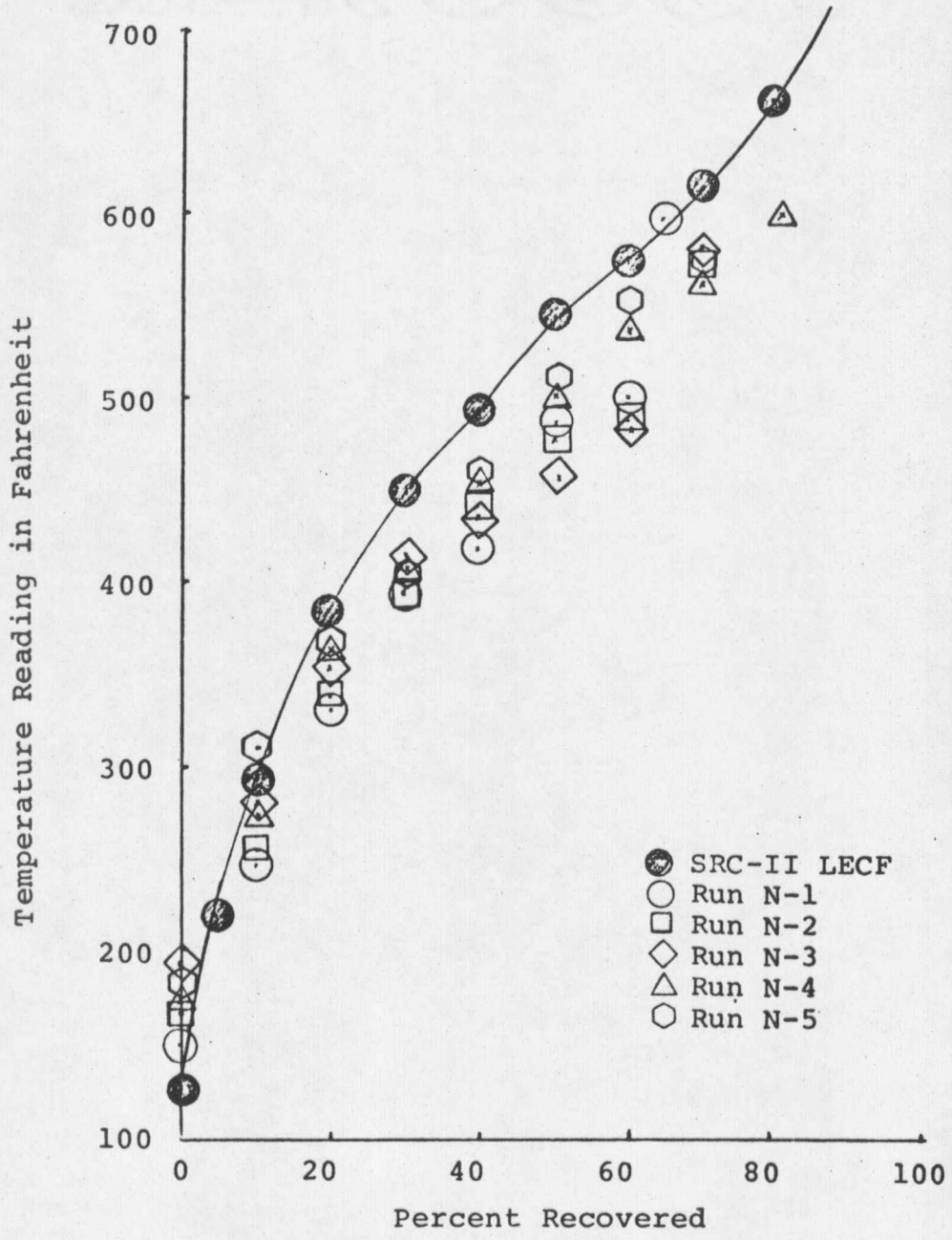


Figure 15. Distillation Data of the Products of Runs N-1 to N-5.

Catalyst Deactivation Tested by the Pore Volume Reduction

Catalyst deactivation due to the carbon laydown on the catalyst was investigated by the pore volume reduction because it might provide information on the activity and life of the catalyst. The water saturation method was used for measuring the pore volume of blank catalyst carrier. A value of 0.77 ml./gm. was obtained compared with the reported value of 0.84 ml./gm. The same measurement method was applied to measure the pore volume of fresh catalyst, MSU-C-49. The pore volume of this fresh catalyst was 0.56 ml./gm. Figure 16 presents the data of calculated pore volumes after regeneration. It was found that the pore volume decreased after the impregnation. It was also noted that there is a slight reduction in the size of the pores of the catalyst after regeneration via burn-off and resulfiding. Comparing runs made with VFF and LECF (H-1 to H-5 and N-1 to N-5) showed that the VFF reduced the pore volume to a greater extent than did LECF. Catalysts of runs N-1 to N-5 had an average pore volume of 0.55 ml./gm., while catalysts of runs H-1 to H-5 had 0.49 ml./gm.

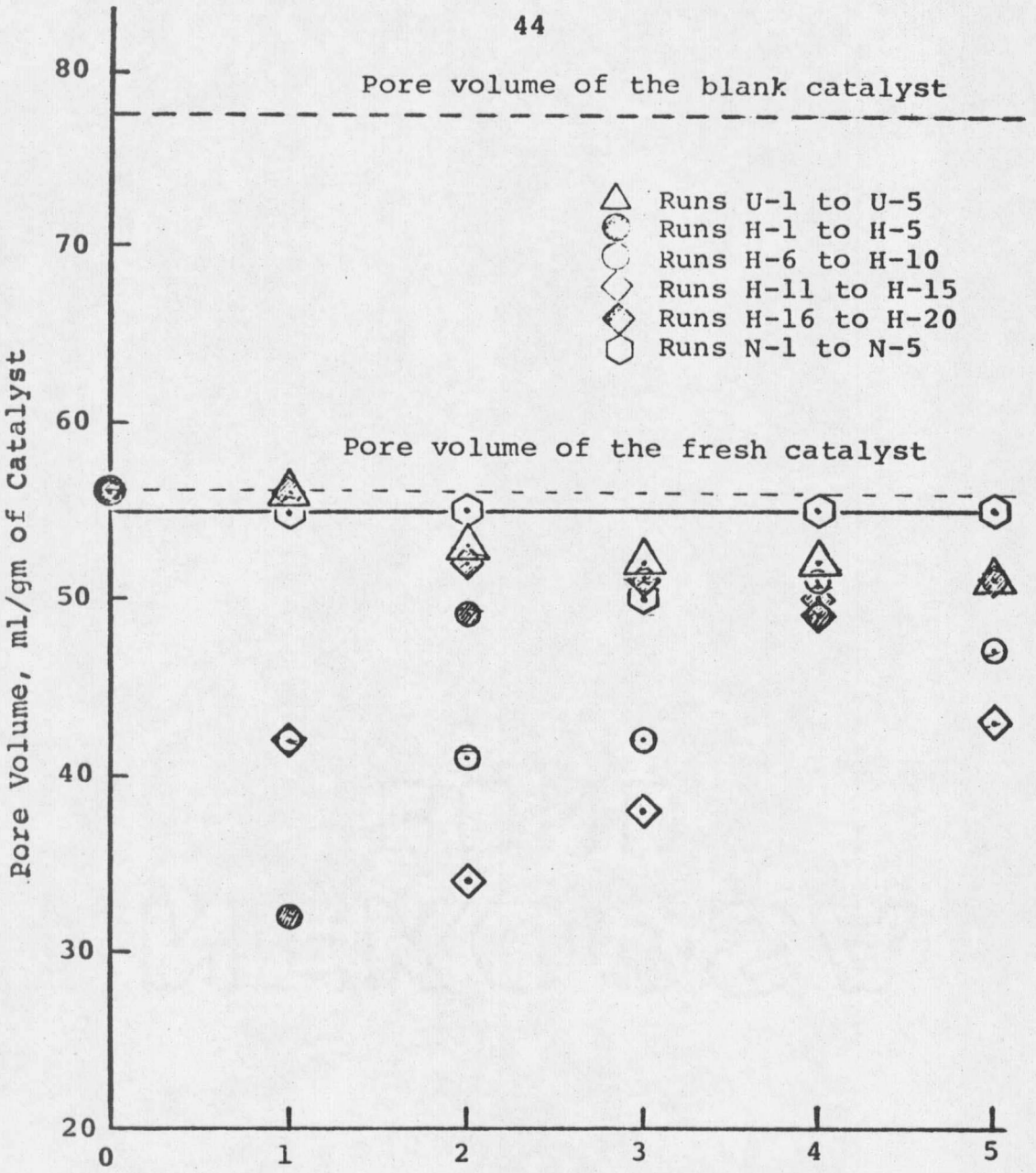


Figure 16. Pore volume vs the five successively used catalysts under the different operating conditions.

CONCLUSIONS

1. Preferred operating conditions for the batch autoclave tests are a temperature of 425 ^{o}C, an initial hydrogen pressure of 2,000 psig, and an operating time of 120 minutes.
2. Sulfur removal was easier from SRC-II LECF than from SRC-II VFF, but nitrogen was little changed.
3. ASTM D-86 distillation results showed a significant improvement in products.
4. A study of regeneration via burn-off and resulfiding showed a slight reduction in the pore volume of the used catalyst following the five successive regenerations, but no progressive deterioration was noted.

RECOMMENDATIONS FOR FUTURE RESEARCH

1. In order to find better operating conditions of the MSU-C-49 catalyst, establishment of statistical models for exploring this catalyst's activity is recommended.
2. The results of batch runs should be confirmed in continuous runs. It is hoped that the operating conditions of 400 ^{o}C and 2,000 psig can be performed in continuous runs.

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APPENDIX

BATCH RUN DATA

Run No. B-1

Catalyst. NALCO-78-6008C-1/32"

Feed Charge: SRC-II Product: 200 ml
Catalyst : 11 gm (25 ml)

Hydrogen Pressure, psig:

	Initial	Final
At Room Temperature:	1100	750
At Run Temperature :	2200	2080

Run Temperature, ^{o}C : 400±10

Time At Run Temperature, min: 60 Residence Time, min: 60

Results:

Sulfur Content of The Liquid Product, % : .69
Weight % Desulfurization : 4.17

Nitrogen Content of The Liquid Product, % : 1.19
Weight % Denitrogenation : 0

Measured Pore Volume, ml/gm : .77
(Blank Catalyst)

ASTM Distillation

Volume of Charge: 44.5 ml Final Volume: 20 ml

Volume % :	IBP	11	23	34	45
^{o}F :	419	450	502	545	594

Run No. B-2

Catalyst. NALCO-78-6008C-1/32"

Feed Charge: SRC-II Product: 200 ml
Catalyst : 11 gm (25 ml)

Hydrogen Pressure, psig:

	Initial	Final
At Room Temperature, :	1100	- -
At Run Temperature :	2250	2110

Run Temperature, ^{o}C : 400±10

Time At Run Temperature, min: 90 Residence Time, min: 60

Results:

Sulfur Content of The Liquid Product, % : .60
Weight % Desulfurization : 6.67

Nitrogen Content of The Liquid Product, % : 1.16
Weight % Denitrogenation : 0

Measured Pore Volume, ml/gm : .77
(Blank Catalyst)

ASTM Distillation

Volume of Charge: 37.6 ml Final Volume: 14 ml

Volume % :	IBP	13	27	37
^{o}F :	412	455	536	606

Run No. U-1

Catalyst. MSU-C-49

Feed Charge: SRC-II Product: 200 ml
Catalyst : 30 gm

Hydrogen Pressure, psig:

	Initial	Final
At Room Temperature:	1100	- -
At Run Temperature :	2900	1780

Run Temperature, ^{o}C : 400±10

Time At Run Temperature, min: 60 Residence Time, min: 44

Results:

Sulfur Content of The Liquid Product, % : .58
Weight % Desulfurization :19.44

Nitrogen Content of The Liquid Product, % : 1.13
Weight % Denitrogenation : 3.42

Calculated Pore Volume, ml/gm : .56

ASTM Distillation

Volume of Charge: 46.3 ml Final Volume: 18.5

Volume % :	IBP	11	22	32	40
^{o}F :	375	425	465	480	550

Run No. U-2

Catalyst. MSU-C-49

Feed Charge: SRC-II Product: 200 ml
Catalyst : 26.6gm

Hydrogen Pressure, psig:

	Initial	Final
At Room Temperature:	1100	- -
At Run Temperature :	3100	2940

Run Temperature, ^{o}C : 400±10

Time At Run Temperature, min: 60 Residence Time, min: 46

Results:

Sulfur Content of The Liquid Product, % : .57
Weight % Desulfurization :20.80

Nitrogen Content of The Liquid Product, % : 1.10
Weight % Denitrogenation : 5.98

Calculated Pore Volume, ml/gm : .53

ASTM Distillation

Volume of Charge: 45.4 ml Final Volume: 21 ml

Volume % :	IBP	11	22	33	46
^{o}F :	350	407	447	490	560

Run No. U-3

Catalyst. MSU-C-49

Feed Charge: SRC-II Product: 200 ml
Catalyst : 23.8 gm

Hydrogen Pressure, psig:

	Initial	Final
At Room Temperature:	1100	- -
At Run Temperature :	2900	2880

Run Temperature, ^{o}C : 400±10

Time At Run Temperature, min: 60 Residence Time, min: 45

Results:

Sulfur Content of The Liquid Product, % : .64
Weight % Desulfurization :11.1

Nitrogen Content of The Liquid Product, % : 1.03
Weight % Denitrogenation :11.97

Calculated Pore Volume, ml/gm : .52

ASTM Distillation

Volume of Charge: 45.4 ml Final Volume: 20 ml

Volume % :	IBP	11	22	33	43
^{o}F :	343	425	465	487	550

Run No. U-4

Catalyst. MSU-C-49

Feed Charge: SRC-II Product: 200 ml
Catalyst : 20.7 gm

Hydrogen Pressure, psig:

	Initial	Final
At Room Temperature:	1150	- -
At Run Temperature :	2720	2600

Run Temperature, ^{o}C : 400±10

Time At Run Temperature, min: 60 Residence Time, min: 47

Results:

Sulfur Content of The Liquid Product, % : 0.60
Weight % Desulfurization :16.7

Nitrogen Content of The Liquid Product, % : 1.15
Weight % Denitrogenation : 1.74

Calculated Pore Volume, ml/gm : 0.52

ASTM Distillation

Volume of Charge: 44.9 ml Final Volume: 17 ml

Volume % :	IBP	11	22	33	38
^{o}F :	335	410	451	550	590

Run No. U-5

Catalyst. MSU-C-49

Feed Charge: SRC-II Product: 200 ml
Catalyst : 18.8 gm

Hydrogen Pressure, psig:

	Initial	Final
At Room Temperature:	1100	- -
At Run Temperature :	2300	2250

Run Temperature, ^{o}C : 400±10

Time At Run Temperature, min: 60 Residence Time, min: 37

Results:

Sulfur Content of The Liquid Product, % : 0.67
Weight % Desulfurization : 6.94

Nitrogen Content of The Liquid Product, % : 1.14
Weight % Denitrogenation : 2.56

Calculated Pore Volume, ml/gm : 0.51

ASTM Distillation

Volume of Charge: 43.9 ml Final Volume: 20 ml

Volume % :	IBP	11	23	34	46
^{o}F :	325	410	437	468	560

Run No. H-1

Catalyst. MSU-C-49

Feed Charge: SRC-II Product: 200 ml
Catalyst : 21 gm (30 ml)

Hydrogen Pressure, psig:

	Initial	Final
At Room Temperature:	1050	- -
At Run Temperature :	1910	1720

Run Temperature, ^{o}C : 400±10

Time At Run Temperature, min: 60 Residence Time, min: 50

Results:

Sulfur Content of The Liquid Product, % : .42
Weight % Desulfurization :41.67

Nitrogen Content of The Liquid Product, % : 1.01
Weight % Denitrogenation :13.60

Calculated Pore Volume, ml/gm : .32

ASTM Distillation

Volume of Charge: 44.4 ml Final Volume: 18 ml

Volume % :	IBP	11	23	34	41
^{o}F :	225	495	497	557	580

Run No. H-2

Catalyst. MSU-C-49

Feed Charge: SRC-II Product: 200 ml
Catalyst : 18.2 gm

Hydrogen Pressure, psig:

	Initial	Final
At Room Temperature:	1050	- -
At Run Temperature :	2050	1900

Run Temperature, ^{o}C : 400±10

Time At Run Temperature, min: 60 Residence Time, min: 55

Results:

Sulfur Content of The Liquid Product, % : .42
Weight % Desulfurization :41.67

Nitrogen Content of The Liquid Product, % : .98
Weight % Denitrogenation :16.23

Calculated Pore Volume, ml/gm : .49

ASTM Distillation

Volume of Charge: 49.5 ml Final Volume: 28 ml

Volume % :	IBP	10	20	30	40	50	57
^{o}F :	200	375	420	450	485	540	620

Run No. H-3

Catalyst. MSU-C-49

Feed Charge: SRC-II Product: 200 ml
Catalyst : 14.7 gm

Hydrogen Pressure, psig:

	Initial	Final
At Room Temperature:	1200	- -
At Run Temperature :	2300	2050

Run Temperature, ^{o}C : 400±10

Time At Run Temperature, min: 60 Residence Time, min: 55

Results:

Sulfur Content of The Liquid Product, % : .66
Weight % Desulfurization : 8.33

Nitrogen Content of The Liquid Product, % : 1.13
Weight % Denitrogenation : 3.41

Calculated Pore Volume, ml/gm : .51

ASTM Distillation

Volume of Charge: 47.6 ml Final Volume: 26 ml

Volume % :	IBP	11	21	32	42	53
^{o}F :	225	410	447	505	555	587

Run No. H-4

Catalyst. MSU-C-49

Feed Charge: SRC-II Product: 200 ml
Catalyst : 10.7 gm

Hydrogen Pressure, psig:

	Initial	Final
At Room Temperature:	1100	- -
At Run Temperature :	2050	1800

Run Temperature, ^{o}C : 400±10

Time At Run Temperature, min: 60 Residence Time, min: 60

Results:

Sulfur Content of The Liquid Product, % : .61
Weight % Desulfurization :12.50

Nitrogen Content of The Liquid Product, % : 1.14
Weight % Denitrogenation : 2.56

Calculated Pore Volume, ml/gm : .49

ASTM Distillation

Volume of Charge: 48.1 ml Final Volume: 27 ml

Volume % :	IBP	10	21	31	42	56
^{o}F :	220	420	450	497	550	592

Run No. H-5

Catalyst. MSU-C-49

Feed Charge: SRC-II Product: 200 ml
Catalyst : 9.8 gm

Hydrogen Pressure, psig:

	Initial	Final
At Room Temperature:	1100	- -
At Run Temperature :	2100	1830

Run Temperature, ^{o}C : 400±10

Time At Run Temperature, min: 60 Residence Time, min: 55

Results:

Sulfur Content of The L-quid Product, % : .65
Weight % Desulfurization : 9.72

Nitrogen Content of The Liquid Product, % : 1.17
Weight % Denitrogenation : 0

Calculated Pore Volume, ml/gm : .51

ASTM Distillation

Volume of Charge: 46.6 ml Final Volume: 17 ml

Volume % :	IBP	11	21	32	37
^{o}F :	220	395	440	475	550

Run. H-6

Catalyst. MSU-C-49

Feed Charge: SRC-II Product: 200 ml
Catalyst : 30 gm

Hydrogen Pressure, psig:

	Initial	Final
At Room Temperature:	1100	540
At Run Temperature :	1780	1550

Run Temperature, ^{o}C : 400±10

Time At Run Temperature, min: 120 Residence Time, min: 49

Results:

Sulfur Content of The Liquid Product, % : 0.40
Weight % Desulfurization : 44.44

Nitrogen Content of The Liquid Product, % : 0.87
Weight % Denitrogenation : 25.64

Calculated Pore Volume, ml/gm : 0.42

ASTM Distillation

Volume of Charge: 49.1 ml Final Volume: 24 ml

Volume % :	IBP	10	20	31	41	49
^{o}F :	305	385	420	490	535	607

Run No. H-7

Catalyst. MSU-C-49

Feed Charge: SRC-II Product: 200 ml
Catalyst : 26.8 gm

Hydrogen Prossure, psig:

	Initial	Final
At Room Temperature:	1100	- -
At Run Temperature :	2340	1900

Run Temperature, ^{o}C : 400±10

Time At Run Temperature, min: 120 Residence Time, min: 43

Results:

Sulfur Content of The Liquid Product, % : 0.46
Weight % Desulfurization : 37.5

Nitrogen Content of The Liquid Product, % : 0.95
Weight % Denitrogenation : 18.8

Calculated Pore Volume, ml/gm : 0.41

ASTM Distillation

Volume of Charge: 46.5 ml Final Volume:22.3 ml

Volume % :	IBP	11	22	32	43	48
^{o}F :	285	425	470	515	578	615

Run No. H-8

Catalyst. C-49

Feed Charge: SRC-II Product: 200 ml
Catalyst : 24.5 gm

Hydrogen Pressure, psig:

	Initial	Final
At Room Temperature:	1150	- -
At Run Temperature :	2350	1680

Run Temperature, ^{o}C : 400±10

Time At Run Temperature, min: 120 Residence Time, min: 55

Results:

Sulfur Content of The Liquid Product, % : 0.42
Weight % Desulfurization : 41.67

Nitrogen Content of The Liquid Product, % : 0.90
Weight % Denitrogenation : 23.08

Calculated Pore Volume, ml/gm : 0.42

ASTM Distillation

Volume of Charge: 45.8 ml Final Volume: 20.7 ml

Volume % :	IBP	11	22	33	44	45
^{o}F :	260	437	485	558	610	635

Run No. H-9

Catalyst. MSU-C-49

Feed Charge: SRC-II Product: 200 ml
Catalyst : 21 gm

Hydrogen Pressure, psig:

	Initial	Final
At Room Temperature:	1100	700
At Run Temperature :	2580	1900

Run Temperature, ^{o}C : 400±10

Time At Run Temperature, min: 120 Residence Time, min: 48

Results:

Sulfur Content of The Liquid Product, % : 0.40
Weight % Desulfurization : 44.44

Nitrogen Content of The Liquid Product, % : 1.04
Weight % Denitrogenation : 11.11

Caculated Pore Volume, ml/gm : 0.51

ASTM Distillation

Volume of Charge: 48.4 ml Final Volume 22.5

Volume % :	IBP	10	21	31	41	46
^{o}F :	275	380	415	482	540	550

Run No. H-10

Catalyst. MSU-C-49

Feed Charge: SRC-II Product: 200 ml
Catalyst : 16.4 gm

Hydrogen Pressure, psig:

	Initial	Final
At Room Temperature:	1100	- -
At Run Temperature :	2700	1920

Run Temperature, ^{o}C : 400±10

Time At Run Temperature, min: 120 Residence Time, min: 48

Results:

Sulfur Content of The Liquid Product, % : 0.38
Weight % Desulfurization : 47.22

Nitrogen Content of The Liquid Product, % : 1.04
Weight % Denitrogenation : 11.11

Calculated Pore Volume, ml/gm : 0.47

ASTM Distillation

Volume of Charge: 47.2 ml Final Volume: 23.5 ml

Volume % :	IBP	11	21	32	42	50
^{o}F :	305	412	460	506	560	578

Run No. H-11

Catalyst. MSU-C-49

Feed Charge: SRC-II Product: 200 ml
Catalyst : 30 gm

Hydrogen Pressure, psig:

	Initial	Final
At Room Temperature:	1100	- -
At Run Temperature :	2000	1230

Run Temperature, ^{o}C : 425±10

Time At Run Temperature, min: 60 Residence Time, min: 56

Results:

Sulfur Content of The Liquid Product,% : 0.37
Weight % Desulfurization : 48.61

Nitrogen Content of The Liquid Product, % : 1.03
Weight % Dennitrogenation : 11.97

Calculated Pore Volume, ml/gm : 0.42

ASTM Distillation

Volume of Charge: 47.3 ml Final Volume: 24.3 ml

Volume % :	IBP	11	21	32	42	51
^{o}F :	195	395	433	468	513	590

Run No. H-12

Catalyst. MSU-C-49

Feed Charge: SRC-II Product: 200 ml
Catalyst : 29.8 gm

Hydrogen Pressure, psig:

	Initial	Final
At Room Temperature:	1100	- -
At Run Temperature :	2700	2100

Run Temperature, ^{o}C : 425±10

Time At Run Temperature, min: 60 Residence Time, min: 52

Results

Sulfur Content of The Liquid Product, % : 0.30
Weight % Desulfurization : 58.33

Nitrogen Content of The Liquid Product, % : 1.0
Weight % Denitrogenation : 14.53

Calculated Pore Volume, ml/gm : 0.34

ASTM Distillation

Volume of Charge: 47.8 ml Final Volume: 24

Volume % :	IBP	10	21	31	42	50
^{o}F :	193	393	440	490	550	603

Run No. H-13

Catalyst. MSU-C-49

Feed Charge: SRC-II Product: 200 ml
Catalyst : 27 gm

Hydrogen Pressure, psig:

	Initial	Final
At Room Temperature:	1100	- -
At Run Temperature :	2460	1860

Run Temperature, ^{o}C : 425±10

Time At Run Temperature, min: 60 Residence Time, min: 45

Results:

Sulfur Content of The Liquid Product, % : 0.37
Weight % Desulfurization : 48.61

Nitrogen Content of The Liquid Product, % : 1.0
Weight % Denitrogenation : 14.53

Calculated Pore Volume, ml/gm : 0.38

ASTM Distillation

Volume of Charge: 47.6 ml Final Volume: 22.7 ml

Volume % :	IBP	11	21	32	42	48
^{o}F :	195	350	410	460	530	605

Run No. H-14

Catalyst. MSU-C-49

Feed Charge: SRC-II Product: 200 ml
Catalyst : 12.4 gm

Hydrogen Pressure, psig:

	Initial	Final
At Room Temperature:	1100	500
At Run Temperature :	2400	1900

Run Temperature, ^{o}C : 425±10

Time At Run Temperature, min: 60 Residence Time, min: 49

Results:

Sulfur Content of The Liquid Product, % : 0.40
Weight % Desulfurization : 44.44

Nitrogen Content of The Liquid Product, % : 1.04
Weight % Denitrogenation : 11.11

Calculated Pore Volume, ml/gm : 0.49

ASTM Distillation

Volume of Charge: 47 ml Final Volume: 25 ml

Volume % :	IBP	11	21	32	43	53
^{o}F :	200	390	420	463	525	585

Run No. H-15

Catalyst. MSU-C-49

Feed Charge: SRC-II Product: 200 ml
Catalyst : 9.3 gm

Hydrogen Pressure, psig:

	Initial	Final
At Room Temperature:	1100	600
At Run Temperature :	2260	1900

Run Temperature, ^{o}C : 425±10

Time At Run Temperature, min: 60 Residence Time, min: 51

Results:

Sulfur Content of The Liquid Product, % : 0.39
Weight % Desulfurization : 45.83

Nitrogen Content of The Liquid Product, % : 1.0
Weight % Denitrogenation : 14.53

Calculated Pore Volume, ml/gm : 0.43

ASTM Distillation

Volume of Charge: 47.4 ml Final Volume: 23 ml

Volume % :	IBP	11	21	32	42	48
^{o}F :	195	405	448	502	570	615

Run No. H-16

Catalyst. MSU-C-49

Feed Charge: SRC-II Product: 200 ml
Catalyst : 30 gm

Hydrogen Pressure, psig:

	Initial	Final
At Room Temperature:	2000	900
At Run Temperature :	4100	2350

Run Temperature, ^{o}C : 400 ± 10

Time At Run Temperature, min: 60 Residence Time, min: 55

Results:

Sulfur Content of The Liquid Product, % : 0.24
Weight % Desulfurization : 66.67

Nitrogen Content of The Liquid Product, % : 0.92
Weight % Denitrogenation : 21.37

Calculated Pore Volume, ml/gm : 0.56

ASTM Distillation

Volume of Charge: 47.8 ml Final Volume: 23 ml

Volume % :	IBP	10	21	31	42	48
^{o}F :	195	415	457	503	508	590

Run No. H-17

Catalyst. MSU-C-49

Feed Charge: SRC-II Product: 200 ml
Catalyst : 23.9 gm

Hydrogen Pressure, psig:

	Initial	Final
At Room Temperature:	1950	- -
At Run Temperature :	3860	2680

Run Temperature, ^{o}C : 400±10

Time At Run Temperature, min: 60 Residence Time, min: 51

Results:

Sulfur Content of The Liquid Product, % : 0.23
Weight % Desulfurization : 68.06

Nitrogen Content of The Liquid Product, % : 0.99
Weight % Denitrogenation : 15.38

Calculated Pore Volume, ml/gm : 0.52

ASTM Distillation

Volume of Charge: 48.2 ml Final Volume: 23.5 ml

Volume % :	IBP	10	21	32	41	49
^{o}F :	193	383	415	453	495	585

Run No. H-18

Catalyst. MSU-C-49

Feed Charge: SRC-II Product: 200 ml
Catalyst : 20.9 gm

Hydrogen Pressure, psig:

	Initial	Final
At Room Temperature:	2000	1100
At Run Temperature :	3740	3100

Run Temperature, ^{o}C : 400±10

Time At Run Temperature, min: 60 Residence Time, min: 44

Results:

Sulfur Content of The Liquid Product, % : 0.40
Weight % Desulfurization : 44.44

Nitrogen Content of The Liquid Product, % : 1.03
Weight % Denitrogenation : 11.97

Calculated Pore Volume, ml/gm : 0.51

ASTM Distillation

Volume of Charge: 46.4 ml Final Volume: 18.5 ml

Volume % :	IBP	11	22	33	40
^{o}F :	200	420	460	465	595

Run No. H-19

Catalyst. MSU-C-49

Feed Charge: SRC-II Product: 200 ml
Catalyst : 19 gm

Hydrogen Pressure, psig:

	Initial	Final
At Room Temperature:	2000	1440
At Run Temperature :	4100	3700

Run Temperature, ^{o}C : 400±10

Time At Run Temperature, min: 60 Residence Time, min: 47

Results:

Sulfur Content of The Liquid Product, % : 0.45
Weight % Desulfurization : 37.50

Nitrogen Content of The Liquid Product, % : 1.06
Weight % Denitrogenation : 9.40

Calculated Pore Volume, ml/gm : 0.50

ASTM Distillation

Volume of Charge: 46.9 ml Final Volume: 22.3 ml

Volume % :	IBP	11	21	32	43	48
^{o}F :	198	393	413	468	525	580

Run No. H-20

Catalyst. MSU-C-49

Feed Charge: SRC-II Product: 200 ml
Catalyst : 17.6 gm

Hydrogen Pressure, psig:

	Initial	Final
At Room Temperature:	2000	1280
At Run Temperature :	3780	3280

Run Temperature, ^{o}C : 400±10

Time At Run Temperature, min: 60 Residence Time, min: 46

Results:

Sulfur Content of The Liquid Product, % : 0.58
Weight % Desulfurization : 19.44

Nitrogen Content of The Liquid Product, % : 1.11
Weight % Denitrogenation : 5.13

Calculated Pore Volume, ml/gm : 0.51

ASTM Distillation

Volume of Charge: 46.6 ml Final Volume: 23.5 ml

Volume % :	IBP	11	21	32	43	50
^{o}F :	193	420	442	467	505	575

Run No. N-1

Catalyst. MSU-C-49

Feed Charge: SRC-II Product (Light Ends Column Feed) : 200 ml
Catalyst : 30 gm

Hydrogen Pressure, psig:

	Initial	Final
At Room Temperature:	1100	350
At Run Temperature :	2200	1300

Run Temperature, ^{o}C : 400±10

Time At Run Temperature, min: 60 Residence Time, min: 49

Results:

Sulfur Content of The Liquid Product, % : 0.19
Weight % Desulfurization : 84.30

Nitrogen Content of The Liquid Product, % : 0.73
Weight % Denitrogenation : 17.05

Calculated Pore Volume, ml/gm : 0.55

ASTM Distillation

Volume of Charge: 49.5 ml Final Volume: 31 ml

Volume % :	IBP	10	20	30	40	51	61	63
^{o}F :	145	243	328	390	415	482	495	595

Run No. N-2

Catalyst. MSU-C-49

Feed Charge: SRC-II Product (Light Ends Column Feed) : 200 ml
Catalyst : 27.8 gm

Hydrogen Pressure, psig:

	Initial	Final
At Room Temperature:	1100	300
At Run Temperature :	2260	1700

Run Temperature, ^{o}C : 400 ± 10

Time At Run Temperature, min: 60 Residence Time, min: 48

Results:

Sulfur Content of The Liquid Product, % : 0.28
Weight % Desulfurization : 76.86

Nitrogen Content of The Liquid Product, % : 0.76
Weight % Denitrogenation : 13.64

Calculated Pore Volume, ml/gm : 0.55

ASTM Distillation

Volume of Charge: 49.4 ml Final Volume: 35 ml

Volume % :	IBP	10	20	30	40	51	61	71
^{o}F :	160	250	330	390	440	475	485	570

Run No. N-3

Catalyst. MSU-C-49

Feed Charge: SRC-II Product (Light Ends Column Feed) : 200 ml
Catalyst : 24.2 gm

Hydrogen Pressure, psig:

	Initial	Final
At Room Temperature:	1150	500
At Run Temperature :	2800	2400

Run Temperature, ^{o}C : 400±10

Time At Run Temperature, min: 60 Residence Time, min: 49

Results:

Sulfur Content of The Liquid Product, % : 0.25
Weight % Desulfurization : 79.34

Nitrogen Content of The Liquid Product, % : 0.79
Weight % Denitrogenation : 10.23

Calculated Pore Volume, ml/gm : 0.50

ASTM Distillation

Volume of Charge: 49.6 ml Final Volume: 35 ml

Volume % :	IBP	10	20	30	40	50	61	71
^{o}F :	190	280	350	415	437	455	485	580

Run No. N-4

Catalyst. MSU-C-49

Feed Charge: SRC-II Product (Light Ends Column Feed) : 200 ml
Catalyst : 23.8 gm

Hydrogen Pressure, psig:

	Initial	Final
At Room Temperature:	1100	500
At Run Temperature :	2120	1700

Run Temperature, ^{o}C : 400 $\pm$10

Time At Run Temperature, min: 60 Residence Time, min: 46

Results:

Sulfur Content of The Liquid Product, % : 0.34
Weight % Desulfurization : 71.90

Nitrogen Content of The Liquid Product, % : 0.81
Weight % Denitrogenation : 7.95

Calculated Pore Volume, ml/gm : 0.55

ASTM Distillation

Volume of Charge: 49.6 ml Final Volume: 40 ml

Volume % :	IBP	10	20	30	40	50	61	71	81
^{o}F :	167	275	360	410	455	500	537	562	600

Run No. N-5

Catalyst. MSU-C-49

Feed Charge: SRC-II Product (Light Ends Column Feed) : 200 ml
Catalyst : 21.8 gm

Hydrogen Pressure, psig:

	Initial	Final
At Room Temperature:	1100	600
At Run Temperature :	2440	2200

Run Temperature, ^{o}C : 400±10

Time At Run Temperature, min: 60 Residence Time, min: 47

Results:

Sulfur Content of The Liquid Product, % : 0.30
Weight % Desulfurization : 75.21

Nitrogen Content of The Liquid Product, % : 0.83
Weight % Denitrogenation : 5.68

Calculated Pore Volume, ml/gm : 0.55

ASTM Distillation

Volume of Charge: 49.3 ml Final Volume: 35 ml

Volume % :	IBP	10	20	30	40	50	60	71
^{o}F :	177	328	365	410	457	497	555	575